SOLVENT EXTRACTION OF ANTIOXIDANTS, PHENOLS AND FLAVONOIDS FROM SAUDI ARABIA DATES

by

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DEDICATION

TO:

My Parents:

Ali Al Udhaib and Sara Al Qahtani

My Sisters and Brothers:

Aisha, Mohammed, Rana, Ruba, Mohanned and Abdulallah

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ABSTRACT

Cultivated dates are a stable food source in many countries, playing significant roles the people's nutrition and their economy. The date palm fruit is the most important crop in the Kingdom of Saudi Arabia and is cultivated in nearly all regions of the Kingdom. Saudi Arabia is the world's second-largest producer of dates, growing 16% of global date production. Saudi Arabia has more than 23.7 million date palm trees cultivated on more than 156,000 hectares and producing about 992,000 tonnes of dates annually. However, there has been a significant interest in recent years regarding the properties of antioxidants, phenols and flavonoids found in dates. These compounds have been found to neutralize free radicals inside the body which originate from metabolic. Free radicals are dangerous because they trigger chemical chain reactions within the body that damage or kill cells and as such they have been linked to a number of diseases. The aim of this study was to optimize extraction conditions of antioxidants, phenols and flavonoids from dates. First, the effects of sample: solvent ratio (1:20, 1:40 and 1:60), reaction temperature (25, 35, 45, 55 and 65°C), reaction time (1, 2, 3 and 4 h), solvent concentration (0, 50 and 75%) and solvent type (acetone and ethanol) on the yield of these compounds from ajwa date were investigated. The optimum extraction condition was used to compare the yields of these compounds from freeze dried ajwa date and fresh ajwa date as well as from 5 dates (Ajwa. Khalas, sukkari, red sukkari and sofry). The three hours reaction time at 55°C with a sample: solvent ratio of 1:20 and 75% ethanol were the optimum extraction conditions for antioxidants yield while 3 h reaction time at 65°C with a sample: solvent ratio of 1:20 and 75% acetone were the optimum conditions for phenols and flavonoids extraction from fresh ajwa date. Freeze drying procedure obtained higher yields for antioxidants (84.60%), phenols (55.70%) and flavonoids (29.99%) than those obtained from fresh ajwa date. Ajwa dates had the highest antioxidants, phenols and flavonoids (73.10 mg/g, 355.24mg/g and 57.52mg/g), followed by khalas (62.85 mg/g, 252.01 mg/g and 52.47 mg/g), sukkari (62.45 mg/g, 204.65 mg/g and 43 mg/g), red sukkari (61.10 mg/g, 200.59 mg/g and 41.65 g/mg) and sofry (60.39 mg/g, 184.05 mg/g and 41.22 mg/g).

LIST OF SYMBOLS AND ABBREVIATIONS USED

ABTS	2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid
ANOVA	Analysis of Variance
BHA	Butylated Hydroxyanisole
BHT	Butylated Hydroxytoluene
°C	Degree Celsius
CONC	Concentration
DF	Degrees of Freedom
DMF	Dimethylformamide
DPL	Date Palm Leaves
DPPH	2,2-diphenyl-1-picrylhydrazyl DPPH
F	F-test, using F-distribution
FAE	Ferrulic Acid
FAO	Food and Agriculture Organization of the United Nations
F-C	Folin-Ciocalteu
g	Gram
h	Hours
kg	kilogram
L	Liter
MDF	Medium Density Fiber Board.
min	Minutes
mg	Miligram
ml	milliliter
MS	Mean of Squares
P	P-value
R	Ratio
S	Solvent
SS	Sum of Squares
T	Time
TA	Total Antioxidants

TE	Trolox
TEMP	Temperature
TF	Total Flavonoid
TP	Total Phenolic

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CHAPTER 1. INTRODUCTION

The date palm (*Phoenix dactylifera L.*) has been an important plant in arid and semi-arid areas for centuries. Cultivated dates are a staple food source in these countries playing significant roles not only in the people's nutrition but also in their economy and religion. Dates are one of the oldest known fruit crops and have been cultivated for over 5,000 years in North Africa and the Middle East. Due to their nutritional value and ease of transportation and storage, dates have had a very important influence on the history of the Middle East (Krueger and Chao, 2007).

Date palm trees produce different types of date fruit which could be eaten fresh or processed into various products including: date syrup, alcohol, animal feed, date powder, different types of bread, marmalade, sweet candy, chocolate date and date paste (Hamada et al., 2002; Krueger and Chao, 2007; Ben Salah el al., 2010). Other parts of the date palm tree are also important in the agricultural and rural economy. For example, the stem is used for making boats, covering the roof of rural houses, making papers and fibers. The foliage is used for making hand crafts such fans and straw hats (Bahman et al., 1997; Khiar et al., 2009; Riahi et al., 2009).

The world production of dates have been increased due to the increases in population and consumer demand. However, lots of dates are not suitable for human consumption for several reasons including: low quality, contamination by insect and hard texture. They are used as animal feed or for production of alcohol (Borchani et al, 2011). Date palm seeds which are considered as a waste is a major issue for the date processing industry. Seeds are used mostly as feed for camel and sheep (Hamada et al, 2002).

According to Habib and Ibrahim (2011), dates have nutritional value. The fruit of the date palm (*Phoenix dactylifera L.)* contains a high percentage of carbohydrates (82%), fat (0.2%), mineral (9%), protein (2.3%), vitamin (4.8%) and dietary fiber (1.7%). The date fruit is a rich source of potassium, magnesium and vitamin B. The weight of the seed is 5.6 - 14.2% of the date fruit and contains 7.7 - 9.7% oil (Al-Shahib et al., 2003 and Balghunaim, 2011). Date palm seed is a source of fatty acids such as oleic, laurie, palmitic, myristic, and stearic acids. These are long chain carboxylic acids which can be extracted from the seeds and used in the health and cosmetic industries as cosmetic creams and as base components (of the oil phase) of many cosmetic formulations (Abdullah et al., 2010; Besbes et al., 2004, Al-Shahib and Marshall, 2003; Souhail et al., 2005; Nehdi et al., 2010).

The date flesh and seeds have active compounds which can be used in production of pharmaceutical. According to Al-Shahib et al., (2003), dates have been identified as a rich source of antioxidants, particularly phenolic acids (Bassam, 2008). These compounds have been found to neutralize free radicals inside the body which originate from internal metabolic processes as well as external processes. Free radicals are dangerous because they trigger chemical chain reactions within the body that damage or kill cells and as such they have been linked to a number of diseases including cancer, neurological disease, pulmonary disease and ocular and pulmonary diseases (Li et al., 2007). The antioxidants are able to mitigate the damage by reacting with the free radicals and converting them into more stable harmless compounds. Extracting and characterizing these antioxidants from date could prove useful in developing food supplements that can positively contribute to human health and wellbeing and improve the economy of the date industry.

CHAPTER 2. OBJECTIVES

The aim of this study was to investigate the possibility of extracting antioxidants (phenolic and flavonoids) from five date varieties (Ajwa, Sukkari, Red Sukkari , Khalas and Sofry) cultivated in the Kingdom of Saudi Arabia. The specific objectives were:

1. To study the effects of the following parameters on the activity of extracted antioxidants from fresh Ajwa date:
   (a) Type of solvent (ethanol and acetone)
   (b) Concentration of solvent (0% , 50% and 75%)
   (c) Sample: solvent ratio (1:20,1:40 and 1:60)
   (d) Reaction temperature (25, 35, 45, 55 and 65°C)
   (e) Reaction time (1, 2, 3, and 4 h)
2. To evaluate the effect of sample preparation (freeze drying vs fresh fruit) on the activity of extracted antioxidant from Ajwa date at the optimum extraction conditions.
3. To compare the activities of the extracted antioxidants from five date varieties at the optimum sample preparation and extraction conditions.

CHAPTER 3. LITERATURE REVIEW

3.1. Origin and Growing Areas of the Date Palm

The botanical name of the date palm is *Phoenix dactylifera L*. There are twelve species of the genus "*Phoenix*". The date palm is classified under the group *Spadiciflora*, the order of *Palmea* which belongs to the sub-family *Coryphyoideae* that fallsunder the tribe *Phoeniceae* which in turn falls under the genus *Phoenix* that falls under the speciece *dactylifera* (Zaid and De Wet, 2002). The date palm is one of the most important crops in many parts of the world (Figure 3.1) including Asia, North Africa, the Middle East and Americas especially in the lower California Peninsula and Mexico (Fki et al., 2001; Awad, 2008; Zaid and De Wet, 2002). The four main areas where palm trees grow are oasis areas, interior areas, coastal areas, and riparian areas. Each of these areas has a specific type of date (Ibrahim and Habib, 2011). According to Zaid and De Wet (2002), date palms grow naturally between 10°N (Somalia) and 39°N (Elche/Spain or Turkmenistan). Other growing regions are situated between 24°N and 34°N (Morocco, Algeria, Tunisia, Libya, Israel, Egypt, Iraq, and Iran). Growing areas in the USA are located between 20° S and 35°N involving arid and subtropical areas in North and South America, Africa and Asia and from 63° W (Venezuela) to 117° W (California). Dates have also been found in the central desert of Australia. However, the best date production is in the arid regions of Peru, Chile, Baja California (Mexico) and the Southwestern USA (Rivera et al., 2013; Zaid and De wet, 2002; Bhaskaran and Smith, 1995). Date palms are grown in 90% of the Arab-speaking world, extending from Mauritania in the west to the Arabian Gulf in the east (Zaid and De Wet, 2002).

Worldwide, date production has increased by 75.31% during the last 50 years, from 1,852,592 tonnes in 1962 to 7,504,984 tonnes in 2011 (FAO, 2013). Table 3.1 shows date production in various countries during the period of 2000 to 2007. Figure 3.2. shows the top ten date-producing countries in 2011 (FAO, 2013). Figure 3.3. shows the top sten date-importing countries in 2010 (FAO, 2013). Globally, date palm trees are about 86 million producing about 6.4 million tonnes of dates. The date trees are most suited for cultivation in dry, semi-dry and subtropical areas. The Arab countries produce 3 million tonnes, which represents 70% of the world's date production (FAO, 2011).

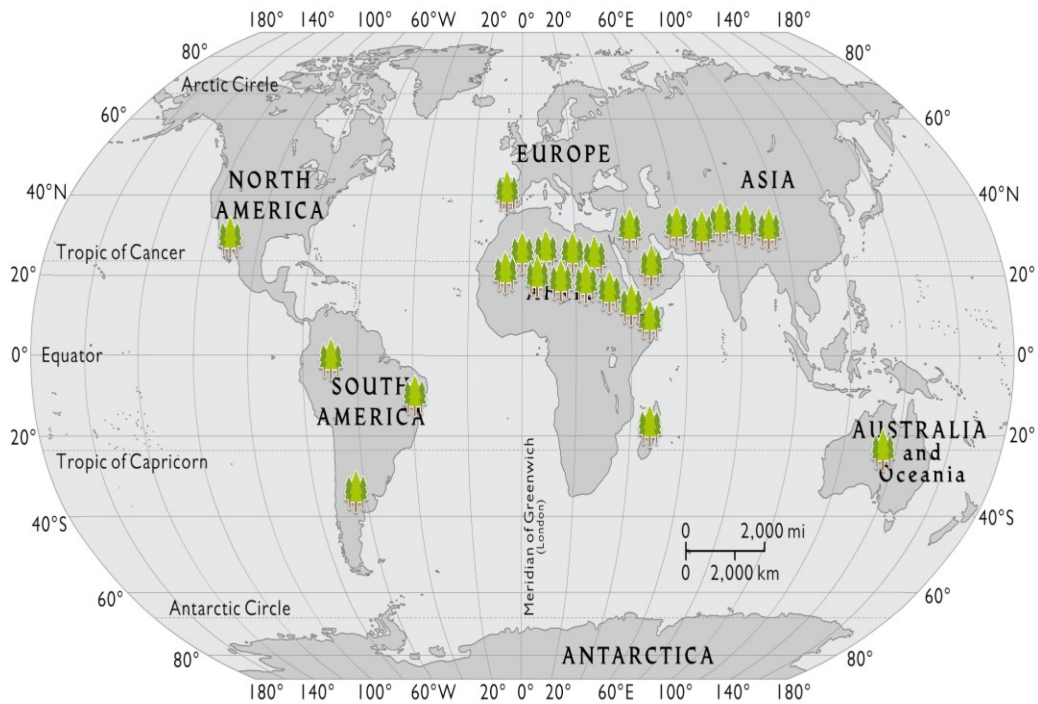


Figure 3.1. World map showing date palm growing regions (Zaid and De Wet, 2002).

Table 3.1. Shows the date production (tonnes) in various countries from 2000 to 2007 (FAO, 2008).

Country	2000	2001	2002	2003	2004	2005	2006	2007
Albenia	-	-	-	-	5800	6590	7900	8000
Algeria	365616	437332	418427	492217	442600	516293	491188	468000
Bahrain	16508	16508	14500	14000	14000	15000	15000	15500
Benin	1000	1000	1000	1100	1100	1150	1100	1150
Cameroon	340	360	380	380	390	390	400	400
Chad	18000	18000	18000	18000	18000	18000	18000	18300
China	125000	117000	130000	120000	125000	130000	125000	128000
Colombia	3	3	3	3	24	70	80	90
Djibouti	72	75	75	80	80	80	80	80
Egypt	1006710	1113270	1090004	1121890	1166182	1170000	1175000	1130000
Iran	869573	874986	879000	885000	989626	996770	1000000	1000000
Iraq	932000	907000	866000	868000	875000	404000	300000	290000
Israel	11732	11700	16830	13430	14475	15975	17869	17900
Jordan	1321	1414	2106	1897	4068	3115	3965	4000
Kenya	1000	1000	1000	1000	1000	1000	1000	1000
Kuwait	10155	10376	12577	15811	16000	15800	14200	14500
Libyan	120000	140000	200000	200000	150000	150000	170000	175000
Mauritania	22000	20000	24000	20000	24000	22000	22000	22000
Mexico	3965	4309	3172	2076	2014	2756	2988	3000
Morocoo	74000	32400	33200	54110	69400	47500	45470	52000
Namibia	500	500	500	500	500	500	500	500
Niger	7600	7700	7700	7800	7800	7800	7800	7800
Occupied Palestinian Territory	3852	3819	5051	3657	5015	3608	2443	2400
Oman	280030	298006	238611	219770	231000	247331	258738	260000
Pakistan	612482	630281	625000	426822	622404	496576	506623	510000
Peru	199	194	248	242	257	276	422	475
Namibia	500	500	500	500	500	500	500	500
Niger	7600	7700	7700	7800	7800	7800	7800	7800
Occupied Palestinian Territory	3852	3819	5051	3657	5015	3608	2443	2400
Country	2000	2001	2002	2003	2004	2005	2006	2007
Albenia	-	-	-	-	5800	6590	7900	8000
Algeria	365616	437332	418427	492217	442600	516293	491188	468000
Qatar	16116	13109	14845	1657	18222	19844	20000	21000
Saudi Arabia	734844	817887	829540	884088	941293	970488	970000	970000
Somalia	10000	11000	11000	12000	12000	10500	12000	12000
Spain	3717	3732	3451	3580	4273	4360	5000	5250
Sudan	332320	330000	330000	328000	336000	328200	330000	330000
Swaziland	200	700	200	200	200	200	280	280
Syrian Arab Republic	3051	3921	1453	4000	3500	3500	3800	3800
Tunisia	105000	112620	120810	116970	122000	125000	125000	120000
Turkey	9200	9300	9400	9400	9400	9400	9400	9400
United Arab Emirates	757601	757601	757601	757601	760000	750000	755000	755000
United States of America	15785	17872	21954	16239	15604	16148	15422	15500
Yemen	29837	31590	32364	33312	28576	29990	50090	50000
World	**6501329**	**6756565**	**6720002**	**6669754**	**7036803**	**6540210**	**6483758**	**6422325**

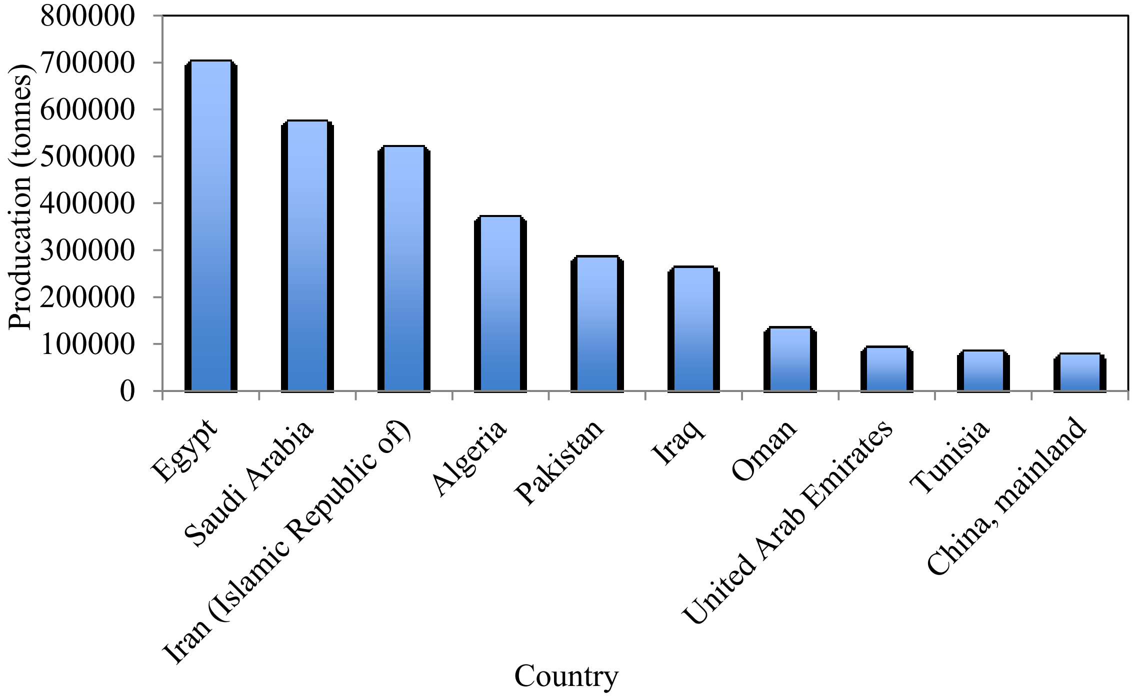


Figure 3.2. The top ten date-producing countries in 2011 (FAO, 2013).

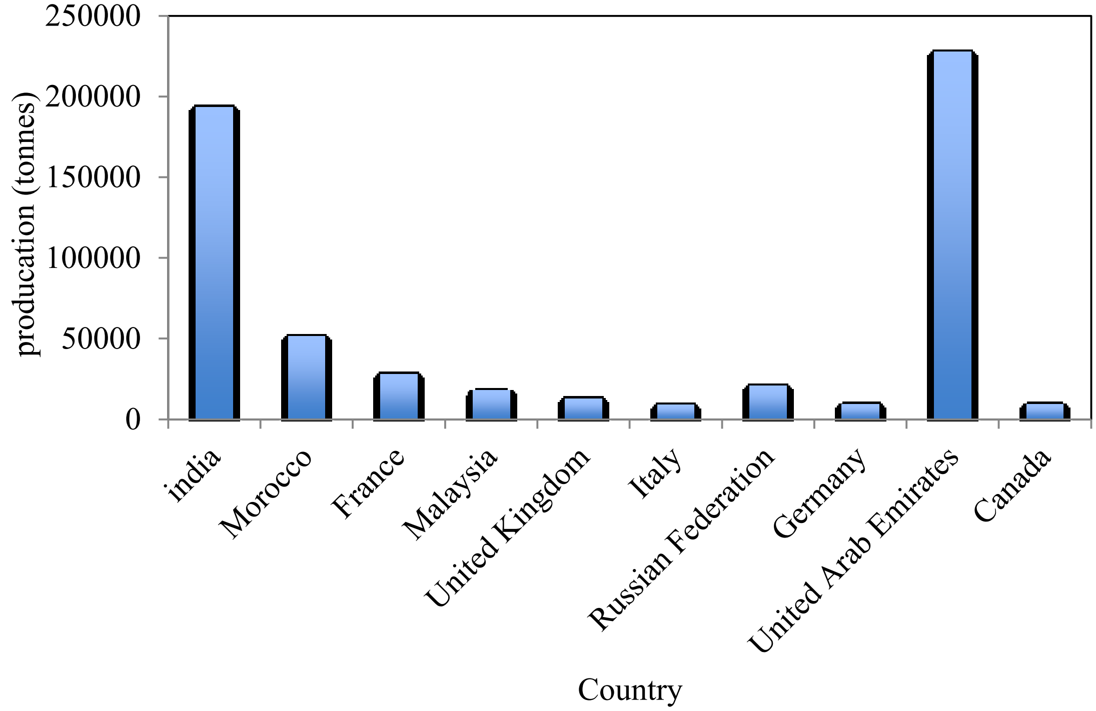


Figure 3.3. The top ten date-importing countries in 2010 (FAO, 2013).

3.2. Botanical Description of the Palm

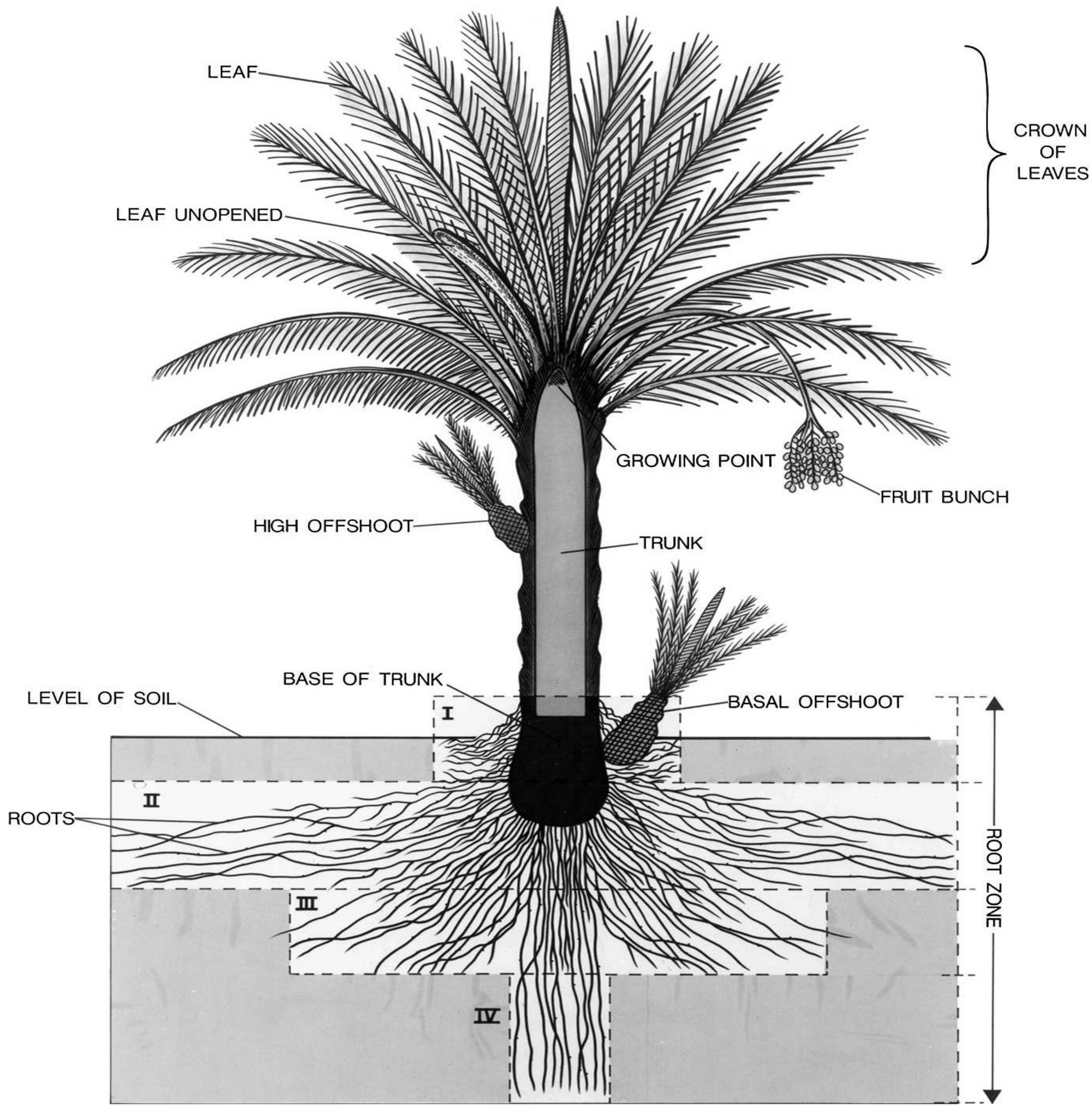
The date palm tree has a root system, a trunk, leaves, florescences and seeds as shown in Figure 3.4.

3.2.1. Root System

The date palm tree is monocotyledon (has a single cotyledon in the seed) with respiratory roots and a fibrous root system (AbdulQadir et al., 2011). Al-Shayeb et al. (1995), Zaid and De Wet (2002), Al-Shayeb et al. (1995) and Barreveld, (1993) divided the root system into four zones as shown in Figure 3.4. The first zone is the respiratory zone, which contains some of the primary and secondary roots and is located near the palm base. The second zone is the nutritional zone, which contains a high percentage of primary and secondary roots and stretches from 0.25 m to 1 m. The function of this zone is the collecton of nutrients and moisture from the soil. In poor soils, the nutrition zone (second zone) will spread into the third zone. The third zone is the water absorbing zone (from underground water) and stretches from 1 m to 2 m. The fourth zone is similar to the third zone. Figure 3.5. shows the roots of a three year old date palm tree.

Osmont et al. (2007) reported that the root system is important for plant growth and survival because of its role in water and nutrient uptake. Waisel et al. (2002) stated that the two primary functions of the root system are acquisition of soil-based resources (water and nutrients) and anchorage and the secondary functions are storage of nutrients and spread and extension of the feeder roots. Hodel and Pittenger (2003) and Carr (2012) found that the good indicators of root growth are the size and the shape of the root, the start zone on the offshoot and the leaf extension.

The roots of the palm tree develop directly from the seed. About 25% of the roots of date palm trees extend from the base of the trunk down into the soil and 75% of the roots extend around the tree. About 50% of secondary roots and offshoots (feeder roots) are concentrated at a depth of 150 cm (Qhaleb, 2008; Al-Obeed, 2005). Waisel et al. (2002) reported that roots are more plentiful in surface soil layers than in deeper layers due to the accessibility of resources in that zone. The roots of the date palm tree can



I: First zone (respiratory roots)
II: Second zone (nutritional roots)
III: Third zone (absorbing roots)
IIII: Fouth zone (absorbing roots)

Figure 3.4. Botanical description of a date palm (Chao and Krueger, 2007).

Figure 3.5. The roots of a three-year-old date palm tree (Al-Jabori and Zayed, 2006).

survive in wet soils for months. However, extended periods of wet conditions will affect the date palm tree and its date production (Zaid and De Wet, 2002). The date palm resists water stress and drought conditions due to the primary roots that allow the plant to use underground moisture.

3.2.2. Trunk

The trunk of the date palm is vertical, cylindrical and extends upward. Shaaban (2012) and Abdul Qadir et al. (2011) steted that the trunk of date palm tree is brown in color and can grow up to 20 meters in height depending on climatic conditions. The date palm is a cotyledon plant that does not contain cambium tissue and does not show secondary growth by the production of concentric annual rings. The trunk is covered by boots from the old leaves as shown in Figure 3.6. The apical bud (phyllophore) is responsible for the growth of the date palm (Qhaleb, 2008). A tree's age can be estimated from the length of the trunk and its special characteristics. Every three petiole segments at the base signify three rows of removed leaves which is equivalent to one year. Several reports mentioned that the length of the date palm depends on type as well as irrigation, fertilization and climate conditions (Al-Shahib and Marshall, 2003; Amer, 1994).

The trunk consists of 45% cellulose, 23% hemicellulose and 32% lignin which make the trunk suitable for plywood manufacturing due to the high amount of cellulose (Al-Jabori and Zayed, 2006). Mansor and Ahmad (1991) reported that that the sugar (glucose, fructose and sucrose) content in the trunk ranged from 2% to 10%. The lignin content was found to be higher in the bottom of the trunk and decrease with increasing tree height.

3.2.3. Leaves

The date palm leaves are pinnately compound with a length at maturity ranging between 2 m to 6 m (Figure 3.7). The average number of leaves produced yearly is between 10 and 20 (Pahsa, 1998). Leaves are divided into three groups depending on their location: (a) outer green leaves, which are photosynthically active and make up 50% of the leaves, (b) fast-growing leaves located in the middle, which make up 10% of the leaves and (c) juvenile leaves, which are white not active photosyntheticaly and are located in the heart (inside) of palm tree and make up 40% of the leaves (Al-Jabori and Zayed, 2006; Zaid and

Figure 3.6. The method used to measure date palm age is shown by the red line (Al-Jabori and Zayed, 2006).

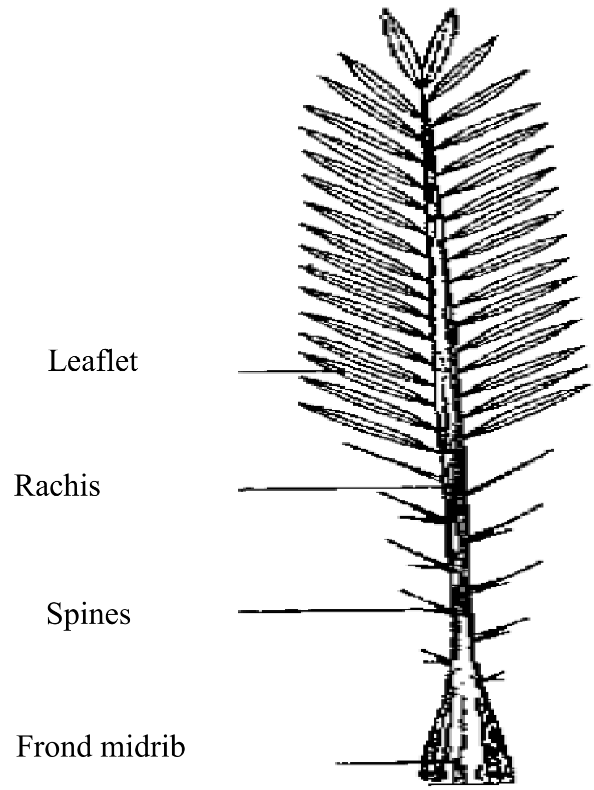


Figure 3.7. The leaf characteristics of a date palm (Zaid and De Wet, 2002).

De Wet, 2002).

The leaf remains green for 3 - 7 years and then dries and loses its green color. The date palm trees do not have the ability to shed their leaves as other trees do and thus the leaves must be removed by humans (Al-Jabori and Zayed, 2006; Kaplan, 1983; Abdul Qadir et al., 2011). The leaf is made of two parts: (a) the leaf blade, which is the top part that contains pinnae area, spines and andrachis and (b) the petiole, which is the bottom part that contains rachis base and fiber sheath. The average thickness of the petiole is between 25 and 50 cm (Al-Jabori and Zayed, 2006; Kaplan, 1983; Davis et al., 1971). Nixon (1951) reported that the palm tree has a special leaf tissue which is thick and tough.

3.2.4. Inflorescence

Azeqour et al. (2002) described palm trees as dioecious. The male flowers are located on a separate tree (a male tree) from the female flowers (located on female tree). Both trees are slow in flowering and fruiting, thus it is not possible to distinguish male trees from female trees until they are around five years old. Figure 3.8 illustrates the differences between male and female flowers. The flower cluster is a group of flowers covered by spathe. The spathe is green at the beginning but later changes to brown and red. The flower cluster contains a spathe that has the flowers inside and a spadix which is made of strands or spikelets. The strand grows inside a spadix that opens under pressure. The male spadix is wider than the female spadix. The length of the spadix is between 25 cm and 100 cm. There are up to 25 spadixes in one female palm tree and more in the male palm tree. The second part of the spadix is made up of approximately 10,000 sessile flowers. The length of strands is 10 - 100 cm. The last part of the spadix is the inflorescence axis, which holds the edge of the strands. Its length is between 25 and 200 cm (Al-Jabori and Zayed, 2006; Zaid and De Wet, 2002; Iqbal et al., 2009; Abul-Soad, 2012; Lim, 2012).

There are three types of flowers: female flowers, male flowers and hermaphrodite flowers. The hermaphrodite flower is similar to the male flower and is made of three carpels, as are natural female flowers, but only one fruit is produced per pollinated flower. If the flower grows, it yields only a small and distorted or sterile fruit. The fruit grows from one fertilized ovule per flower (Al-Jabori and Zayed, 2006; Demason, 1980; Zaid and De Wet, 2002).

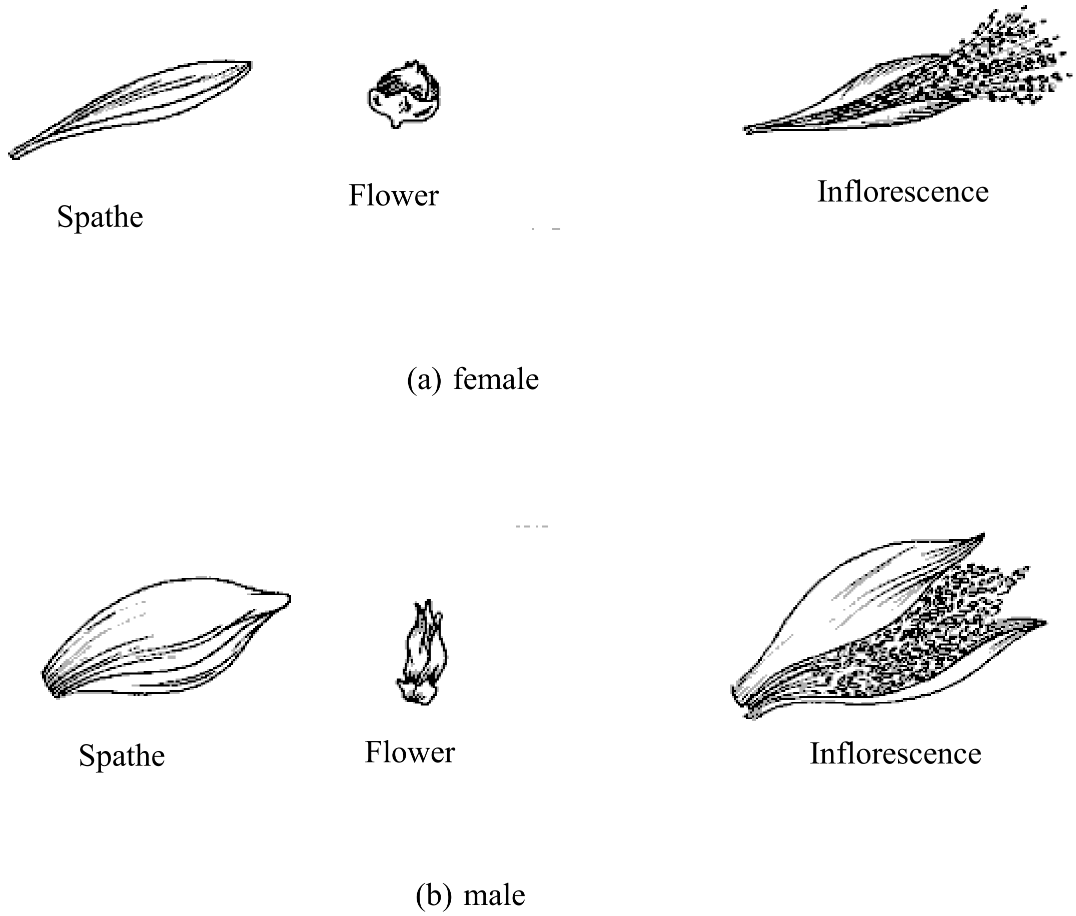


Figure 3.8. Differences between male and female flowers (Barreveld, 1993).

3.2.5. Date Flesh

The fruit is composed of a seed and flesh. The flesh constitutes 85 - 90% of date fruit weight (Elleuch et al., 2008; Hussein et al., 1998).The date palm fruit main saturated fatty acid is lauric acid (15.0 - 17.8%). Also found in the fruit are capric, myristic, myristoleic, stearic, linoleic, palmitoleic (Ibrhim, 2012; Lim, 2012; Besbes et al., 2004).

3.2.6. Seeds

Depending on the type of date palm, seeds vary in the amount of oil, starch and other components. A seed is usually oblong and ventrally grooved with a small embryo and a hard endosperm made of a cellulosic deposit on the inside of the cell walls as shown in Figure 3.9 (Zaid and De Wet, 2002). The weight of the seed is 5.6 - 14.2% of the entire weight of the date fruit and has 6.46% moisture, 5.22% protein, 16.20% fiber, 8.49% fat, 62.51% carbohydrate and 1.12% ash (Al-Shahib and Marshall., 2003; Lim, 2012). The highest mineral concentration is potassium, followed by magnesium, calcium, phosphorus, sodium and iron (Nehdi et al., 2010; Lim, 2012; Beses et al,, 2004). Saafi-Ben Salah et al. (2012) reported that the oil of the date palm seeds contains 18 fatty acids with oleic acid (30.77-42.50%) and lauric acid (18.51-27.48%) as the main unsaturated and saturated fatty acids.

3.3. Growth Stages of the Date Fruit

The growth stages of the date fruit include both internal and external changes. These changes start after fertilization and are usually categorized by the color and chemical composition of the fruit. There are five different stages of date development: Hababouk, Kimri, Khalal, Rutab and Tamar (Zaid and De Wet, 2002).

3.3.1. Hababouk

The Hababouk stage starts after fertilization and features the loss of two unfertilized carpels. The fruit is thus totally covered by the calyx and just the sharp end of the ovary is visible (Figure 3.10). The weight of the fruit is one gram, which is similar in size to a pea. Hababouk last approximately 4 -5 weeks (Zaid and De Wet , 2002; Al- Juburi, 2012; Smarawira, 1983; Zaid and Arias-Jimenez, 1999; Lim, 2012; Golshan Tafti and Fooladi, 2006).

Figure 3.9. Seeds in a date palm fruit (Ibrhim, 2012).

Figure 3.10. The Hababouk stage (Alebidi, 2008).

3.3.2. Kimri

The Kimri stage is the longest and lasts about 9 - 14 weeks, depending on the species. In this stage, the fruit is green, hard and essentially inedible. The average length of the fruit is 27.5 mm and the average weight is 5.8 g. At this stage, the dates contain 5.6% protein, 0.5% fat and 3.7% ash (Smarawira, 1983; Golshan Tafti and Fooladi, 2006). During the first 4-5 weeks of Kimri (Figure 3.11), the average weekly growth is 90%. However, the average weekly growth decreases to 20% during the remaing 5 – 9 weeks, when the fruit size starts to increase from a small green berry to nearly a full-sized fruit (Zaid and De Wet, 2002; Al-Juburi, 2012; Zaid and Arias-Jimenez, 1999; Lim, 2012; Al-Shahib and Marshall, 2003; Golshan Tafti and Fooladi., 2006).

3.3.3. Khalal

In Khalal stage, the fruit starts to change color from green to yellow or red (depending on the date type) as shown in Figure 3.12. This stage continues for up to 5 weeks, depending on the variety. Changes in size and weight, accumulation of sugars and increased acidity due to the drop in water content. In this stage, the length increases to 21mm and the nutrionat content swells to 2.7% protein, 0.3% fat and 2.8% ash. Some varieties such as Barhee, Hallawi, Hayani and Zaghloul can be eaten in this stage,. The average weekly growth is decreased at the end of this stage and the average weight of the fruit increases. (Zaid and De Wet, 2002; Al-Juburi, 2012; Golshan Tafti and Fooladi, 2006; Smarawira, 1983; Zaid and Arias-Jimenez, 1999; Lim, 2012; Al-Shahib and Marshall, 2003).

3.3.4. Rutab

In the Rutab stage, the fruit turns brown or black (Figure 3.13) becomes soft, and the astringent taste disappears. This stage lasts around 2 - 4 weeks. The weight decreases due to loss of moisture and the date fruits become very sweet. The Fruits in this stage of growth must be harvested and stored in cool temperatures to avoid spoilase. (Golshan Tafti and Fooladi, 2006; Zaid and De Wet, 2002; Al-Juburi, 2012; Smarawira, 1983; Zaid and Arias-Jimenez, 1999; Lim, 2012; Al-Shahib and Marshall, 2003).

Figure 3.11. The first 4 -5weeks of the Kimri stage.

(a) Color changing from green to red first phase of Kimri stage.

(b) Color changing from green to red and yellow.

Figure 3.12. Colors in the Khalal stage (Ministry of Agriculture in the Kingdom of Saudi Arabia, 2010).

(a) Hayani dates

(b) Halawi dates

(c) Barhee dates

(d) Zaghloul dates

(e) Majhoul dates (black in color)

(f) Sukari dates (brown in color)

Figure 3.13. Varieties of dates (Ministry of Agriculture in the Kingdom of Saudi Arabia, 2010).

3.3.5. Tamar

In the Tamar stage, the fruit is fully ripened. It is brown or black and the skin is soft in moist dates and hard in dry dates. At this stage, the date has lost a lot of water and the low moisture content prevents fermentation and spoilage. The fruit weight continues to decrease if the harvest is delayed. Dates in the Tamar stage are more suitable for storage (Zaid and De Wet, 2002; Al-Juburi, 2012; Hussien, 2004; Smarawira, 1983; Golshan Tafti and Fooladi, 2006; Zaid and Arias-Jimenez, 1999; Lim, 2012; Al-Shahib and Marshall, 2003; Allaith, 2008).

3.4. Composition of Date Fruit Flesh

Date fruit has been used for centuries as a staple food in Arab countries due to its high nutritive and medicinal values (Mustafa et al., 1983; Besbes et al., 2004). It is very rich in sugar, minerals, vitamins, protein, amino acids, fat, pectin, tannin compounds, antitoxins and pigments (Besbes et al., 2003; Devshony et al., 1992; Borchani et al., 2011). Table 3.2. Shows the nutritional components of dates.

3.4.1 Carbohydrates

Carbohydrates are the major chemical constituents of dates and include: (a) reducing sugars such as glucose and fructose, (b) non-reducing sugars such as sucrose, and small amounts of polysaccharides such as cellulose and starch (Hashempoor, 1999). Glucose ($C_6 H_{12} O_6$), fructose ($C_6 H_{12} O_6$) and sucrose ($C_{12} H_{22} O_{11}$) are the most types of sugars found in the date fruit and their content depends on the date varieties. Bacha et al. (1987) and Eltayeb et al. (1999) noticed that during the Kimri, Khalal and Tamer stages of the date fruit, the total soluble solids and total sugars rise progressively due to the decline in moisture content.

One of the most important commercial significant characteristics for both fresh consumption and fruit processing is the sugar content. Dates comprise about 50 - 88% of their total weight sugar, depending on cultivar, stage of ripening and water content (Hashempoor, 1999). Several studies showed that one kilogram of dates provides 3,000 calories which is equal to 10 kg of meat (Hashempoor, 1999; Rohani, 1988). Depending on the types of sugars, dates can be classifies into two types: reducing sugar-containing dates or sucrose-containing dates (Sawaya et al., 1983). Morton (1987) and Golshan Tafti

Table 3.2. Nutritional components of dates (Balghunaim, 2011).

Components	Percentage
Water (%)	22.5-24.5
Protein (%)	2.3-5.6
Energy (calories)	274
Glucose (%)	44-88
Raw fiber (%)	6.5-11.5
Ash (mg)	1.9
Fat (%)	0.2-0.5
Vitamin A (intl.unit)	50
Calcium (mg)	59
Phosphorus (mg)	63
Iron (mg)	3
Sodium (mg)	1
Potassium (mg)	5.9
Thiamine (mg)	0.09
Riboflavin (mg)	0.10
Niacin (mg)	2.2

and Fooladi (2006) reported that during the Kimri stage, there is a fast decrease in total sugars while sucrose content increases during the Khalal stage. Eltayeb et al. (1999) found that the maximum sugar buildup occurs between the Rutab and Tamer stages which shows high photosynthesis activity, a high rate of photosynthetic translocation and high invertase activity. Borchani et al. (2011) found that the major sugars found in the date flesh samples were fructose, glucose and sucrose.

Ismail et al. (2006) studied five date cultivars grown in the United Arab Emirates (Khalas, Barhee, Fard, Boumaan, Ruzeiz) at the Tamar stage and found that the date pulp contains 68.4 - 76.2% reducing sugar (dry weight basis). The total sugar of date reported by Al-Hooti et al. (1997) was 81.6 - 88.4%, reported by Elleuch et al. (2008) was 72.80 - 79.10%, reprted by Besbes et al. (2004) was 78.30 - 87.55%. The variation of the sugar content is due a difference between the varieties and the local cultural conditions which affect the composition of both the flesh and the seeds (Ismail et al., 2006; Saafi et al., 2008). Table 3.3. shows the total sugar content in four date varieties.

3.4.2. Fiber

Nutritional fiber can be used as an element in food products (dairy, soup, meat, bakery products and jam) to change textural properties and stabilize high fat food. The consumption of 100 g dates daily provides 50 - 100% of the recommended daily amount of fiber (Ashraf and Hamidi-Esfahani, 2011). Borchani et al. (2010) noticed that the fiber amount differs among varieties due to climatic or growing conditions (season, geographic origin) and the use of different analytical methods. Al-Shahib and Marshall (2002), measured the total dietary fiber contents of 13 varieties of dates and found that the percentage of total dietary fiber was in the range of 6.4 - 11.5 % fresh weight, depending on variety and degree of ripeness. The water activities (aw) of the fiber ranged from 0.17 - 0.195 (Borchani et al., 2010; Ahmed et al., 2013). Borchani et al. (2010) reported that the total fiber contents vary from 8.09% in Deglet Nour to20.25 % in Kentichi.

3.4.3. Minerals

Dates are known to be a reasonably good source of 15 minerals (Hui, 2006; Ibrahim et al., 2001). The concretion of each mineral in dried dates varies from 0.1 to 916 mg/100 g date, depending on the type of mineral and type of date (Al-shahib and Marshall, 2003). Potassium is found in dates in large quantities (dates are considered an important source

Table 3.3. The total sugar content in four varieties (Chaira et al., 2007; Borchani et al. 2010).

Date Variety	Total Sugar (% dry matter)
Deglet Nour	72.82±0.25
Alig	60.28±0.59
Bajo	79.93±0.31
Goundi	84.79±0.91

source of potassium), followed in descending order by phosphorous, magnesium, sodium and iron (Al-shahib and Marshall, 2003; Al-Farsi and Lee, 2008). Eating seven dates (approximately 100 g) provides an adult person with the daily requirements of for magnesium, manganese, copper and sulfur, half of the daily need for iron, and a quarter of the daily need for calcium and potassium (Ali et al., 2008; Al-Shahib and Marshall, 2003; Al-Farsi and Lee, 2008; Ahmed et al., 1995).

Al-shahib and Marshall (2003) found that potassium can be found in many varieties at a concentration as high as 0.9% in the fruit flesh. Raynes et al. (1994) and Chaira et al. (2007) reported that the minerals in date flesh were potassium, calcium, phosphorus and manganese. Gasim (1994) reported that the iron content in date fruit is high, but zinc and copper are relatively low. On average, dates contain a higher percentage of potassium, phosphorous and iron than other types of fruit. Al-Hooti et al. (2002) and Borchani et al. (2010) stated that the mineral composition were relatively low in sodium but rich in potassium. The fluorine in the fruit is useful for preventing tooth decay. Also, the selenium content of date has beneficial effects for preventing cancer and strengthening the human immune system (Al-shahib and Marshall, 2003). Al-shahib and Marshall (2003) and Raynes et al. (1994) reported that the differences in the minerals content are due to variety, soil type and amount of fertilizer.

3.4.4. Vitamins

Dates in the Khalal stage have considerable amounts of ascorbic acid and β-carotene (Hui, 2006). Al-shahib and Marshall (2003), Balghunaim (2011) and El-Sohaimy and Hafez (2010) reported that dates contain six vitamins: vitamin A, B1 (thiamine), B2 (riboflavin), niacin (nicotinic acid), vitamin C and folic acid. The dates are an ideal food because they offer important nutrients and potential health benefits as reported by El-Sohaimy and Hafez (2010). Table 3.4 shows the vitamin content of dates.

3.4.5. Enzymes

Enzymes play a role in the change processes that takes place during the growth and ripening of the date fruit. There are four enzymes that have an impact on the final product quality: (a) invertase, which is responsible for the inversion of sucrose into glucose and fructose, (b) polygalacturonase, (c) pectinesterase, which converts insoluble pectin to

Table 3.4. Vitamin content of dates (Balghunaim, 2011; Al-Shahib and Marshall, 2003).

Vitamin	% 100 / gm
Vitamin A	4.8 - 6
Vitamin C	0.77 - 2.7
Vitamin B1	0.07 - 0.1
Vitamin B2	0.03 - 0.05
Vitamin B3	0.33 - 2.2
Folic acid (folacin)	0.004 - 0.007
(Niacin)	0.0004 – 0.0007

soluble pectin and (d) cellulase which helps in the conversion of cellulose to simpler compounds (Al-shahib and Marshall, 2003). Abbès et al. (2011) reported that pectinase and cellulase reduce the weight of the food molecules in three varieties of date (Deglet Nour, Kentichi and Allig). Kanner et al. (1978) found that invertase activity occurs at the late stages of fruit development and the marked differences in invertase activity between two cultivars higher in Khadrawi fruits than in Deglet Noor. The difference may be attributable to genetic features. During ripening, the cellulase activity of dates rises almost 2 - 4 times higher than in other fruits (El-Zoghbi, 1994).

3.4.6. Protein

Dates contain about 1 - 7% protein and the important amino acids needed by the human body. Ashraf and Hamidi-Esfahani (2011) reorted that the protein content of dates is highest in the Kimri stage (5.5 - 6.4%). Date protein contains 17 - 23 types of amino acids, some of which are not present even in more popular fruits such as oranges, apples and bananas. The amino acids found in some date cultivars include: lysine, histidine, arginine, aspartic acid, threonine, glutamic acid, erine, prolin, glycine, alanine, cystine, valine, methionine, isoleucine, leucine, styrosine, and phenylalanine (Ashraf and Hamidi-Esfahani, 2011; El-Sohaimy and Hafez, 2010). Al-Shahib and Marshall (2003) reported 800 times more isoleucine in dates than apples and 2000 times more lysine than in apple and about 5000 times more of lysine than in orange. Table 3.5 shows the types of amino acids.

3.4.7. Fat

According to Al-Shahib and Marshall (2003), fat is more important for the protection of the fruit than for its nutritional value. Date fruit contains a small amount of fat which usually decreases from 0.5% at the Kimri stage to 0.1% at the Tamer stage (Ashraf and Hamidi-Esfahani, 2011). Sahari et al. (2007) reported that the fat differs from 0.22 % to 0.62 % (fresh weight basis). The results obtained from several reports indicate that the amount of the lipids in date flesh were almost similar (Boukouada andYoussif, 2009; Ahmed et al., 1995; Aidoo et al., 1996; Al-Shahib and Marshall, 2003; Besbes et al., 2004; Sahari et al., 2007). Table 3.6. shows the amount of fat in date flesh.

Table 3.5. The types of amino acids in date flesh (Chaira et al., 2007; Borchani et al., 2010).

Flesh	Amino acid (mg/100 g dry date)
Alanine	8 – 342
Arginine	2 – 261
Aspartamine	230 – 450
Aspartic acid	2 – 467
a-Amino butyric acid	266 – 337
Cysteine	11 -114
Cytine	0.73 – 122
Glutamine	65 – 87
Glutamic acid	40 -631
Glycine	4 – 349
Histidine	0.1 – 76
Isoleucine	0.2 - 465
Leucine	0.5 – 264
Leucine and isoleucine	254
Lysine	3 – 282
Methionine	0.2 – 219
Phenylalanine	0.8 - 173
Proline	12 – 369
Serine	6 – 238
Threonine	1 – 264
Tryptophan	100
Tyrosine	1 – 181
Valine	0.5 - 271

Table 3.6. The amount of fat in date flesh (Sahari et al., 2007)

Variety	Fat g/100 g*
Zahedi	0.28 ± 0.050
Khanizi	0.368 ± 0.040
Halilei	0.323 ± 0.020
Barhi	0.272 ± 0.060
Maktoom	0.339 ± 0.040

*(fresh weight basis)

3.4.8. Antioxidants

Antioxidants play an important role in minimizing or inhibiting the risk of many human diseases (Benzie, 2003; Kris-Etherton, 2004; Allaith, 2008). Ragab et al. (2013) studied the protective effect of Madinah Ajwa against the toxic effects of lead acetate poisoning. Al-Farsi et al. (2005) reported that the majority of antioxidants and carotenoids. It can be used as functional food or as one of its ingredients (Mansouri et al., 2005; Al-Farsi et al., 2005). Al-Farsi et al. (2008) reported that fresh dates are a rich source of antioxidants such as trolox and anthocyanins. Al-Farsi and Lee (2008) added the carotenoids to the list of antioxidants.

Several researchers studied the antioxidants activity of date fruit from Oman (Al-Farsi et al., 2005), Kuwait (Vayalil, 2002), Saudi Arabia (Allaith, 2008) and the USA (Vinson et al., 2001) and found them to have a positive impact on heath. Saleh et al. (2011), Vayalil (2002) and Al-Humaid et al. (2010) stated that date fruit can be considered a rich source of hydrophilic antioxidants and its decrease was related with the presence of polyphenols specially flavanols and phenolics.

Biglari et al. (2008) stated that in Kimri stage, the phenolic compounds are high and they decrease in Tamer stage. Gil et al. (2002) stated that the differences in antioxidants are due to the factors affecting the antioxidant properties include genetics, growing and environmental conditions, diseases and processing. According to Ragab et al. (2013), when the date fruit has a dark color it contains more antioxidants (Table 3.7).

3.5. Composition of Date Seeds

The date seed weighs 5.6 – 14.2 % of the date fruit depending on the type of date and contains carbohydrates, fibers, protein, minerals, vitamins and fat (Al shahib and Marshall, 2003).

3.5.1. Carbohydrates

Besbes et al. (2004), Hussein and El-Zeid (1975) and Devshony et al. (1992). reported that the date seeds contain 83.0% carbohydrates. Al-Whaibi et al. (1985) studied four types of date seeds (Sukkeri, Khedhri, Nabt- Saif and Menifi) and found Sukkeri to contain higher sugar (fructose, glucose and sucrose). The dates carbohydrates are made of about 42% cellulose, 18% hemicellulose, 25% sugar and other compounds,

Table 3.7. Individual phenols analyzed by HPLC (mg/kg) (Saleh et al., 2011).

Phenol type	Date Varieties		
	Ajwa	Sukhari	Khalas
Rutin	6.50 ± 1.70	8.10 ± 0.20	3.60 ± 0.07
Catechin	7.30 ± 0.14	7.50 ± 0.70	5.00 ± 0.18
Caffeic acid	5.70 ± 0.77	5.40 ± 0.64	7.40 ± 1.61

11% lignin, and 4% ash. The moisture content is about 5- 10 % (Ahmed and Theydan, 2012).

3.5.2. Fiber

Date seed contains 10% - 20% crude fiber (Boukouada and Yousfi, 2009). According to Al-Farsi and Lee (2008) the total dietary fiber in date seeds was 57.87 g/100g. Al-Farsi et al. (2007) reprted total dietary fiber of 80.2 g/100g. These differences dietary fiber content is due to the stage of maturation and different varities.

3.5.3. Protein

Akasha et al. (2012) found that the yield of protein in date seeds is between 27 and 52%. Protein content is usually determined by the Kjeldahl method (Besbes et al., 2004; Barminas et al., 1999). The protein of Siwi date seeds was 7.37% which is similar to that of Ruzeiz date seed (Sawaya et al., 1984; Nezam El-Din and Abd El-Hameed; 1997). Saafi-Ben Salah et al. (2012) reported that the date seeds contain considerable amounts of lipids and proteines. Nezam El-Din and Abd El-Hameed (1997) reported that the date seed contains different types of amino acids including threonine, Valine, cysteine, methionine and Leucine.

3.5.4. Minerals

Besbes et al. (2004) studied the date seed of two verities (Deglet Nour and Allig) and found them to contain high amounts of magnesium (51.7 % in Deglet Nour and 58.4 % in Allig). Calcium, phosphorus, sodium, potassium, aluminum, cadmium, sulfur and lead are also present in date seeds (Besbes et al., 2004; Ali-Mohamed and Khamis, 2004, Baliga et al., 2011).

3.5.5. Vitamins

Seeds contain vitamin C, A and E (Besbes et al., 2004). riboflavin (B_{12}), thiamine(B_1) (Al Faris and Lee, 2008), biotin and ascorbic acid (Baliga et al., 2011).

3.5.6. Fat

The amount of lipid contents in date seeds is 5.05 - 12% (Boukouada and Yousfi, 2009). Sawaya et al. (1984) and Besbes et al. (2004) reported that date seed content has only a small amount of oil compared to olive, sunflower and cotton seeds, which are generally in the range of 30 - 45%. Oil from date seeds can be used in production of health products as well as industrial and pharmaceutical applications. The major fatty acid found was oleic acid (Besbes et al., 2004; Devshony et al., 1992). Table 3.8. show the characteristics of date seed oil.

3.6. Utilization of the date palm

Traditionally, dates have been eaten fresh or used in making food products. Figure 3.14 shows the derived date products. With the steady increase the cultivation of date palms waste production is becoming a major envirmental concern (Al-Shahib and Marshall, 2003). The date waste from palm trees contain numerous nutritional elements that can be used as feed for human consumption or animals. The leaves can also be used as construction materials (Bassam, 2008; Al-Shahib and Marshall, 2003; Mustafa et al., 1983; Besbes et al., 2004).

3.6.1. Date Fruit

Most people consume the date as a fruit. Eating dates is by far the most popular usage of the date fruit. In preparing date fruit for mass consumption, workers harvest the fruit and then transport it to a factory. There, the dates are weighed, cleaned and exposed to methyl bromide gas (insecticide) for four hours. The dates are classified according to shape, size and color, then washed with water and dehydrated in order to prevent rot. Finally, the dates are stored at $3 - 11$ ℃ and sent to market (Barreveld, 1993; Zaid and De Wet, 2002; Alhmdainm, 2000).

3.6.2. Date-based Products.

The most common edible products from the date palm are date dough, date syrup, molasses and food supplements and health products.

3.6.2.1. Date Dough: Date dough can be made by removing the seed from the date without the loss of date pulp. Factories use lower quality dates to make date dough which can later be used to make molasses, cookies, breakfast cereals and baby food (Harlow, 1985; Frame, 1994). Balghunaim (2011) reported that the date dough contains 22.5% water, 2.3% protein, 1.9% ash, 0.2 - .05% fat and 44 - 88% sugar.

3.6.2.2. Date Syrup or Molasses: Date molasses or syrup is known as a thick sugar extracte from the fruit of date palms. Almost all varieties of dates are useful for extracting molasses

Table 3.8. Characteristics of date seed oil and selected vegetable oils (Devshony et al., 1992).

Analysis	Date seed	Palm	Coconut
Iodine value (%)	-	53.3	9.5
Saponification value (%)	221.0	195.7	256.0
Caprylic C_s (%)	-	-	8.0
Capric C_{10} (%)	0.3		7.0
Lauric C_{12} (%)	21.8	0.2	48.2
Myristic C_{14} (%)	10.9	1.1	18.0
Palmitic C_{16} (%)	9.6	44.0	8.5
Stearic C_{18} (%)	1.5	4.5	2.3
Oleic $c_{18:1}$ (%)	42.3	39.2	5.7

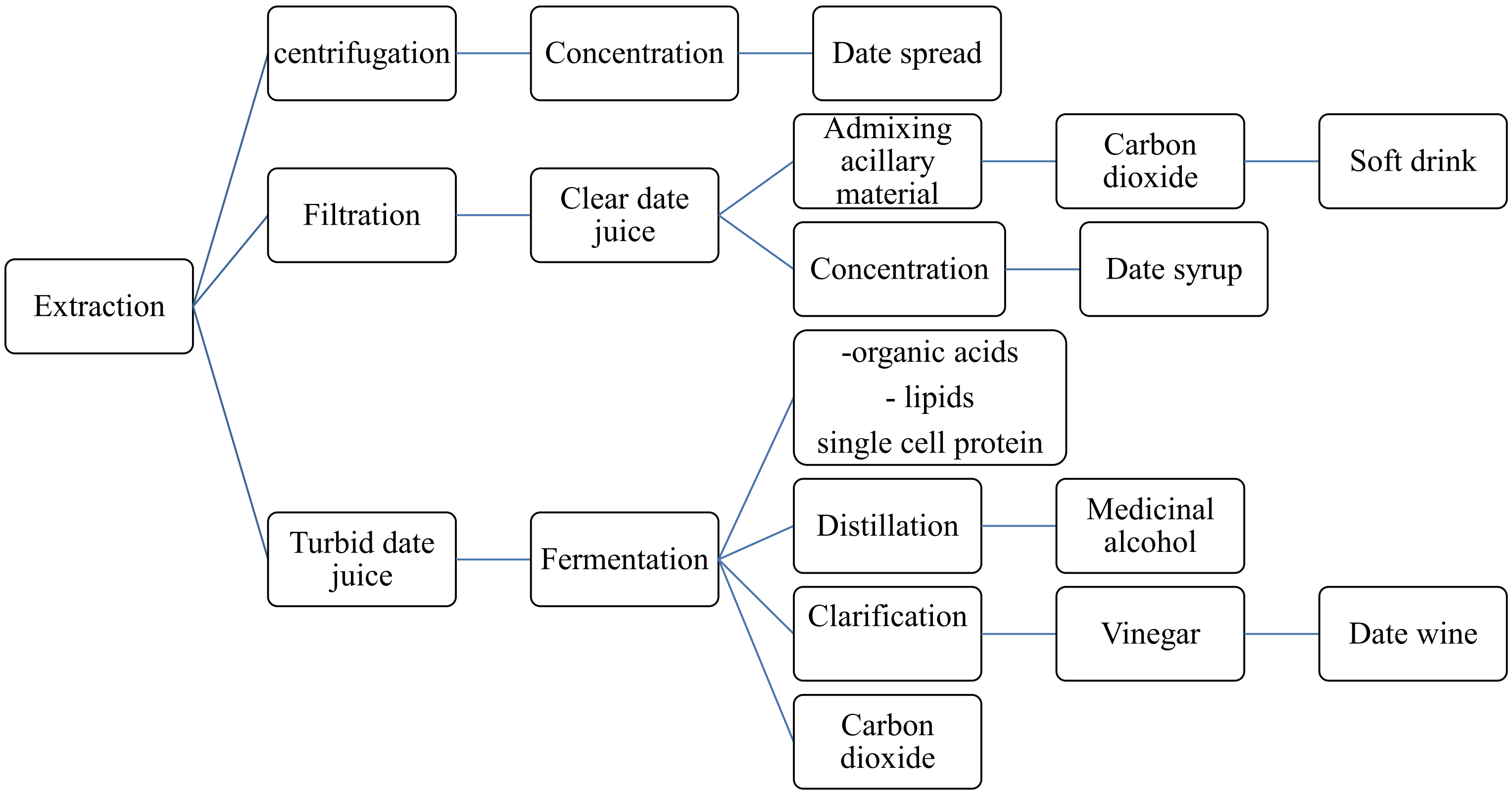


Figure 3.14. Shows the derived date products (Ashraf and Hamidi-Esfahani, 2011)

(Zaid and De Wet, 2002; Aleid, 2011; Al Eid, 2006). Most people simply eat the molasses pure or add sesame oil to it. Molasses can also be added as a substitute for sugar to many food products such as bread, cakes, biscuits, soft drinks and ice cream. Hamad et al. (1983) used date syrup as a sweetener in ice cream. Yousif et al. (1996) mixed the date syrup with milk. Al Eid (2006) reported that date molasses consisted of 13.5% moisture, 81% sugar (sucrose 1%, fructose 41% and glucose 39%), 1.5% ash, 2.2% protein and 1.8% pectin. Al Eid (2006) and Sawaya et al. (1983) stated that date molasses has high amounts of potassium, sodium and calcium.

3.6.2.3. Food Supplements and Health Products: Chaira et al. (2007) reported that dates are a source of vitamins including: ascorbic acid (2.4–17.5 mg/100 g), thiamine (0.08–0.13 mg/100 g) and riboflavin (0.13–17.5 mg/100 g). Alhamdain (2000) reported on the production of date capsules for use as vitamins and dietary supplements. Phenolic compounds of date seeds (mainly phenolic acids and flavonoids) have been shown to possess benefits as antioxidants (Peterson and Dwyer, 1998), anti-carcinogenics (Bailey and Williams,1993; Block, 1992), antimicrobials (Takechi et al., 1985), anti-mutagenics (Liverio et al., 1994) and anti-inflammatories (Landolfi et al., 1984) and have the ability to inhibit cancer (Ishrud and Kennedy, 2005). They have also been shown to treat cardiovascular diseases (Diplock et al., 1998; Halliwell, 1997). Polysaccharide aqueous and ethanol extracts of date were effective in mitigating gastric ulcers in rats (Al-Qarawi et al. 2005). Puri et al. (2000) reported that date extract stimulates the immune system of women after childbirth. The antibacterial and antifungal properties of date fruits were also investigated by Sallal and Ashkenani (1989) and Shraideh et al. (1998). Another study by Bastway Ahmed et al. (2010) on rats showed that the ameliorative activity of aqueous extract from date flesh (Phoenix dactylifera L.) and ascorbic acid on thioacetamide-induced hepatotoxicity. This study suggests that thioacetamide-induced liver damage in rats can be ameliorated by the administration of the extract of date flesh and ascorbic acid. Almana and Mahmaud (1994) evaluated date seed as an alternative source of dietary fiber in comparison with wheat bran and suggested that they may provide a valuable contribution to dietary fiber intakes.

3.6.3. Livestock Feed

Vandepopuliere et al. (1995) reported that in the Middle East, date seeds are already used in animal feed. After the production of molasses, the remaing material can be used as animal feed. Following the extraction of oil from date palm seeds, the remaining biomass can be used to feed animals as a source of fiber (Barreveld, 1993; Besbes et al., 2004; Amani et al., 2013).

Date leaves are used as a source of roughage in rations for fattening goats. Bahman et al. (1997) reprted that the date palm leaflets are used as a roughage for dairy cows in Kuwait (Table 3.9) Despite the very low nutritive value of date palm leaves (DPL), it may be an acceptable alternative to barley straw in high concentrate diets for dairy cows, since low quality roughages contribute little to the energy value of the feed and their primary function is to stimulate rumen activity. Dayani and Malmstadt (2012) stated that date palm pulp has the potential as an alternative feed resource for small ruminant nutrition.

3.6.4. Vinegar

Vinegar is an acetic acid solution diluted with water. Several researches (Aleid, 2011; Ashraf and Hamidi –Esfahani, 2011 and Chao and Krueger, 2007) reported on the production of vinger from the sugary solution of date by alcoholic fermentation.

3.6.5. Yeast Production

The yeast *Saccharomyces cervisiae*, can be cultivated by aerobic fermentation of sugars and other carbon sources such as date syrup. Aleid (2011) reported that the dry matter of the yeast cell is 40 - 45% protein, 39% carbohydrates, 7% lipids, 6 - 10% minerals. Aleid et al. (2012) found that to manufacture 1 kg of yeast, about 3 mg D-Biotin, 150 mg D-Pantothenic acid and 2 g m-Inositol, 2.5 kg of the date syrup or 3.4 kg of the molasses are needed.

3.6.6. Paper Production

Khiar et al. (2009) investigated the valorisation of two lignocellulosic materials largely available in Tunisia as a source of cellulosic fibres namely: Posidonia oceanica balls and date palm. The studies showed that the amount of holocellulose, lignin and cellulose found in the date palm was similar to those found in softwood and hardwood. Table 3.10 shows the number of palm trees needed to produce wood.

Table 3.9. The mean milk, composition, live weight gain and food intake of cows fed either barley straw or date palm leaves as roughage (Bahman el al., 1997).

Contents	Barley Straw	Date Palm Leaves
Milk yield (kg/day)	17.0	16.0
Milk fat (%)	34.9	31.7
Milk protein (%)	35.6	34.7
Live weight Gain (kg/day)	0.72	0.66
Feed intake		
Roughage (kg/day)	4.4	4.1
Total (kg/day)	16.9	16.6

Initial cow weight = 356 kg

3.6.7. Bio-ethanol

Fuel in the form of alcohol ethanol CH_3CH_2OH produced from plant starch and cellulose from crops such as sugar cane, corn and potatoes is known as bio-ethanol fuel. It can replace gasoline directly in gasoline engines after an amendment to the engine (Balat and Balat, 2009; Algaym, 2007; Cardona and Sanchez, 2007; Coté et al. 2004). Bio-ethanol is one of the most important discoveries in the field of alternative energy that aims to reduce the amount of toxic gases rising from car exhaust which are harmful to the ozone layer and cause global warming. The bio-ethanol plays a role in reducing 50% emissions of carbon dioxide in the atmosphere (Bassam, 2008).

Bio-ethanol can be produced from the sugary material of fresh date fruit or the cellulosic parts of the trunk of palm tree. A series of biological processes including hydrolysis and fermentation by the microorganisms converts sugar into ethanol molecules (Bassam, 2008; Aleid, 2011). Aleid (2011) reported that 1 metric ton of dates can produce 300–400 kg of bio-ethanol which is about 380–500 L. Table 3.11 shows the amount of bio-ethanol produced from one tonne of raw material.

3.6.8. Bio-diesel

Fuel made from vegetable oils extracted from soybeans, palm or other plants is known as biodiesel. It may be used directly or mixed with diesel in diesel engines (Moradi and Ismail, 2007). Palm oil contains fatty acids including: acid albalmtk, citric acid, miricitric acid, oleic acid and linoleic acid. Palm oil can be used for the production of bio-diesel which is useful in reducing carbon dioxide emissions to the atmosphere (Devshony, 1992).

3.7. Date Waste Production and Utilization

3.7.1. Wash Water

Water is used in various date processing operations including soaking, washing, rinsing, blanching, heating, cooling, sanitation and disinfection purposes. Ashraf and Hamidi-Esfahani (2011) reported that date processing starts with removing the cap and in some cases the pits of the dates. Washing is done by automatic machines with water sprays to remove dust and any foreign materials using clean water. Reuse of date processing water has been primarily limited to non-food uses such as irrigation of palm trees, general facility

Table 3.10. The number of palm trees needed to produce wood (Mansour, 2010).

Raw Material Used	Number of Palm Tree Needed
100% palm tree waste	3,650,000 Palms
90% palm tree waste + 10% wood waste	3,300,000 Palms
80% palm tree waste + 20% wood waste	2,900,000 Palms
70% palm tree waste + 30% wood waste	2,500,000 Palms
60% palm tree waste + 40% wood waste	2,200,000 Palms
50% palm tree waste + 50% wood waste	1,800,000 Palms

Each one m^3 from MDF needs to 73 palm tree waste
MDF= Medium density fiber board.

Table 3.11. The amount of bio-ethanol produced from one tonne of raw material (Bassam, 2008).

Raw Material	Sugar Content (%)	Bio-ethanol Produced (t/l)
Sugarcane (seasonal crop)	13	60
Beet (seasonal crop)	18	116
Dates (permanent crop)	65%	280

cleaning, cooling process and fire extinguishing purposes (Katsuyama, 1979 and Casani et al., 2005).

3.7.2. Under-Utilized Date

The under utilized date seem to exhibit promising properties that can open new pathways for the efficient production of cost-effective xanthan gum. Therefore, they can be considered as a strong candidate for future industrial and commercial applications related to xanthan gum (Ben Salah et al., 2010; Moosavi-Nasab et al., 2010) and production of activated carbon (Haimour and Emeish, 2006). The date can also be an inexpensive source of sugars (reducing sugars such as fructose and glucose), natural antioxidants and dietary fiber (Al-Farsi and Lee, 2008). Sánchez-Zapata et al. (2011) reported on addition of date paste (15%) to a cooked meat for production of bologna with lower fat content and higher dietary fiber content.

3.7.3. Date Seeds

The date palm seed is a solid waste from the date production industry and date consumption. The annual seed production accounts for about one million tonnes (Mirghani, 2012). Pulverized and ground date seeds are being used on a small scale on dirt roads as a replacement to base gravels. The date seed oil can be used in cosmetics (Besbes et al., 2004). Finding a way to make a profit from the seeds would substantially benefit date farmers.

3.7.4. Date Leaves

Date palm leaves could be used in water and wastewater treatment. The leaves have a fibrous structure with four types of fibers: leaf fibers in the peduncle, baste fibers in the stem, wood fibers in the trunk and surface fibers around the trunk. Riahi et al. (2009) showed that using date-palm fiber in filtration could be a potential technology for tertiary wastewater treatment as it provides a green engineering solution. Figure 3.15. shows the date palm fiber filter set-up. Elderly leaves are used to make fans, traditional ceilings and walls in rural area. The palm leave waste is also used to make compressed wood because of its high cellulose and lignin contents. The sticks are used to make furniture, fishing nets, baskets, boxes, children’s cradles and ropes (Barreveld, 1993; Ibrhim and Nadefe, 2004). The soft growing point or terminal bud of date tree leaves been used as food because of

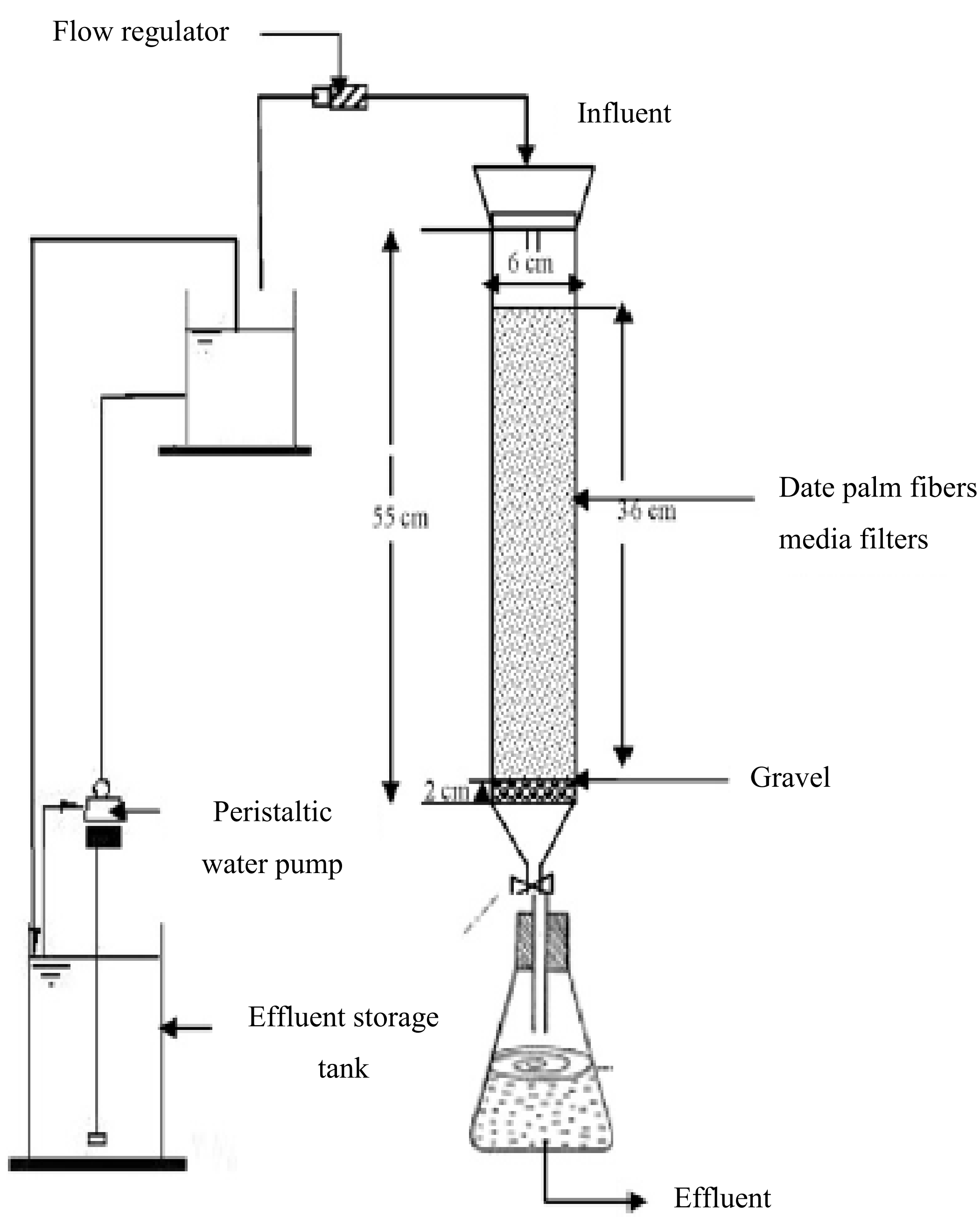


Figure 3.15. Date-palm fiber filter set-up (Riahi el al., 2009).

their sweet taste (added to a salad).

3.7.5. Date Trunk

Humans have been using wood for centuries for thermal power generation and construction of housing. Currently, there is now a lack of wood in many countries which is prompting researchers to look for wood alternatives. The palm tree is being used to develop wood panels or medium density fiber board (MDF) (Mansoor, 2010). The trunk can also be used in the ceiling or in other aspects of housing construction. Farmers use the trunk as a tunnel to transport water around their farms Alkoaik et al. (2011) reported that the date palm trunk can be converted to fertilizer and soil conditioner.

3.8. Antioxidants

Oxidation occurs as result of a chemical process in which electrons are transferred to an oxidizing agent. According to Scandalios (2004) and Halliwell and Cross (1994), all aerobic organisms exist in an environment in which they are faced with exposure to reactive oxygen radicals. These reactions can occur in the body as a result of metabolic processes that result in the reactive elements such as superoxide radicals, hydroxyl radicals and peroxyl radicals. The date fruit is rich in phenolics, carotenoids, sterols and flavonoids (Baliga et al., 2011). The amounts of these chemicals depend on the date variety, stages of picking and the environmental conditions. Frei and Higdon (2003), Scalbert et al. (2005) and Willcox et al. (2004) reported that date antioxidants play an important role in human health.

3.8.1. Classification of Antioxidants

Antioxidants are divided in to two basic categories based on their sources: synthetic and natural. The synthetic antioxidants such as butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) are compounds with phenolic structures of various degrees of alkyl substitution. The natural antioxidants such as (tocopherols, flavonoids, and phenolic acids), nitrogen compounds (alkaloids, chlorophyll derivatives, amino acids, and amines), or carotenoids as well as ascorbic acid) are divided to eight groups: vitamins, flavonoids anthocyanidins, flavon-3-ols, flavones, flavan-3-ols, flavnones, aflavins and hydroxycinnamates (Rice-Evans et al., 1997, Larson, 1988; Pratt and Hudson, 1990; Hall and Cuppett, 1997; Velioglu et al., 1998, Halifeoglu et al., 2003). The antioxidants can also be divided into two groups based on their action. The first group is made of chemical substances that cut the propagation from the radical chain by hydrogen by donation of electrons to the radicals (Figure 3.16). The molecule that loses an electron to the free radicals causes on-going chain reaction. Such mode of action is demonstrated by tocopherols, gallusans, and hydrochinons. The second group has a synergistic approach of action (Grajek et al., 2005). It contains oxygen scavengers and chelators that bind the ions involved in free radical formation. Their activity consists of hydrogen delivery to phenoxy radicals that leads to the reconstitution of the primary function of antioxidants. This group includes citric acid, amino acids, flavonoids, β-caroten and selenium.

Chaira et al. (2009) reported that the korkobbi date of Tunisia contains a high amount of flavonoids. Rice-Evans et al. (1997) reported on the importance of flavonoids, phenylpropanoids and phenolic acids in dates as antioxidants. Hong et al. (2006) studied the flavonoids in the deglet noor date variety in the khalal stage and presented the structure shown in Figure 3.17. Boudries et al. (2007) reported that dates contain lutein, β-carotene and neoxanthin. Di Mascio (1991) reported that carotenoids are soluble pigments and essential sources of vitamin A that can protect the cell from deleterious effects. The author presented the structures of carotenoids shown in Figure 3.18.

3.8.2. The Benefits of Antioxdants

The use of antioxidant supplements has shown some improvement in health (Khare, 2007). Many studies show that consumation of fruit and vegetables (rich in antioxidants) can result in a lower cancer rate (Bjelakovic et al., 2004, Weisburger, 1991, Van Poppel, 1996, Rice-Evans et al., 1997 and Valko et al., 2007). Free radicals can cause cancer and cardiovascular and other diseases that the antioxidants have the ability to prevent as shown in Figure 3.19. Figure 3.20 shows pharmacological activities of date fruit (Shklar, 1998; Surh, 1999; Kris-Etherton et al., 2004; Ferrari and Torres, 2003).

Nordberg et al. (2002) found that antioxidants treated neurological disease (Alzheimer). Tapas et al. (2008) sated that the flavonoids have the ability to protect health from chronic disease. Santosa and Jones (2005) found that antioxidants can reduce or prevent ocular disease. Bauza et al. (2002) reported that the consumption of the date fruit by itself can inhibit wrinkle formation and in the skin.

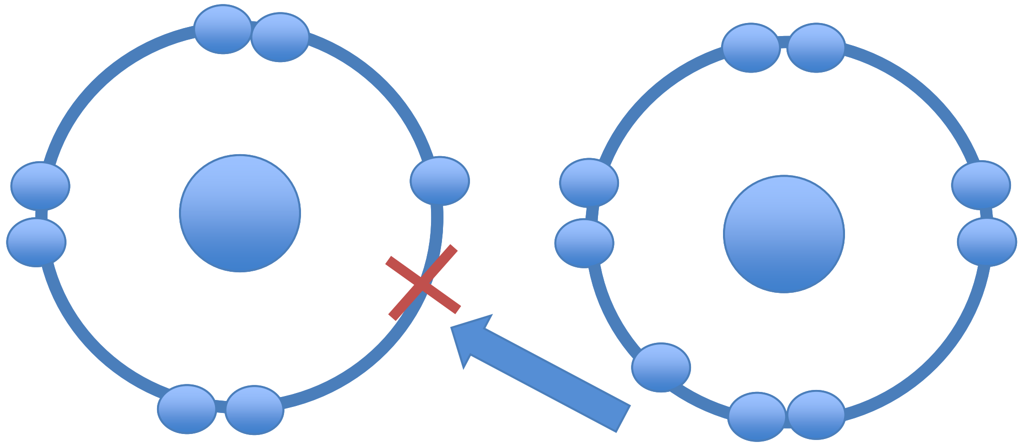
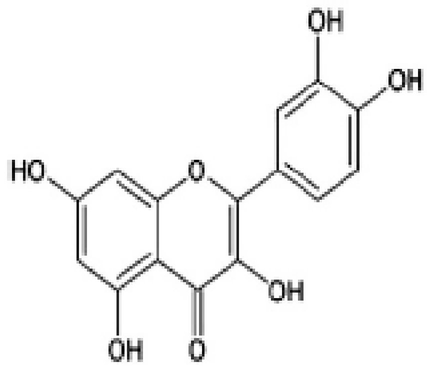
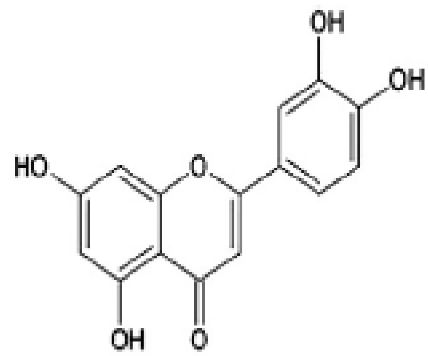


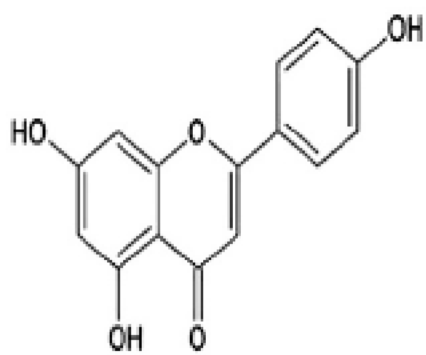
Figure 3.16. Antioxidant molecule donating an electron to a free radical (Keeley, 2014).



Quercetin

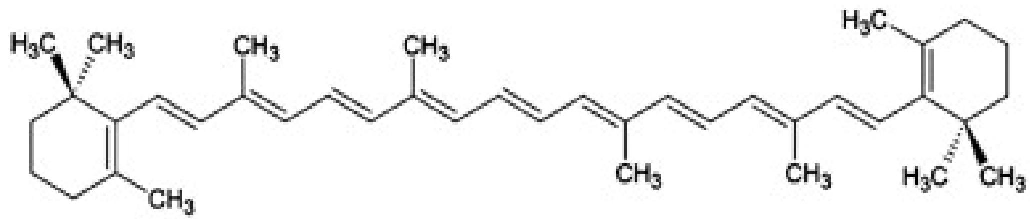


Lutecolin

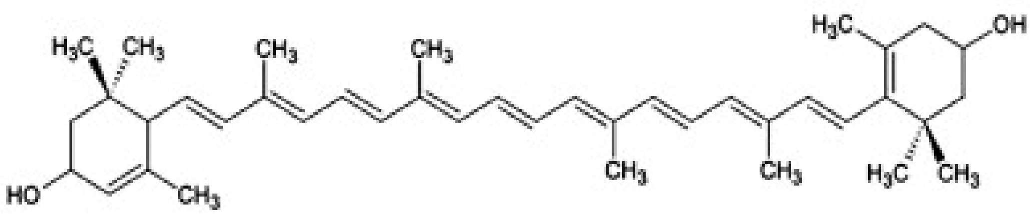


Apigenin

Figure 3.17. Structures of flavonoid in Deglet Noor (Baliga et al., 2011).



β-Carotene



Lutein

Figure 3.18. Structures of carotenoids (Baliga et al., 2011).

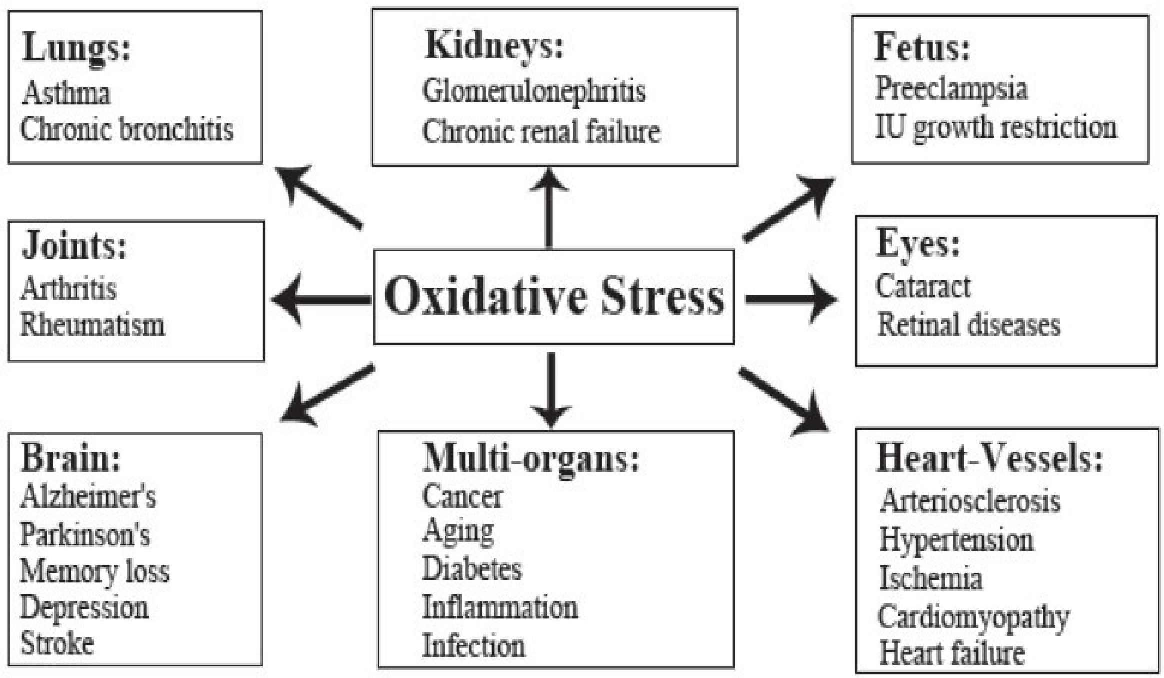


Figure 3.19. Oxidative stress-induced in humans (Pham-Huy et al., 2008).

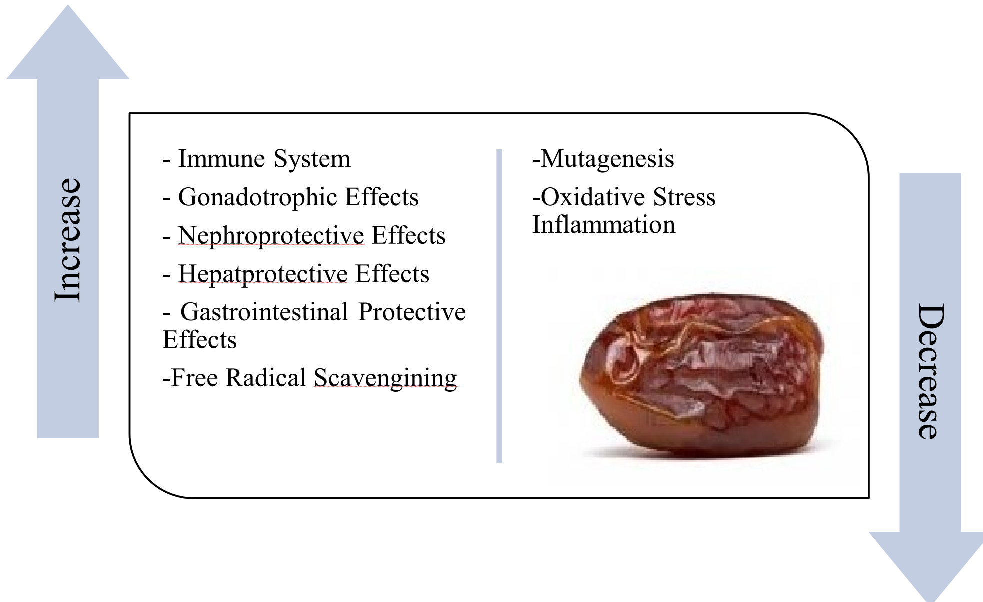


Figure 3.20. Shows pharmacological activities of date fruit (Baliga et al., 2011).

Vayalil (2012) and Al-Farsi et al. (2007) reported that the aqueous extract from date fruit contain a high amount of superoxide and hydroxyl radicals which prevent iron-induced lipid peroxidation and protein oxidation on rats. Yeh et al. (2008) found positive relationship between the amount of date eaten by rats and the amount of antioxidant enzyme in the rat heart tissues.

The date seeds contain elements (oleic acid) that reduce the wrinkling in skin. Zaid (1999) reported that boiled date, black pepper and cardamom was able to treat headaches, coughs and fever and consuming the dates every morning increases the immune system and cure the asthma.

3.8.3. Extraction of Antioxidants

Different methods are used for the extraction of antioxidants. These include: decoction methods, maceration method, hydro-alcoholic method, ultrasound method and solvents method. Regardless of the method used, drying is not desirable because it can affect the chemical compositions of the date fruit. Therefore, it is best to use fresh date when extracting antioxidants (Jiang et al., 2007; Li et al., 2007).

3.8.3.1. Decoction Method: In this method, the fruit is boiled in a process called decoction method (Figure 3.21) in order to gain a liquid rich in antioxidants (Li et al., 2007, Kaneria et al., 2012). 5g of sample is mixed with 100 ml deionized water and boiled at 100 ^{o}C for 30 minutes in water bath. Then, the mixture is centrifuged at 10000 rpm for 10 min. The liquid portion is filtered using muslin cloth. The filtrate is used to determine the antioxidants.

This decoction method does not damage the antioxidant because of the short heating duration. Li et al. (2007) stated that the use of water in this method is preferable over methanol as a decoction liquid containg water is completely nontoxic. The authors found that high temperature may have a negative impact on antioxidants activities. Lim and Murtijaya (2007) recommended the decoction method over other methods because the extracted herbal antioxidant yields are higher when using boiling water. Vongsak et al. (2013) reported that the decoction method decreased the total phenols extracted from tea extract. Kaneria et al. (2012) found the decoction methods to be the best method to extract antioxidants from pomegranate. Vongsak et al. (2013) studied two different methods

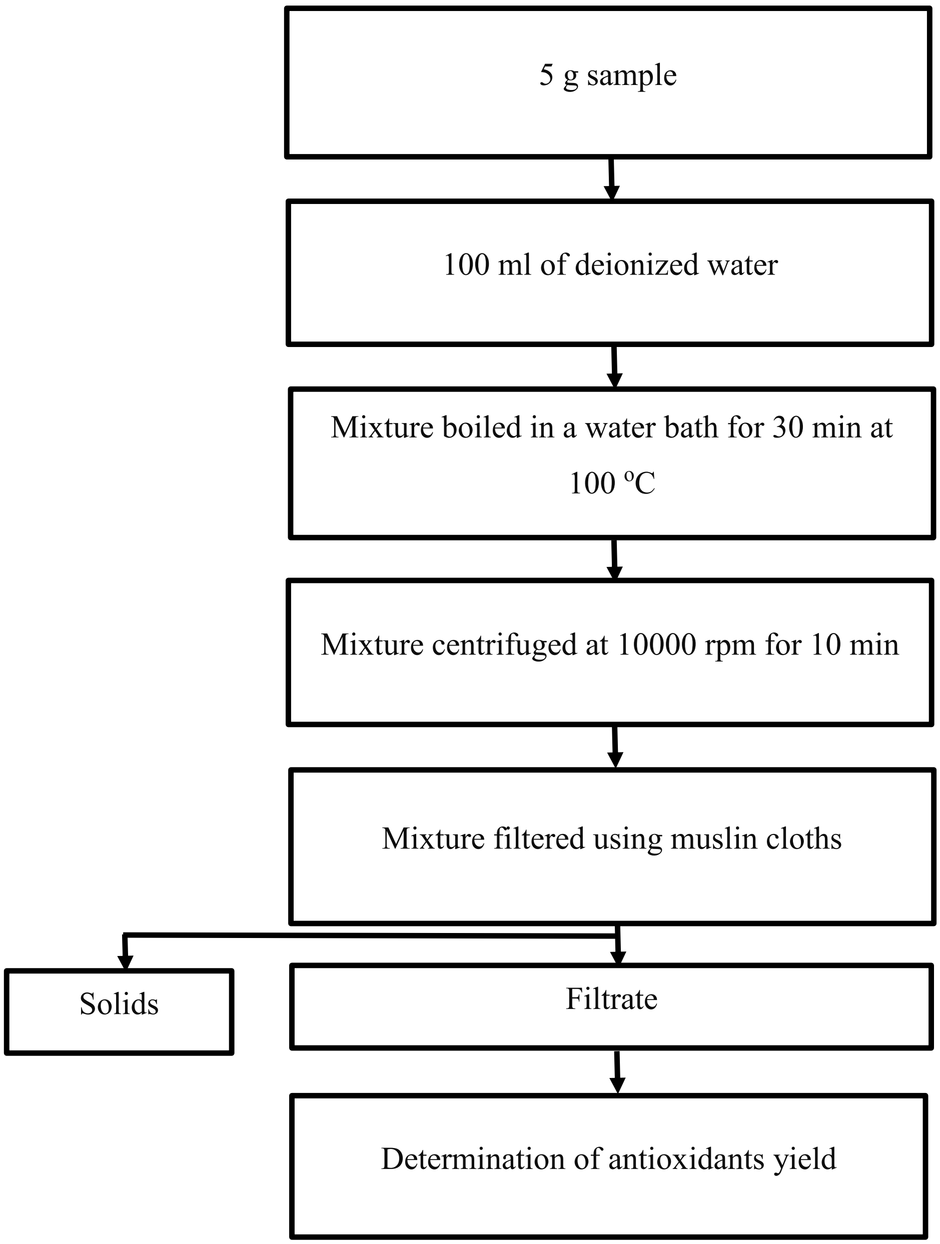


Figure 3.21. The decoction method extraction procedure (Li et al., 2007, Kaneria et al., 2012).

(maceration and decoction method) to extract antioxidants from Moringa oleifera leaf and found that 70% ethanol produced better results than hot water. Gabrieli et al. (2005) used the decoction method to extract beverages from the *Sideritis raeseri* from the mountain areas of North Greece which are used as anti-inflammatory and for their tonic actions. They stated that decoction method preserved the chemical compound of the extract. Li and Zhou (2007) extract the antioxidant by the decoction method from *Lycium barbarum*.

3.8.3.2. Hydro-alcoholic Method: Antioxidants can be extracted by the hydro-alcoholic cold separation method (Figure 3.22). In this method, 10 g sample is mixed with petroleum ether ethanol 90% at a ratio of 1:1 (v/v) in flask capped by cotton wool and shaken on a rotary shaker for 24 hours. The solution is centrifuged at 10000 rpm for 10 min and filtered using eight layers of muslin cloth. The combined filtrate is concentrated in a rotary evaporator at 60°C under reduced pressure to evaporate the petroleum ether and yield a thick solution (Parekh and Chanda, 2007, Kaneria et al., 2012, Bhandare et al., 2010).

Milovanović et al. (2007) used 3 g of Serbian Equisetum with 8 ml of 80% ethanol and obtained high amount of antioxidants. Durling et al. (2007) found that the hydro-alcoholic mixtures of 75% ethanol to be the most appropriate solvent systems for the extraction due to the polarities of the active constituents and suitability of this solvent system for human consumption. Miguel et al. (2010) reported that the hydro-alcoholic method achieved higher amount of total phenols and flavanones than methanolic and aqueous method. However, Kaneria et al. (2012) found that higher yield of antioxidants was achieved by the decoction method compared to the hydro-alcoholic method.

3.8.3.3. Ultrasound Method: The ultrasound procedure is shown in Figure 3.23. In this method, 2.5 g sample and 20 ml 80% methanol (solvent) are placed in 25 ml flask. The mixture is sonicated at 20 khz for 30 minutes at a temperature of 23°C and then filtered using eight layers of muslin cloth. The combined filtrate is concentrated in a rotary evaporator at 60°C under reduced pressure to evaporate the methanol (Albu et al., 2004, Goli et al., 2005, Kim & Lee, 2002).

Pan et al. (2011) studied the yields, activities and extraction kinetics of antioxidants from pomegranate peel using ultrasound-assisted extractions and they obtained high amount of antioxidants Lieu and Li (2010) stated that the advantage of using ultrasound

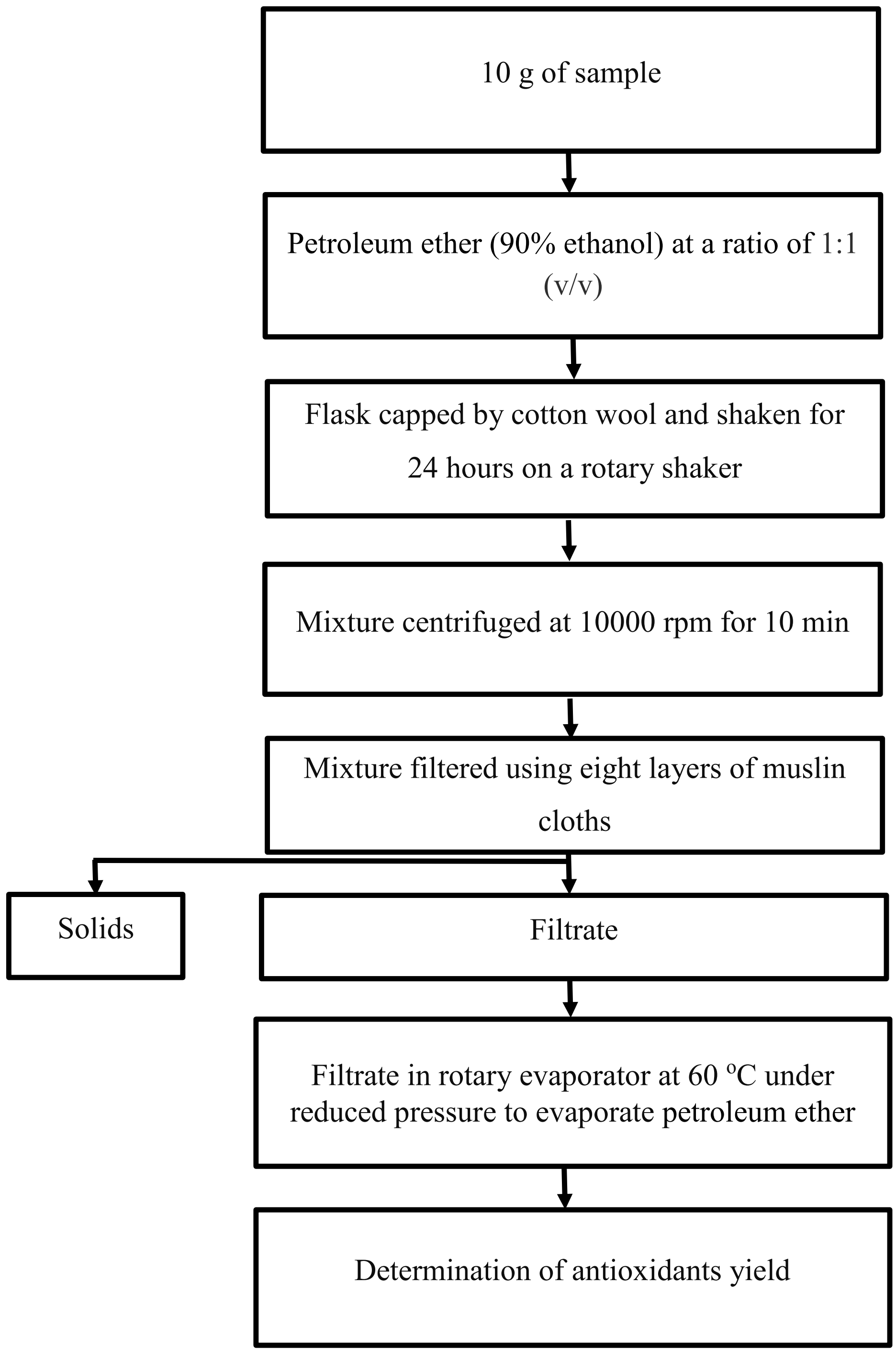


Figure 3.22. The hydro-alcoholic extraction procedure (Parekh and Chanda, 2007, Kaneria et al., 2012, Bhandare et al., 2010).

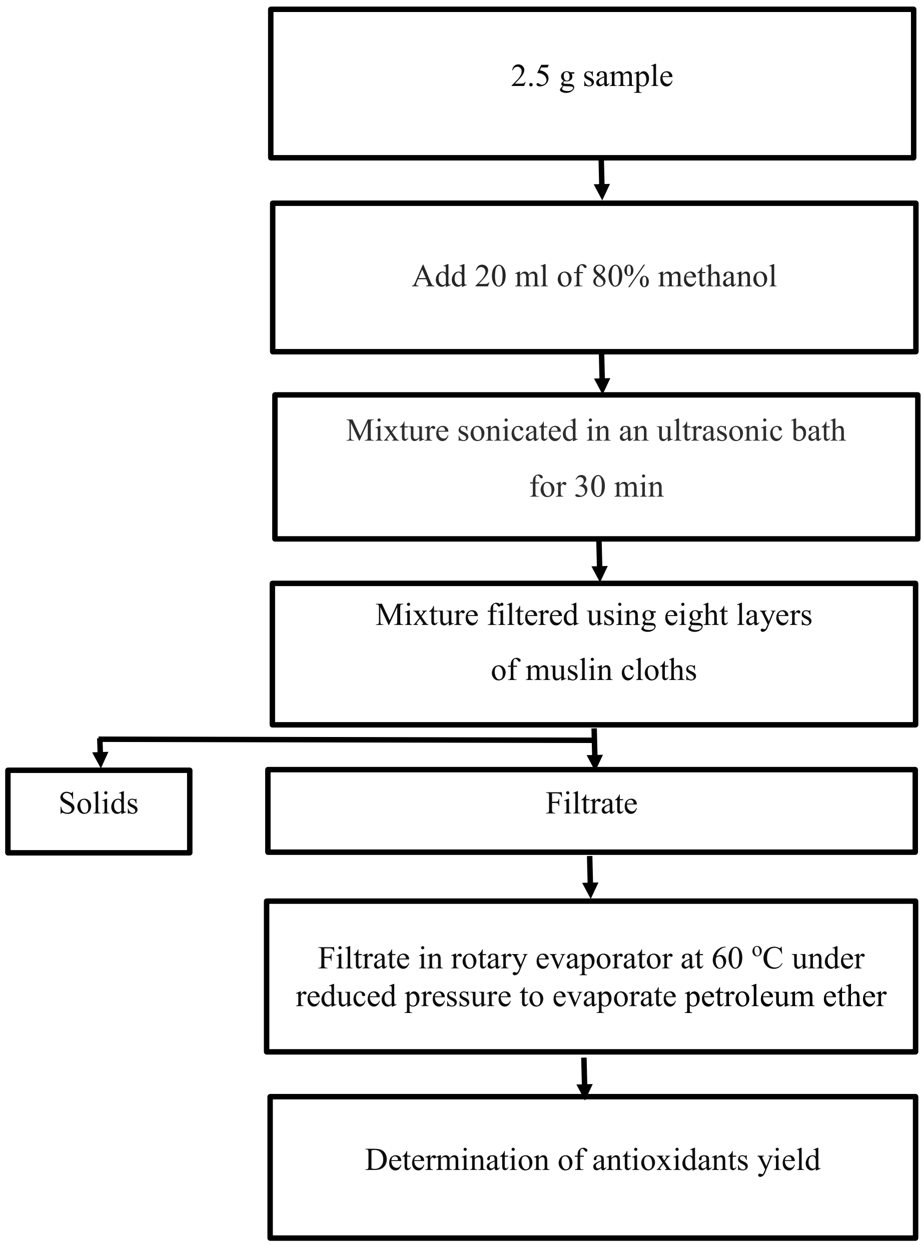


Figure 3.23. The ultrasound extraction procedure (Albu et al., 2004, Goli et al., 2005, Kim & Lee, 2002).

method are better yield and reduced time. Paniwnyk et al. (2001) stated that the effect of ultrasound depends on the solvent used in the extraction. Chaovanalikit and Wrolstad (2004) used acetone:water with chloroform to extract antioxidants from cherries (10:10) and obtained high yields of phenol.

3.8.3.4. Solvents Method: In the solvent extraction method (Figure 3.24), 1 g sample is mixed with 20 ml of solvents (acetone, DMF, methanol, Ethyl acetate and ethanol). These solvents can be mixed with water to create more diluted solutions. The mixture is kept in water bath at a temperature of 60^{o}C for 4 hours. Then, the mixture is centrifuge at 10000 rpm for 10 min and filtered using eight layers of muslin cloths. The filtrate is concentrated in a rotary evaporator at 60°C under reduced pressure to evaporate the petroleum ether and yield a thick solution.

Katalinic et al. (2004) and Turkmen et al. (2006) stated that the antioxidant extraction yield is influenced by the solvent type and concentration. Selection of proper solvents can affect the quantity of phenols extracted. Table 3.12. shows the level of extraction for different solvents. For fruits that contain anthocyanins, an acid is added to the solvent which is normally methanol or ethanol. This compound solution breaks down the cell membranes and dissolves and stabilizes the antioxidant (Dai and Mumper, 2010). Turkmen et al. (2006) reported that using DMF as a solvent to extract polyphenol from tea produced a higher yield than acetone. Methanol is the best option for molecules that have a higher molecular weight. However, methanol is not safe for consumption (Song et al., 2007). Ethanol can be used for the polyphenol extraction and can be safely ingested (Pariza and Foster, 1983).

3.8.4. Factors Effecting Antioxidants Extraction Yield

The main factors that affect the extraction process are: reaction temperature, type of solvent, solvent concentration, extraction time and solvent: material ratio.

3.8.4.1. Reaction Temperature: Extraction temperature is an important parameter and different studies suggest different temperature ranges. According to Wu et al. (2012), the best temperature for optimum antioxidant yield was 30 - 50oC from *Salvia miltiorrhiza* Bunge while Rababah et al. (2012) found the best temperature to be 60 ^{o}C from olive oil. Pinelo et al. (2005) found that 50 ^{o}C is a good temperature to extract high amount of

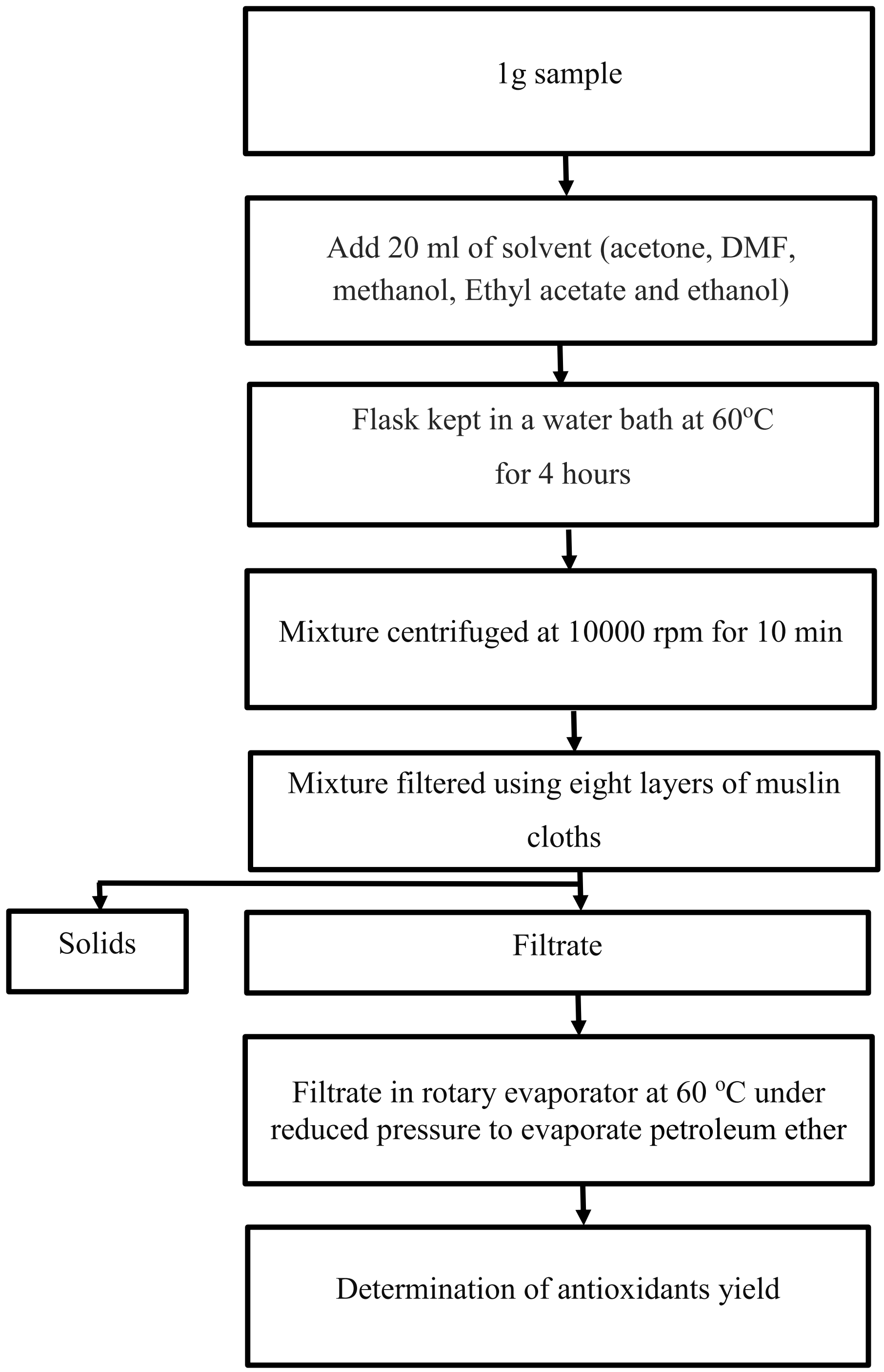


Figure 3.24. The solvent extraction procedure (Katalinic et al., 2004).

Table 3.12. Antioxidant extraction ability for different solvents in the Fard Date Cultivar (Al-Farsi et al., 2005).

Extraction Solvent	Antioxidant Activity (μmol of TE/g)	Total Phenolic Compound (mg of FAE/100 g)
H_2O	9177 ± 798	276 ± 5
phosphate buffer (75 mM, pH 7.4)	9986 ± 765	292 ± 9
methanol (containing 0.1% formic acid)/ H_2O (88:12, v/v)	8552 ± 650	248 ± 12
methanol/HCl (99.9:0.1, v/v)	2005 ± 191	308 ± 9
acetone/H_2O (70:30, v/v)	9406 ± 115	280 ± 9
acetone (containing 7% cyclodextrin)/ H_2O (50:50, v/v)	7312 ± 484	314 ± 8
methanol/H_2O (50:50, v/v)	5840 ± 343	343 ± 7

Data are expressed as mean ± SD (n) on a fresh weight basis.

phenols from dried red and white grape marc (pomase). Spigno et al. (2007) found that 45°C is the best temperature to extract phenol from grape marc. Bucic-Kojić et al. (2009) studied the effect of temperature on extraction of antioxidants from grape seed using a temperature range of 25 -80°C and found that 80°C to be the best. Thoo et al. (2010) stated that 65°C is the best temperature to extract antioxidants from mengkudu (Morinda citrifolia). These differences in the extraction temperature may be due to the different solvent used in the process. However, Mokbel et al. (2012) and Mohan et al. (2008) reported that high temperature is the main cause of decreased yield and breakdown of antioxidants. Hossain et al. (2013) found that high extraction temperature resulted in reduced antioxidants.

3.8.4.2. Type of Solvent: Different studies used different solvents. Acetone, water, ethanol, DMF and methanol are common solvents (Zuo et al., 2002). According to Wu et al. (2012), the best solvent was hot water and ethanol. Spigno et al. (2007) and Bazykina et al. (2002) stated that the ethanol is a polar solvent which makes it the best solvent to extract the flavonoids. Thoo et al. (2010) found that ethanol is the best solvent to extract antioxidants from mengkudu which is in agreement with Herrero et al. (2005) who used it for *Spirulina platensis* microalga.

3.8.4.3. Solvent Concentration: The solvent concentration affects the total yield of antioxidants. Al-Farsi and Lee (2008) reported that the acetone concentration affected the total phenol yields and 50% acetone was the best to extract phenols. Thoo et al. (2010) found that 40% ethanol was the best to extract antioxidants from mengkudu. Turkmen et al. (2006) found that 70% acetone was the best solvent for phenolic extraction from black tea. Chen et al. (2007) found 95% ethanol is the best to extract antitoxidnts from Ganoderma atrum. It appears that the concentration of solvent depends on the type of solvent used.

3.8.4.4. Reaction Time: Reaction time affects antioxidant yield. However, the reaction time depends on the extraction method used. Using ultrasound method will reduce the time (Pérez-Cid et al., 1998). Perva –Uzunalic et al. (2006) suggest using longer time with reduced temperature to avoid the degradation of catechines. Bonilla et al. (1999) stated that 30 min of extraction time has an influence on total phenol yield from red grape marc. Pinelo et al. (2005) and Pekic et al. (1998) recommended longer time of extraction while Bonilla et al. (1999) used shorter time of extraction. Spigno et al. (2007) stated that the main parameters are temperature and time that need to be elevated in order to reduce the energy cost.

3.8.4.5. Sample: Solvent Ratio: The ratio of sample: solvent ratio is dependent on the type of solvent used in the extraction (Al-Farsi and Lee, 2008). Generally, increasing the ratio of solvent: material will increase the phenols yield (Pinelo et al., 2005, Durling et al., 2007, Perva –Uzunalic et al., 2006). Al-Farsi and Lee (2008) and Chirinos et al. (2007) found that the ratio of 60:1 to be the best for the extraction of antioxidants from mashua. Naidu et al. (2008) found that the best sample: solvent ratio to extract antioxidants from green coffee is 60:40 (isopropanol: water).

3.8.5. Assay Methods for Antioxidant Content

There are a number of methods that have been developed to assess the presence of antioxidants (phenols, flavonoids) and measure their total content. These methods include: Folin-Ciocalteu method, aluminum chloride colorimetric method, DPPH method and ABTS method.

3.8.5.1. Folin-Ciocalteu method: The Folin-Ciocalteu method is the most popular method for the determination of the phenolic content (Singlenton and Rossi, 1965; Krishnaiah et al., 2011). The process works as the electrons are transferred in the alkaline medium from phenolic compounds to phosphomolybdic/phosphotungstic acid complexes to form blue complexes. These compounds are then examined using a spectrophotometer at about 765 nm. The most commonly used comparison standard is gallic acid.

This procedure will give a measurement of the total phenolic and other oxidations substrates within the sample. However, because of other substrates in the sample, there is the possibility of interference, inhibition or enhancement of the final reading. Therefore, in order to prevent inhibition effects because the oxidants are competing with the reagent or oxidization from the air, the F-C reagent must be added to the solution before the alkali. Additive effects can occur if there are unanticipated phenols, aromatic amines, high sugar levels or ascorbic acid present in the testing material. (Singleton et al., 1999).

In this procedure, 1 ml of sample extract is add to 1ml of the Folin-Ciocalteu reagent followed by stirring for 5 minutes. 10 ml of 7% sodium carbonate ($Na_2 CO_3$) solution (w/v) is added, followed by 13 ml of deionized water to raise the volume to 25 ml. The mixture is kept for one hour at room temperature. Antioxidants are determined using a spectrophotometer at 765 nm. Figure 3.25. shows a schematic of the Folin-Ciocalteu method. Gallic acid is used for preparation of standard curve (Krishnaiah et al., 2011).

Naidu et al. (2008) used the Folin-Ciocalteu method to measure the total phenol yield from green coffee extract. Blainski et al. (2013) used Folin-Ciocalteu method to determine the total phenolic content from Limonium brasiliense. Wojdyło et al. (2007) followed Folin-Ciocalteu method procedure to determine the total phenols content in Echinacea purpurea.

***3.8.5.2. Aluminum chloride method*:** Aluminum chloride method was used for flavonoids determination by Chang et al. (2002) and Zhishen et al. (1999). In this procedure (Figure 3.26), 5 ml of deionized water and 1 mL of sample are added to 10 ml volumetric flask. 0.3 ml of 5% sodium nitrite are added after 5 minutes. 0.3 ml of 10% aluminum chloride is added and the mixture is stirred for 6 minutes. Then, 2 ml of 1M sodium hydroxide are added Followed by deionized water with mixing to raise the volume to 10 ml. Antioxidants are determined using a spectrophotometer at 510 nm. Catechin is used as a standard for the standard curve.

Chang et al. (2002) used the aluminum chloride method to determine total flavonoids from propolis. Meda et al. (2005) used the aluminum chloride procedure to determine the total flavonoids from Burkina Fasan honey. Ghasemi et al. (2009) used the aluminum chloride method to determine the total flavonoid content in peels and tissues from 13 citrus species and found that flavonoids are higher in peels than tissues.

3.8.5.3. DPPH method: Blois in 1958 was the first one to use the DPPH procedure 2,2-diphenyl-1-picrylhydrazyl DPPH. The DPPH method (Figure 3.27) is the most used method with plant samples. The DPPH method is based on antioxidant decolorizing the DPPH solution and the absorbance is measured by the spectrophotometer at 515 nm (Krishnaiah et al., 2011). In this procedure, 4 ml from the DPPH methanol solution (50 ml DPPH solution was added to 3ml methanol) are added to 1 ml of the sample solution. After stirring for 30 minutes at room temperature the color changes from deep violet to light

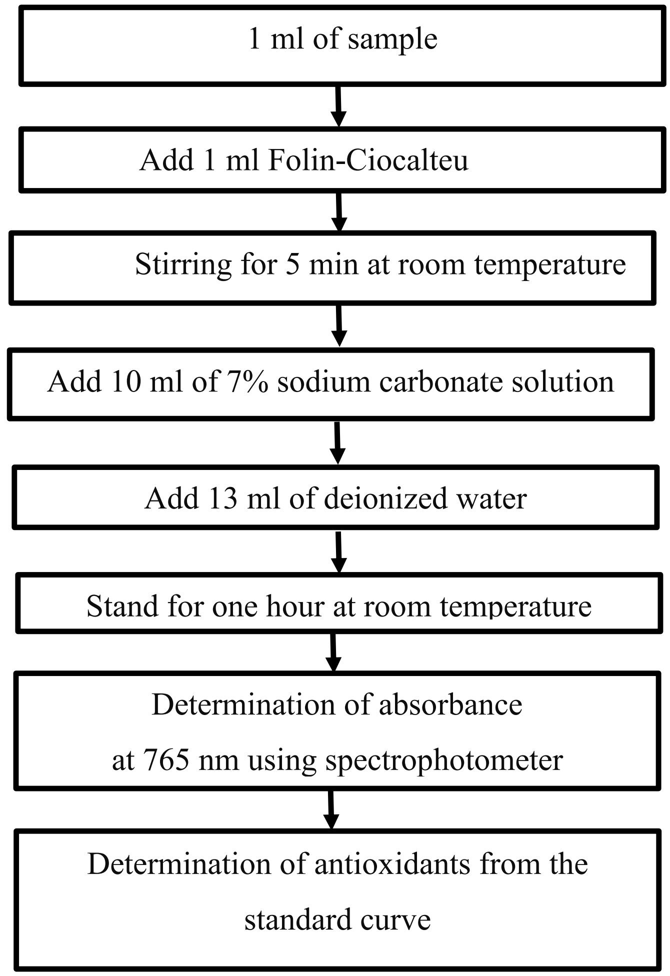


Figure 3.25. Schematic of the folin-ciocalteu method (Krishnaiah et al., 2011).

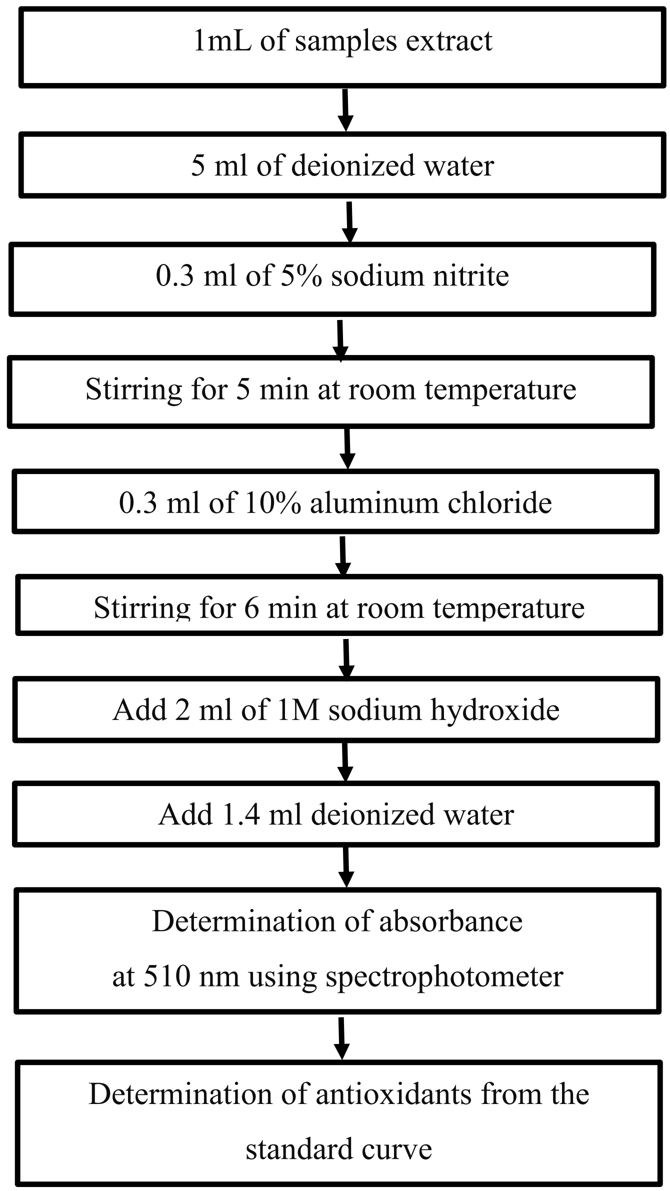


Figure 3.26. Schematic of the aluminum chloride method (Chang et al., 2002 and Zhishen et al., 1999).

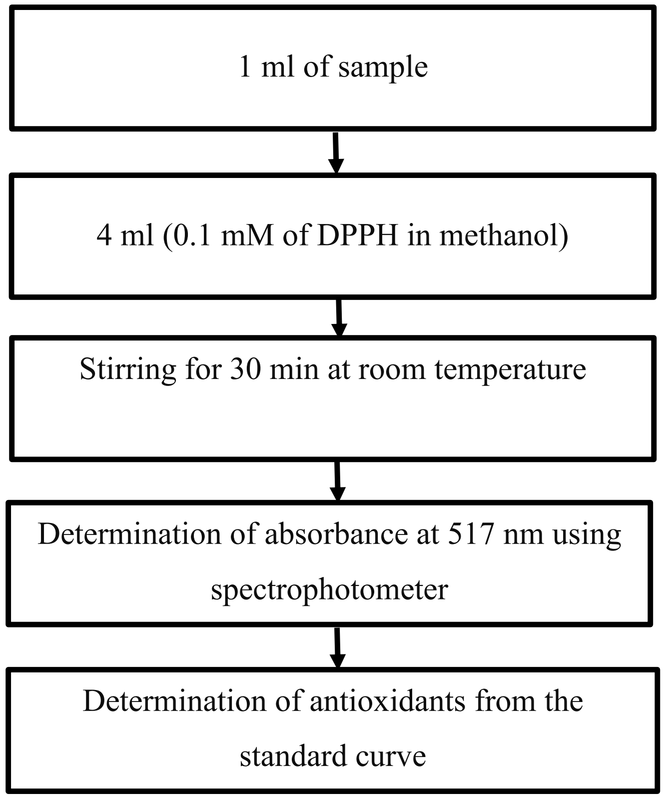


Figure 3.27. Schematic of the DPPH method (Krishnaiah et al., 2011).

yellow and measuring the absorbance at 517 nm. The blank is prepared by adding 3.3 ml ethanol to 0.5 ml sample. A control solution was prepared by mixing 3.5 mL ethanol with 0.3 mL of DPPH radical solution. The absorbance was measured at 517 nm using Spectrophotometer. The scavenging activity percentage (SA%) is determined by the following equation:

$$SA\% = 100 - [\frac{(Abs_{sample} - Abs_{blank}) \times 100}{Abs_{Control}}] \quad (1)$$

Naidu et al. (2008) used DPPH method to measure the antioxidants from green coffee. Leong and Shui (2002) used DPPH method to investigate the antioxidants from Singapore fruits. Prior et al. (2005) used the DPPH method to determination of antioxidant capacity in foods and dietary supplements. Krishnaiah et al. (2011) stated that when there is a large decrease in the absorbance in the mixture, indicates an important free radical scavenging activity of the compound.

3.8.5.4. ABTS method: Rice-Evans and Miller (1997) created the 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid (ABTS) method and Re et al. (1999) improved it. The ABTS radical cation is generated by the oxidation of ABTS with potassium persulfate. This method measured the total antioxidant capacity in both lipophilic and hydrophilic substances (Krishnaiah et al., 2011).

In this procedure (Figure 3.28), the ABTS radical solution is prepared by adding 1 mM of peroxyl radical, 2,2-azobis (2-amidinopropane) dihydrochloride (AAPH) is prepared by adding 2.5 mM of 2,2′- azino - bis (3 - ethylbenzthiazoline - 6-sulphonic acid) (ABTS) and 100 mM potassium phosphate buffer (pH 7.4). The mixture is heated for 30 min in a water bath set at 70 ^{O}C. The ABTS mixture is adjusted with phosphate buffer solution to an absorbance of 0.650 at 734 nm. Then, 20 µl of the sample solution is added to 980 µl of the ABTS radical solution and kept in a water bath for 10 min at 37°C. The absorbance is measured at 734 nm and this value is compared to the ABTS radical solution of the control and used to determine the antioxidant capacity. A control solution was prepared by mixing 2 mL of the ABTS solution with 50 mL methanol and the absorbance measured at 734 nm using a spectrophotometer. The scavenging activity percentage (SA%) is determined by the following equation:

$$SA\% = [\frac{(A_{control} - A_{sample})}{A_{Control}} \times 100] \quad (2)$$

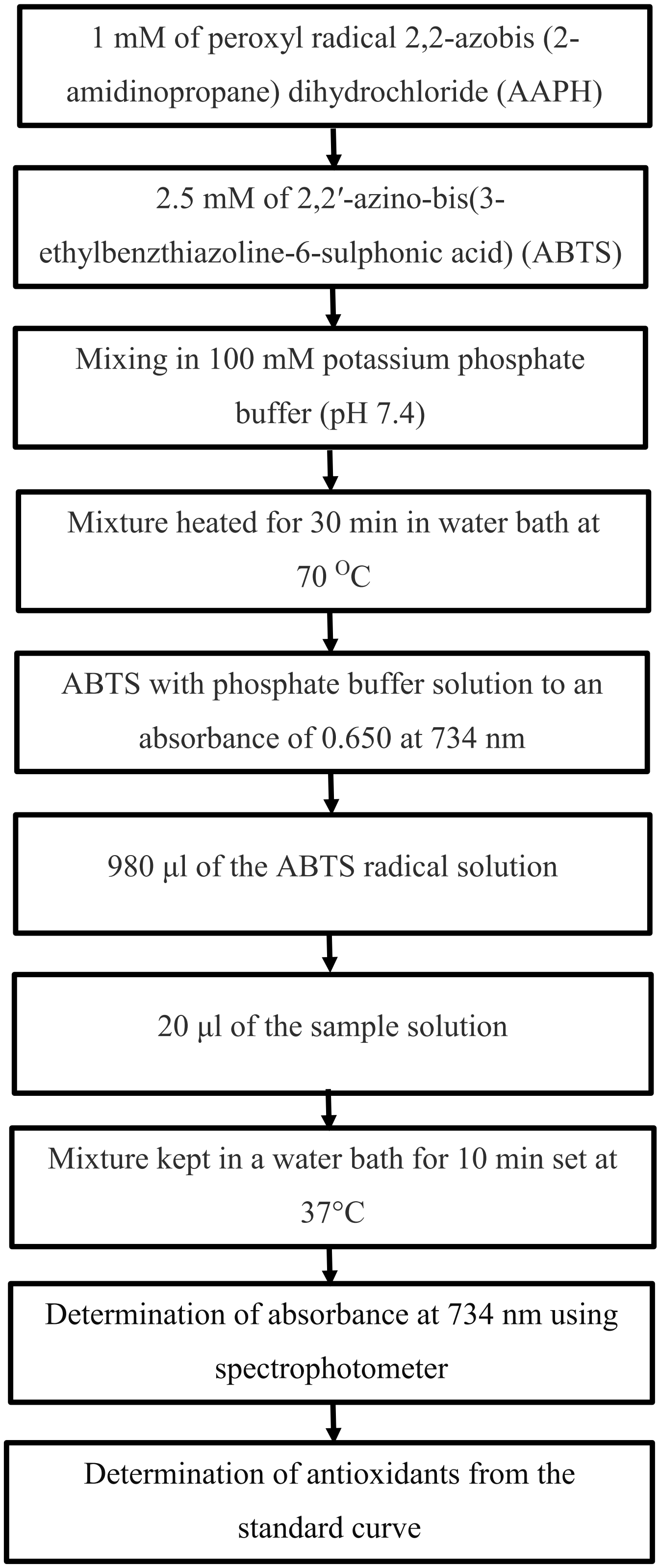


Figure 3.28. Schematic of the ABTS method (Re et al., 1999).

Wojdyło et al. (2007) used the ABTS procedure to determine hirsutum from Onagraceae and observed the high antioxidant activity. Long et al. (2000) used the ABTS procedure to study the antioxidant activities of seasonings used in Asian cooking. Sun et al. (2007) used ATBS procedure to evaluate the antioxidant activity of asparagus, broccoli and their juices. Cai et al. (2004) tested the antioxidant activity of 112 traditional Chinese medicinal plants associated with anticancer properties using the ABTS procedure.

CHAPTER 4. MATERIALS AND METHODS

4.1. Selection of Dates

The date palm fruit is the most important crop in the Kingdom of Saudi Arabia and is cultivated in nearly all regions of the Kingdom. Saudi Arabia is the world's second-largest producer of dates, growing 16% of global date production. Saudi Arabia has more than 23.7 million date palm trees cultivated on more than 156,000 hectares and producing about 992,000 tonnes of dates annually. There are 400 species of dates in the Kingdom of Saudi Arabia. The most popular and consumed dates in the Kingdom of Saudi Arabia are ajwa, sukkari, red sukkari, Khalas and sofry (Figure 4.1) (FAO, 2010).

4.1.1 Ajwa

The cultivation areas of Ajwa in Saudi Arabia are Madinah, Makkah and Hail (International Informatics Network, 2012). The fruit shape is ovoid elongated and medium in size. The color is dark red in the rutab stag and turns to dark brown in the tamer stage with wrinkles. The weights of Ajwa flesh and seed increase during the rutab stage and then decrease during the tamer stage (Gasim, 1994). Ajwa has a sugar contet of 77% (0.5% sucrose, 34.5% glucose and 25.6% fructose) and high proportion of minerals (3%) compared to other varieties of dates (1.5 - 2.7 %), especially calcium (1.223 g / 100 g dry matter) (Gasim, 1994). Ajwa is the best varieties of dates for fresh eating but need to be further studies to explore their properties as they can be used for treatment of many diseases and toxins (Date Palm Journal, 2008; Al-Kahtani et al. 1998; Al Khalifah and Saleh, 2013).

4.1.2. Sukkari

Sukkari is an important cultivar in Al Qassim, Riyadh, North Region, Hail, Madinah, and Njrain (International Informatics Network, 2012). The fruit ripens in August and is consumed in the rutab stage or tamar stage. The color of the fruit changes during the stages from yellow to yellowish brown to brown in the tamar stage (Moawad and Al-Ghamdi, 2013). The shape is ovoid elongated and the fruit is a medium size. The percentage of sugar is 76.2% (sucrose 34.7 %, 21.7% glucose and 19.8% fructose) (Aqidi, 2010; Eleid, 2008; Moawad and Al-Ghamdi, 2013). The fatty acid content varies among varieties between 0.285% and 7.80% and the fiber content varies from 8.78% to 9.2% (Moawad and

(a) Red Sukkari

(c) Sofry

(b) Sukkari

(d) Khalas

(e) Ajwa

Figure 4.1. The poplar types of date in the Kingdom of Saudi Arabia (Kingdom of Saudi Arabia Ministry of Agriculture, 2012).

Al-Ghamdi, 2013, Al-Shahib and Marshall, 2003).

4.1.3. Red Sukkari

Red sukkari is an important cultivar in Al Qassim, Riyadh, North Region, Hail, Madinah, and Njrain (International Informatics Network, 2012). The red sukkari is similar to sukkari but the color darker. The fruit ripens in August and is consumed in the rutab or tamer stage. The color of the fruit changes during the ripeness stages from red to dark red to brown in the tamar stage (Moawad and Al-Ghamdi, 2013). The shape is ovoid elongated and the fruit is a medium size. The percentage of sugar is 78.5% (sucrose 3.2 %, 52.3% glucose and 48.2% fructose) (Eleid, 2008, Moawad and Al-Ghamdi, 2013, Assirey, 2015). The fatty acid content is 0.52% (Al-Shahib and Marshall, 2003), Moawad and Al-Ghamdi , 2013) and the fiber content is 2.67% (Moawad and Al-Ghamdi, 2013, Al-Shahib and Marshall, 2003 , Assirey, 2015).

4.1.4. Khalas

The regions of this cultivator in Saudi Arabia are Riyadh, Eastern Region, Makkah, Qassim, Hail, Njran and Asser (Allaith, 2008; Al-Shahib and Marshall, 2003; Sahari et al., 2007). The fruit shape is constant ovoid elongated and is medium in size. The color varies between yellowish brown in the rutab stage to brown in the tamer stage. It contains 57 % sugar, 2.1% protein 1.4% ash, 22.3% moisture and 280 – 150 mg tannins per 100 g in tamer stage (Al-Shahib and Marshall, 2003; Aqidi, 2010).

4.1.5. Suferi (Sofry)

Suferi is an important cultivar in Al Qassim, Riyadh, North Region, Hail and Madinah (International Informatics Network, 2012).The fruit shape is ovoid. The color is dark red in the rutab stag and turns to dark brown in the tamer stage with wrinkles.The sofry contains 9.2 fiber in dry matter, 15.7 moisture content and 68.4 - 76.2% reduced sugar. The protein content consists of 17 - 23 types of amino acids and the fat content is about 0.22 - 0.62 %. (Al-Shahib and Marshall, 2003; Al-Shahib and Marshall, 2002).

4.2. Experimental Materials

4.2.1. Glassware

The glassware used included test tubes, volumetric flasks, Erlenmeyer flasks, funnel, beakers, reagent bottles, pyrex bottles, centrifuge tubes and pipettes. All glassware were washed with acetone and left to dry overnight in an oven at 105°C (HeraTherm Oven, OMS100, Thermo Fisher Scientific, Ontario, Canada).

4.2.2. Chemicals

Folin-Ciocalteu reagent, sodium nitrite, 10% aluminum chloride, sodium hydroxide, potassium acetate, acetone, ascorbic acid, gallic acid and catechin were obtained from Sigma-Aldrich Ltd, (Oakville, Ontario, Canada). Potassium phosphate buffer (pH 7.4), 7% sodium carbonate solution, acetone and ethanol was obtained from Fisher Scientific Company (Ottawa, Ontario, Canada).

4.2.3. Equipment

Several equipment were used in the experiments. Analytical Balance (PI-314, Denver Instruments, Bohemia, New York, USA) was used to weigh out the samples. A UV-Vis spectrophotometer (Genesys 10S UV-Vis, Thermo Fisher Scientific, Burlington, Ontario, Canada) was used to measure the amount of phenols and flavonoids and antioxidants. Yamato RE-51 rotary evaporator (HiTEC RE-51, Yamato Scientific America, Los Angeles California, USA) was used in this experiment to remove the solvent from the extraction. An oven (OMS100, Thermo Fisher Scientific, Burlington, Ontario, Canada) was used to dry all glassware after it was washed. A centrifuge Sorvall T1 (Thermo Scientific, Marietta, Ohio, USA) and freeze dryer (LABCONCO, Kansas, Missouri, USA) were also used.

4.3. Experimental Design

The experimental work was performed on three steps as shown in Figure 4.2. In the first step, ajwa date was used to evaluate the effects of various parameters on the antioxidants activates as shown in Tables 4.1 – 4.5. There are two solvents (acetone and ethanol), three concentrations (0%, 50% and 75%), three sample: solvent ratios (1:20, 1:40 and 1:60), four reaction temperatures (25, 35, 45, 55 and 65°C) and four reaction times (1, 2, 3, and 4 hours).

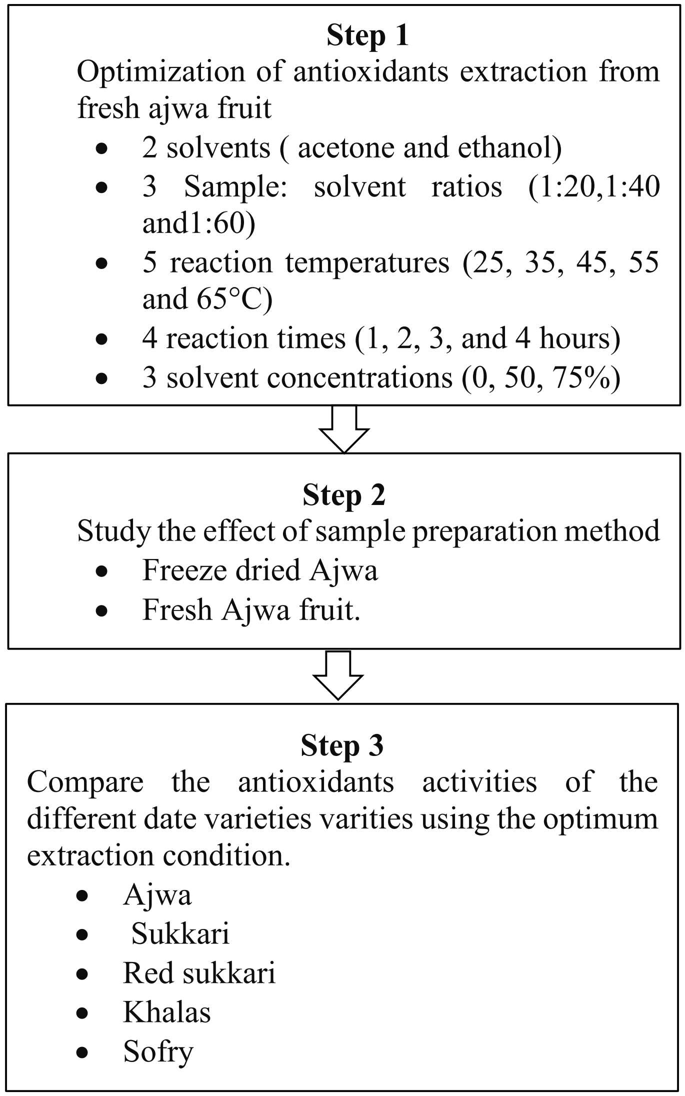


Figure 4.2. Schematic of the experimental plan for the research.

Table 4.1. Experimental plan for optimization of the total antioxidants from fresh date fruit using water 0%.

Sample: Solvent Ratio	Reaction Temperature (ºC)	Reaction Time (h)
1:20	25	1
		2
		3
		4
	35	1
		2
		3
		4
	45	1
		2
		3
		4
	55	1
		2
		3
		4
	65	1
		2
		3
		4
1:40	25	1
		2
		3
		4
	35	1
		2
		3
		4
	45	1
		2
		3
		4
	55	1
		2
		3
		4
	65	1
		2
		3
		4
1:60	25	1
		2
		3
		4
	35	1
		2
		3
		4
	45	1
		2
		3
		4
	55	1
		2
		3
		4
	65	1
		2
		3
		4

Table 4.2. Experimental plan for optimization of the total antioxidants from fresh date fruit using acetone at 50%.

Sample: Solvent Ratio	Reaction Temperature (ºC)	Reaction Time (h)
1:20	25	1
		2
		3
		4
	35	1
		2
		3
		4
	45	1
		2
		3
		4
	55	1
		2
		3
		4
	65	1
		2
		3
		4
1:40	25	1
		2
		3
		4
	35	1
		2
		3
		4
	45	1
		2
		3
		4
	55	1
		2
		3
		4
	65	1
		2
		3
		4
1:60	25	1
		2
		3
		4
	35	1
		2
		3
		4
	45	1
		2
		3
		4
	55	1
		2
		3
		4
	65	1
		2
		3
		4

Table 4.3. Experimental plan for optimization of the total antioxidants from fresh date fruit using ethanol at 50%.

Sample: Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)
1:20	25	1
		2
		3
		4
	35	1
		2
		3
		4
	45	1
		2
		3
		4
	55	1
		2
		3
		4
	65	1
		2
		3
		4
1:40	25	1
		2
		3
		4
	35	1
		2
		3
		4
	45	1
		2
		3
		4
	55	1
		2
		3
		4
	65	1
		2
		3
		4
1:60	25	1
		2
		3
		4
	35	1
		2
		3
		4
	45	1
		2
		3
		4
	55	1
		2
		3
		4
	65	1
		2
		3
		4

Table 4.4. Experimental plan for optimization of the total antioxidants from fresh date fruit using acetone at 75%.

Sample: Solvent Ratio	Reaction Temperature (ºC)	Reaction Time (h)
1:20	25	1 2 3 4
	35	1 2 3 4
	45	1 2 3 4
	55	1 2 3 4
	65	1 2 3 4
1:40	25	1 2 3 4
	35	1 2 3 4
	45	1 2 3 4
	55	1 2 3 4
	65	1 2 3 4
1:60	25	1 2 3 4
	35	1 2 3 4
	45	1 2 3 4
	55	1 2 3 4
	65	1 2 3 4

Table 4.5. Experimental plan for optimization of the total antioxidants from fresh date fruit using ethanol at 75%.

Sample: Solvent Ratio	Reaction Temperature (ºC)	Reaction Time (h)
1:20	25	1 2 3 4
	35	1 2 3 4
	45	1 2 3 4
	55	1 2 3 4
	65	1 2 3 4
1:40	25	1 2 3 4
	35	1 2 3 4
	45	1 2 3 4
	55	1 2 3 4
	65	1 2 3 4
1:60	25	1 2 3 4
	35	1 2 3 4
	45	1 2 3 4
	55	1 2 3 4
	65	1 2 3 4

In step two, the effect of sample preparation on the antioxidants yield was evaluated at the optimum extraction condition. Fresh fruit and freeze dried fruits of ajwa date were used. The fruit was cut into small size pieces (1 cm).

In step three, the antioxidants activities obtained from five date varieties (ajwa, sukkari, red sukkari, khalas and sofry) at the optimum extraction condition were evaluated

4.4. Sample Preparation

The date flesh of ajwa was used in the experiments. The dates obtained from Saudi Arabia were stored in a refrigerator at 4 ℃ until used. Fresh Ajwa dates were used to optimize the total extract of antioxidant in the first and second steps of the experiments while the five varieties were used in the third step of the experiments. The dates were pitted and the flesh was cut by knife to half then to small pieces (1 cm). In the freeze drying method, the cut date fruits parts were kept in a freeze dryer for a week.

4.5. Extraction Procedure

The extraction procedure was carried out on the flesh part of the date fruits according to the procedure shown in Figure 4.3 and used by Al-Farsi and Lee (2008), Mokbel et al. (2012), Wu et al. (2012), Kahkonen et al. (2001) and Perva –Uzunalic et al. (2006) Deionized water was added to the sample using the ratio of water to sample of 20:1 (v/w). This was done by adding 20 ml of water (0% solvent) to 1 g of the sample (fresh date) in a 125 mL Erlenmeyer flask. The flask was placed in a water bath set to 25°C for 1 h. The contents of the flask was transferred to a centrifuge tube and centrifuged (IEC Centra CL2, Thermo Electron Corporation, Mississauga, Ontario, Canada) at 1000 rpm for 10 min. The contents of the tube was filtered using Whatman (No.4 coleparme, Maidstone, England) in order to separate the solids. The solvent was evaporated using a rotary evaporator (HiTEC RE-51, Yamato Scientific America, California, USA) set to 60°C. Finally, the samples was analyzed for total antioxidants (TA), total phenolic (TP) and total flavonoid (TF) using a spectrophotometer (X-Sight, Oxford Instruments, Oxfordshire, UK). The same procedure was followed with all solvents, solvent : sample ratios, reaction tempreatures and reaction times.

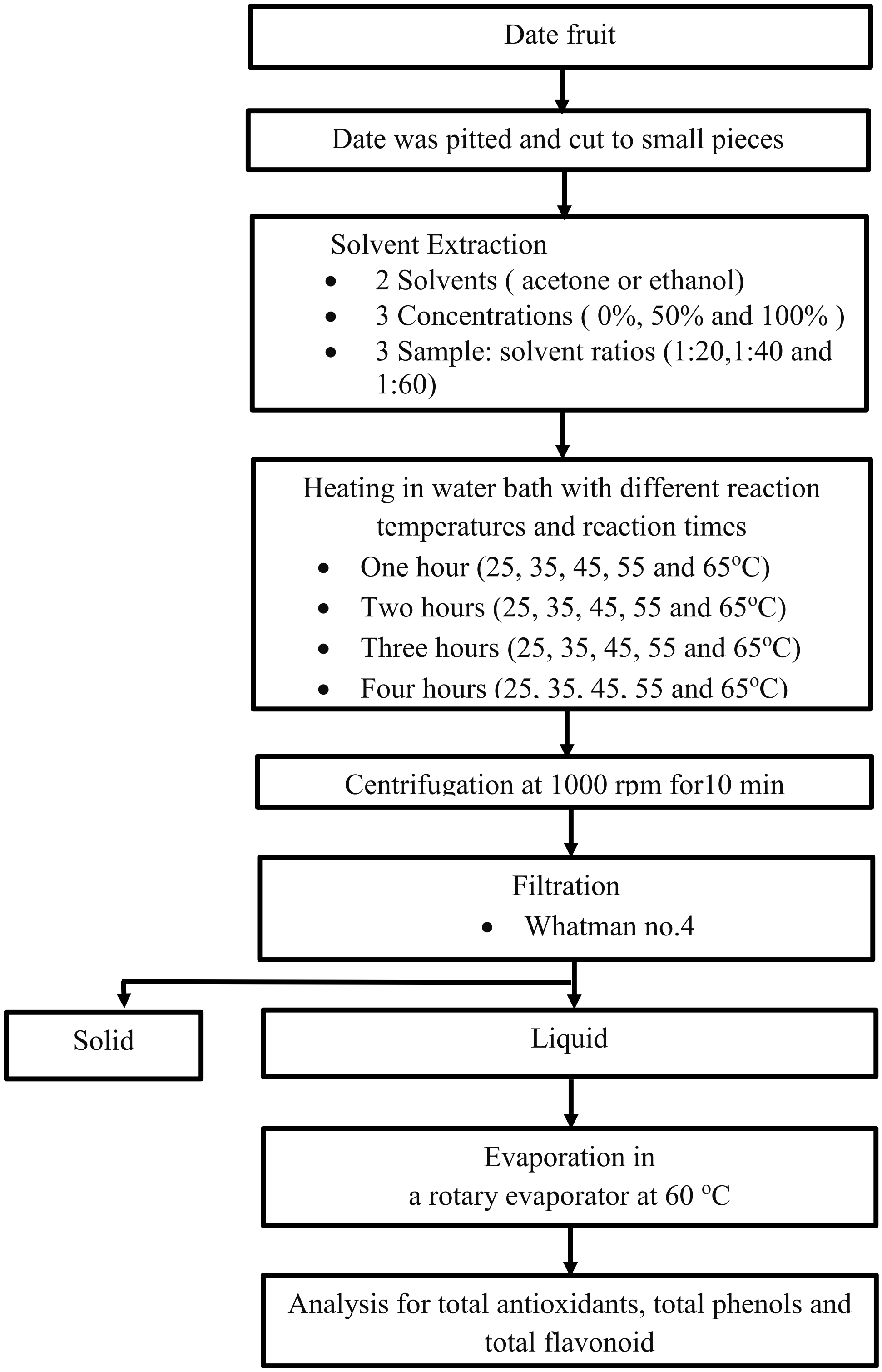


Figure 4.3. Flow diagram of phenol extraction from date fruit.

4.6. Experimental Analyses

The extracted samples were analyzed for total antioxidants (TA), total phenols (TP) and total flavonoids (TF).

4.6.1. Total Antioxidants

The total antioxidants activity was determining according to the phosphomolybdenum method descibed by Prieto et al. (1999). 0.3 ml sample and 3 ml reagent solution (0.6 M sulfuric acid, 28 mM sodium phosphate and 4 mM ammonium molybdate) were placed in 10 ml tube. The tube was kept in water bath at 95°C for 90 min. The absorbance was measured at 695 nm using a spectrophotometer (X-Sight, Oxford Instruments, Oxfordshire, UK).

A standered carve was prepared using five concentrations of ascorbic acid in the range of 9 – 34 mg/100 ml (50% ethanol and water to dilute to volume) as shown in Table 4.6 and Figue 4.4. The absorbance was measured at 695 nm using a spectrophotometer (X-Sight, Oxford Instruments, Oxfordshire, UK). The total antioxidants are expressed as mg/ml of ascorbic acid equivalents per 1g of date fruit.

4.6.2. Total Phenolic

The Folin-Ciocalteu method (F-C) described by Singlenton and Rossi (1965); Krishnaiah et al.(2011) and Yoo et al. (2004) is the most popular method and was used in this study to determine the phenolics content. 1 ml of sample extract was add to 1 ml of the Folin-Ciocalteu reagent followed by stirring for 5 minutes. Then, 10 ml of 7 % sodium carbonate solution (w/v) was added followed by 13 ml of deionized water to raise the volume to 25 ml. The mix was kept for one hour at room temperature.

A standard curve was prepared using five concentrations of gallic acid in the range of 50 –350 mg/ L of 50% ethanol (50% ethanol and water to dilute to volume); were used to prepare the standard curve as shown in Table 4.7 and Figue 4.5. The absorbance was measured at 765 nm using a spectrophotometer (X-Sight, Oxford Instruments, Oxfordshire, UK). The total phenols are expressed as mg/l of Gallic acid equivalents per 1g of date fruit.

Table 4.6. Ascorbic acid concentration and absorbance at 695nm.

Ascorbic Acid Concentration (mg/mL)	Absorbance (at 695 nm)
0	0.000
9	0.202
17	0.404
24	0.606
34	0.808

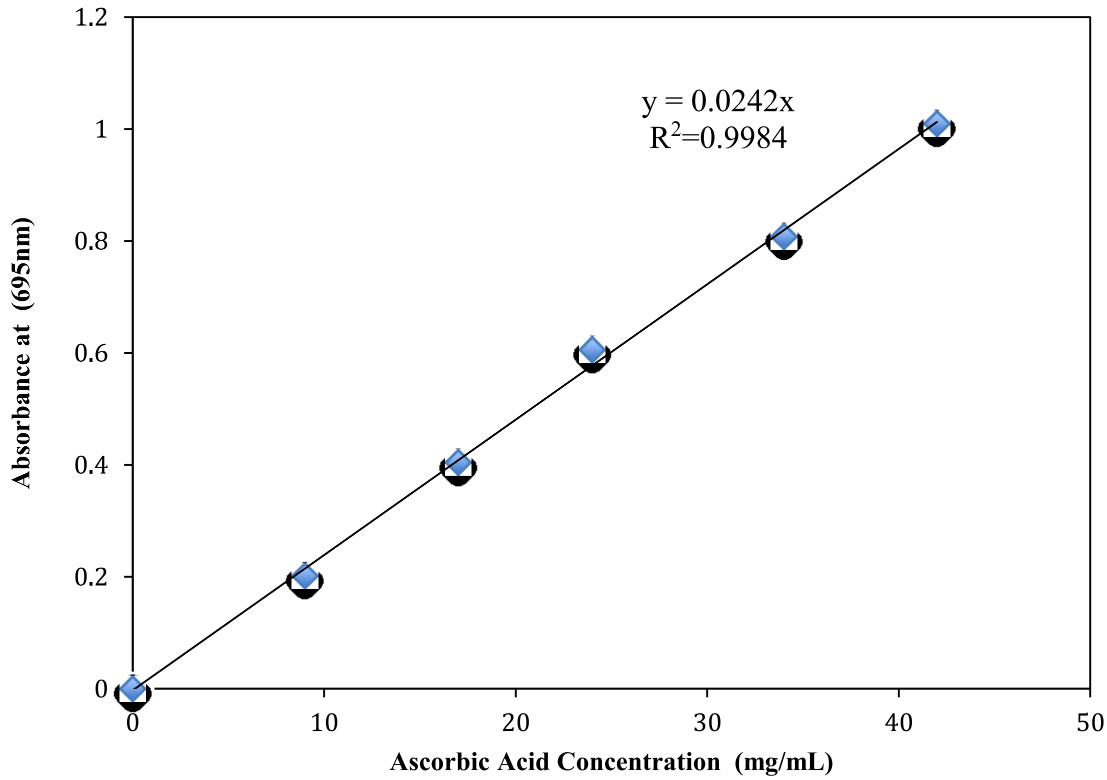


Figure 4.4. The standard curve for Ascorbic Acid.

Table 4.7. Galic acid concentration and absorbance at 765 nm.

Gallic Acid Concentration (mg/L)	Absorbance (at 765nm)
0	0
50	0.04
100	0.14
200	0.28
350	0.46

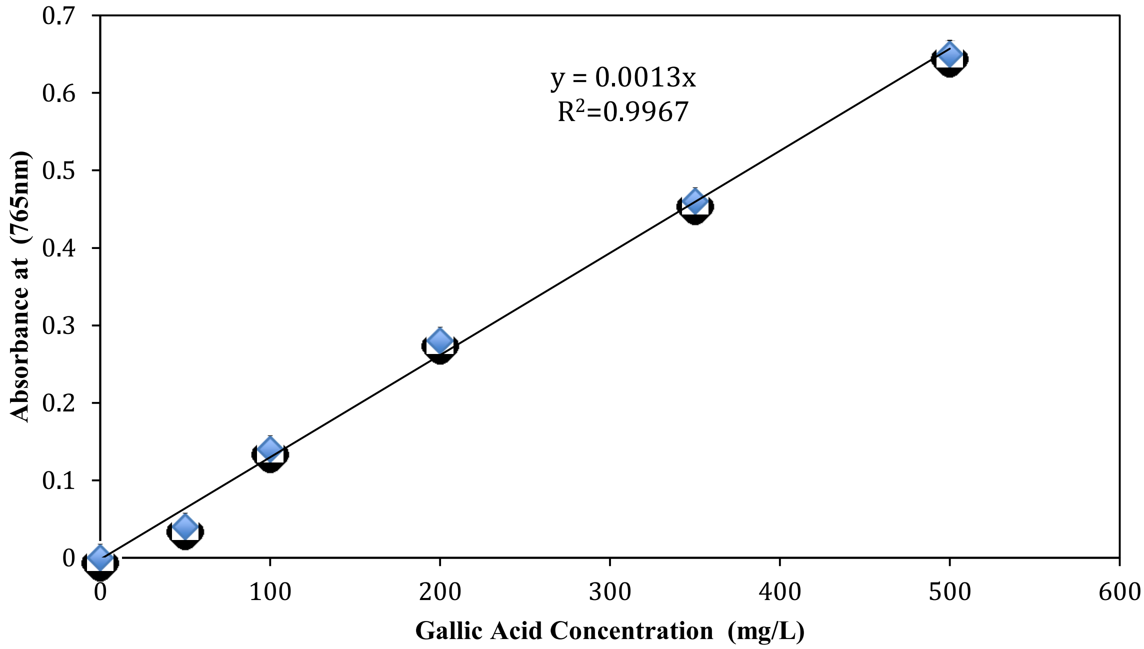


Figure 4.5. The standard curve for Gallic Acid.

4.6.3. Total Flavonoid

The aluminum chloride method described by Chang et al. (2002) and Zhishen et al. (1999) was used for flavonoids determination. 5 ml of deionized water, 1 mL of samples extract, and 0.3 ml of 5% sodium nitrite were added to a 10 ml volumetric flask. 0.3 ml of 10% aluminum chloride was added after 5 min. The mixture in the flask was stirred for 6 min. Then, 2 ml of 1M sodium hydroxide was added followed by deionized water to raise the volume to 10 ml with continuous mixing.

A standard curve was prepared using five concentrations of catechin in the range of 20 – 80 mg/1 L of ethanol as shown in Table 4.8 and Figure 4.6 (50% ethanol and water to dilute to volume). The absorbance was measured at 510 nm using a spectrophotometer (X-Sight, Oxford Instruments, Oxfordshire, UK. The total flavonoids are expressed as grams of catechin per 1g of date fruit.

Table 4.8. Catechin concentration and absorbance at 510 nm.

Catechin Concentration (mg/L)	Absorbance (at 510nm)
0	0.000
20	0.041
40	0.071
60	0.101
80	0.131

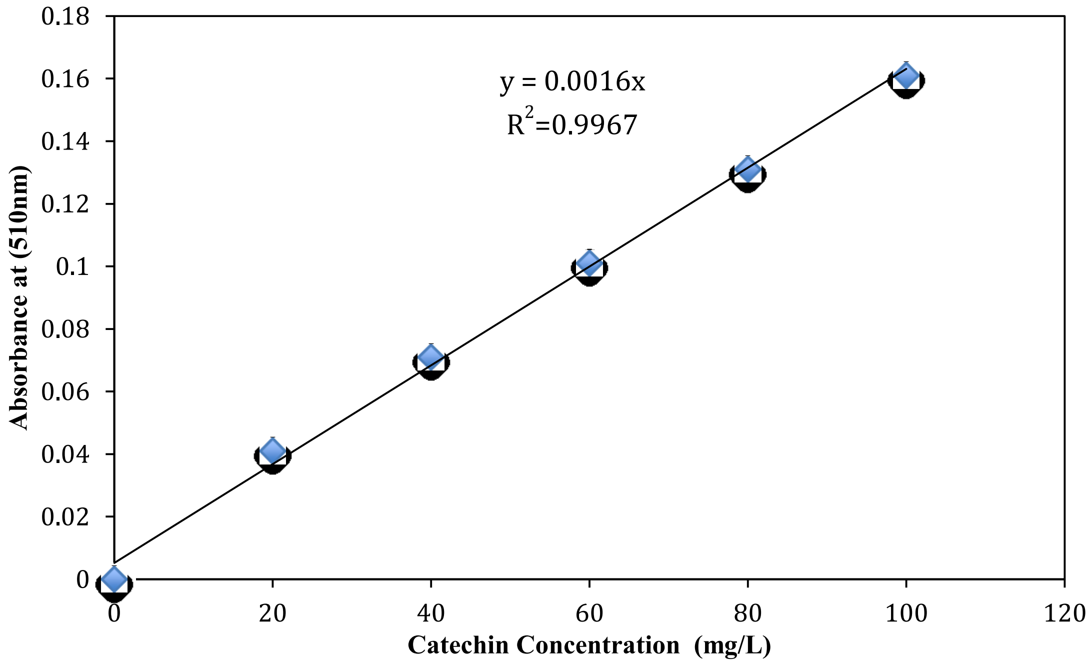


Figure 4.6. The standard curve for Catechin.

CHAPTER 5. RESULTS

5.1. Optimization of the Extraction Process Using Fresh Ajwa Date

The antioxidants, phenol and flavonoid yields from fresh ajwa date fruit were determined at various reaction temperatures (25, 35, 45, 55 and 65˚C), solvent: sample ratios (20:1, 40:1 and 60:1), reaction times (1, 2, 3 and 4 hours), solvents types (acetone and ethanol) and solvent concentrations (0, 50 and 75%).

5.1.1. Total Antioxidants

The antioxidants results are illustrated in Tables 5.1-5.5. Analysis of the variance (ANOVA) was performed on the total antioxidants data using Minitab statics software (Minitab® 17.1.0., Minitab Inc., Canada) and the results are shown in Table 5.6. The results obtained from Tukey's grouping are shown in Table 5.7.

The main effects of reaction time (Ti), solvents: sample ratio (R), solvent concentration (C), solvent type (S) and temperature (Te) on the total antioxidants yield were significant at the 0.001 level. There were also significant interactions between the various parameter. However, the two way interactions between reaction time and ample: solvent ratio and between the reaction time and solvent type were not significant. The three-way interaction were not significant at the 0.001 level except the interactions between the reaction time, the reaction temperature and sample: solvent ratio, between the reaction temperature, sample: solvent ratio and solvent type, between the reaction temperature, sample: solvent ratio and solvent concentration, between sample: solvent ratio, solvent concentration and solvent type, between reaction temperature, solvent type and solvent concentration. The four-way interaction were not significant except the interaction between the reaction time, reaction temperature, sample: solvent ratio and solvent type and between reaction temperature, sample: solvent ratio, solvent type and solvent concentration. The five-way interaction were not significant at the 0.001 level.

All the sample: solvent ratios (1:20, 1:40 and 1:60) were significantly different from one another at the 0.05 level. The results indicated that the highest average yield of the total antioxidants were reached at the sample: solvent ratio of 20:1 (121.161 mg/ml). The reaction times 1 and 2 h were significantly different from one another at the 0.05 level,

Table 5.1. Average antioxidant yield from ajwa date fruit using water at different solvent: sample ratio, reaction temperature and reaction time.

Sample Ratio :Solvent	Reaction Temperature (^{o}C)	Reaction Time (h)	Total Antioxidants Yield* (mg/ml)
1:20	25	1	80.61 ± 0.61
		2	82.58 ±16.42
		3	84.43 ± 0.17
		4	74.54 ±19.89
	35	1	81.05 ± 4.76
		2	83.20 ± 4.79
		3	85.70 ± 2.36
		4	81.67 ± 1.92
	45	1	88.86 ± 3.15
		2	90.76 ± 8.85
		3	93.76 ± 4.98
		4	83.70 ± 3.32
	55	1	98.08 ± 2.86
		2	113.37 ± 0.64
		3	124.98 ± 1.22
		4	103.90 ± 0.81
	65	1	93.53 ± 2.27
		2	97.35 ± 2.89
		3	107.95 ± 4.26
		4	90.00 ± 3.18
1:40	25	1	50.27 ± 0.81
		2	54.44 ± 3.85
		3	56.63 ± 3.59
		4	55.75 ± 0.43
	35	1	57.79 ± 6.01
		2	67.77 ± 1.78
		3	72.64 ± 0.78
		4	68.88 ± 2.13
	45	1	61.63 ± 3.44
		2	73.12 ± 5.02
		3	74.46 ± 2.71
		4	70.52 ± 2.01
	55	1	65.35 ± 2.68
		2	74.60 ± 3.03
		3	75.46 ± 8.21
		4	67.34 ± 1.34
	65	1	55.06 ± 7.30
		2	66.16 ± 2.07
		3	72.40 ±28.72
		4	57.17± 1.46
1:60	25	1	30.60 ± 0.75
		2	36.34 ± 5.84
		3	40.27 ± 1.98
		4	32.52 ± 3.30
	35	1	33.22 ± 3.12
		2	37.59 ± 1.72
		3	39.52 ±14.55
		4	37.11 ± 8.15
	45	1	38.97 ± 0.61
		2	40.12 ± 0.08
		3	45.92 ± 0.70
		4	40.21 ± 0.49
	55	1	48.88 ± 2.95
		2	50.08 ± 0.26
		3	51.71 ± 1.11
		4	47.95 ± 5.66
	65	1	34.23 ± 1.16
		2	39.44 ± 0.46
		3	40.97 ± 2.86
		4	38.92 ± 1.13

*average of two replicates

Table 5.2. Average antioxidants yield from ajwa date fruit using 50% acetone at different solvent: sample ratio, reaction temperature and reaction time.

Sample Ratio: Solvent	Reaction Temperature (ºC)	Reaction Time (h)	Total Antioxidants Yield* (mg/ml)
1:20	25	1	91.90 ± 5.05
		2	105.19 ± 5.90
		3	124.45 ± 0.87
		4	117.73 ± 5.05
	35	1	116.70 ±10.86
		2	120.80 ± 5.49
		3	134.03 ± 9.58
		4	119.11 ± 4.32
	45	1	119.36 ±12.15
		2	126.45 ± 5.46
		3	144.75 ±11.60
		4	127.12 ± 3.62
	55	1	126.43 ± 3.21
		2	132.75 ± 0.93
		3	151.18 ± 1.87
		4	128.64 ± 2.54
	65	1	103.02 ± 2.01
		2	108.24 ± 8.76
		3	119.65 ± 1.57
		4	102.48 ± 2.71
1:40	25	1	53.18 ±48.76
		2	57.50 ± 0.00
		3	74.40 ± 1.63
		4	71.37 ± 0.75
	35	1	69.21 ± 2.83
		2	80.80 ± 1.92
		3	90.11 ± 3.27
		4	77.75 ±11.30
	45	1	75.19 ±13.09
		2	81.42 ± 1.13
		3	92.64 ± 0.61
		4	88.57 ± 4.20
	55	1	80.63 ± 2.95
		2	84.98 ±16.65
		3	96.70 ± 0.75
		4	92.80 ± 1.02
	65	1	75.25 ± 4.82
		2	79.57 ± 1.84
		3	82.93 ± 2.36
		4	72.29 ± 5.52
1:60	25	1	44.61 ± 3.38
		2	50.93 ±11.86
		3	59.44 ± 0.70
		4	52.24 ± 5.96
	35	1	53.90 ±10.28
		2	57.46 ± 1.69
		3	71.38 ± 5.72
		4	70.80 ± 3.68
	45	1	69.23 ± 3.03
		2	75.72 ± 3.06
		3	81.08 ± 0.26
		4	75.73 ± 1.98
	55	1	74.38 ± 2.95
		2	78.35 ± 7.62
		3	85.14 ± 2.98
		4	79.29 ± 4.41
	65	1	71.20 ± 0.14
		2	72.58 ± 1.22
		3	78.08 ± 1.22
		4	68.64 ± 1.34

*average of two replicates

Table 5.3. Average antioxidants yield from ajwa date fruit using 50% ethanol at different solvent: sample ratio, reaction temperature and reaction time.

Sample Ratio: Solvent	Reaction Temperature (°C)	Reaction Time (h)	Total Antioxidants Yield* (mg/ml)
1:20	25	1	72.66 ± 1.753
		2	88.14 ± 5.580
		3	95.14 ± 9.817
		4	90.02 ± 8.882
	35	1	92.85 ± 0.43
		2	99.03 ± 3.38
		3	105.33 ± 9.20
		4	100.43 ± 4.14
	45	1	109.88 ± 7.10
		2	116.61 ± 8.73
		3	122.10 ±19.22
		4	125.88 ± 7.27
	55	1	118.42 ± 1.69
		2	122.51 ± 8.97
		3	125.53 ± 3.33
		4	119.14 ± 0.23
	65	1	103.33 ± 3.15
		2	114.81 ± 9.93
		3	120.06 ± 2.04
		4	107.46 ± 2.68
1:40	25	1	57.13 ± 9.75
		2	63.99 ± 4.90
		3	77.25 ± 0.40
		4	74.13 ± 6.80
	35	1	75.13 ± 1.75
		2	87.04 ± 2.92
		3	95.10 ± 0.17
		4	89.65 ± 0.52
	45	1	80.94 ± 1.66
		2	90.33 ± 2.60
		3	96.42 ±10.98
		4	90.79 ± 4.77
	55	1	98.01 ± 8.38
		2	100.37 ± 4.38
		3	108.95 ± 4.47
		4	102.68 ± 2.68
	65	1	77.91 ± 1.37
		2	84.54 ± 2.30
		3	91.40 ± 1.08
		4	84.80 ± 1.51
1:60	25	1	47.07 ± 8.44
		2	53.90 ± 8.64
		3	77.75 ± 9.35
		4	58.74 ±21.79
	35	1	55.99 ± 3.71
		2	68.53 ± 4.49
		3	82.29 ± 0.49
		4	72.42 ± 2.10
	45	1	73.99 ± 0.58
		2	85.38 ±18.52
		3	92.79 ±19.81
		4	85.33 ±19.83
	55	1	89.01 ±27.20
		2	90.74 ±23.17
		3	98.30 ±27.55
		4	92.06 ±29.89
	65	1	72.89 ± 9.67
		2	82.11 ±21.06
		3	90.60 ±25.42
		4	81.98 ±14.08

*average of two replicates

Table 5.4. Average antioxidants yield from ajwa date fruit using 75% acetone at different solvent: sample ratio, reaction temperature and reaction time.

Solvent : Sample Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Antioxidants Yield* (mg/ml)
1:20	25	1	111.18 ± 1.81
		2	130.86 ± 2.92
		3	140.00 ± 1.31
		4	130.12 ± 8.26
	35	1	129.75 ± 4.41
		2	135.64 ± 7.01
		3	155.83 ±16.45
		4	138.84 ± 6.34
	45	1	146.34 ± 6.25
		2	170.08 ±21.06
		3	199.34 ± 1.31
		4	187.62 ± 5.43
	55	1	169.90 ±31.84
		2	181.80 ± 9.08
		3	201.18 ± 2.42
		4	188.08 ± 9.93
	65	1	146.49 ±25.27
		2	155.87 ±31.41
		3	194.55 ± 4.35
		4	177.30 ±10.40
1:40	25	1	100.27 ± 0.08
		2	103.57 ± 2.80
		3	113.16 ± 5.61
		4	99.77 ± 5.08
	35	1	105.26 ± 2.74
		2	113.93 ±10.78
		3	121.88 ± 4.61
		4	109.94 ± 0.64
	45	1	110.58 ± 4.41
		2	120.45 ± 9.02
		3	134.07 ± 3.62
		4	118.70 ± 2.92
	55	1	151.65 ± 1.02
		2	153.64 ±22.17
		3	160.38 ±24.62
		4	150.60 ±16.71
	65	1	137.46 ± 0.52
		2	140.52 ± 1.22
		3	152.23 ± 2.07
		4	139.43 ±10.19
1:60	25	1	76.94 ± 9.43
		2	84.54 ± 2.42
		3	97.60 ± 1.02
		4	87.97 ± 4.93
	35	1	85.70 ± 8.73
		2	93.37 ± 9.17
		3	101.74 ± 2.60
		4	92.08 ±23.60
	45	1	97.15 ±29.94
		2	99.73 ± 0.87
		3	107.91 ± 1.98
		4	99.01 ± 6.57
	55	1	117.37 ± 3.18
		2	122.29 ± 4.67
		3	130.74 ± 2.01
		4	127.29 ± 2.80
	65	1	105.50 ± 5.37
		2	120.35 ±13.93
		3	129.61 ± 0.99
		4	120.27 ±14.49

*average of two replicates

Table 5.5. Average antioxidants yield from ajwa date fruit using 75% ethanol at different solvent: sample ratio, reaction temperature and reaction time.

Solvent : Sample Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Antioxidants Yield* (mg/ml)
1:20	25	1	142.85 ± 4.20
		2	146.51 ± 8.41
		3	152.69 ±11.89
		4	141.86 ± 6.80
	35	1	163.16 ±30.56
		2	170.16 ±29.77
		3	173.24 ±45.61
		4	171.49 ±27.72
	45	1	176.88 ±16.12
		2	188.28 ± 2.16
		3	204.39 ± 0.75
		4	197.46 ± 3.27
	55	1	178.22 ±28.40
		2	181.82 ± 5.11
		3	203.39 ± 1.78
		4	193.82 ± 3.21
	65	1	161.61 ±44.29
		2	174.28 ±41.78
		3	198.18 ± 1.84
		4	190.40 ±21.00
1:40	25	1	109.94 ±33.89
		2	120.83 ± 5.93
		3	133.72 ± 9.90
		4	128.14 ± 8.09
	35	1	111.57 ± 5.87
		2	122.17 ± 5.14
		3	153.76 ± 1.84
		4	152.23 ± 7.80
	45	1	136.40 ±16.97
		2	147.79 ±10.81
		3	164.57 ± 1.05
		4	167.66 ±15.77
	55	1	161.96 ± 1.84
		2	170.09 ± 1.16
		3	177.85 ± 7.85
		4	174.88 ± 1.24
	65	1	141.14 ± 8.00
		2	156.85 ± 1.25
		3	169.26 ± 0.40
		4	159.67 ± 0.75
1:60	25	1	88.33 ± 2.68
		2	97.02 ± 3.30
		3	105.10 ± 3.68
		4	100.44 ± 3.38
	35	1	104.17 ± 6.04
		2	115.25 ± 3.65
		3	122.17 ± 2.80
		4	113.16 ±15.25
	45	1	118.90 ± 1.57
		2	130.58 ±13.06
		3	135.45 ± 4.47
		4	130.42 ± 2.51
	55	1	160.51 ±17.15
		2	169.65 ± 9.05
		3	174.49 ± 2.62
		4	171.41 ± 6.13
	65	1	136.96 ± 7.30
		2	150.35 ±27.14
		3	163.19 ± 1.98
		4	151.83 ±11.54

*average of two replicates

Table 5.6. Analysis of the variance for total antioxidants capacity.

Source	DF	SS	MS	F	P
Total	719	1425798			
Model					
Time (Ti)	3	12008	4003	34.68	0.001
Ratio (R)	2	244106	122053	1057.46	0.001
Temperature (Te)	4	83946	20987	181.83	0.001
Solvent (S)	1	12642	12642	109.53	0.001
Concentration (C)	2	808850	404425	3503.92	0.001
Ti*R	6	914	152	1.32	0.248
Ti*Te	12	3885	324	2.80	0.001
Ti*S	3	249	83	0.72	0.540
Ti*C	6	6959	1160	10.05	0.001
Te*R	8	7485	936	8.11	0.001
Te*S	4	6252	1563	13.54	0.001
Te*C	8	55695	6962	60.32	0.001
R*S	2	3540	1770	15.34	0.001
R*C	4	11747	2937	25.44	0.001
S*C	2	18190	9095	78.80	0.001
Ti*Te*R	6	7561	315	2.73	0.001
Ti*R*S	6	892	149	1.29	0.262
Ti*R*C	12	3263	272	2.36	0.006
Ti*Te*S	12	2429	202	1.75	0.054
Ti*S*C	6	353	59	0.51	0.801
Te*R*S	8	7958	995	8.62	0.001
Te*R*C	16	15071	942	8.16	0.001
R*C*S	4	7092	1773	15.36	0.001
Te*S*C	8	10284	1286	11.14	0.001
Ti*Te*C	24	4453	186	1.61	0.037
Ti*Te*R*S	24	3510	146	1.27	0.182
Ti*Te*R*C	48	13092	273	2.36	0.001
Ti*Te*S*C	24	5186	216	1.87	0.008
Ti*R*S*C	12	974	81	0.70	0.749
Te*R*S*C	16	19066	1192	10.32	0.001
Ti*Te*R*S*C	48	6596	137	1.19	0.191
Error	360	41551	115		

Reaction Time (Ti)
Reaction Temperature (Te)
Solvent: Sample Ratio (R)
Solvent Type (S)
Solvent Concentration (C)
R^2= 97.09%

Table 5.7. Tukey's grouping of total antioxidant yield.

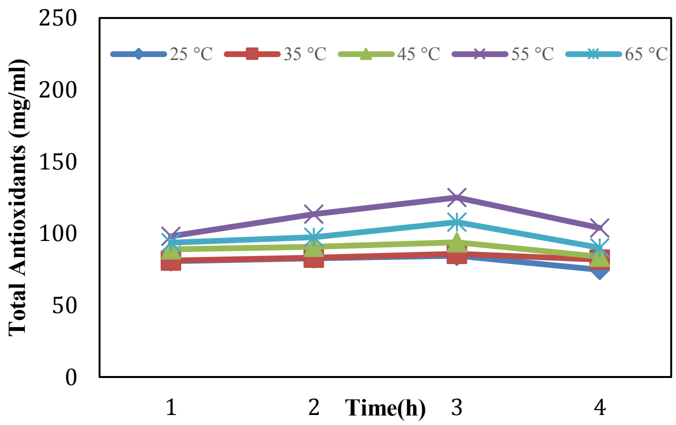
Factors	level	N	Mean	Tukey's Grouping
Ratio	20	240	121.161	A
	40	240	95.767	B
	60	240	76.183	C
Time	1	180	91.346	A
	2	180	96.990	B
	3	180	101.331	C
	4	180	101.238	C
Temperature	25˚C	144	85.610	A
	35˚C	144	89.181	B
	45˚C	144	93.085	C
	55˚C	144	113.808	D
	65˚C	144	106.834	E
Concentration	0%	240	60.899	A
	50%	240	90.238	B
	75%	240	141.974	C
Solvent	Acetone	360	93.513	A
	Ethanol	360	101.897	B

Groups with the same letter are not significantly different from each other at the 0.05 level.

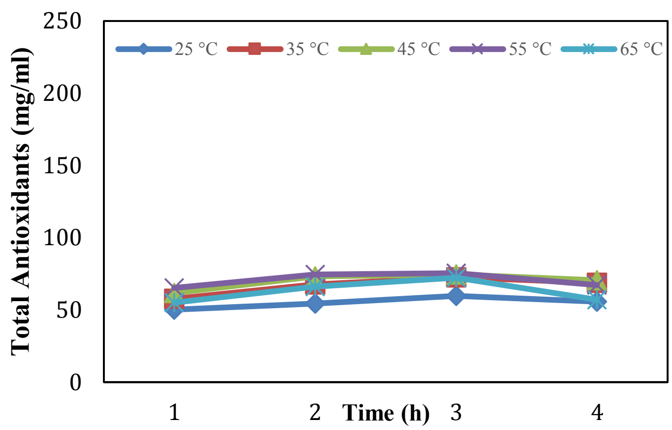
but the reaction times 3 and 4 hours were not significantly different from one another at the 0.05 level. The highest average yield of the total antioxidants (101.331 mg/ml) was obtained after 3 hours. All reaction temperatures (25, 35, 45, 55 and 65 ˚C) were significantly different from one another at the 0.05 level. The highest average yield of the total antioxidants was achieved a temperature of 55 ˚C (113.808 mg/ml). All solvent concentrations (0, 50 and 75%) were significantly different from one another at the 0.05 level. The highest average yield of total antioxidants was achieved at a solvent concentration of 75% (141.974 mg/ml). The solvent types (acetone and ethanol) were significantly different from one another at the 0.05 level. The highest average yield of the total antioxidants was obtained with ethanol (101.897mg/ml).

5.1.1.1. Effect of Reaction Time: Figures 5.1-5.5 show the effect of reaction time on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios (1:20, 1:40 and 1:60) and reaction temperatures (25, 35, 45, 55 and 65 ^oC) using acetone and ethanol at different concentrations (0, 50 and 75%). Generally, there was an increase in the total antioxidants yield when the reaction time was increased from 1 to 3 h at all reaction temperatures, sample: solvent ratios and solvent concentration for both solvents. This was followed by a decrease in the antioxidants yield with a further increase in reaction time from 3 to 4 h.

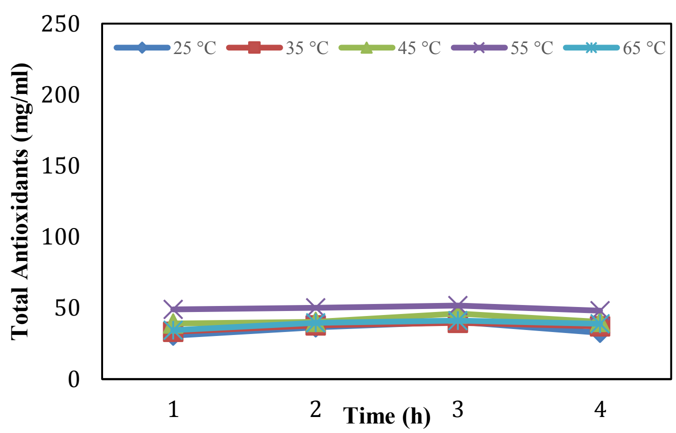
When water was used as a solvent, increasing the reaction time from 1 to 3 h, increased the antioxidants yield from 80.61 to 84.43 mg/ml (4.74%), from 81.05 to 85.70 mg/ml (5.73%), from 88.86 to 93.78 mg/ml (5.51%), from 98.08 to 124.98 mg/ml (27.43%) and from 93.53 to 107.95 (15.42%) at the sample: solvent ratio of 1:20, from 50.27 to 56.63 mg/ml (12.65%), from 57.79 to 72.64 mg/ml (25.70%), from 61.63 to 74.46 mg/ml (20.82%), from 65.35 to 75.46 mg/ml (15.47%) and from 55.06 to 72.40 (31.49%) at the sample: solvent ratio of 1:40 and from 30.60 to 40.27 mg/ml (31.60%) and from 33.22 to 39.52 mg/ml (18.96%), from 38.97 to 45.92 mg/ml (17.83%), from 48.88 to 51.71 mg/ml (5.79%) and from 34.23 to 40.97 (19.69%) at the sample: solve ratio 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^oC, respectively. A further increase the reaction time from 3 to 4 h, decreased the antioxidants yield from 82.43 to 74.54 mg/ml (11.71%), from 85.70 to 81.67 mg/ml (4.7%), from 93.76 to 83.70 mg/ml (5.81%), from 124.98 to 103.90



(a) Sample: solvent ratio 1:20

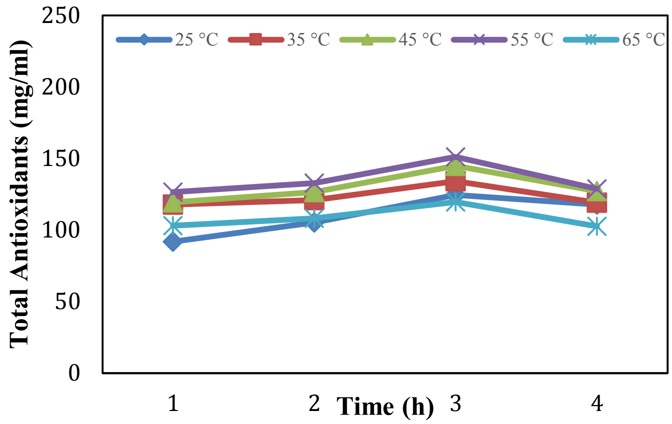


(b) Sample: solvent ratio 1:40

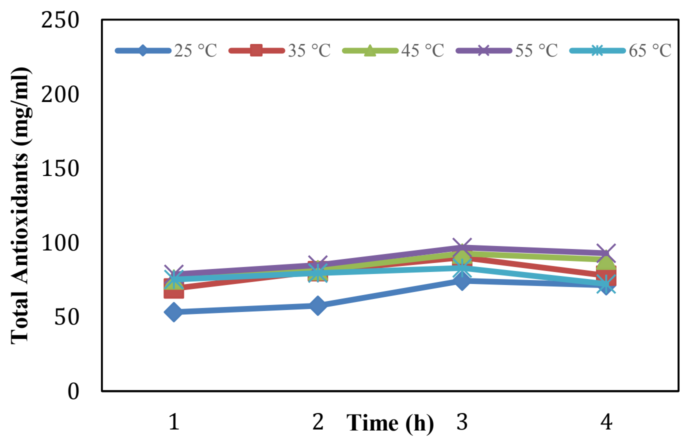


(c) Sample: solvent ratio 1:60

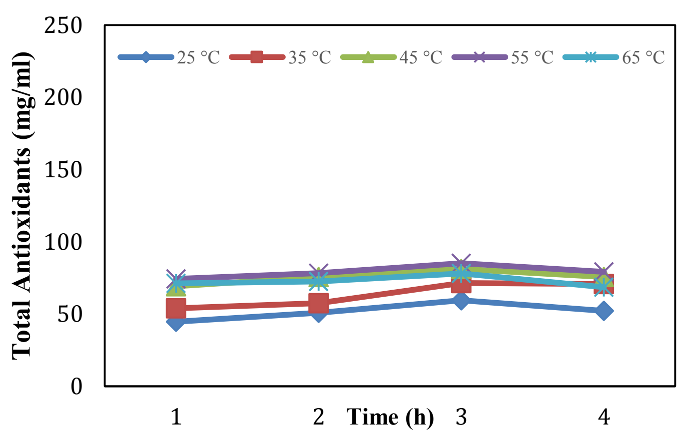
Figure 5.1. The effect of reaction time on the total antioxidants yield from ajwa date fruit using water at different temperatures.



(a) Sample: solvent ratio 1:20

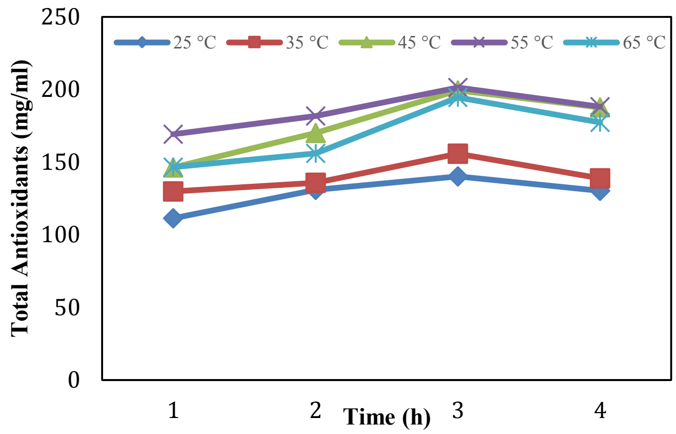


(b) Sample: solvent ratio 1:40

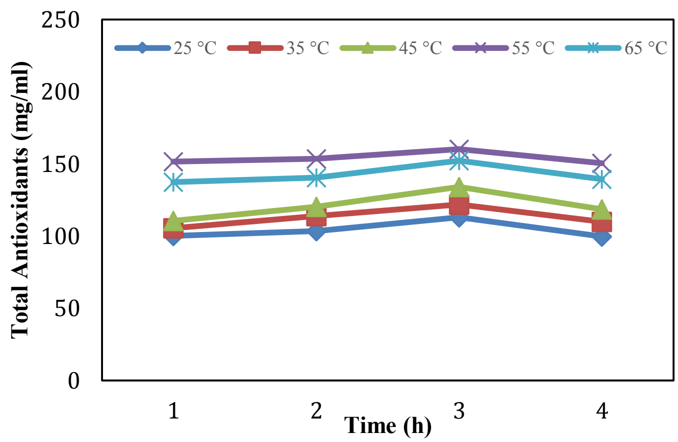


(c) Sample: solvent ratio 1:60

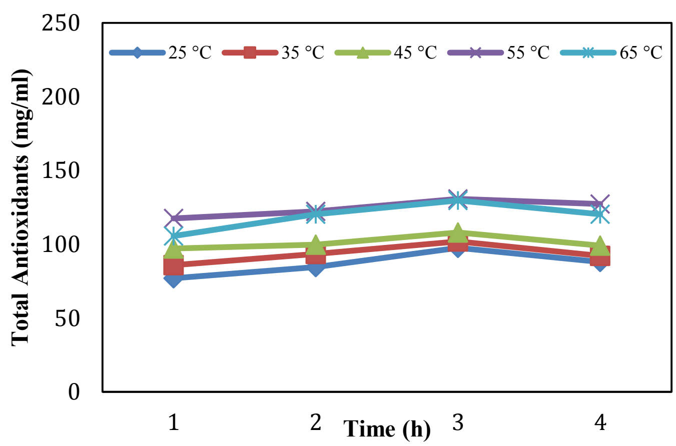
Figure 5.2. The effect of reaction time on the total antioxidants yield from ajwa date fruit using 50% acetone at different temperatures.



(a) Sample: solvent ratio 1:20

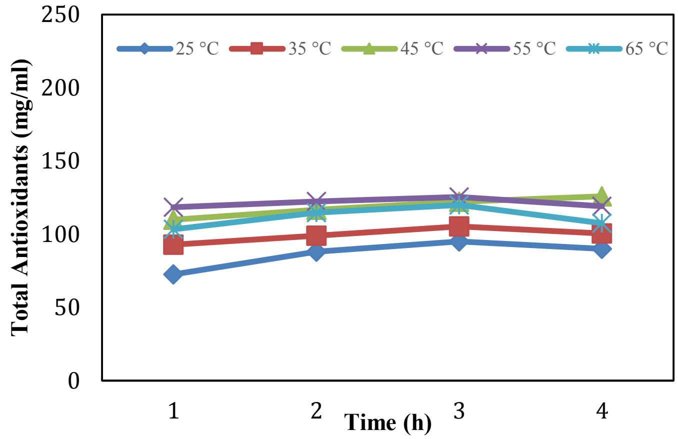


(b) Sample: solvent ratio 1:40

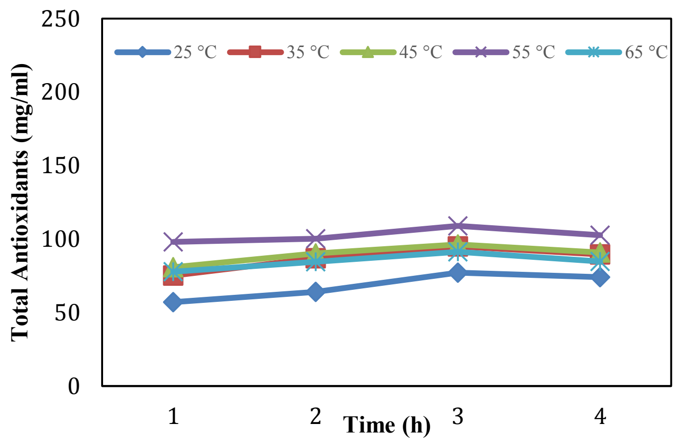


(c) Sample: solvent ratio 1:60

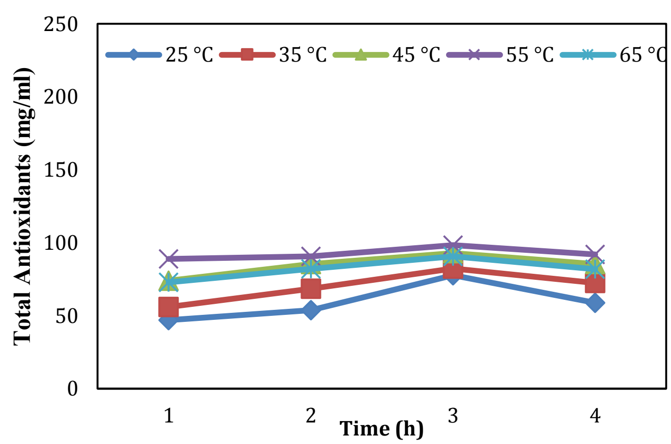
Figure 5.3. The effect of reaction time on the total antioxidants yield from ajwa date fruit using 75% acetone at different temperatures.



(a) Sample: solvent ratio 1:20

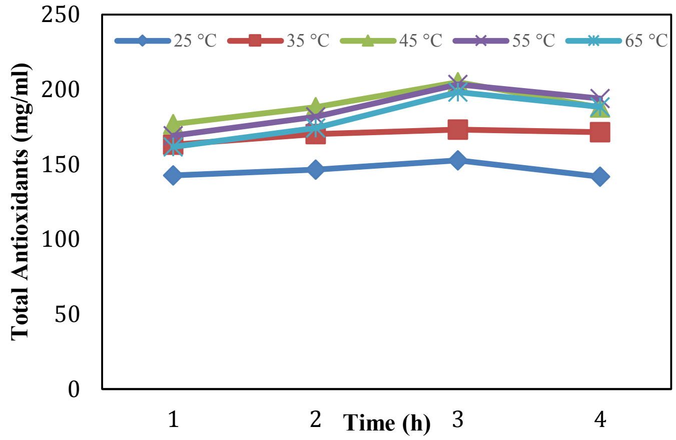


(b) Sample: solvent ratio 1:40

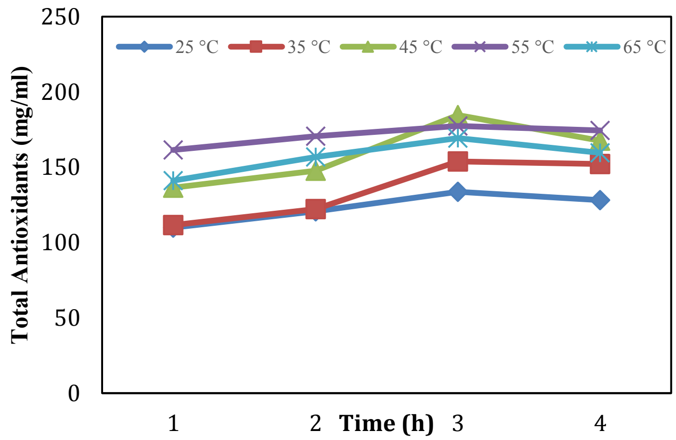


(c) Sample: solvent ratio 1:60

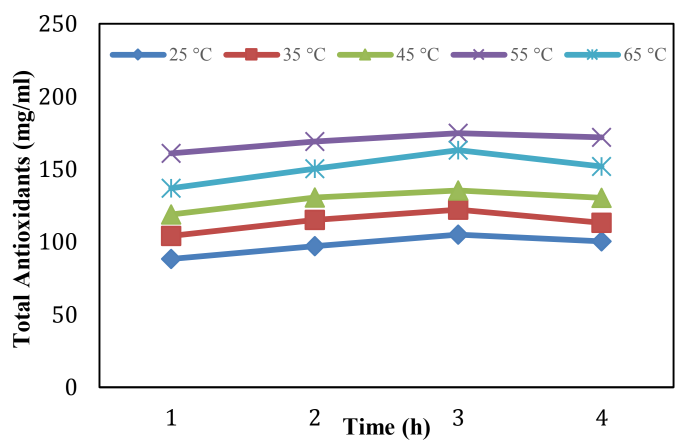
Figure 5.4. The effect of reaction time on the total antioxidants yield from ajwa date fruit using 50% ethanol at different temperatures.



(a) Sample: solvent ratio 1:20



(b) Sample: solvent ratio 1:40



(c) Sample: solvent ratio 1:60

Figure 5.5. The effect of reaction time on the total antioxidants yield from ajwa date fruit using 75% ethanol at different temperatures.

mg/ml (16.87%) and from 107.95 to 90.00 (16.63%) at the sample: solvent ratio of 1:20, from 56.63 to 55.75 mg/ml (1.55%), from 72.64 to 68.88 mg/ml (5.18%), from 74.46 to 70.52 mg/ml (5.29%), from 75.46 to 67.34 mg/ml (10.76%) and from 72.40 to 57.17 (21.04%) at the sample: solvent ratio of 1:40 and from 40.27 to 32.52 mg/ml (19.25%), from 39.52 to 37.11 mg/ml (6.10%), from 45.92 to 40.21 mg/ml (12.43%), from 51.71 to 47.95 mg/ml (7.27%) and from 40.97 to 38.92 (5%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^{o}C, respectively.

when acetone was used as solvent at 50% concentration, increasing the reaction time from 1 to 3 h, increased the antioxidants yield from 91.90 to 124.45 mg/ml (35.42%), from 116.70 to 134.03 mg/ml (14.85%), from 119.36 to 144.75 mg/ml (21.27%), from 126.43 to 151.18 mg/ml (19.58%) and from 103.02 to 119.65 (16.14%) at the sample: solvent ratio of 1:20, from 53.18 to 74.40 mg/ml (39.90%), from 69.21 to 90.11 mg/ml (30.20%), from 75.19 to 92.64 mg/ml (23.21%), from 55.63 to 96.70 mg/ml (73.83%) and from 75.25 to 82.93 (10.21%) at the sample: solvent ratio of 1:40 and from 44.61 to 59.44 mg/ml (33.24%), from 53.90 to 71.38 mg/ml (32.43%), from 69.23 to 81.08 mg/ml (17.12%), from 74.38 to 85.14 mg/ml (14.47%) and from 71.20 to 78.08 (9.66%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^{o}C, respectively. A further increase in reaction time from 3 to 4 h, decreased the antioxidants yield from 124.45 to 117.73 mg/ml (5.40%), from 134.03 to 119.11 mg/ml (11.13%), from 144.75 to 127.12 mg/ml (12.18%), from 151.18 to 128.64 mg/ml (14.91%) and from 119.65 to 102.48 (14.35%) at the sample: solvent ratio of 1:20, from 74.40 to 71.37 mg/ml (4.07%), from 90.11 to 77.75 mg/ml (13.72%), from 92.64 to 88.57 mg/ml (4.39%), from 96.70 to 92.80 mg/ml (4.03%) and from 82.93 to 72.29 (12.83%) at the sample: solvent ratio of 1:40 and from 59.44 to 52.24 mg/ml (12.11%), from 71.38 to 70.80 mg/ml (0.81%), from 81.08 to 75.73 mg/ml (6.60%), from 85.14 to 79.29 mg/ml (6.87%) and from 78.08 to 68.64 (12.09%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^{o}C, respectively.

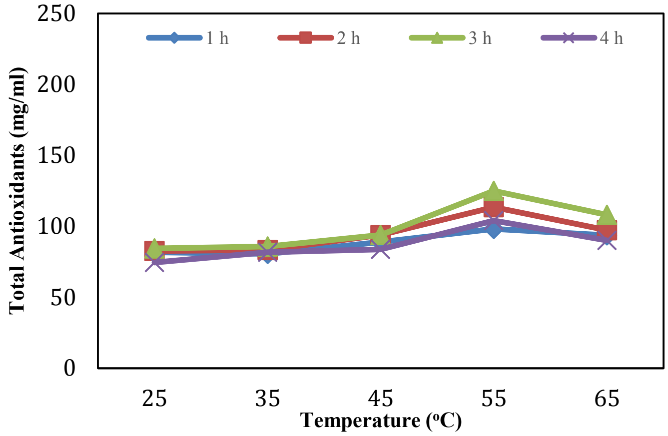
When acetone was used as solvent at 75% concentration, increasing the reaction time from 1 to 3 h, increased the antioxidants yield from 111.18 to 140.00 mg/ml (25.92%), from 129.75 to 155.83 mg/ml (20.10%), from 146.34 to 199.34 mg/ml (36.22%), from 173.24 to 201.18 mg/ml (16.13%) and from 146.49 to 194.55 (32.81%) at the sample:

solvent ratio of 1:20, from 100.27 to 113.16 mg/ml (12.86%), from 105.26 to 121.88 mg/ml (15.79%), from 110.58 to 134.07 mg/ml (21.24%), from 151.65 to 160.38 mg/ml (5.76%) and from 137.46 to 152.23 (10.74%) at the sample: solvent ratio of 1:40 and from 76.94 to 97.60 mg/ml (26.85%), from 85.70 to 101.74 mg/ml (18.72%), from 97.15 to 107.91 mg/ml (11.08%), from 117.37 to 130.74 mg/ml (11.39%) and from 105.50 to 129.61 (22.85%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^oC, respectively. A further increase in reaction time from 3 to 4 h, decreased the antioxidants yield from 140.00 to 130.12 mg/ml (7.06%), from 155.83 to 138.84 mg/ml (10.90%), from 204.34 to 197.62 mg/ml (3.29%), from 203.18 to 193.08 mg/ml (4.97%) and from 194.55 to 177.33 (8.85%) at the sample: solvent ratio of 1:20, from 113.16 to 99.77 mg/ml (11.83%), from 121.88 to 109.94 mg/ml (9.80%), from 134.07 to 118.70 mg/ml (11.46%), from 160.38 to 150.60 mg/ml (6.10%) and from 152.23 to 139.43 (8.41%) at the sample: solvent ratio of 1:40 and from 97.60 to 87.97 mg/ml (9.87%), from 101.74 to 92.08 mg/ml (9.49%), from 107.91 to 99.01 mg/ml (8.25%), from 130.74 to 127.29 mg/ml (2.64%) and from 129.61 to 120.27 (7.21%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^oC, respectively.

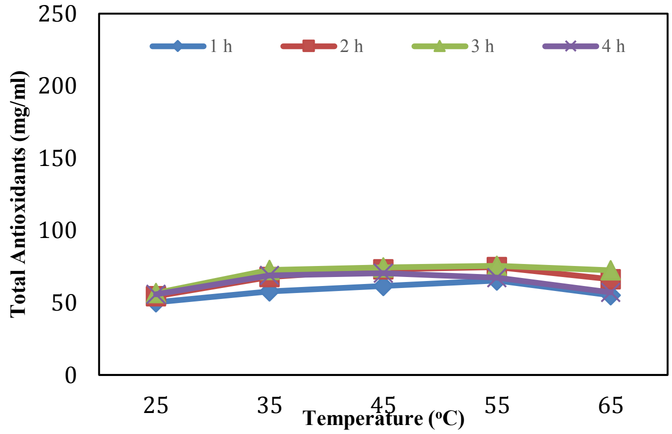
When ethanol was used as a solvent at 50% concentration increase in the reaction time from 1 to 3 h, increased the antioxidants yield from 72.66 to 95.14 mg/ml (30.94%), from 92.85 to 105.33 mg/ml (13.44%), from 109.88 to 122.10 mg/ml (11.12%), from 118.42 to 125.53 mg/ml (6.00%) and from 103.33 to 120.06 (16.19%) at the sample: solvent ratio of 1:20, from 57.13 to 77.25 mg/ml (35.22%), from 75.12 to 95.10 mg/ml (26.58%), from 80.94 to 96.42 mg/ml (19.13%), from 98.01 to 108.95 mg/ml (11.16%) and from 77.91 to 91.40 (17.31%) at the sample: solvent ratio of 1:40 and from 47.07 to 77.75 mg/ml (65.18%), from 55.99 to 82.29 mg/ml (46.97%), from 73.99 to 92.79 mg/ml (25.41%), from 89.01 to 98.30 mg/ml (10.44%) and from 72.89 to 90.60 (24.30%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^oC, respectively. A further increase in the reaction time from 3 to 4 h, decreased the antioxidants yield from 95.14 to 90.02 mg/ml (5.38%), from 105.33 to 100.43 mg/ml (4.65%), from 122.10 to 125.88 mg/ml (3.10%), from 125.53 to 119.14 mg/ml (5.09%) and from 120.06 to 107.46 (10.49%) at the sample: solvent ratio of 1:20, from 77.25 to 74.13 mg/ml (4.04%), from 95.10 to 89.65 mg/ml (5.73%), from 96.42 to 90.79 mg/ml (5.84%), from 108.95 to 102.68 mg/ml (5.75%) and from 91.40 to 84.80 (7.22%) at the sample: solvent ratio of 1:40 and from 77.75 to 58.74 mg/ml (24.45%), from 82.29 to 72.42 mg/ml (11.99%), from 92.79 to 85.33 mg/ml (8.04%), from 98.30 to 92.06 mg/ml (6.35%) and from 90.60 to 81.98 (9.51%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^oC, respectively.

When ethanol was used as a solvent at 75% concentration, increasing the reaction time from 1 to 3 h, increased the antioxidants yield from 142.85 to 152.69 mg/ml (6.89%), from 163.16 to 173.24 mg/ml (6.89%), from 176.88 to 204.74 mg/ml (15.54%), from 169.22 to 203.39 mg/ml (20.19%) and from 161.61 to 198.18 (22.63%) at the sample: solvent ratio of 1:20, from 109.94 to 133.72 mg/ml (21.63%), from 111.57 to 153.76 mg/ml (37.81%), from 136.40 to 184.57 mg/ml (35.32%), from 161.96 to 177.85 mg/ml (9.81%) and from 141.14 to 169.26 mg/ml (19.92%) at the sample: solvent ratio of 1:40 and from 88.33 to 105.10 mg/ml (18.99%), from 104.17 to 122.17 mg/ml (17.28%), from 118.90 to 135.45 mg/ml (13.92%), from 160.51 to 174.49 mg/ml (8.71%) and from 136.96 to 163.19 mg/ml (19.15%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^oC, respectively. A further increase in the reaction time from 3 to 4 h, decreased the antioxidants yield from 152.69 to 141.86 mg/ml (7.09%), from 173.24 to 171.49 mg/ml (1.01%), from 204.74 to 197.46 mg/ml (3.56%), from 203.39 to 193.82 mg/ml (4.71%) and from 198.18 to 177.40 (10.49%) at the sample: solvent ratio of 1:20, from 133.72 to 128.14 mg/ml (4.17%), from 153.76 to 152.23 mg/ml (1%), from 184.57 to 167.66 mg/ml (9.16%), from 177.85 to 174.88 mg/ml (1.67%) and from 169.26 to 159.67 (5.67%) at the sample: solvent ratio of 1:40 and from 105.10 to 100.44 mg/ml (4.43%), from 122.17 to 113.16 mg/ml (7.37%), from 135.45 to 130.42 mg/ml (3.71%), from 174.49 to 171.41 mg/ml (1.77%) and from 163.19 to 151.83 (6.96%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^oC respectively.

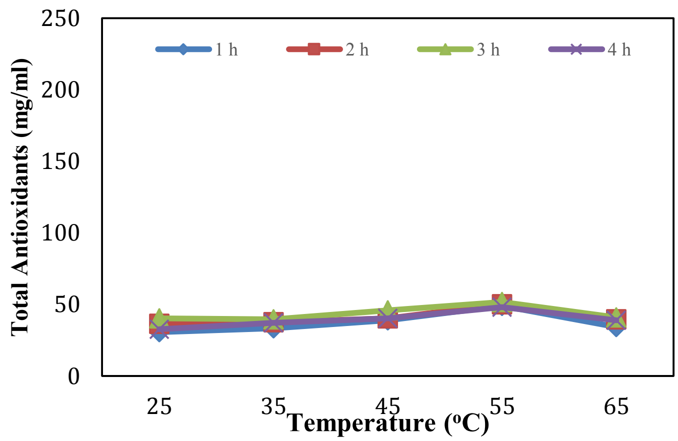
5.1.1.2. Effect of Reaction Temperature: Figures 5.6-5.10 show effect of reaction temperature on the total antioxidants yield from ajwa date fruit at different reaction times (1, 2, 3 and 4 h) and sample: solvent ratios (1:20, 1:40 and 1:60) using acetone and ethanol at different concentrations (0, 50 and 75%). Generally, there was an increase in the total antioxidants yield when the reaction temperature was increased from 25 to 55^oC for both solvents at all sample: solvent ratios and solvent concentrations. This was followed by a



(a) Sample: solvent ratio 1:20

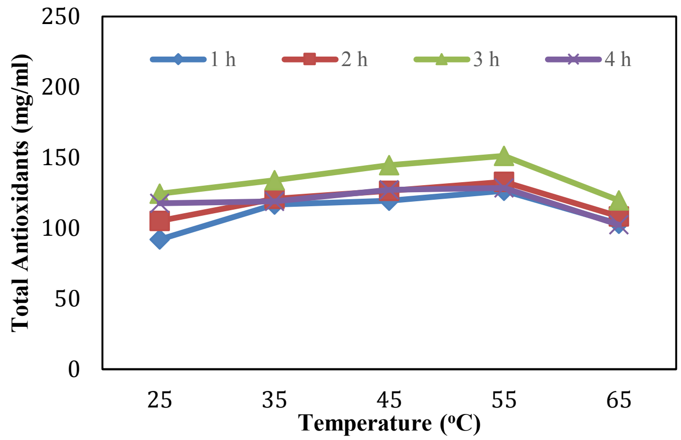


(b) Sample: solvent ratio 1:40

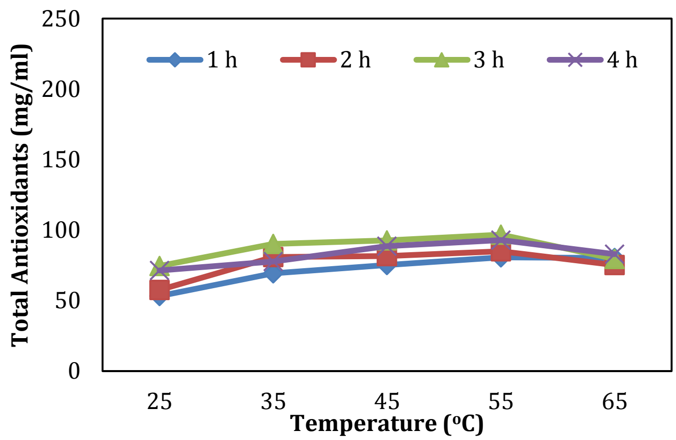


(c) Sample: solvent ratio 1:60

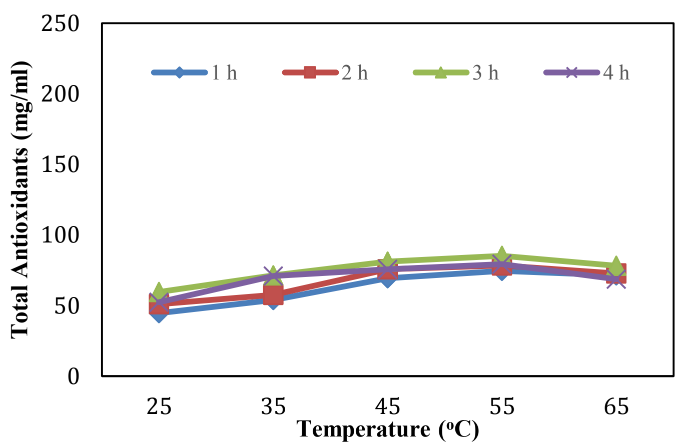
Figure 5.6. The effect of reaction temperature on the total antioxidants yield from ajwa date fruit using water at different reaction times.



(a) Sample: solvent ratio 1:20

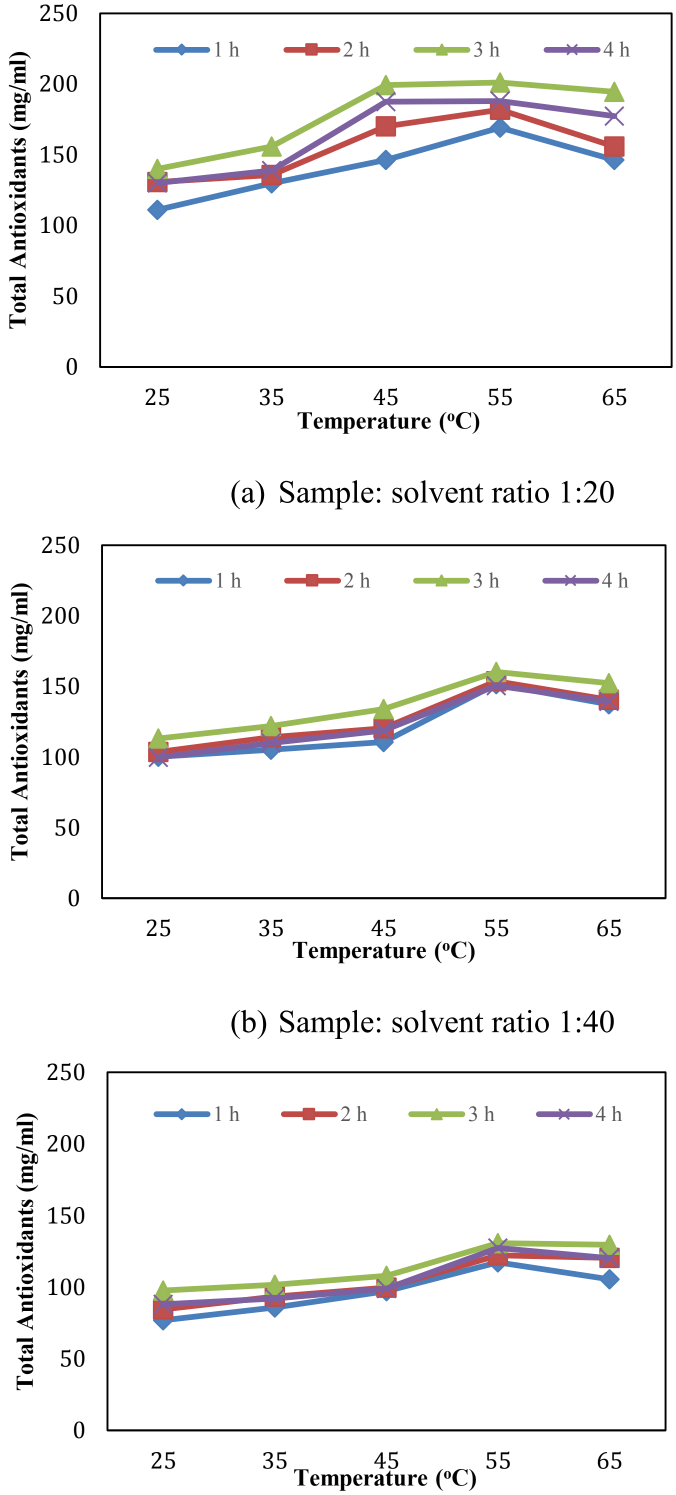


(b) Sample: solvent ratio 1:40



(c) Sample: solvent ratio 1:60

Figure 5.7. The effect of reaction temperature on the total antioxidants yield from ajwa date fruit using 50% acetone at different reaction times.

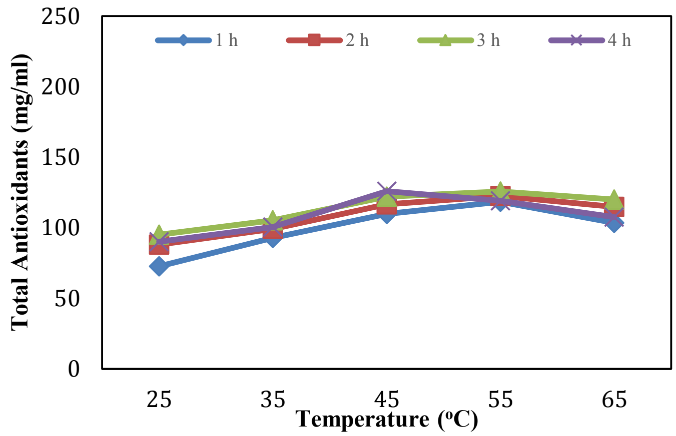


(a) Sample: solvent ratio 1:20

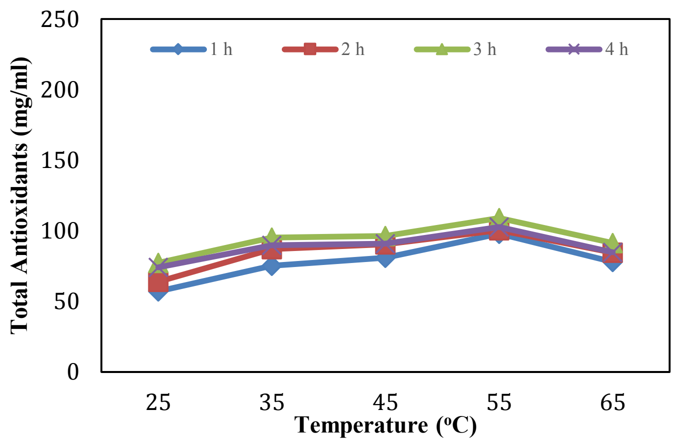
(b) Sample: solvent ratio 1:40

(c) Sample: solvent ratio 1:60

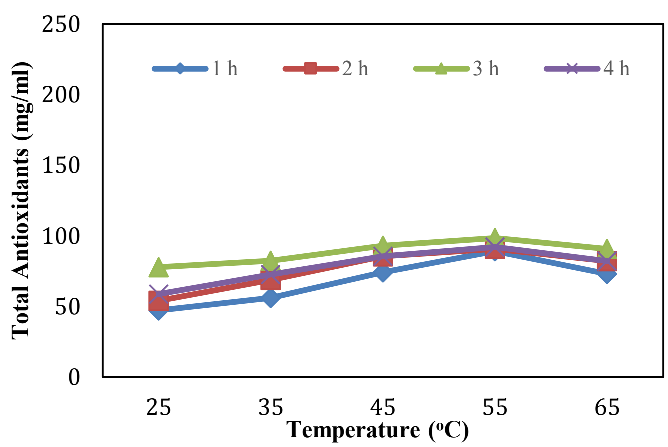
Figure 5.8. The effect of reaction temperature on the total antioxidants yield from ajwa date fruit using 75% acetone at different reaction times.



(a) Sample: solvent ratio 1:20

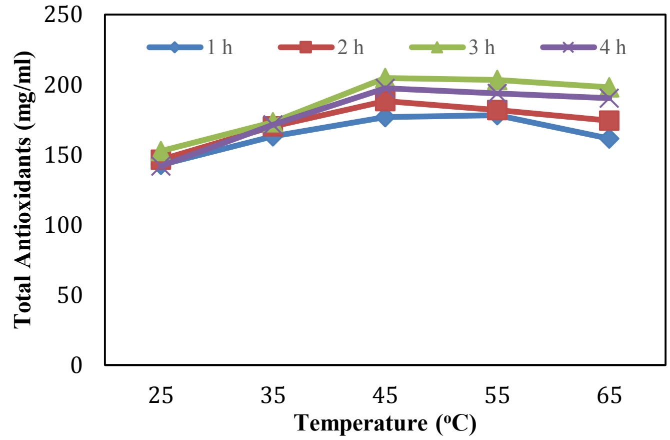


(b) Sample: solvent ratio 1:40

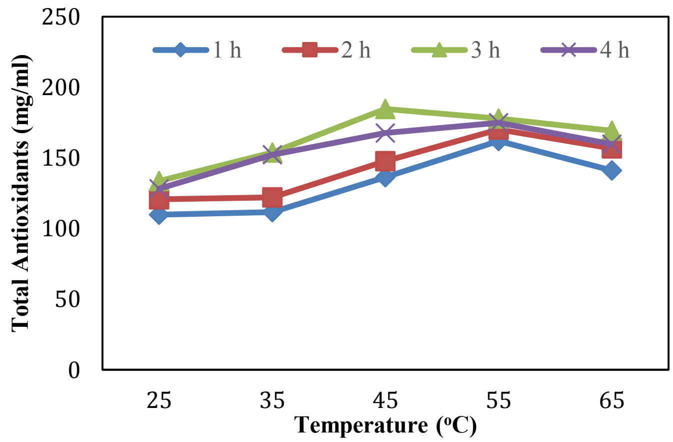


(c) Sample: solvent ratio 1:60

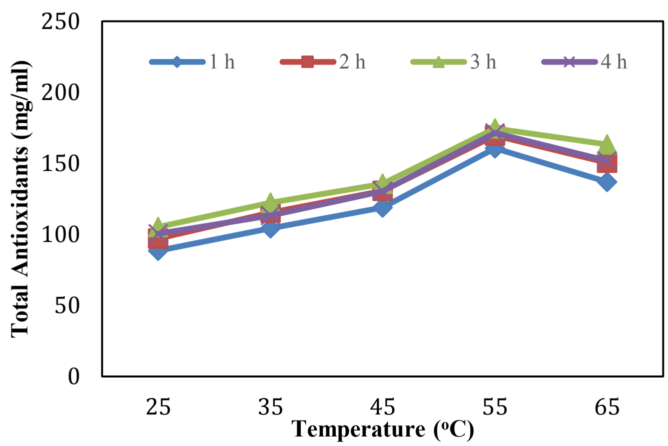
Figure 5.9. The effect of reaction temperature on the total antioxidants yield from ajwa date fruit using 50% ethanol at different reaction times.



(a) Sample: solvent ratio 1:20



(b) Sample: solvent ratio 1:40



(c) Sample: solvent ratio 1:60

Figure 5.10. The effect of reaction temperature on the total antioxidants yield from ajwa date fruit using 75% ethanol at different reaction times.

decrease in the total antioxidants with a further increase in reaction temperatures from 55 to 65 ^{o}C.

When water was used as a solvent, increasing the reaction temperature from 25 to 55^{o}C, increased the antioxidants yield from 80.61 to 98.08 mg/ml (21.67%), from 82.58 to 113.37 mg/ml (37.29%), from 84.43 to 124.98 mg/ml (48.03%) and from 74.54 to 103.90 mg/ml (39.39%) at the sample: solvent ratio of 1:20, from 50.27 to 65.35 mg/ml (30%), from 54.44 to 74.60 mg/ml (37.03%), from 56.63 to 75.46 mg/ml (33.25%) and from 55.75 to 67.34 mg/ml (20.79%) at the sample: solvent ratio of 1:40 and from 30.60 to 48.88 mg/ml (59.74%), from 36.34 to 50.08 mg/ml (37.81%), from 40.27 to 51.71 mg/ml (28.41%) and from 32.52 to 47.95 mg/ml (47.45%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively. A further increase in the reaction temperature from 55 to 65^{o}C, decreased the antioxidants yield from 98.08 to 93.53 mg/ml (4.64%), from 113.37 to 97.35 mg/ml (14.13%), from 124.98 to 107.95 mg/ml (13.63%) and from 103.90 to 90.00 mg/ml (13.38%) at the sample: solvent ratio of 1:20, from 65.35 to 55.06 mg/ml (15.75%), from 74.60 to 66.16 mg/ml (11.31%), from 75.46 to 72.40 mg/ml (4.06%) and from 67.34 to 57.17 mg/ml (15.10%) at the sample: solvent ratio of 1:40 and from 48.88 to 34.23 mg/ml (29.97%), from 50.08 to 39.44 mg/ml (21.25%), from 51.71 to 40.97 mg/ml (20.77%) and from 47.95 to 38.92 mg/ml (18.83%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h respectively.

When acetone was used as solvent at 50 % concentration, increasing the reaction temperature from 25 to 55^{o}C, increased the antioxidants yield from 91.90 to 126.43 mg/ml (37.57%), from 105.19 to 132.75 mg/ml (26.20%), from 124.45 to 151.18 mg/ml (21.48%) and from 117.73 to 128.64 mg/ml (9.27%) at the sample: solvent ratio of 1:20, from 53.18 to 55.63 mg/ml (4.61%), from 57.50 to 84.98 mg/ml (47.79%), from 74.40 to 96.70 mg/ml (29.80%) and from 71.37 to 92.80 mg/ml (30.03%) at the sample: solvent ratio of 1:40 and from 44..61 to 74.38 mg/ml (66.73%), from 50.93 to 78.35 mg/ml (53.84%), from 59.44 to 85.14 mg/ml (43.24%) and from 52.24 to 79.29 mg/ml (51.78%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively. A further increase in the reaction temperature from 55 to 65^{o}C, decreased the antioxidants yield from 126.43 to 103.02 mg/ml (18.52%), from 132.75 to 108.24 mg/ml (18.46%), from 151.18 to 119.65 mg/ml (20.86%) and from 128.64 to 102.48 mg/ml (20.34%) at the sample: solvent ratio of 1:20, from 80.63 to 75.25 mg/ml (6.67%), from 84.98 to 79.57 mg/ml (6.37%), from 96.70 to 82.93 mg/ml (14.24%) and from 92.80 to 72.29 mg/ml (22.10%) at the sample: solvent ratio of 1:40 and from 74.38 to 71.20 mg/ml (4.28%), from 78.35 to 72.58 mg/ml (7.36%), from 85.14 to 78.08 mg/ml (8.29%) and from 79.29 to 68.64 mg/ml (13.43%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

When acetone was used as solvent at 75 % concentration, increasing the reaction temperature from 25 to 55^{o}C, increased the antioxidants yield from 111.18 to 169.24 mg/ml (52.22%), from 130.86 to 181.80 mg/ml (38.93%), from 140.00 to 201.18 mg/ml (43.70%) and from 130.12 to 188.08 mg/ml (44.54%) at the sample: solvent ratio of 1:20, from 100.27 to 151.65 mg/ml (51.24%), from 103.57 to 153.64 mg/ml (48.34%), from 113.16 to 160.38 mg/ml (41.73%) and from 99.77to 150.60 mg/ml (50.95%) at the sample: solvent ratio of 1:40 and from 76.94 to 117.37 mg/ml (52.55%), from 84.54 to 122.29 mg/ml (44.65%), from 97.60 to 130.74 mg/ml (33.95%) and from 87.97 to 127.29 mg/ml (44.70%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively. A further increase in the reaction temperature from 55 to 65^{o}C, decreased the antioxidants yield from 169.24 to 146.49 mg/ml (13.44%), from 181.80 to 155.87 mg/ml (14.26%), from 201.18 to 194.55 mg/ml (3.30%) and from 188.08 to 177.33 mg/ml (5.72%) at the sample: solvent ratio of 1:20, from 151.65 to 137.46 mg/ml (9.36%), from 153.64 to 140.52 mg/ml (8.54%), from 160.38 to 152.23 mg/ml (5.08%) and from 150.60 to 139.43 mg/ml (7.42%) at the sample: solvent ratio of 1:40 and from 117.37 to 105.50 mg/ml (10.11%), from 122.29 to 120.35 mg/ml (1.59%), from 130.74 to 129.61 mg/ml (0.86%) and from 127.29 to 120.27 mg/ml (5.51%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

When ethanol was used as solvent at 50 % concentration, increasing the reaction temperature from 25 to 55^{o}C, increased the antioxidants yield from 72.66 to 118.42 mg/ml (62.98%), from 88.14 to 122.51 mg/ml (38.99%), from 95.14 to 125.53 mg/ml (31.94%) and from 90.02 to 119.14 mg/ml (32.35%) at the sample: solvent ratio of 1:20, from 57.13 to 98.01 mg/ml (71.56%), from 63.99 to 100.37 mg/ml (56.85%), from 77.25 to 108.95 mg/ml (41.04%) and from 74.13 to 102.68 mg/ml (38.51%) at the sample: solvent ratio of 1:40 and from 47.07 to 89.01 mg/ml (89.10%), from 53.90 to 90.74 mg/ml (68.35%), from 77.75 to 98.30 mg/ml (26.43%) and from 58.74 to 92.06 mg/ml (56.72%) at the sample:

solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively. A further increase in the reaction temperature from 55 to 65°C, decreased the antioxidants yield from 118.42 to 103.33 mg/ml (12.74%), from 122.51 to 114.81 mg/ml (8.53%), from 125.53 to 120.06 mg/ml (4.36%) and from 119.14 to 107.46 mg/ml (9.80%) at the sample: solvent ratio of 1:20, from 98.01 to 77.91 mg/ml (20.05%), from 100.37 to 84.54 mg/ml (15.77%), from 108.95 to 91.40 mg/ml (16.11%) and from 102.68 to 84.80 mg/ml (17.41%) at the sample: solvent ratio of 1:40 and from 89.01 to 72.89 mg/ml (18.11%), from 90.74 to 82.11 mg/ml (9.51%), from 98.30 to 90.60 mg/ml (7.83%) and from 92.06 to 81.98 mg/ml (10.95%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

When ethanol was used as solvent at 75 % concentration, increasing the reaction temperature from 25 to 55°C, increased the antioxidants yield from 25 to 55°C, increased the antioxidants yield from 142.85 to 178.22 mg/ml (24.76%), from 146.51 to 181.82 mg/ml (24.10%), from 152.69 to 203.39 mg/ml (33.20%) and from 141.86 to 193.80 mg/ml (36.61%) at the sample: solvent ratio of 1:20, from 109.94 to 161.96 mg/ml (47.32%), from 120.83 to 170.09 mg/ml (40.77%), from 133.72 to 177.85 mg/ml (33%) and from 128.14 to 174.88 mg/ml (36.48%) at the sample: solvent ratio of 1:40 and from 88.33 to 160.51 mg/ml (81.72%), from 97.02 to 169.65 mg/ml (74.86%), from 105.10 to 174.49 mg/ml (66.02%) and from 100.44 to 171.41 mg/ml (70.66%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively. A further increase in the reaction temperature from 55 to 65°C, decreased the antioxidants yield from 178.22 to 161.61 mg/ml (9.32%), from 181.82 to 174.28 mg/ml (4.15%), from 203.39 to 198.18 mg/ml (2.56%) and from 193.80 to 190.40 mg/ml (1.75%) at the sample: solvent ratio of 1:20, from 161.96 to 141.14 mg/ml (12.86%), from 170.09 to 156.85 mg/ml (13.24%), from 177.85 to 169.26 mg/ml (8.59%) and from 174.88 to 159.67 mg/ml (8.70%) at the sample: solvent ratio of 1:40 and from 160.51 to 136.96 mg/ml (14.67%), from 169.65 to 150.35 mg/ml (11.38%), from 174.49 to 163.19 mg/ml (6.40%) and from 171.41 to 151.83 mg/ml (11.42%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

5.1.1.3. Effect of Sample: Solvent Ratio: Figures 5.11-5.15 effect of sample: solvent ratio on the total antioxidants yield from ajwa date fruit at different reaction times (1, 2, 3 and 4

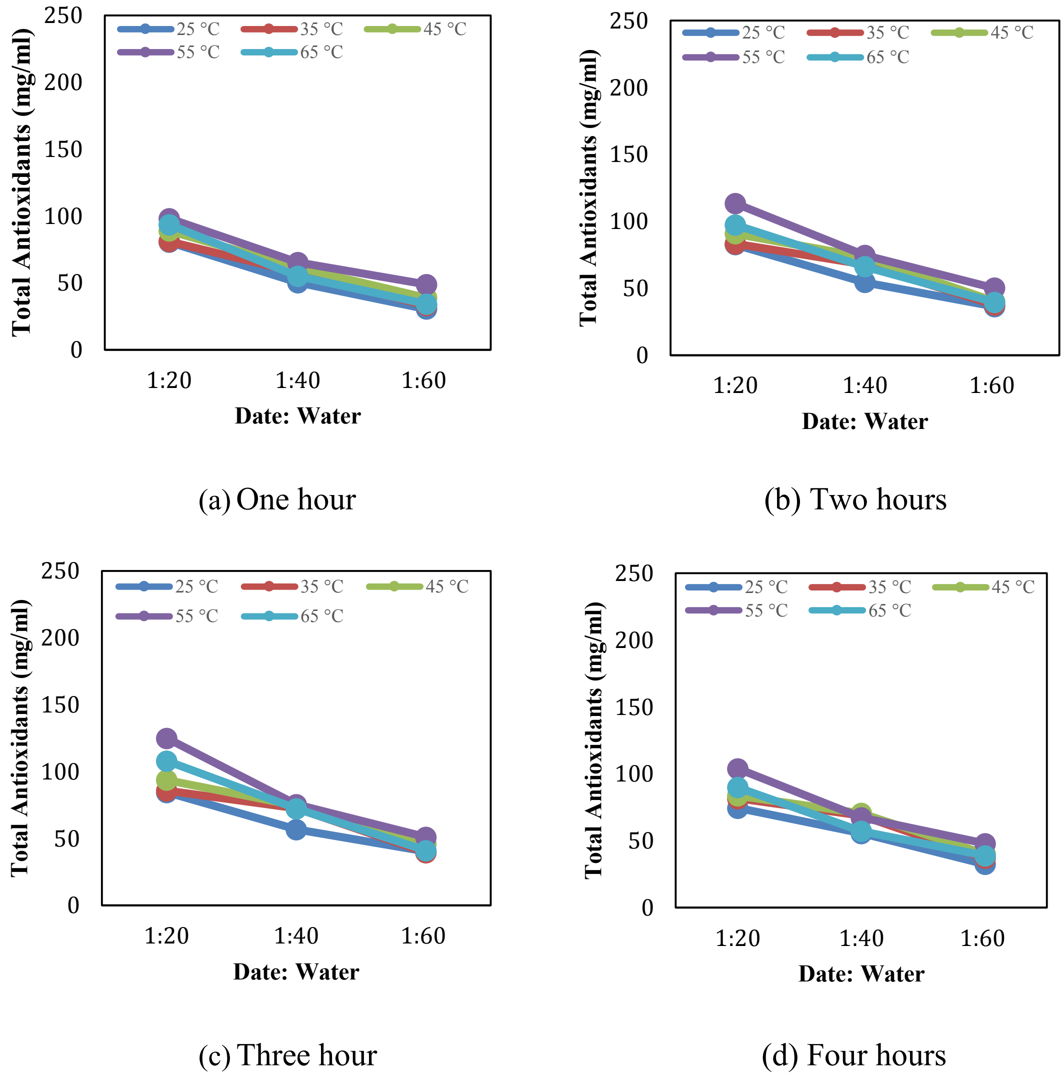
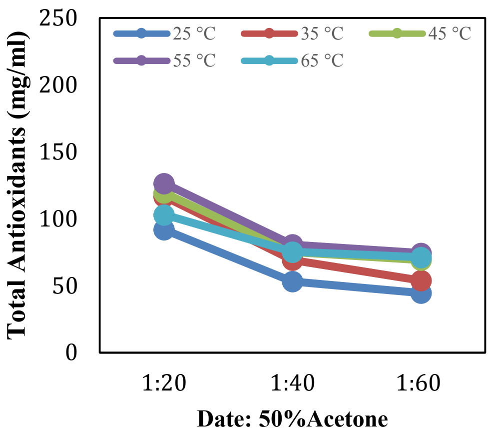
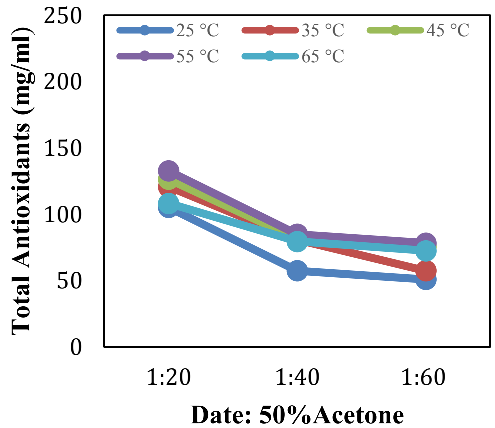


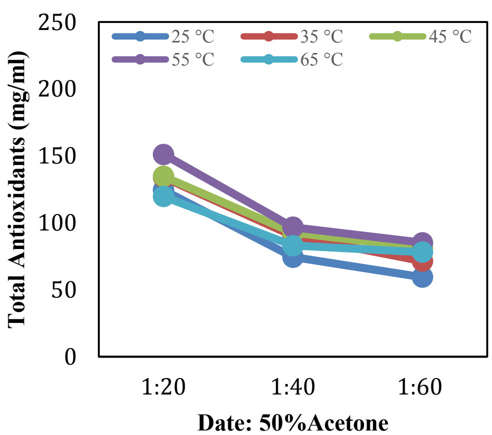
Figure 5.11. The effect of sample: solvent ratio on the total antioxidants yield from ajwa date fruit using water at different reaction temperatures.



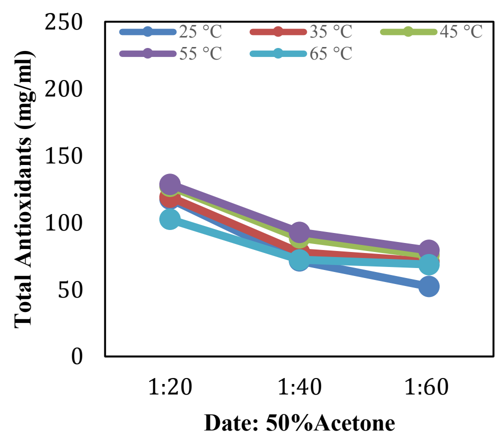
(a) One hour



(b) Two hours

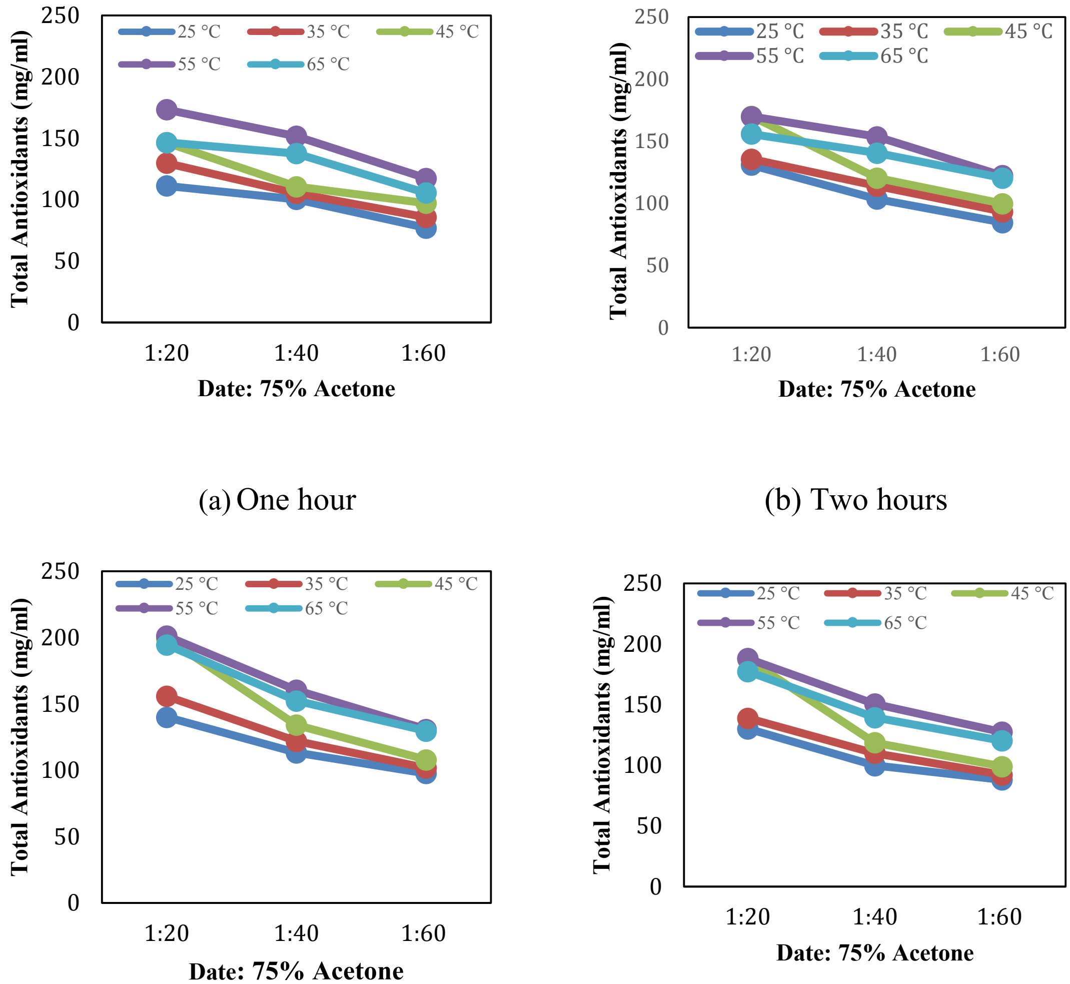


(c) Three hour



(d) Four hours

Figure 5.12. The effect of sample: solvent ratio on the total antioxidants yield from ajwa date fruit using 50% acetone at different reaction temperatures.



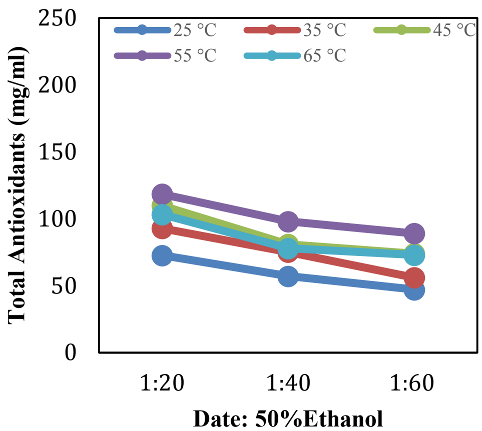
(a) One hour

(b) Two hours

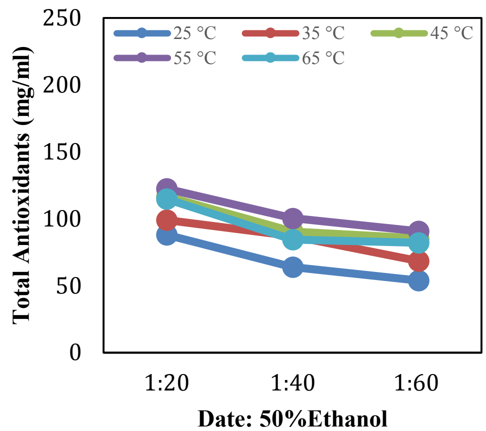
(c) Three hour

(d) Four hours

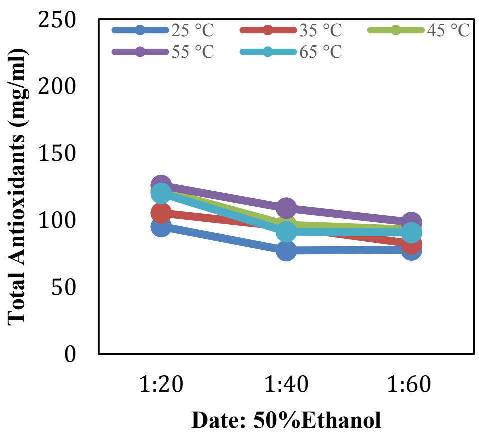
Figure 5.13. The effect of sample: solvent ratio on the total antioxidants yield from ajwa date fruit using 75% acetone at different reaction temperatures.



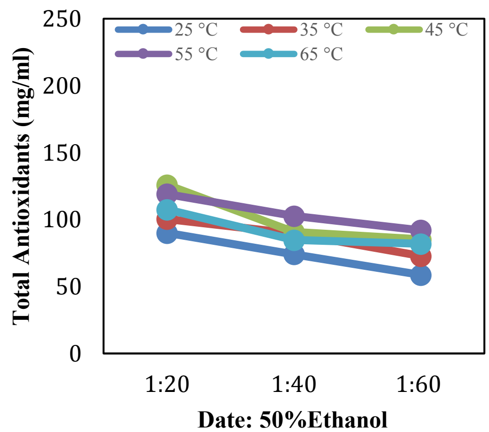
(a) One hour



(b) Two hours

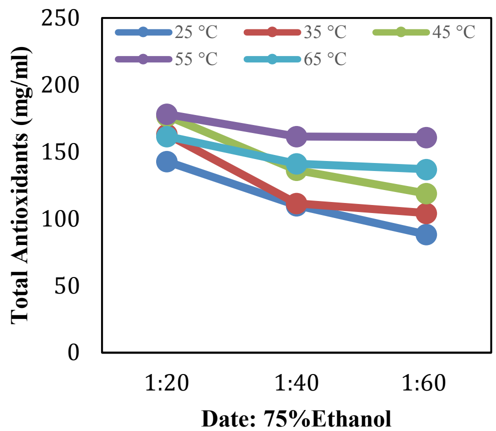


(c) Three hour

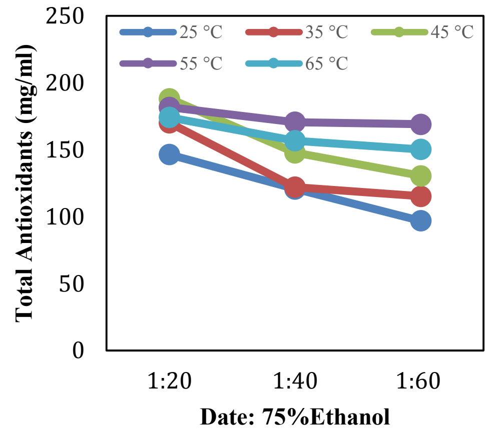


(d) Four hours

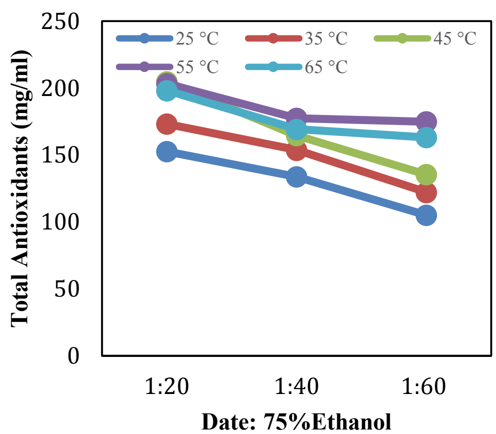
Figure 5.14. The effect of sample: solvent ratio on the total antioxidants yield from ajwa date fruit using 50% ethanol at different reaction temperatures.



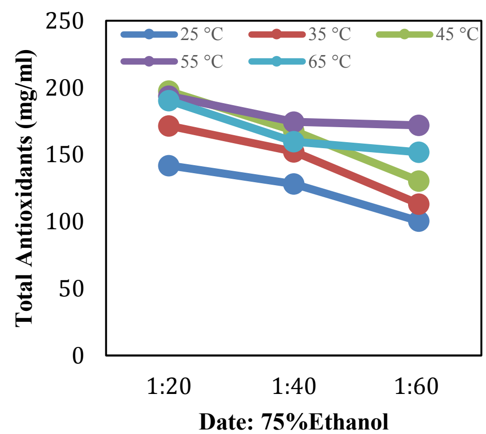
(a) One hour



(b) Two hours



(c) Three hour



(d) Four hours

Figure 5.15. The effect of sample: solvent ratio on the total antioxidants yield from ajwa date fruit using 75% ethanol at different reaction temperatures.

h) and reaction temperatures (25, 35, 45, 55 and 65°C) using (acetone and ethanol) at different concentrations (0, 50 and 75%). Generally, there was a decrease in the total antioxidants yield when sample: solvent ratio was increased from 1:20 to 1:60 at all reaction temperatures, solvents, solvent concentration and reaction times.

When water was used as solvent, increasing the sample: solvent ratio from 1:20 to 1:60, decreased the antioxidants yield from 80.61 to 30.60 mg/ml (62.04%), from 81.05 to 33.22 mg/ml (59.01%), from 88.86 to 38.97 mg/ml (56.14%), from 98.08 to 48.88 mg/ml (50.16%) and from 93.53 to 34.23 mg/ml (63.40%) at the reaction time of 1 h, from 82.58 to 36.34 mg/ml (55.99%), from 83.20 to 37.59 mg/ml (54.82%), from 90.76 to 40.12 mg/ml (55.8%), from 113.37 to 50.08 mg/ml (55.83%) and from 97.35 to 39.44 mg/ml (59.49%) at the reaction time of 2 h, from 84.43 to 40.27 mg/ml (52.30%), from 85.70 to 39.52 mg/ml (53.89%), from 93.76 to 45.92 mg/ml (51.02%), from 124.98 to 51.71 mg/ml (58.63%) and from 107.95 to 40.97 mg/ml (62.05%) at the reaction time of 3 h and from 74.54 to 32.52 mg/ml (56.37%), from 81.67 to 37.11 mg/ml (54.56%), from 83.70 to 40.21 mg/ml (51.96%), from 103.90 to 47.95 mg/ml (53.85%) and from 90.00 to 38.92 mg/ml (56.76%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65 °C, respectively.

When acetone was used as solvent at 50 % concentration, increasing the sample: solvent ratio from 1:20 to 1:60, decreased the antioxidants yield from 91.90 to 44.61 mg/ml (51.46%), from 116.70 to 53.90 mg/ml (53.81%), from 119.36 to 69.23 mg/ml (42%), from 126.43 to 74.38 mg/ml (41.17%) and from 103.02 to 71.20 mg/ml (30.89%) at the reaction time of 1 h, from 105.19 to 50.93 mg/ml (51.58%), from 120.80 to 57.46 mg/ml (52.43%), from 126.45 to 75.72 mg/ml (40.12%), from 132.75 to 78.35 mg/ml (40.98%) and from 108.24 to 72.58 mg/ml (32.95%) at the reaction time of 2 h, from 124.45 to 59.44 mg/ml (52.24%), from 134.03 to 71.38 mg/ml (46.74%), from 144.75 to 81.08 mg/ml (43.99%), from 151.18 to 85.14 mg/ml (43.68%) and from 119.65 to 78.08 mg/ml (34.74%) at the reaction time of 3 h and from 117.73 to 52.24 mg/ml (55.63%), from 119.11 to 70.80 mg/ml (40.56%), from 127.12 to 75.73 mg/ml (40.43%), from 128.64 to 79.29 mg/ml (38.36%) and from 102.48 to 6.64 mg/ml (33.02%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65 °C, respectively.

When acetone was used as solvent at 75% concentration, increasing the sample: solvent ratio from 1:20 to 1:60, decreased the antioxidants yield from 111.18 to 76.94 mg/ml (30.8%), from 129.75 to 85.70 mg/ml (33.95%), from 146.34 to 97.15 mg/ml (33.61%), from 169.24 to 117.37 mg/ml (30.65%) and from 146.49 to 105.50 mg/ml (27.98%) at the reaction time of 1 h, from 130.86 to 84.54mg/ml (35.40%), from 135.64 to 93.37 mg/ml (31.16%), from 170.08 to 99.73 mg/ml (41.36%), from 181.80 to 122.29 mg/ml (32.73%) and from 155.87 to 120.35 mg/ml (22.79%) at the reaction time of 2 h, from 140.00 to 97.60 mg/ml (30.29%), from 155.83 to 101.74 mg/ml (34.71%), from 199.34 to 107.91 mg/ml (45.87%), from 201.18 to 130.74 mg/ml (35.01%) and from 194.55 to 129.61 mg/ml (33.38%) at the reaction time of 3 h and from 130.12 to 87.97 mg/ml (32.39%), from 138.84 to 92.08 mg/ml (33.68%), from 187.62 to 99.01 mg/ml (89.50%), from 188.08 to 127.29 mg/ml (32.32%) and from 177.33 to 120.27 mg/ml (32.18%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

When ethanol was used as solvent at 50% concentration, increasing the sample: solvent ratio from 1:20 to 1:60, decreased the antioxidants yield from 72.66 to 47.07 mg/ml (35.22%), from 92.85 to 55.99 mg/ml (39.70%), from 109.88 to 73.99 mg/ml (32.66%), from 118.42 to 89.01 mg/ml (24.84%) and from 103.33 to 72.89 mg/ml (29.46%) at the reaction time of 1 h, from 88.14 to 53.90 mg/ml (38.85%), from 99.03 to 68.53 mg/ml (30.80%), from 116.61 to 85.38 mg/ml (26.78%), from 122.51 to 90.74 mg/ml (25.93%) and from 114.81 to 82.11 mg/ml (28.48%) at the reaction time of 2 h, from 95.14 to 77.75 mg/ml (18.28%), from 105.33 to 82.29 mg/ml (21.87%), from 122.10 to 92.79 mg/ml (24%), from 125.53 to 98.30 mg/ml (21.69%) and from 120.06 to 90.60 mg/ml (24.54%) at the reaction time of 3 h and from 90.02 to 58.74 mg/ml (34.75%), from 100.43 to 72.42 mg/ml (27.89%), from 125.88 to 85.33 mg/ml (32.21%), from 119.14 to 92.06 mg/ml (22.73%) and from 107.46 to 81.98 mg/ml (23.71%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65 ^{o}C, respectively.

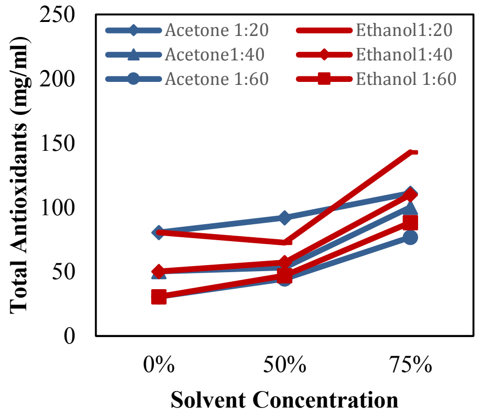
When ethanol was used as solvent at 75% concentration, increasing the sample: solvent ratio from 1:20 to 1:60, decreased the antioxidants yield from 142.85 to 88.33 mg/ml (38.17%), from 163.16 to 104.17 mg/ml (36.15%), from 176.88 to 118.90 mg/ml (32.78%), from 178.22 to 160.51 mg/ml (9.94%) and from 161.61 to 136.96 mg/ml

(15.25%) at the reaction time of 1 h, from 146.51 to 97.02 mg/ml (33.78%), from 170.16 to 115.25 mg/ml (32.27%), from 188.28 to 130.58 mg/ml (57.70%), from 181.82 to 169.65 mg/ml (6.69%) and from 174.28 to 150.35 mg/ml (13.73%) at the reaction time of 2 h, from 152.69 to 105.10 mg/ml (31.17%), from 173.24 to 122.17 mg/ml (29.98%), from 204.78 to 135.45 mg/ml (33.86%), from 203.39 to 174.49 mg/ml (14.21%) and from 198.18 to 163.19 mg/ml (17.66%) at the reaction time of 3 h and from 141.86 to 100.44 mg/ml (29.20%), from 171.49 to 113.16 mg/ml (34.01%), from 197.46 to 130.42 mg/ml (33.95%), from 193.80 to 171.41 mg/ml (11.55%) and from 190.40 to 151.83 mg/ml (20.26%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65 ^{o}C, respectively.

5.1.1.4. Effect of Solvent Concentration: Figures 5.16-5.20 show the effect of solvent (acetone and ethanol) concentration on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios (1:20, 1:40 and 1:60), reaction times (1, 2, 3 and 4 h) for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C. Generally, there was an increase in the total antioxidants yield when solvent concentration was increased from 0 to 75% for all reaction temperatures, sample: solvent ratios and reaction times for both solvents.

Increasing the concentration of acetone from 0 to 75% at the temperature of 25^{o}C increased the total antioxidants yield from 80.61 to 111.18 mg/ml (37.92%), from 50.24 to 100.27 mg/ml (99.46%) and from 30.60 to 76.94 mg/ml (151.44%) at the reaction time of 1 h, from 82.58 to 130.86 mg/ml (58.46%), from 54.44 to 103.57 mg/ml (47.44%) and from 36.34 to 84.54 mg/ml (132.64%) at the reaction time of 2 h, from 84.43 to 140.00 mg/ml (65.82%), from 56.63 to 113.16 mg/ml (99.82%) and from 40.27 to 97.60 mg/ml (142.36%) at the reaction time of 3 h and from 74.54 to 130.12 mg/ml (74.56%), from 55.75 to 99.77 mg/ml (78.96%) and from 32.52 to 87.97 mg/ml (170.52%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 35^{o}C increased the total antioxidants yield from 81.05 to 129.75 mg/ml (60.09%), from 57.79 to 105.26 mg/ml(82.14%) and from 33.22 to 85.70 mg/ml (157.98%) at the reaction time of 1 h, from 83.20 to 135.64 mg/ml (63.03%), from 67.77 to 113.93 mg/ml (68.11%) and from 37.59 to 93.37 mg/ml (148.39%) at the reaction time of 2 h, from 85.70 to 155.83 mg/ml (81.83%), from 72.64 to 121.88 mg/ml (67.79%) and from 39.52 to 101.74 mg/ml



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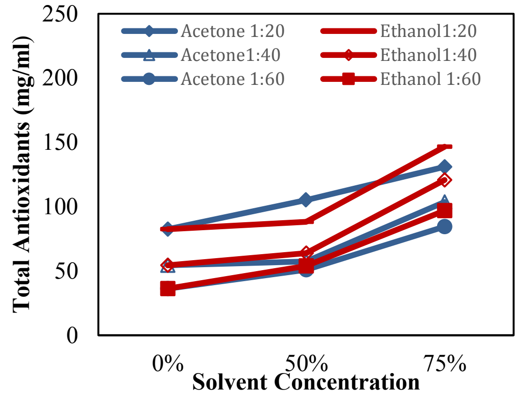

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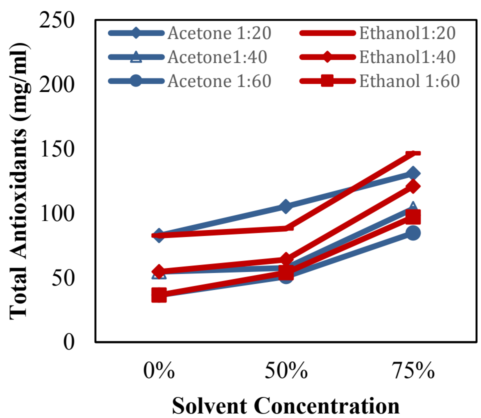
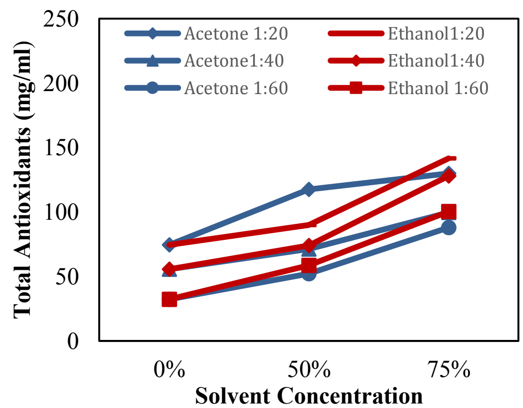
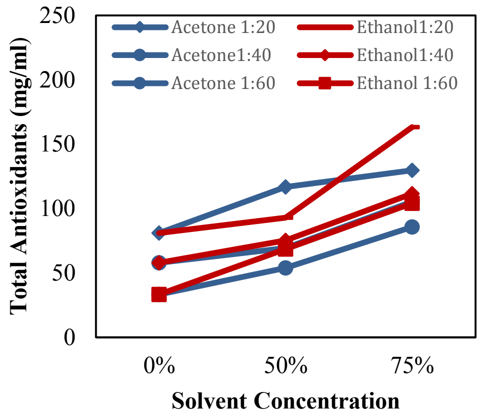


(c) Three hours

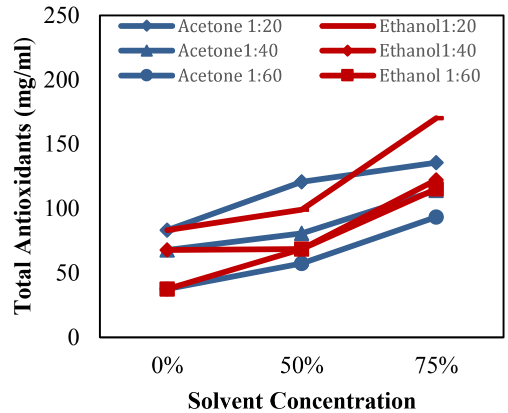


(d) Four hours

Figure 5.16. The effect of solvent (acetone and ethanol) concentration on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 25°C.



(a) One hour



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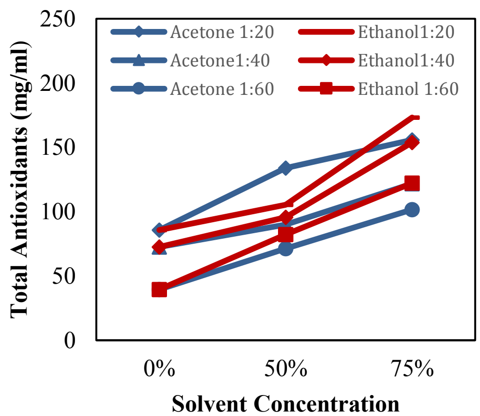

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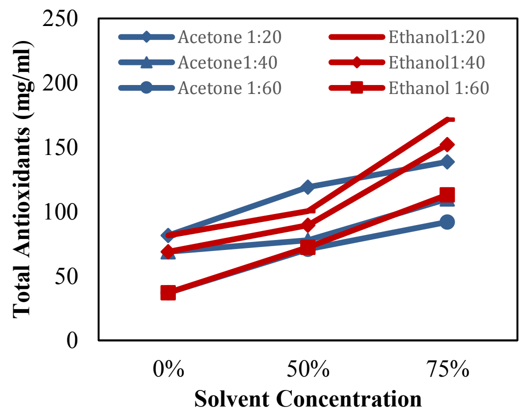
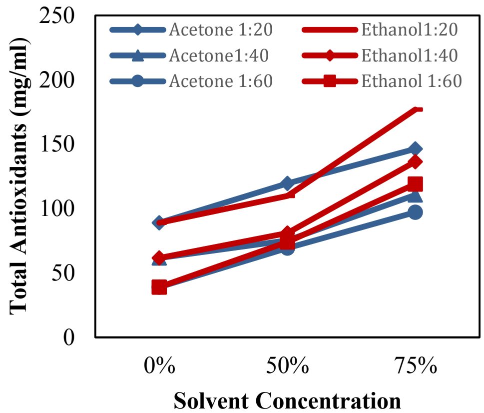
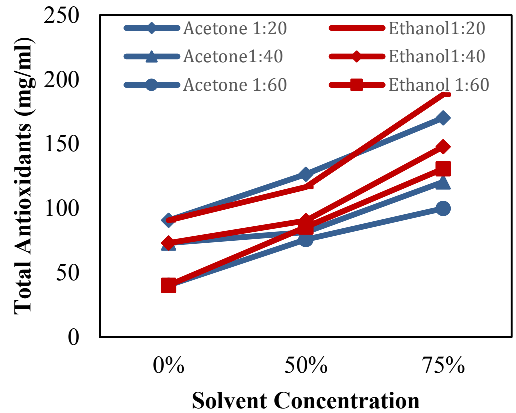


(d) Four hours

Figure 5.17. The effect of solvent (acetone and ethanol) concentration on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 35°C.



(a) One hour



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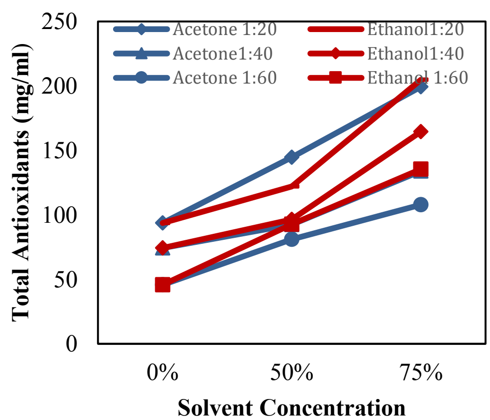

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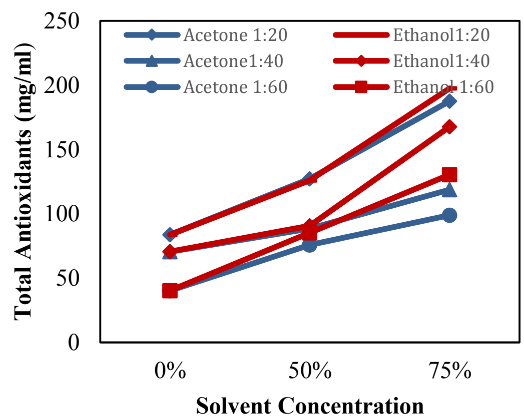
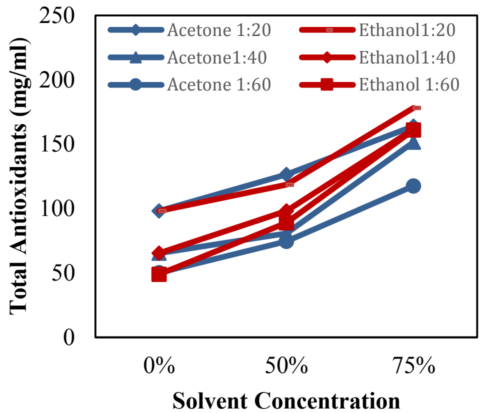
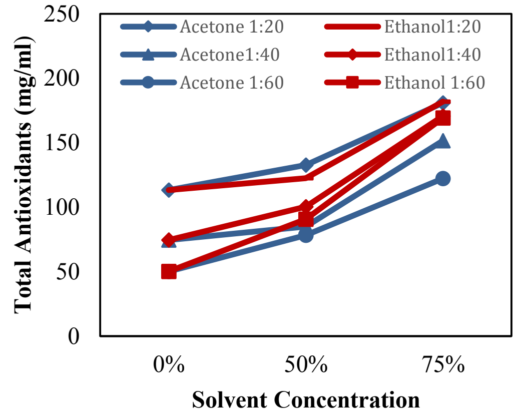


(d) Four hours

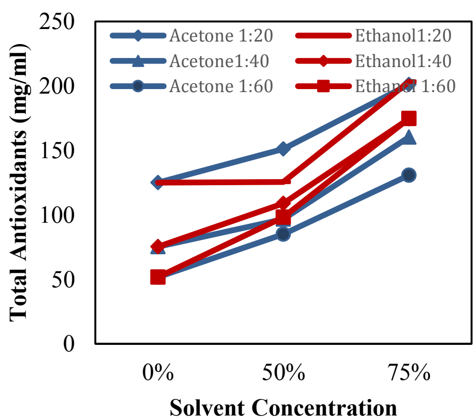
Figure 5.18. The effect of solvent (acetone and ethanol) concentration on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 45^{o}C.



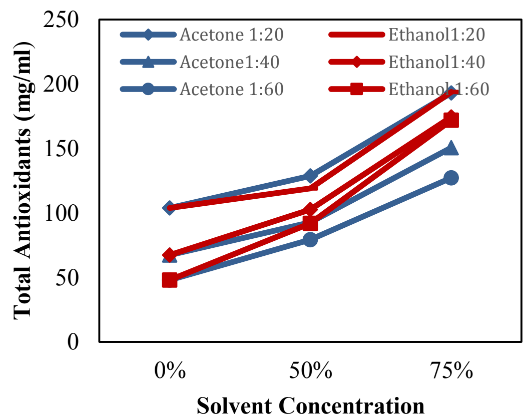
(a) One hour



(b) Two hours

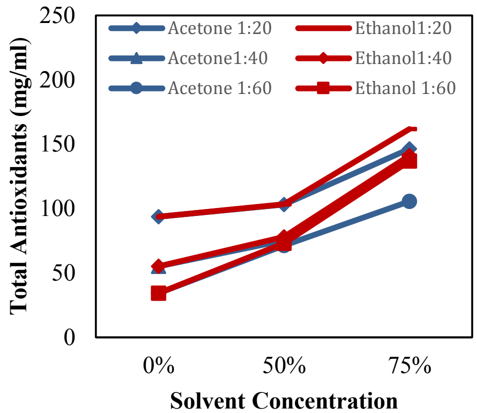


(c) Three hours

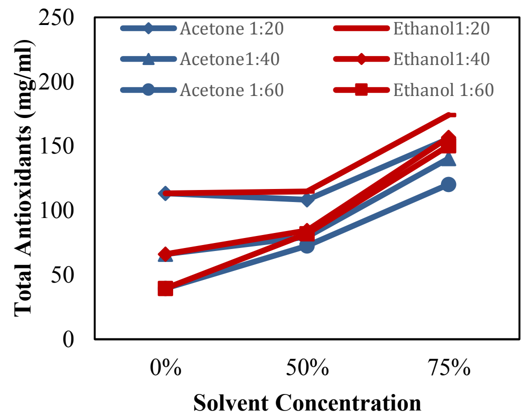


(d) Four hours

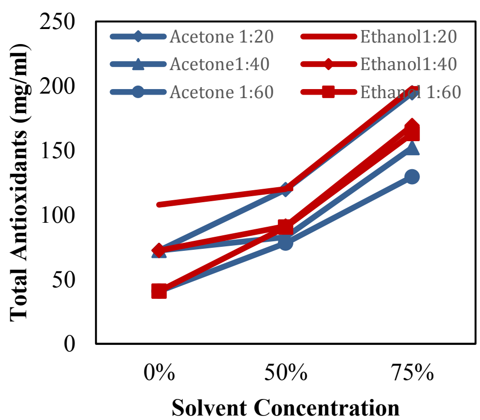
Figure 5.19. The effect of solvent (acetone and ethanol) concentration on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 55°C.



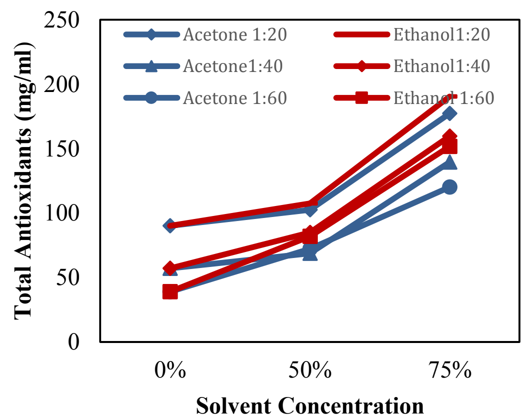
(a) One hour



(b) Two hours



(c) Three hours



(d) Four hours

Figure 5.20. The effect of solvent (acetone and ethanol) concentration on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 65ºC.

(157.44%) at the reaction time of 3 h and from 81.67 to 138.84 mg/ml (70%), from 68.88 to 109.94 mg/ml (59.61%) and from 37.11 to 92.08 mg/ml (148.13%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 45^{o}C increased the total antioxidants yield from 88.86 to 146.34 mg/ml (64.69%), from 61.63 to 110.58 mg/ml (79.43%) and from 38.97 to 97.15 mg/ml (149.29%) at the reaction time of 1 h, from 90.76 to 170.08 mg/ml (87.40%), from 73.12 to 120.45 mg/ml (64.73%) and from 40.12 to 99.73 mg/ml (148.58%) at the reaction time of 2 h, from 93.76 to 199.34 mg/ml (112.61%), from 74.46 to 134.07 mg/ml (80.06%) and from 45.92 to 107.91 mg/ml (135%) at the reaction time of 3 h and from 83.70 to 187.62 mg/ml (124.16%), from 70.52 to 118.70 mg/ml (68.32%) and from 40.21 to 99.01 mg/ml (146.23%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 55^{o}C increased the total antioxidants yield from 93.53 to 169.24 mg/ml (80.95%), from 65.35 to 151.65 mg/ml (132.06%) and from 48.88 to 117.37 mg/ml (140.12%) at the reaction time of 1 h, from 113.37 to 181.80 mg/ml (60.36%), from 74.60 to 153.64 mg/ml (105.95%) and from 50.08 to 122.29 mg/ml (144.19%) at the reaction time of 2 h, from 124.98 to 201.18 mg/ml (60.97%), from 75.46 to 160.38 mg/ml (112.54%) and from 51.71 to 130.74 mg/ml (152.83%) at the reaction time of 3 h and from 103.90 to 188.08 mg/ml (81.02%), from 67.34 to 150.60 mg/ml (123.64%) and from 47.95 to 127.29 mg/ml (165.46%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 65^{o}C increased the total antioxidants yield from 93.53 to 146.49 mg/ml (56.62%), from 55.06 to 137.46 mg/ml (149.65%) and from 34.23 to 105.50 mg/ml (208.21%) at the reaction time of 1 h, from 97.35 to 155.87 mg/ml (60.11%), from 66.16 to 140.52 mg/ml (112.39%) and from 39.44 to 120.35 mg/ml (205.15%) at the reaction time of 2 h, from 107.95 to 194.55 mg/ml (80.22%), from 72.40 to 152.23 mg/ml (110.26%) and from 40.97 to 129.61 mg/ml (216.35%) at the reaction time of 3 h and from 90 to 177.33 mg/ml (97.03%), from 57.17 to 139.43 mg/ml (143.89%) and from 38.92 to 120.27 mg/ml (209.02%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 25^{o}C increased the total antioxidants yield from 80.61 to 142.85 mg/ml (77.21%), from 50.24 to 109.94 mg/ml (118.70%) and from 30.60 to 88.33 mg/ml (188.6%) at the reaction time of 1 h, from 82.58 to 146.51 mg/ml (77.42%), from 54.44 to 120.83 mg/ml (121.95%) and from 36.34 to 97.02 mg/ml (166.98%) at the reaction time of 2 h, from 84.43 to 152.69 mg/ml (80.85%), from 56.63 to 133.72 mg/ml (136.13%) and from 40.27 to 105.10 mg/ml (160.99%) at the reaction time of 3 h and from 74.54 to 141.86 mg/ml (90.31%), from 55.75 to 128.14 mg/ml (129.85%) and from 32.52 to 100.44 mg/ml (208.86%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 35^{o}C increased the total antioxidants yield from 81.05 to 163.16 mg/ml (101.31%), from 57.79 to 111.57 mg/ml (93.06%) and from 33.22 to 104.17 mg/ml (213.58%) at the reaction time of 1 h, from 83.20 to 170.16 mg/ml (104.52%), from 67.77 to 122.17 mg/ml (80.27%) and from 37.59 to 93.37 mg/ml (148.39%),at the reaction time of 2 h, from 85.70 to 173.24 mg/ml (102.15%), from 72.64 to 153.76 mg/ml (111.67%) and from 39.52 to 122.17 mg/ml (209.13%) at the reaction time of 3 h and from 81.67 to 171.49 mg/ml (109.98%), from 68.88 to 152.23 mg/ml (121.01%) and from 37.11 to 113.16 mg/ml (204.93%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

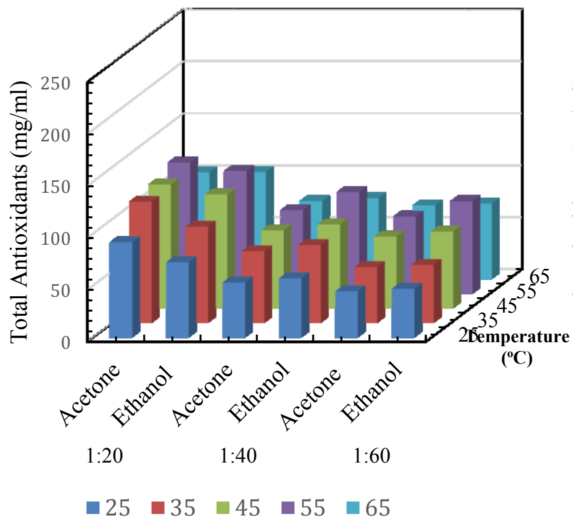
Increasing the concentration of ethanol from 0 to 75% at the the temperature of 45^{o}C increased the total antioxidants yield from 88.86 to 176.88 mg/ml (99.05%), from 61.63 to 136.40 mg/ml (121.32%) and from 38.97 to 118.90 mg/ml (205.11%) at the reaction time of 1 h, from 90.76 to 188.28 mg/ml (107.45%), from 73.12 to 147.79 mg/ml (102.12%) and from 40.12 to 130.58 mg/ml (225.47%) at the reaction time of 2 h, from 93.76 to 204.78 mg/ml (118.41%), from 74.46 to 164.57 mg/ml (121.02%) and from 45.92 to 135.45 mg/ml (194.97%) at the reaction time of 3 h and from 83.70 to 197.46 mg/ml (135.91%), from 70.52 to 167.66 mg/ml (137.75%) and from 40.21 to 130.42 mg/ml (224.35%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 55^{o}C increased the total antioxidants yield from 93.53 to 178.22 mg/ml (90.55%), from 65.35 to 161.96 mg/ml (147.83%) and from 48.88 to 160.51 mg/ml (228.38%) at reaction time of 1 h, from 113.37 to 181.82 mg/ml (60.38%), from 74.60 to 170.09 mg/ml (128%) and from 50.08 to 169.65 mg/ml (238.76%) at reaction time of 2 h, from 124.98 to 203.39 mg/ml (62.74%), from 75.46 to 177.85 mg/ml (135.69%) and from 51.71 to 174.49 mg/ml (237.44%) at the reaction time of 3 h and from 103.90 to 193.80 mg/ml (86.53%), from 67.34 to 174.88 mg/ml (159.70%) and from 47.95 to 171.41 mg/ml (257.48%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

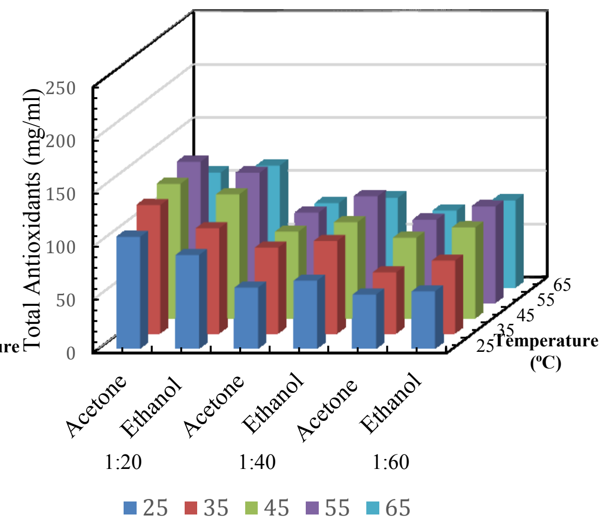
Increasing the concentration of ethanol from 0 to 75% at the temperature of 65^{o}C increased the total antioxidants yield from 93.53 to 161.61 mg/ml (72.79%), from 55.06 to 141.14 mg/ml (156.34%) and from 34.23 to 136.96 mg/ml (300.12%) at the reaction time of 1 h, from 97.35 to 174.28 mg/ml (79.02%), from 66.16 to 156.85 mg/ml (137.08%) and from 39.44 to 150.35 mg/ml (281.21%) at the reaction time of 2 h, from 107.95 to 198.18 mg/ml (83.58%), from 72.40 to 169.26 mg/ml (133.78%) and from 40.97 to 163.19 mg/ml (298.32%) at the reaction time of 3 h and from 90 to 190.40 mg/ml (111.56%), from 57.17 to 159.67 mg/ml (179.29%) and from 38.92 to 151.83 mg/ml (290.11%) at the reaction time of 4 h for sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

5.1.1.5. Effect of Solvent Type: Figures 5. 21 and 5.22 show the effect of solvent type on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios (1:20, 1:40 and 1:60), reaction times (1, 2, 3 and 4 h) and reaction temperatures (25, 35, 45, 55 and 65^{o}C). Generally, when ethanol was used as a solvent, higher total antioxidants yield was obtained compared to that obtained with acetone at the same concentration at all reaction temperatures, sample: solvent ratios and reaction times with 50 and 75% concentrations except for the 50% concentration with sample: solvent ratio of 1:20 at the temperatures of 25, 35, 45 and 55^{o}C, respectively.

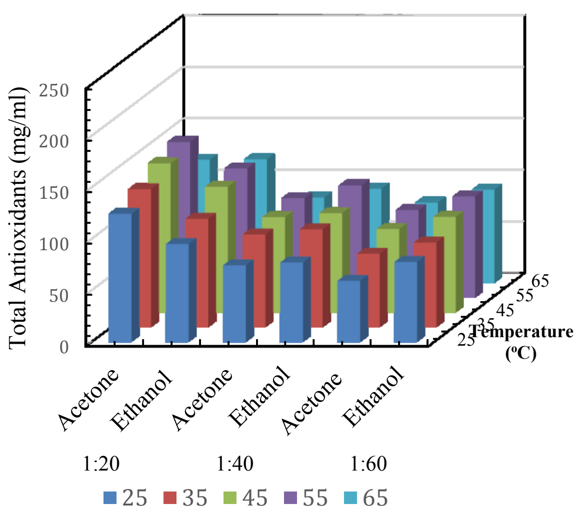
Replacing acetone with ethanol at 50% concentration at the sample: solvent ratio of 1:20 decreased the total antioxidants yield from 91.90 to 72.66 mg/ml (20.94%), from 116.70 to 92.85 mg/ml (20.44%), from 119.36 to 109.88 mg/ml (7.94%) and from 126.43 to 118.42 mg/ml (6.34%) at the reaction time of 1 h, from 105.19 to 88.14 mg/ml (16.21%), from 120.80 to 99.03 mg/ml (21.77%), from 126.45 to 116.61 mg/ml (7.78%) and from 132.75 to 122.51 mg/ml (7.71%) at the reaction time of 2 h, from 124.45 to 95.14 mg/ml (23.55%), from 134.03 to 105.33 mg/ml (21.41%), from 144.75 to 122.10 mg/ml (15.65%) and from 151.18 to 125.53 mg/ml (16.97%) at the reaction time of 3 h and from 117.73



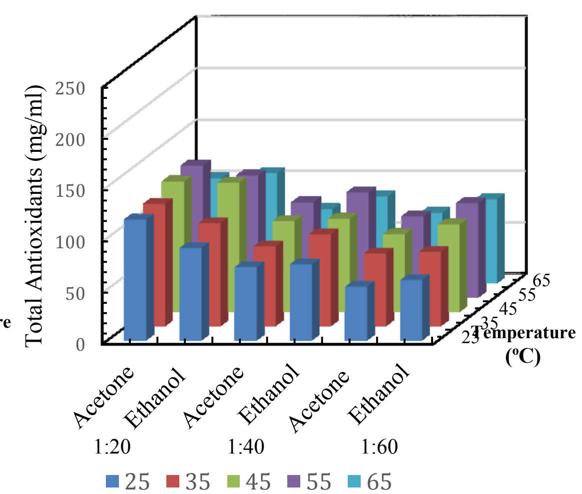
(a) One hour



(b) Two hours

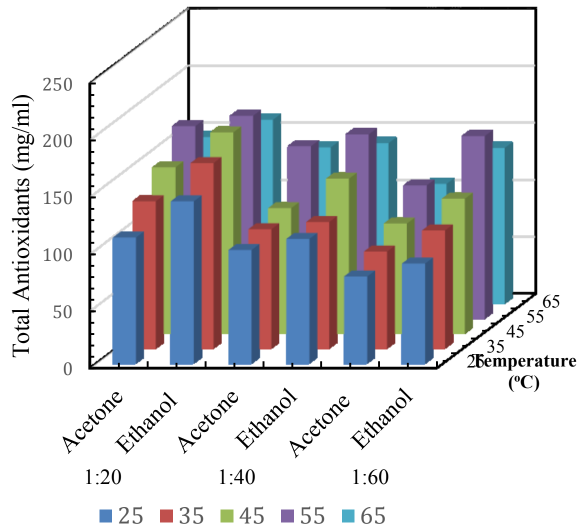


(c) Three hours

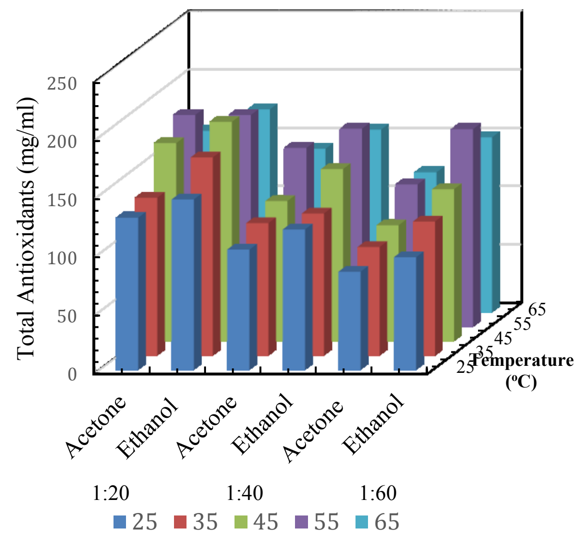


(d) Four hours

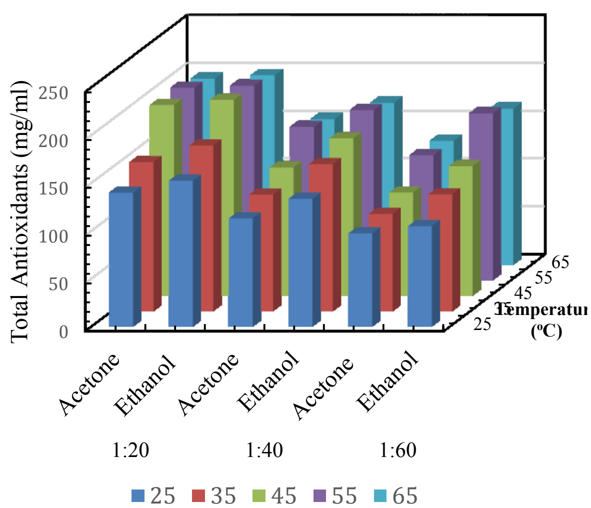
Figure 5.21. The effect of solvent (acetone and ethanol) at the concentration of 50% on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios and different reaction temperatures.



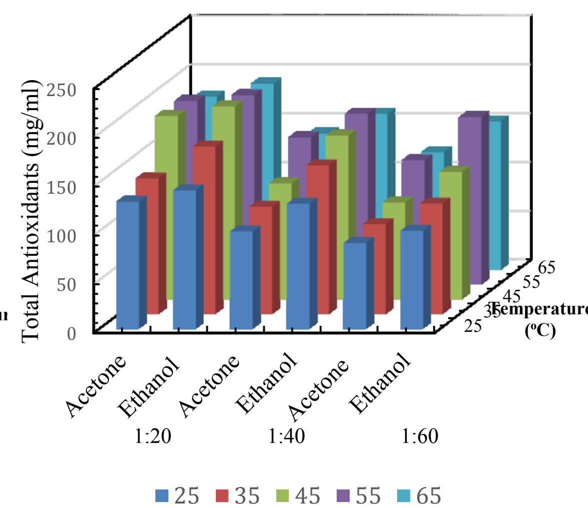
(a) One hour



(b) Two hours



(c) Three hours



(d) Four hours

Figure 5.22. The effect of solvent (acetone and ethanol) at the concentration of 75% on the total antioxidants yield from ajwa date fruit at different sample: solvent ratios and different reaction temperatures.

to 90.02 (27.71%), from 119.11 to 100.43 mg/ml (15.68%), from 127.12 to 125.88 mg/ml (0.98%) and from 128.64 to 119.14 mg/ml (7.38%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45 and 55^{o}C respectively. However, the reaction temperature of 65^{o}C, the total antioxidants yield increased from 103.02 to 103.33 mg/ml (0.13%), from 108.24 to 114.81 mg/ml (6.07%), from 119.65 to 120.06 mg/ml (0.34%) and from 102.48 to 107.46 mg/ml (4.86%) at the reaction times of 1, 2, 3 and 4 h, respectively.

Using ethanol at the 50% concentration with the sample: solvent ratio of 1:40 increased the total antioxidants yield over that obtained with acetone from 53.18 to 57.13 mg/ml (7.43%), from 69.21 to 75.13 mg/ml (8.55%), from 75.19 to 72.91 mg/ml (3.21%), from 80.63 to 98.01 mg/ml (17.73%) and 75.25 to 76.91 mg/ml (2.21%) at the reaction time of 1 h, from 57.50 to 63.99 mg/ml (11.29%), 80.80 to 87.04 mg/ml (7.72%), 81.42 to 90.33 mg/ml (10.94%), 84.98 to 100.37 mg/ml (18.11%) and 79.57 to 84.54 mg/ml (6.25%) at the reaction time of 2 h, from 74.40 to 77.25 mg/ml (3.83%), from 90.11 to 95.10 mg/ml (5.54%), from 92.64 to 96.42 mg/ml (4.08%), 96.70 to 108.95 mg/ml (12.67%) and from 82.93 to 91.40 mg/ml (10.21%) at the reaction time of 3 h, from 71.37 to 74.13 mg/ml (3.87%), from 77.75 to 89.65 mg/ml (15.31%), from 88.57 to 90.79 mg/ml (2.51%), from 92.80 to 102.68 mg/ml (10.65%) and from 72.29 to 84.80 mg/ml (17.31%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

Using ethanol at the 50% concentration with the sample: solvent ratio of 1:60 increased the total antioxidants yield over that obtained with acetone from 44.61 to 47.07 mg/ml (5.51%) , from 53.90 to 55.99 mg/ml (3.88%), from 69.23 to 73.99 mg/ml (6.88%), from 74.38 to 89.01 mg/ml (19.67%) and from 71.20 to 72.89 mg/ml (2.37%) at the reaction time of 1 h, from 50.93 to 53.90 mg/ml (5.83%), from 57.46 to 68.53 mg/ml (19.27%), from 75.72 to 85.38 mg/ml (12.76%), from 78.35 to 90.74 mg/ml (15.81%) and from 72.58 to 82.11 mg/ml (13.13%) at the reaction time of 2 h, from 59.44 to 77.75 mg/ml (30.80%), from 71.38 to 82.29 mg/ml (15.28%), from 81.08 to 92.79 mg/ml (14.44%), from 85.14 to 98.30 mg/ml (15.46%) and from 78.08 to 90.60 mg/ml (16.03%) at the reaction time of 3 h and from 52.24 to 58.74 mg/ml (12.44%), from 70.80 to 72.42 mg/ml (2.29%), from 75.73 to 85.33 mg/ml (12.68%), from 79.29 to 92.06 mg/ml (16.11%) and from 68.64 to 81.98 mg/ml (19.43%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

Using ethanol at the 75% concentration with the sample: solvent ratio of 1:20 increased the total antioxidants yield over that obtained with acetone from 111.18 to 142.85 mg/ml (28.49%), from 129.75 to 163.16 mg/ml (25.75%), from 146.34 to 176.88 mg/ml (20.87%), from 169.24 to 169.22 mg/ml (5.31%) and from 146.49 to 161.61 mg/ml (10.32%) at the reaction time of 1 h, from 130.86 to 146.51 mg/ml (11.96%), from 135.64 to 170.16 mg/ml (25.45%), from 170.08 to 188.28 mg/ml (10.7%), from 181.80 to 181.82 mg/ml (0.01%) and 155.87 to 174.28 mg/ml (11.81%) at the reaction time of 2 h, from 140 to 152 mg/ml (9.06%), from 155.83 to 173.24 mg/ml (11.17%), 199.34 to 204.78 mg/ml (2.73%), 201.18 to 203.39 mg/ml (1.10%) and from 194.55 to 198.18 mg/ml (1.87%) at the reaction time of 3 h and from 130.12 to 141.86 mg/ml (9.02%), from 138.84 to 171.49 mg/ml (23.52%), from 187.62 to 197.46 mg/ml (5.24%), from 188.08 to 193.80 mg/ml (3.04%) and from 177.33 to 190.40 mg/ml (7.37%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65ºC, respectively.

Using ethanol at the 75% concentration with acetone with the sample: solvent ratio of 1:40 increased the total antioxidants yield over that obtained from 100.27 to 109.94 mg/ml (9.64%), from 105.26 to 111.57 mg/ml (5.99%), from 110.58 to 136.40 mg/ml (23.35%), from 151.65 to 161.96 mg/ml (6.80%) and from 137.46 to 141.14 mg/ml (2.68%) at the reaction time of 1 h, from 103.57 to 120.83 mg/ml (16.67%), from 113.93 to 122.17 mg/ml (7.23%), from 120.45 to 147.79 mg/ml (22.70%), from 153.64 to 170.09 mg/ml (10.71%) and from 140.52 to 156.85 mg/ml (11.62%) at the reaction time of 2 h, from 113.16 to 133.72 mg/ml (18.17%), from 121.88 to 153.76 mg/ml (26.16%), from 134.07 to 164.57 mg/ml (22.75%), 160.38 to 177.85 mg/ml (10.89%) and from 152.23 to 169.26 mg/ml (11.19%) at the reaction time of 3 h, from 99.77 to 128.14 mg/ml (28.44%), from 109.94 to 152.23 mg/ml (38.47%), from 118.70 to 167.66 mg/ml (41.25%), from 150.60 to 174.88 mg/ml (16.12%) and from 139.43 to 159.67 mg/ml (14.52%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65ºC, respectively.

Using ethanol at the 75% concentration with acetone with the sample: solvent ratio 1:60 increased the total antioxidants yield over that obtained from 76.94 to 88.33 mg/ml (14.80%), from 85.70 to 104.17 mg/ml (21.55%), from 97.15 to 118.90 mg/ml (22.39%), from 117.37 to 160.51 mg/ml (36.76%) and from 105.50 to 136.96 mg/ml (29.82%) at the reaction time of 1 h, from 84.54 to 97.02 mg/ml (14.76%), from 93.37 to 115.25 mg/ml

(23.43%), from 99.73 to 130.58 mg/ml (30.93%), from 122.29 to 169.65 mg/ml (38.73%) and from 120.35 to 150.35 mg/ml (24.93%) at the reaction time of 2 h, from 97.60 to 105.10 mg/ml (7.68%), from 101.74 to 122.17 mg/ml (20.08%), from 107.91 to 135.45 mg/ml (25.52%), from 130.74 to 174.49 mg/ml (33.46%) and from 129.61 to 163.19 mg/ml (25.91%) at the reaction time of 3 h and from 87.97 to 100.44 mg/ml (14.18%), from 92.08 to 113.16 mg/ml (22.89%), from 99.01 to 130.42 mg/ml (31.72%), from 127.29 to 171.41 mg/ml (34.66%) and from 120.27 to 151.83 mg/ml (26.24%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

5.1.2. Total phenols

The phenolic results are shown in Tables 5.8-5.12. Analysis of the variance (ANOVA) was performed on the total phenols data using Minitab Software (Minitab® 17.1.0., Minitab Inc., Canada).The results are shown in Table 5.13. The results obtained from Tukey’s grouping are shown in Table 5.14.

The main effects of reaction time (Ti), sample: solvents ratio (R), reaction temperature (Te) solvent concentration (C) and solvent type (S) on the total phenolic yield were significant at the 0.001 level. The two way interactions between reaction time and sample: solvent ratio, between reaction times and reaction temperature and between the reaction time and solvent type were not significant. The three-way interactions between the reaction time, reaction temperature and sample: solvent ratio, between the reaction time, sample: solvent ratio and solvent type, between the reaction time, sample: solvent ratio, solvent concentration, between reaction time, reaction temperature and solvent type and between reaction time, sample: solvent ratio and solvent concentration are not significant. The four-way interactions between the reaction time, reaction temperature, sample: solvent ratio and solvent type, between the reaction time, reaction temperature, solvent type and solvent concentration, between the reaction time, sample: solvent ratio, solvent type and solvent concentration were not significant at the 0.001 level. The five-way interactions were not significant at the 0.001 level.

All the sample: solvent ratios (1:20, 1:40 and 1:60) were significantly different from one another at the 0.05 level. The highest average yield of total phenolic (617.673 mg/l) was obtained at the sample: solvent ratio of 1:20. The reaction times 3 and 4 hours were not significantly different from one another, but they were significantly different from

Table 5.8. Average phenols yield from ajwa date fruit using water at different sample: solvent ratio, reaction temperature and reaction time.

Sample: Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Phenols Yield* (mg/l)
1:20	25	1	241.62 ± 4.35
		2	264.69 ± 24.76
		3	317.46 ± 95.73
		4	283.07 ± 95.73
	35	1	321.23 ± 8.15
		2	349.30 ± 6.86
		3	425.76 ± 4.14
		4	385.15 ± 1.08
	45	1	401.23 ± 4.89
		2	408.92 ± 4.89
		3	466.23 ± 2.63
		4	405.69 ± 6.52
	55	1	412.00 ± 3.80
		2	413.53 ± 5.98
		3	509.30 ± 0.87
		4	457.76 ± 3.26
	65	1	421.61 ± 20.66
		2	440.84 ± 18.49
		3	512.38 ± 38.07
		4	480.07 ± 35.89
1:40	25	1	124.69 ± 3.26
		2	147.38 ± 0.54
		3	151.00 ± 8.70
		4	122.84 ± 0.54
	35	1	144.30 ± 4.89
		2	150.76 ± 18.49
		3	167.23 ± 1.63
		4	155.76 ± 1.63
	45	1	179.69 ± 1.63
		2	180.46 ± 9.24
		3	213.15 ± 1.08
		4	195.46 ± 11.96
	55	1	182.76 ±180.58
		2	240.00 ± 28.28
		3	255.53 ± 28.82
		4	223.38 ± 13.59
	65	1	188.53 ± 8.70
		2	252.76 ± 22.30
		3	265.00 ± 53.84
		4	243.53 ± 16.86
1:60	25	1	105.07 ± 19.03
		2	116.61 ± 8.15
		3	124.30 ± 9.24
		4	117.76 ± 13.05
	35	1	138.53 ± 9.79
		2	142.76 ± 1.63
		3	178.92 ± 16.86
		4	139.30 ± 13.05
	45	1	140.53 ± 62.00
		2	146.23 ± 72.34
		3	180.46 ± 5.98
		4	176.46 ± 94.09
	55	1	155.23 ± 14.68
		2	178.84 ± 21.21
		3	188.38 ± 22.3
		4	183.46 ± 11.96
	65	1	158.61 ± 31.54
		2	180.38 ± 2.17
		3	198.00 ± 17.40
		4	188.15 ± 3.26

*average of two replicates

Table 5.9. Average phenols yield from ajwa date fruit using 50% acetone at different sample: solvent ratio, reaction temperature and reaction time.

Sample: Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Phenols Yield* (mg/l)
1:20	25	1	470.46 ±173.51
		2	518.54 ± 22.84
		3	598.92 ± 64.72
		4	542.00 ± 76.69
	35	1	650.08 ±180.58
		2	818.53 ± 21.75
		3	898.46 ±264.34
		4	814.30 ± 10.33
	45	1	756.62 ± 44.05
		2	891.23 ±147.40
		3	920.38 ±373.13
		4	895.53 ±166.98
	55	1	787.00 ± 5.43
		2	895.15 ± 10.87
		3	940.07 ± 12.51
		4	898.07 ± 76.69
	65	1	815.46 ± 55.48
		2	897.69 ± 3.26
		3	963.92 ± 37.53
		4	940.53 ± 77.23
1:40	25	1	212.38 ± 79.41
		2	223.54 ±106.06
		3	263.53 ± 36.44
		4	243.00 ± 57.65
	35	1	228.84 ± 39.70
		2	259.69 ± 32.09
		3	299.30 ± 52.21
		4	283.15 ± 16.31
	45	1	321.61 ± 0.01
		2	407.00 ± 60.92
		3	475.46 ± 14.14
		4	433.15 ± 34.81
	55	1	424.30 ±113.68
		2	451.23 ± 4.89
		3	589.30 ± 7.61
		4	580.92 ± 8.70
	65	1	430.92 ± 3.26
		2	458.92 ± 1.63
		3	592.61 ± 27.19
		4	538.15 ± 15.77
1:60	25	1	176.00 ± 21.21
		2	198.53 ± 21.75
		3	222.00 ± 2.71
		4	205.15 ± 57.65
	35	1	232.31 ± 15.77
		2	258.61 ± 8.70
		3	261.53 ± 6.52
		4	240.46 ± 20.12
	45	1	250.88 ±189.12
		2	287.53 ± 36.44
		3	293.61 ± 52.76
		4	253.00 ± 8.15
	55	1	253.54 ± 18.49
		2	289.61 ± 29.37
		3	293.38 ± 0.54
		4	254.30 ± 4.89
	65	1	263.92 ± 23.93
		2	291.30 ± 38.61
		3	296.15 ± 22.30
		4	271.07 ± 18.49

*average of two replicates

Table 5.10. Average phenols yield from ajwa date fruit using 50% ethanol at different sample: solvent ratio, reaction temperature and reaction time.

Sample: Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Phenols Yield* (mg/l)
1:20	25	1	320.08 ± 45.69
		2	325.46 ± 35.89
		3	343.53 ± 31.00
		4	336.61 ± 34.26
	35	1	328.69 ± 4.89
		2	364.38 ± 36.44
		3	443.15 ± 79.95
		4	397.76 ± 39.70
	45	1	432.76 ± 15.77
		2	458.92 ± 23.38
		3	476.62 ± 88.66
		4	414.15 ± 80.50
	55	1	456.38 ± 31.54
		2	460.15 ± 10.87
		3	534.69 ± 28.28
		4	510.07 ± 45.96
	65	1	486.54 ± 79.95
		2	525.46 ± 6.52
		3	589.69 ± 88.66
		4	578.07 ± 9.79
1:40	25	1	198.92 ± 8.15
		2	223.30 ± 3.26
		3	260.46 ± 70.16
		4	218.23 ± 3.26
	35	1	208.46 ± 5.98
		2	213.53 ± 19.03
		3	266.15 ± 32.09
		4	230.84 ± 35.89
	45	1	220.83 ± 0.01
		2	233.53 ± 2.71
		3	273.08 ± 32.63
		4	258.07 ± 9.79
	55	1	240.46 ± 37.53
		2	311.23 ± 8.15
		3	317.00 ± 16.31
		4	297.38 ± 4.89
	65	1	280.15 ± 129.99
		2	339.38 ± 56.56
		3	380.00 ± 214.30
		4	323.15 ± 31.54
1:60	25	1	162.69 ± 8.70
		2	178.92 ± 47.32
		3	213.53 ± 45.14
		4	173.46 ± 35.89
	35	1	167.77 ± 28.82
		2	191.92 ± 73.43
		3	248.61 ± 43.51
		4	236.53 ± 8.15
	45	1	169.46 ± 41.88
		2	198.38 ± 36.44
		3	262.15 ± 175.68
		4	248.23 ± 100.08
	55	1	178.45 ±140.33
		2	201.69 ±113.13
		3	268.84 ±157.73
		4	252.84 ±128.36
	65	1	180.62 ±165.35
		2	212.84 ±147.40
		3	270.84 ±163.17
		4	261.07 ±132.71

*average of two replicates

Table 5.11. Average phenols yield from ajwa date fruit using 75% acetone at different sample: solvent ratio, reaction temperature and reaction time.

Sample: Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Phenols Yield* (mg/l)
1:20	25	1	675.08 ±136.52
		2	745.46 ± 55.48
		3	905.46 ± 66.35
		4	833.31 ± 23.93
	35	1	750.85± 78.32
		2	870.07±187.11
		3	922.34± 23.38
		4	842.76±114.76
	45	1	891.62 ± 91.38
		2	876.15 ± 38.07
		3	963.53 ±181.12
		4	859.30 ± 53.30
	55	1	930.31 ± 80.50
		2	975.53 ±192.00
		3	970.07 ± 97.90
		4	959.30 ± 71.79
	65	1	998.92 ±178.40
		2	1032.61 ± 46.23
		3	1052.39 ±344.30
		4	1003.92 ±163.70
1:40	25	1	407.77 ± 72.88
		2	438.07 ± 52.21
		3	450.69 ± 35.89
		4	420.07 ± 52.21
	35	1	509.31 ± 3.26
		2	550.46 ± 91.92
		3	590.15 ±157.73
		4	581.23 ± 1.63
	45	1	525.85 ± 48.40
		2	558.61 ± 21.21
		3	636.23 ± 89.20
		4	595.84 ± 27.74
	55	1	619.69 ± 60.37
		2	680.84 ± 72.88
		3	719.30 ± 78.32
		4	673.84 ±250.20
	65	1	708.92 ± 52.76
		2	754.46 ±207.78
		3	880.00 ± 36.98
		4	783.84 ± 13.59
1:60	25	1	353.50 ± 7.07
		2	386.61 ± 15.77
		3	390.38 ± 8.15
		4	290.07 ± 11.96
	35	1	351.62 ± 64.18
		2	395.84 ± 81.04
		3	466.23 ± 75.06
		4	412.38 ± 137.07
	45	1	439.69 ± 72.34
		2	448.53 ± 64.18
		3	458.92 ± 98.45
		4	445.85 ± 16.86
	55	1	441.92 ± 10.33
		2	453.69 ± 38.61
		3	466.15 ± 116.94
		4	448.61 ± 121.29
	65	1	483.53 ± 32.09
		2	486.53 ± 27.74
		3	490.30 ± 137.61
		4	453.00 ± 54.39

*average of two replicates

Table 5.12. Average phenols yield from ajwa date fruit using 75% ethanol at different sample: solvent ratio, reaction temperature and reaction time.

Sample :Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Phenols Yield* (mg/l)
1:20	25	1	583.23 ± 61.463
		2	589.53 ±199.622
		3	610.84 ±281.755
		4	575.07 ±127.823
	35	1	588.92 ±573.112
		2	591.30 ± 62.007
		3	629.69 ± 67.991
		4	579.69 ± 61.463
	45	1	598.92 ±101.171
		2	603.00 ±124.560
		3	653.15 ±566.773
		4	599.69 ±262.717
	55	1	629.31 ± 47.865
		2	640.07 ± 306.77
		3	685.38 ± 62.55
		4	606.53 ± 331.25
	65	1	637.00 ± 90.292
		2	653.07 ± 47.865
		3	688.92 ± 75.606
		4	612.46 ±223.555
1:40	25	1	402.77 ± 42.970
		2	430.15 ±103.890
		3	433.38 ±124.016
		4	418.30 ±139.790
	35	1	454.31 ± 67.991
		2	459.30 ± 72.886
		3	462.23 ± 31.547
		4	431.46 ± 28.828
	45	1	462.77 ± 52.761
		2	467.15± 32.635
		3	471.07± 83.765
		4	449.46± 9.246
	55	1	478.08± 26.108
		2	483.00± 37.531
		3	490.15± 35.899
		4	451.23± 69.622
	65	1	490.38± 23.388
		2	503.38± 84.308
		3	512.07± 81.589
		4	483.38± 49.497
1:60	25	1	275.85 ± 38.618
		2	382.76 ± 69.078
		3	386.61 ± 66.903
		4	260.69 ± 57.112
	35	1	293.23 ± 39.162
		2	386.30 ± 27.196
		3	389.30 ± 22.845
		4	264.30 ± 63.095
	45	1	302.61 ± 14.142
		2	391.23 ± 24.476
		3	397.30 ± 65.815
		4	273.23 ± 66.359
	55	1	323.54 ± 43.514
		2	396.53 ± 5.439
		3	402.15 ± 2.175
		4	275.69 ± 52.217
	65	1	338.15 ± 19.037
		2	406.76 ± 62.551
		3	410.92 ± 2.175
		4	281.53 ± 2.175

*average of two replicates

Table 5.13. Analysis of the variance for total phenols yield.

Source	DF	SS	MS	F	P
Total	719	56808050			
Model					
Ti	3	767286	255762	35.25	0.001
R	2	10478848	5239424	722.01	0.001
Te	4	4819102	1204776	166.02	0.001
S	1	461507	461507	63.60	0.001
C	2	14150448	7075224	975.00	0.001
Ti*R	6	74680	12447	1.72	0.116
Ti*Te	12	104149	8679	1.20	0.284
Ti*S	3	27616	9205	1.27	0.285
Ti*C	6	205493	34249	4.72	0.001
Te*R	8	3092408	386551	53.27	0.001
Te*S	4	178637	44659	6.15	0.001
Te*C	8	4208502	526063	72.49	0.001
R*S	2	3387000	1693500	233.37	0.001
R*C	4	2390580	597645	82.36	0.001
S*C	2	237305	118652	16.35	0.001
Ti*Te*R	24	274420	11434	1.58	0.043
Ti*R*S	6	81681	13614	1.88	0.084
Ti*R*C	12	131682	10973	1.51	0.117
Ti*Te*S	12	150053	12504	1.72	0.060
Ti*S*C	6	41770	6962	0.96	0.453
Te*R*S	8	645743	80718	11.12	0.001
Te*R*C	16	3133387	195837	26.99	0.001
R*C*S	4	1842238	460560	63.47	0.001
Te*S*C	8	584204	73026	10.06	0.001
Ti*Te*C	24	417301	17388	2.40	0.001
Ti*Te*R*S	24	186626	7776	1.07	0.374
Ti*Te*R*C	48	657012	13688	1.89	0.001
Ti*Te*S*C	24	163279	6803	0.94	0.550
Ti*R*S*C	12	118955	9913	1.37	0.180
Te*R*S*C	16	867125	54195	7.47	0.001
Ti*Te*R*S*C	48	316613	6596	0.91	0.647
Error	360	2612401	7257		

Reaction Time (Ti)
Reaction Temperature (Te)
Sample: Solvent Ratio(R)
Solvent Type (S)
Solvent Concentration (C)
R^2= 95.40%

Table 5.14. Tukey's grouping of total phenols yield.

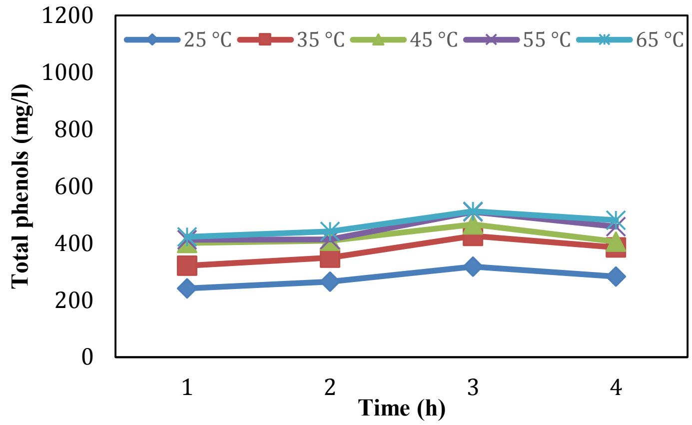
Factors	level	N	Mean	Tukey's Grouping
Sampl: Solvent Ratio	20	240	617.673.	A
	40	240	384.619	B
	60	240	343.802	C
Reaction Time	1	180	404.091	A
	2	180	431.679	B
	3	180	486.009	C
	4	180	473.013	C
Reaction Temperature	25˚C	144	329.238	A
	35˚C	144	381.685	B
	45˚C	144	516.307	C
	55˚C	144	468.955	D
	65˚C	144	547.304	E
Solvent Concentration	0%	240	304.968	A
	50%	240	402.299	B
	75%	240	638.827	C
Solvent Type	Acetone	360	474.016	A
	Ethanol	360	423.380	B

Groups with the same letter are not significantly different from each other at the alpha significance level of 0.05.

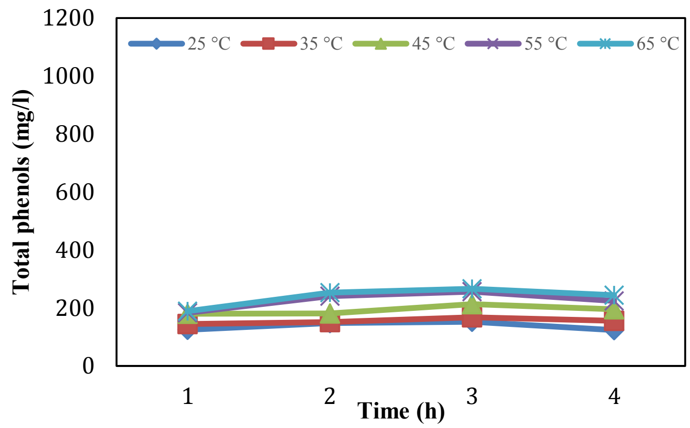
reaction time 1 and 2 h at the 0.05 level. The highest yield of total phenolic average yield of total phenolic (486.009 mg/l) was obtained after 3 hours. All reaction temperatures (25, 35, 45, 55 and 65 ˚C) were significantly different from one another at the 0.05 level. The highest average yield of total phenolic (547.304 mg/l) was obtained a temperature of 65 ˚C All solvent concentrations (0, 50 and 75%) were significantly different from one another at the 0.05 level. The highest average yield of total phenolic (638.827 mg/l) was obtained at the concentration of 75%. The solvent types (acetone and ethanol) were significantly different from one another at the 0.05 level. The highest average yield of total phenolic (474.016mg/l) was obtained with acetone.

5.1.2.1. Effect of Reaction Time: Figures 5.23-5.27 show the effect of reaction time on the total phenols yield from ajwa date fruit at different sample: solvent ratios (1:20, 1:40 and 1:60) and reaction temperatures (25, 35, 45, 55 and 65 ºC) using acetone and ethanol at different concentrations (0, 50 and 75%). Generally, there was an increase in the total phenols yield when the reaction time was increased from 1 to 3 h at all reaction temperatures, sample: solvent ratios and solvent concentration for both solvents. This was followed by a decrease in the antioxidants yield with a further increase in reaction time from 3 to 4 h.

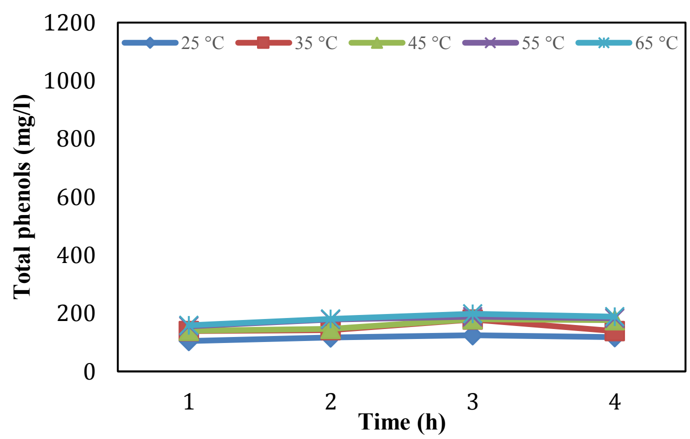
When water was used as a solvent, increasing the reaction time from 1 to 3 h, increased the phenols yield from 241.62 to 317.46 mg/l (31.39%), from 321.23 to 125.76 mg/l (32.54%), from 401.23 to 466.23 mg/l (16.20%), from 412 to 509.30 mg/l (23.62%) and from 421.61 to 512.38 (21.53%) at the sample: solvent ratio of 1:20, from 124.69 to 151 mg/l (21.10%), from 144.30 to 167.23 mg/l (15.89%), from 179.69 to 213.15 mg/l (18.62%), from 182.76 to 255.53 mg/l (39.82%) and from 188.53 to 265 mg/l (40.56%) at the sample: solvent ratio of 1:40 and from 105.07 to 124.30 mg/l (18.30%), from 138.53 to 178.92 mg/l (29.16%), from 140.53 to 180.46 mg/l (28.41%), from 155.23 to 188.38 mg/l (21.36%) and from 158.61 to 198 mg/l (24.83%) at the sample: solve ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65ºC, respectively. A further increase the reaction time from 3 to 4 h decreased the phenols yield from 317.46 to 283.07 mg/l (10.83%), from 425.76 to 385.15 mg/l (9.54%), from 466.23 to 405.69 mg/l (12.99%), from 509.30 to 457.76 mg/l (10.12%) and from 512.38 to 480.07 mg/l (6.30%) at the sample: solvent ratio of 1:20, from 151 to 122.84 mg/l (18.65%), from 167.23 to 155.76



(a) Sample: solvent ratio 1:20

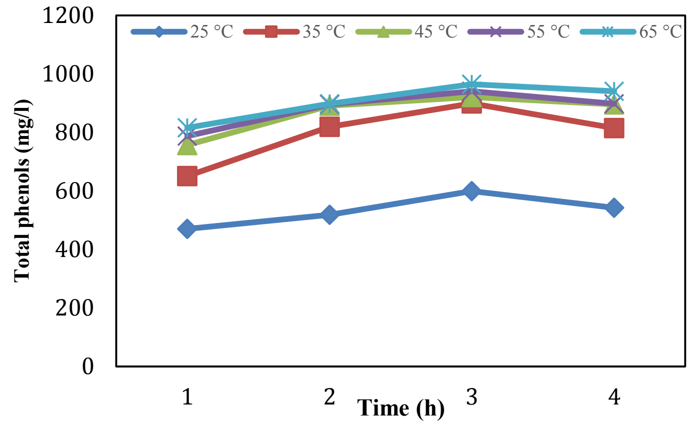


(b) Sample: solvent ratio 1:40

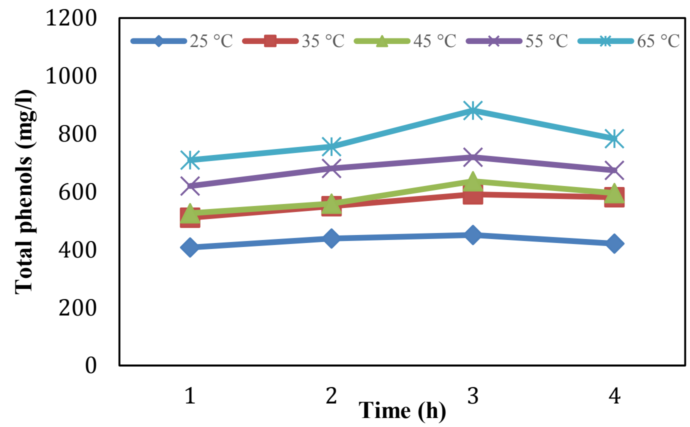


(c) Sample: solvent ratio 1:60

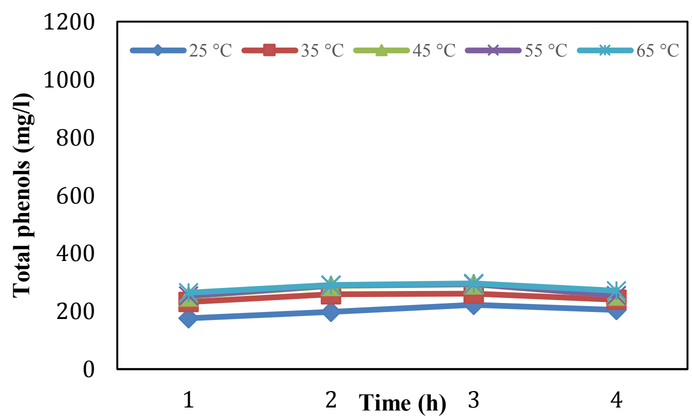
Figure 5.23. The effect of reaction time on the total phenols yield from ajwa date fruit using water at different temperatures.



(a) Sample: solvent ratio 1:20

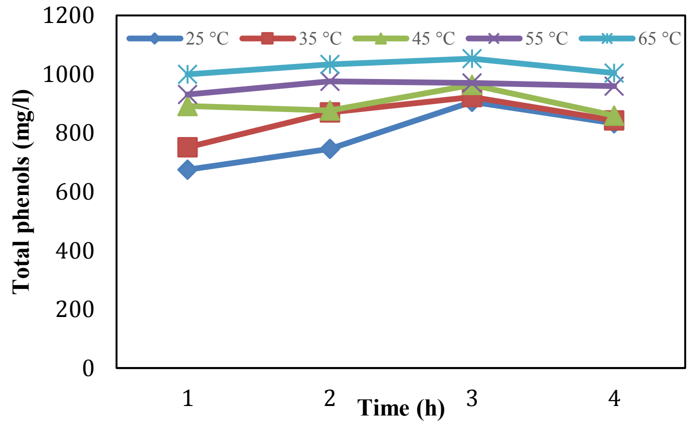


(b) Sample: solvent ratio 1:40

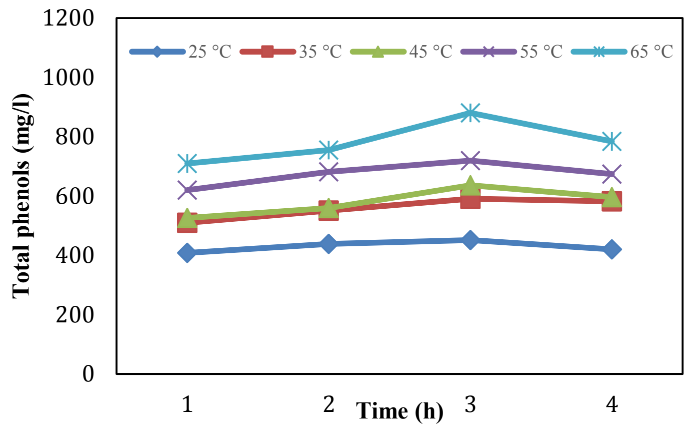


(c) Sample: solvent ratio 1:60

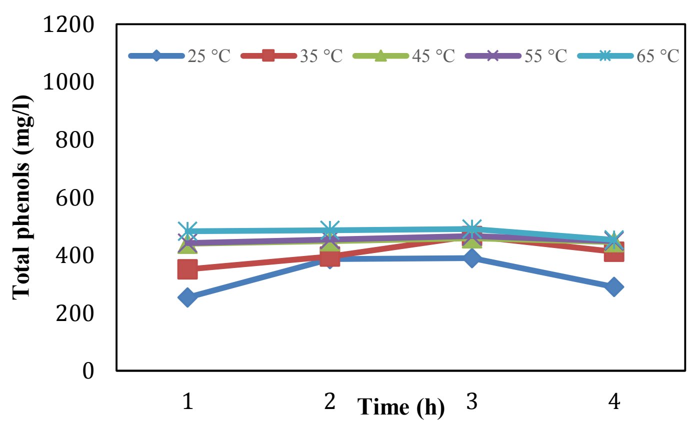
Figure 5.24. The effect of reaction time on the total phenols yield from ajwa date fruit using 50% acetone at different temperatures.



(a) Sample: solvent ratio 1:20

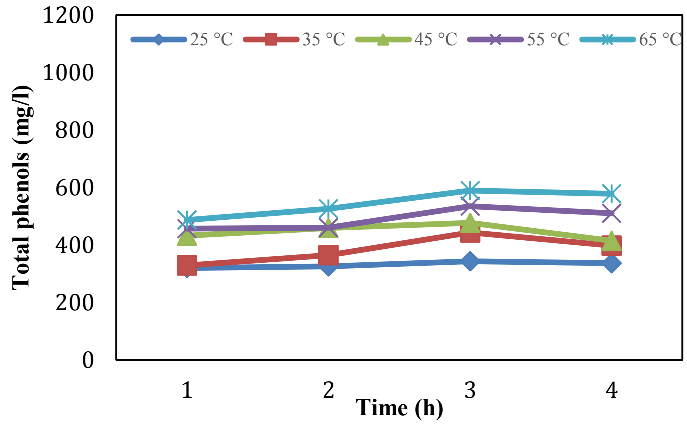


(b) Sample: solvent ratio 1:40

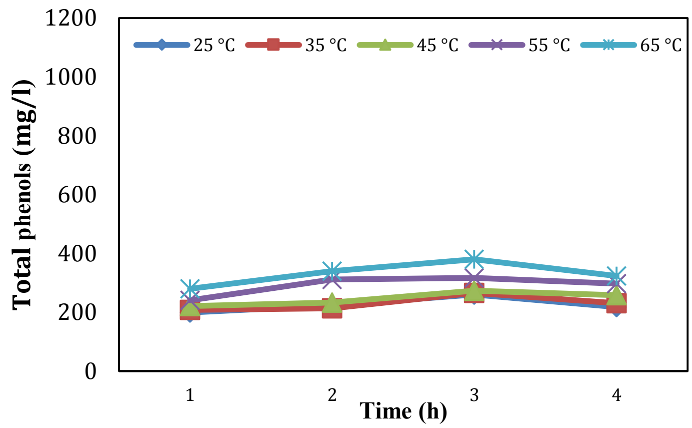


(c) Sample: solvent ratio 1:60

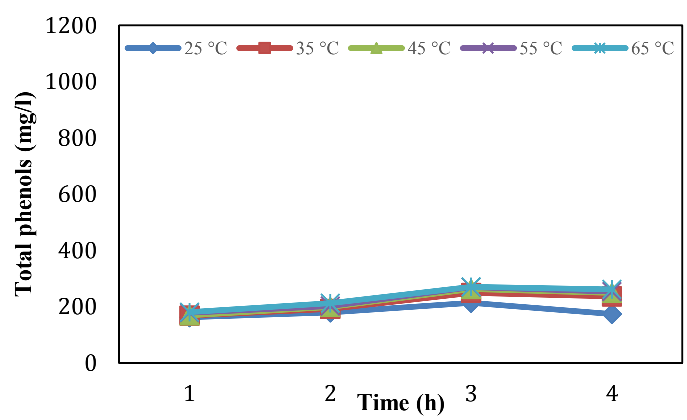
Figure 5.25. The effect of reaction time on the total phenols yield from ajwa date fruit using 75% acetone at different temperatures.



(a) Sample: solvent ratio 1:20

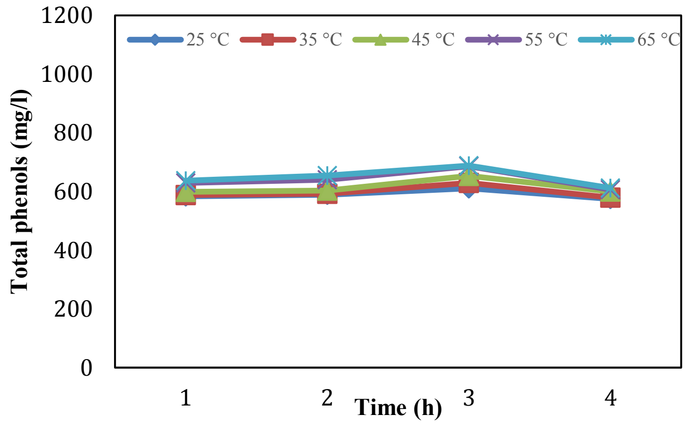


(b) Sample: solvent ratio 1:40

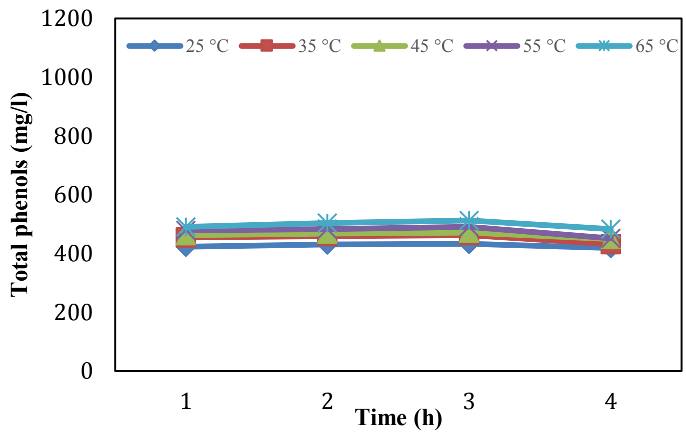


(c) Sample: solvent ratio 1:60

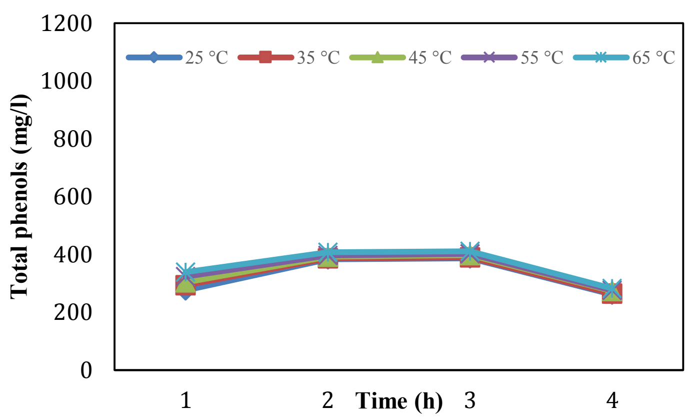
Figure 5.26. The effect of reaction time on the total phenols yield from ajwa date fruit using 50% ethanol at different temperatures.



(a) Sample: solvent ratio 1:20



(b) Sample: solvent ratio 1:40



(d) Sample: solvent ratio 1:60

Figure 5.27. The effect of reaction time on the total phenols yield from ajwa date fruit using 75% ethanol at different temperatures.

mg/l (6.86%), from 213.15 to 195.46 mg/l (8.30%), from 255.53 to 223.38 mg/l (12.58%) and from 265 to 243.53 mg/l (8.10%) at the sample: solvent ratio of 1:40 and from 124.30 to 117.76 mg/l (5.26%), from 178.92 to 139.30 mg/l (22.14%), from 180.46 to 176.46 mg/l (2.22%), from 188.38 to 183.46 mg/l (2.61%) and from 198 to 188.15 mg/l (4.97%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

when acetone was used as solvent at the 50% concentration, increasing the reaction time from 1 to 3 h, increased the phenols yield from 470.46 to 598.92 mg/l (27.31%), from 650.08 to 898.46 mg/l (38.21%), from 756.62 to 920.38 mg/l (21.64%), from 787 to 940.07 mg/l (19.45%) and from 815.46 to 963.92 (18.21%) at the sample: solvent ratio of 1:20, from 212.38 to 263.53 mg/l (24.08%), from 228.84 to 299.30 mg/l (30.79%), from 321.61 to 475.46 mg/l (47.84%), from 424.30 to 589.30 mg/l (38.89%) and from 430.92 to 592.61 mg/l (37.52%) at the sample: solvent ratio of 1:40 and from 176 to 222 mg/l (26.14%), from 232.31 to 261.53 mg/l (12.58%), from 250.88 to 293.61 mg/l (17.03%), from 253.54 to 293.38 mg/l (15.71%) and from 263.92 to 296.15 mg/l (12.21%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively. A further increase in reaction time from 3 to 4 h decreased the phenols yield from 598.92 to 542 mg/l (9.50%), from 898.46 to 814.30 mg/l (9.37%), from 920.38 to 895.53 mg/l (2.70%), from 940.07 to 898.07 mg/l (4.47%) and from 963.92 to 940.53 mg/l (2.43%) at the sample: solvent ratio of 1:20, from 263.53 to 243 mg/l (7.79%), from 299.30 to 283.15 mg/l (5.40%), from 475.46 to 433.15 mg/l (8.90%), from 589.30 to 580.92 mg/l (1.42%) and from 592.61 to 538.15 mg/l (9.19%) at the sample: solvent ratio of 1:40 and from 222 to 205.15 mg/l (7.59%), from 261.53 to 240.46 mg/l (8.06%), from 293.61 to 253 mg/l (13.83%), from 293.38 to 254.30 mg/l (13.32%) and from 296.15 to 271.07 mg/l (8.47%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65 ^{o}C, respectively.

When acetone was used as solvent at the 75% concentration, increasing the reaction time from 1 to 3 h, increased the phenols yield from 675.08 to 905.46 mg/l (34.13%), from 750.85 to 922.34 mg/l (22.84%), from 891.62 to 963.53 mg/l (8.07%), from 930.31 to 970.07 mg/l (4.27%) and from 998.92 to 1052.39 mg/l (5.35%) at the sample: solvent ratio of 1:20, from 407.77 to 450.69 mg/l (10.53%), from 509.31 to 590.15 mg/l (15.87%), from

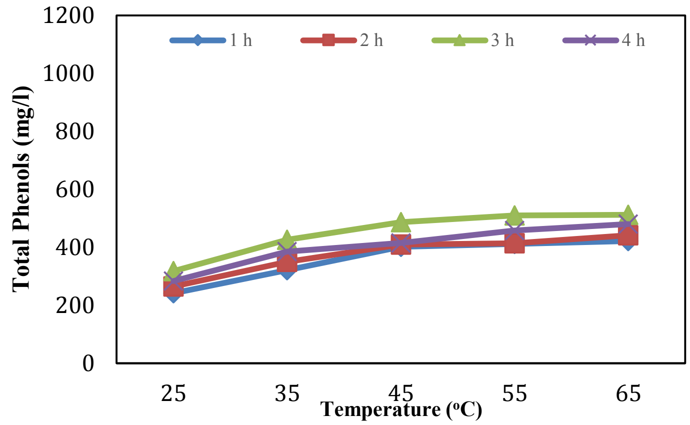
525.85 to 636.23 mg/l (20.99%), from 619.69 to 719.30 mg/l (16.07%) and from 708.92 to 880.00 mg/l (24.13%) at the sample: solvent ratio of 1:40 and from 253.50 to 337.38 mg/l (33.09%), from 351.62 to 466.23 mg/l (32.59%), from 439.69 to 458.92 mg/l (4.37%), from 441.92 to 466.15 mg/l (5.48%) and from 483.53 to 490.30 mg/l (1.40%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively. A further increase in reaction time from 3 to 4 h decreased the phenols yield from 905.46 to 833.31 mg/l (7.97%), from 922.34 to 842.76 mg/l (8.63%), from 963.53 to 859.30 mg/l (10.82%), from 970.07 to 959.30 mg/l (1.11%) and from 1052.39 to 1003.92 mg/l (4.61%) at the sample: solvent ratio of 1:20, from 450.69 to 420.07 mg/l (6.79%), from 590.15 to 581.23 mg/l (1.51%), from 636.23 to 595.84 mg/l (6.35%), from 719.30 to 673.84 mg/l (6.32%) and from 880.00 to 783.84 mg/l (10.93%) at the sample: solvent ratio of 1:40 and from 337.38 to 290.07 mg/l (14.02%), from 466.23 to 412.38 mg/l (11.55%), from 458.92 to 445.85 mg/l (2.85%), from 466.15 to 448.61 mg/l (3.76%) and from 490.30 to 453.00 mg/l (6.31%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

When ethanol was used as a solvent at the 50% concentration, increasing in the reaction time from 1 to 3 h, increased the phenols yield from 320.08 to 343.53 mg/l (7.33%), from 328.69 to 443.15 mg/l (34.82%), from 432.76 to 476.62 mg/l (10.13%), from 456.38 to 534.69 mg/l (17.16%) and from 486.54 to 589.69 mg/l (21.20%) at the sample: solvent ratio of 1:20, from 198.92 to 260.46 mg/l (30.94%), from 208.46 to 266.15 mg/l (27.67%), from 220.83 to 273.08 mg/l (23.66%), from 240.46 to 317.00 mg/l (31.83%) and from 280.15 to 380.00 mg/l (35.71%) at the sample: solvent ratio of 1:40 and from 162.69 to 213.53 mg/l (31.25%), from 167.77 to 248.61 mg/l (48.19%), from 169.46 to 262.15 mg/l (54.70%), from 178.45 to 268.84 mg/l (50.65%) and from 180.62 to 270.84 mg/l (49.95%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively. A further increase in the reaction time from 3 to 4 h decreased the phenols yield from 343.53 to 336.61 mg/l (2.01%), from 443.15 to 397.76 mg/l (10.24%), from 476.62 to 414.15 mg/l (13.11%), from 534.69 to 510.07 mg/l (4.60%) and from 589.69 to 578.07 mg/l (1.97%) at the sample: solvent ratio of 1:20, from 260.46 to 218.23 mg/l (16.21%), from 266.15 to 230.84 mg/l (13.27%), from 273.08 to 258.07 mg/l (5.50%), from 317.00 to 297.38 mg/l (6.19%) and from 380.00 to 323.15 mg/l

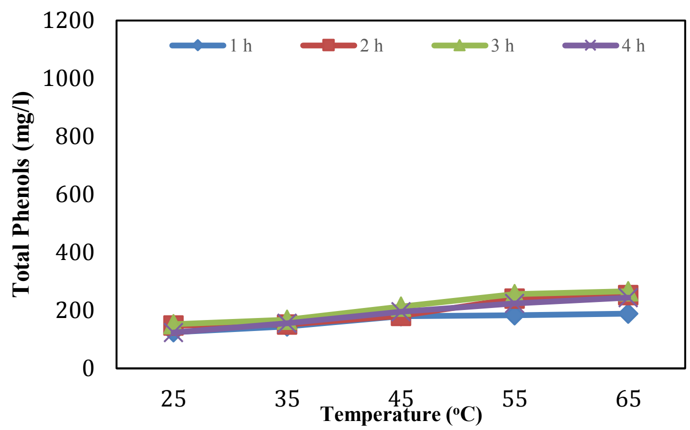
(14.96%) at the sample: solvent ratio of 1:40 and from 213.53 to 173.46 mg/l (18.77%), from 248.61 to 236.53 mg/l (4.86%), from 262.15 to 248.23 mg/l (5.31%), from 268.84 to 252.84 mg/l (5.95%) and from 270.84 to 261.07 mg/l (3.61%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

When ethanol was used as a solvent at the 75% concentration, increasing the reaction time from 1 to 3 h, increased the phenols yield from 583.23 to 610.84 mg/l (4.73%), from 588.92 to 629.69 mg/l (6.92%), from 598.92 to 653.15 mg/l (9.05%), from 629.31 to 685.38 mg/l (8.91%) and from 637.00 to 688.92 mg/l (8.15%) at the sample: solvent ratio of 1:20, from 422.77 to 433.38 mg/l (2.51%), from 454.31 to 462.23 mg/l (1.74%), from 462.77 to 471.07 mg/l (1.79%), from 478.08 to 490.15 mg/l (2.52%) and from 490.38 to 512.07 mg/l (4.42%) at the sample: solvent ratio of 1:40 and from 275.85 to 386.61 mg/l (40.15%), from 293.23 to 389.30 mg/l (32.76%), from 302.61 to 397.30 mg/l (31.29%), from 323.54 to 402.15 mg/l (24.30%) and from 338.15 to 410.92 mg/l (21.52%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively. A further increase in the reaction time from 3 to 4 h decreased the phenols yield from 610.84 to 575.07 mg/l (5.86%), from 629.69 to 579.69 mg/l (7.94%), from 653.15 to 599.69 mg/l (8.18%), from 685.38 to 606.53 mg/l (11.50%) and from 688.92 to 612.46 mg/l (3.85%) at the sample: solvent ratio of 1:20, from 433.38 to 418.30 mg/l (3.48%), from 462.23 to 431.46 mg/l (5.03%), from 471.07 to 449.46 mg/l (4.58%), from 490.15 to 451.23 mg/l (7.94%) and from 512.07 to 483.38 mg/l (1.43%) at the sample: solvent ratio of 1:40 and from 386.61 to 260.69 mg/l (32.57%), from 389.30 to 264.30 mg/l (32.11%), from 397.30 to 273.23 mg/l (31.23%), from 402.15 to 275.69 mg/l (31.45%) and from 410.92 to 281.53 mg/l (31.49%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C respectively.

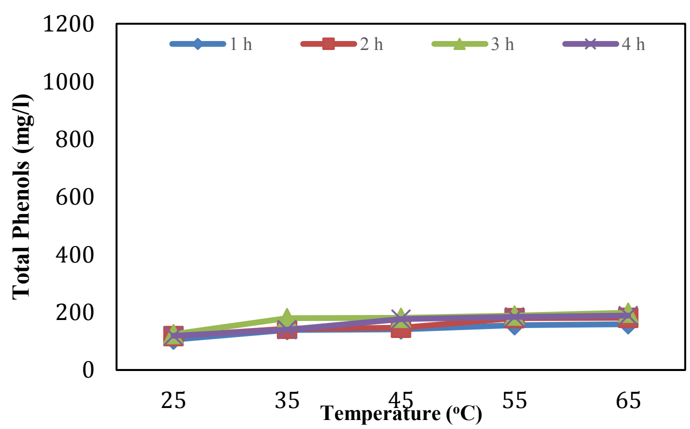
5.1.2.2. Effect of Reaction Temperature: Figures 5.25-5.32 show the effect of reaction temperature on the total phenols yield from ajwa date fruit at different reaction times (1, 2, 3 and 4 h) and sample: solvent ratios (1:20, 1:40 and 1:60) using acetone and ethanol at different concentrations (0, 50 and 75%). Generally, there was an increase in the total phenols yield when the reaction temperature was increased from 25 to 65^{o}C at all sample: solvent ratio at all sample: solvent ratios and solvent concentrations for both solvents.



(a) Sample: solvent ratio 1:20

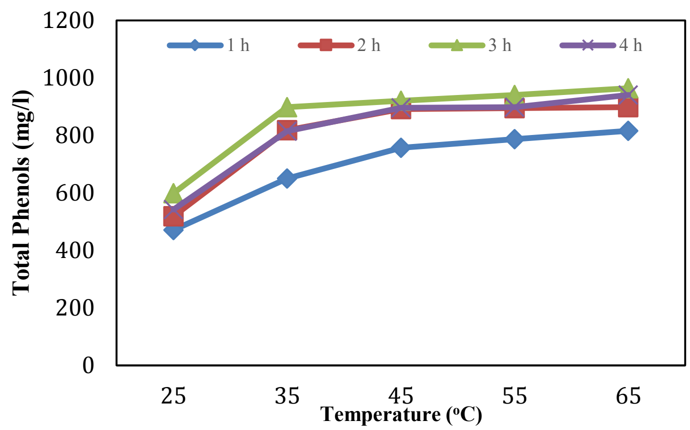


(b) Sample: solvent ratio 1:40

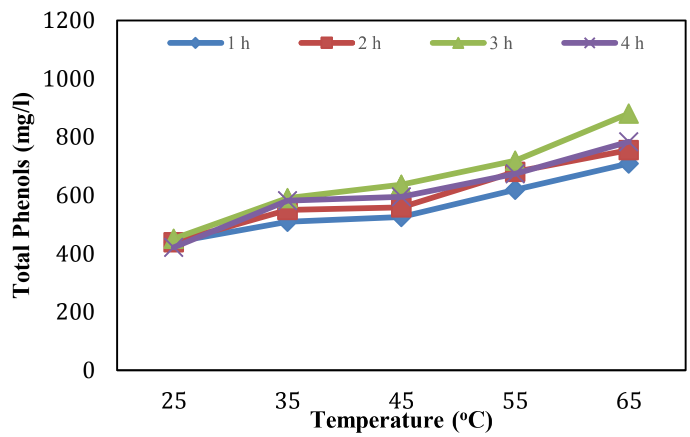


(c) Sample: solvent ratio 1:60

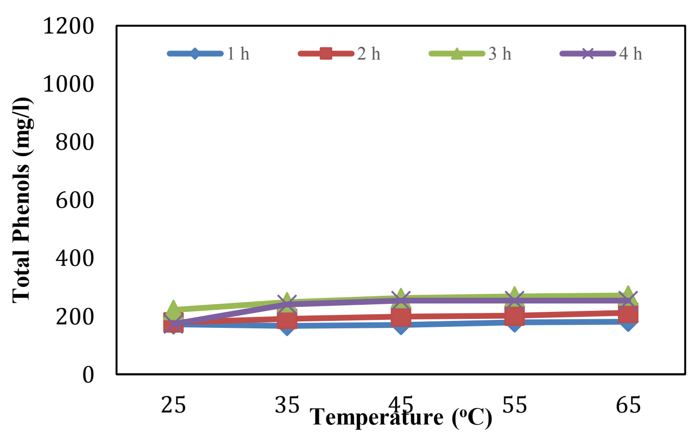
Figure 5.28. The effect of reaction temperatures on the total phenols yield from ajwa date fruit using water at different reaction times.



(a) Sample: solvent ratio 1:20

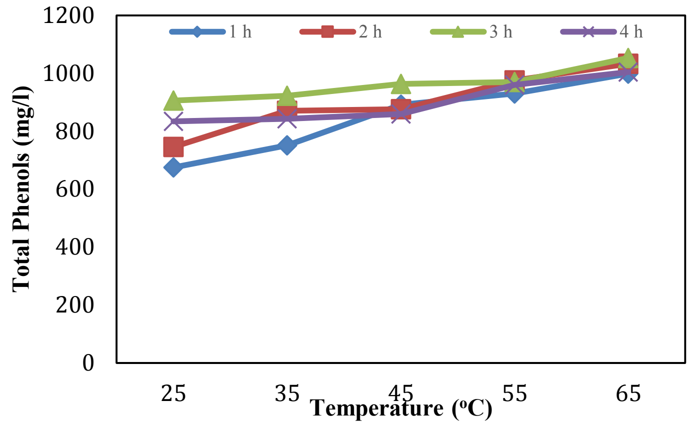


(b) Sample: solvent ratio 1:40

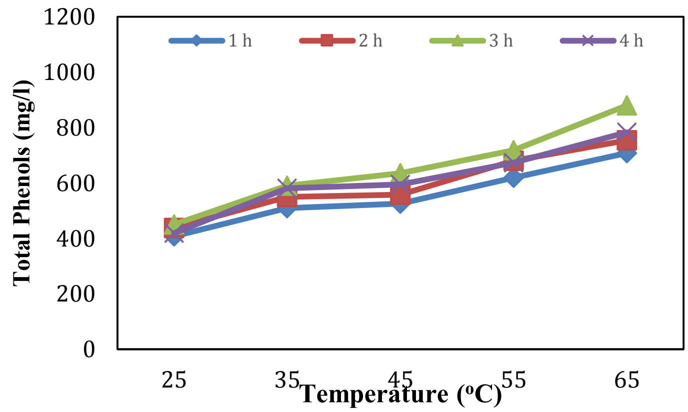


(c) Sample: solvent ratio 1:60

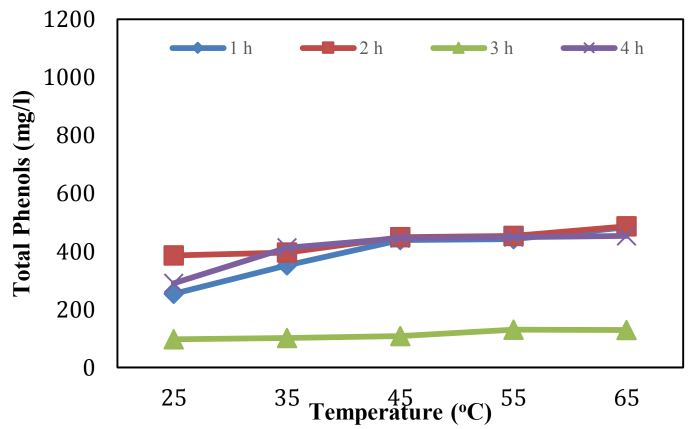
Figure 5.29. The effect of reaction temperatures on the total phenols yield from ajwa date fruit using 50% acetone at different reaction times.



(a) Sample: solvent ratio 1:20

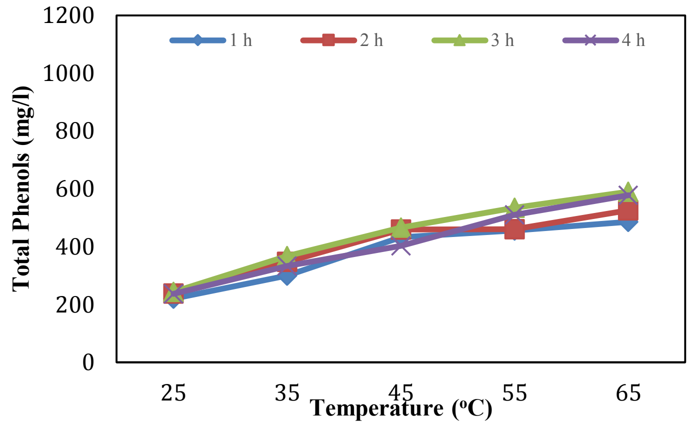


(b) Sample: solvent ratio 1:40

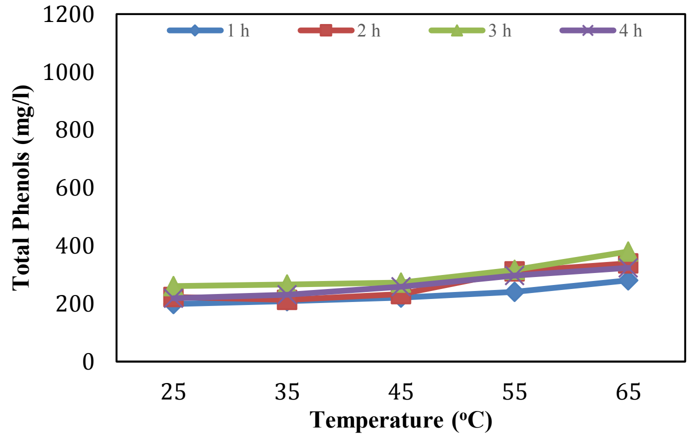


(a) Sample: solvent ratio 1:60

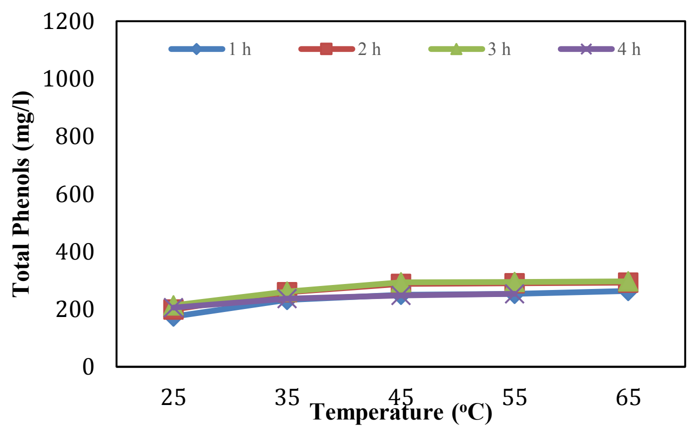
Figure 5.30. The effect of reaction temperatures on the total phenols yield from ajwa date fruit using 75% acetone at different reaction times.



(b) Sample: solvent ratio 1:20

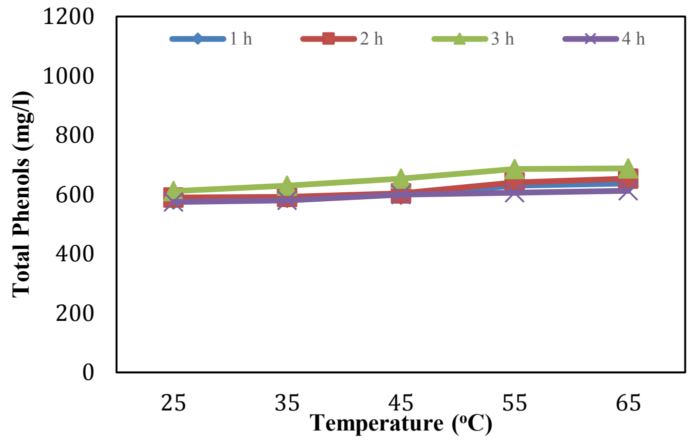


(c) Sample: solvent ratio 1:40

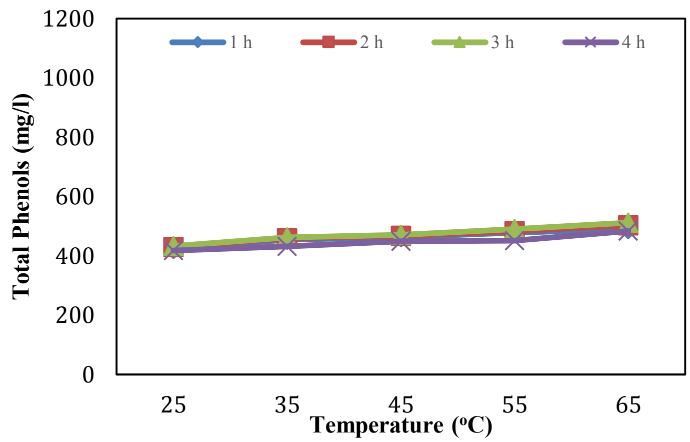


(c) Sample: solvent ratio 1:60

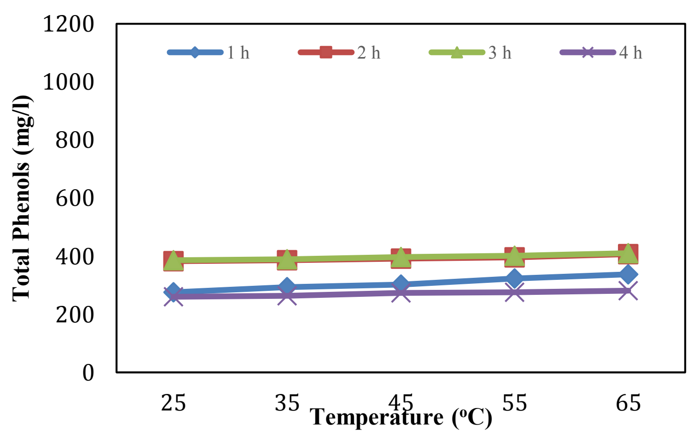
Figure 5.31. The effect of reaction temperatures on the total phenols yield from ajwa date fruit using 50% ethanol at different reaction times.



(a) Sample: solvent ratio 1:20



(b) Sample: solvent ratio 1:40



(c) Sample: solvent ratio 1:60

Figure 5.32. The effect of reaction temperatures on the total phenols yield from ajwa date fruit using 75% ethanol at different reaction times.

When water was used as a solvent, increasing the reaction temperature from 25 to 65°C, increased the phenols yield from 241.62 to 421.61 mg/l (74.49%), from 264.69 to 440.84 mg/l (66.55%), from 317.46 to 512.38 mg/l (61.40%) and from 283.07 to 480.07 mg/l (69.59%) at the sample: solvent ratio of 1:20, from 124.69 to 188.53 mg/l (51.20%), from 147.38 to 252.76 mg/l (71.50%), from 151.00 to 265.00 mg/l (75.50%) and from 122.84 to 243.53 mg/l (98.25%) at the sample: solvent ratio of 1:40 and from 105.07 to 158.61 mg/l (50.96%), from 116.61 to 180.38 mg/l (54.69%), from 124.30 to 198.00 mg/l (59.29%) and from 117.76 to 188.15 mg/l (59.77%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

When acetone was used as solvent at the 50 % concentration, increasing the reaction temperature from 25 to 65°C increased the phenols yield from 470.46 to 815.46 mg/l (73.33%), from 518.54 to 897.69 mg/l (73.12%), from 598.92 to 963.92 mg/l (60.94%) and from 542.00 to 940.53 mg/l (73.53%) at the sample: solvent ratio of 1:20, from 212.38 to 430.92 mg/l (102.90%), from 223.54 to 458.92 mg/l (105.30%), from 263.53 to 592.61 mg/l (124.87%) and from 243.00 to 538.15 mg/l (121.46%) at the sample: solvent ratio of 1:40 and from 176.00 to 263.92 mg/l (49.95%), from 198.53 to 291.30 mg/l (46.73%), from 222.00 to 296.15 mg/l (33.40%) and from 205.15 to 271.07 mg/l (46.34%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

When acetone was used as solvent at the 75 % concentration, increasing the reaction temperature from 25 to 65°C increased the phenols yield from 675.08 to 998.92 mg/l (47.97%), from 745.46 to 1032.61 mg/l (38.52%), from 905.46 to 1052.39 mg/l (16.23%) and from 833.31 to 1003.92 mg/l (20.47%) at the sample: solvent ratio of 1:20, from 407.77 to 708.92 mg/ml (73.85%), from 438.07 to 754.46 mg/l (72.22%), from 450.69 to 880.00 mg/l (95.26%) and from 420.07 to 783.84 mg/l (86.60%) at the sample: solvent ratio of 1:40 and from 253.50 to 483.53 mg/l (90.74%), from 386.61 to 486.53 mg/l (25.85%), from 337.38 to 490.30 mg/l (45.33%) and from 290.07 to 453.00 mg/l (56.17%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

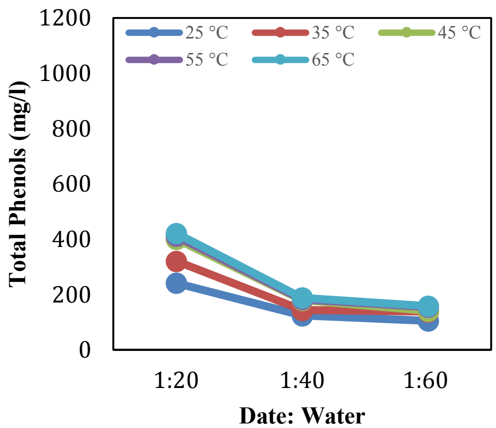
When ethanol was used as solvent at the 50 % concentration, increasing the reaction temperature from 25 to 65°C increased the phenols yield from 320.08 to 486.54 mg/l (52.01%), from 325.46 to 525.46 mg/l (61.45%), from 343.53 to 589.69 mg/l (71.66%) and from 336.61 to 578.07 mg/l (71.73%) at the sample: solvent ratio of 1:20, from 198.92 to

280.15 mg/l (40.84%), from 223.30 to 339.38 mg/l (51.98%), from 260.46 to 380.00 mg/l (45.90%) and from 218.23 to 323.15 mg/l (48.08%) at the sample: solvent ratio of 1:40 and from 162.69 to 180.62 mg/l (11.02%), from 178.92 to 212.84 mg/l (18.96%), from 213.53 to 270.84 mg/l (26.84%) and from 173.46 to 261.07 mg/l (50.51%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

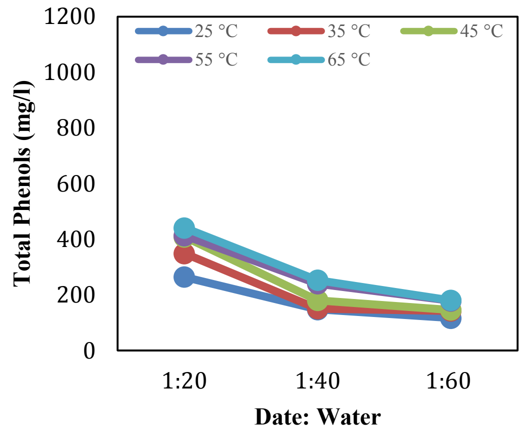
When ethanol was used as solvent at the 75 % concentration, increasing the reaction temperature from 25 to 65°C increased the phenols yield from 583.23 to 637.00 mg/l (9.22%), from 589.53 to 653.07 mg/l (10.78%), from 610.84 to 688.92 mg/l (2.78%) and from 575.07 to 612.46 mg/l (6.50%) at the sample: solvent ratio of 1:20, from 422.77 to 490.38 mg/l (15.99%), from 430.15 to 503.38 mg/l (17.02%), from 433.38 to 512.07 mg/l (18.16%) and from 418.30 to 483.38 mg/l (15.56%) at the sample: solvent ratio of 1:40 and from 275.85 to 338.15 mg/l (22.58%), from 382.76 to 406.76 mg/l (6.27%), from 386.61 to 410.92 mg/l (6.29%) and from 260.69 to 281.53 mg/l (7.99%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

5.1.2.3. Effect of Sample: Solvent Ratio: Figures 5.33-5.37 shown the effect of sample: solvent ratio on the total phenols yield from ajwa date fruit at different reaction times (1, 2, 3 and 4 h) and reaction temperatures (25, 35, 45, 55 and 65°C) using acetone and ethanol at different concentrations (0, 50 and 75%). Generally, there was a decrease in the total phenols yield when sample: solvent ratio was increased from 1:20 to 1:60 at all reaction temperatures, solvent concentration and reaction times for both solvents.

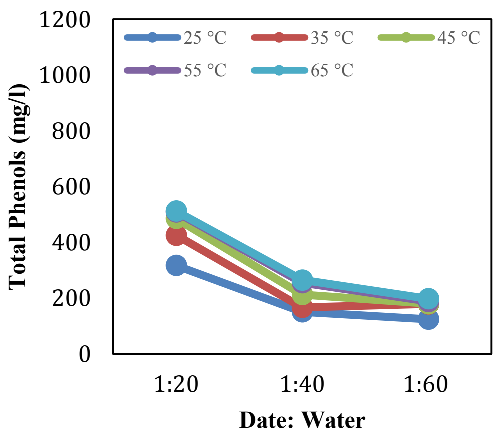
When water was used as solvent, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the phenols yield from 241.62 to 105.07 mg/l (56.51%), from 321.23 to 138.53 mg/l (56.88%), from 401.23 to 140.53 mg/l (64.98%), from 412.00 to 155.23 mg/l (62.32%) and from 421.61 to 158.61 mg/ml (62.38%) at the reaction time of 1 h, from 264.69 to 116.61 mg/l (55.94%), from 349.30 to 142.76 mg/l (59.13%), from 408.92 to 146.23 mg/l (64.24%), from 412.00 to 178.84 mg/l (56.75%) and from 440.84 to 180.38 mg/l (59.08%) at the reaction time of 2 h, from 317.46 to 124.30 mg/l (60.85%), from 425.76 to 178.92 mg/l (57.98%), from 466.23 to 180.46 mg/l (61.29%), from 509.30. to 188.38 mg/l (63.01%) and from 512.38 to 198.00 mg/l (61.36%) at the reaction time of 3 h and from 283.07 to 117.76 mg/l (58.40%), from 385.15 to 139.30 mg/l (63.83%), from 405.69 to 176.46 mg/l (56.50%), from 457.76 to 183.46 mg/l (59.92%) and from 480.07 to



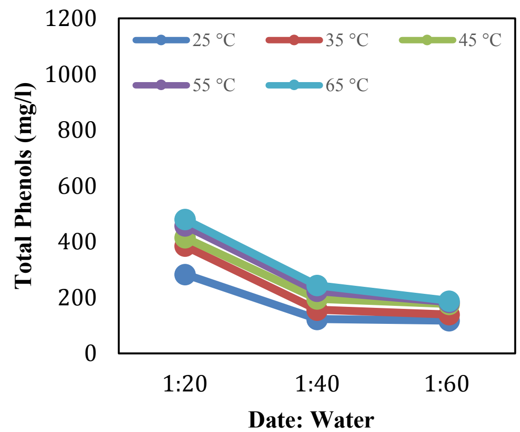
(a) One hour



(b) Two hours



(c) Three hours



(d) Four hours

Figure 5.33. The effect of sample: solvent ratio on the total phenols yield from ajwa date fruit using water at different reaction temperatures.

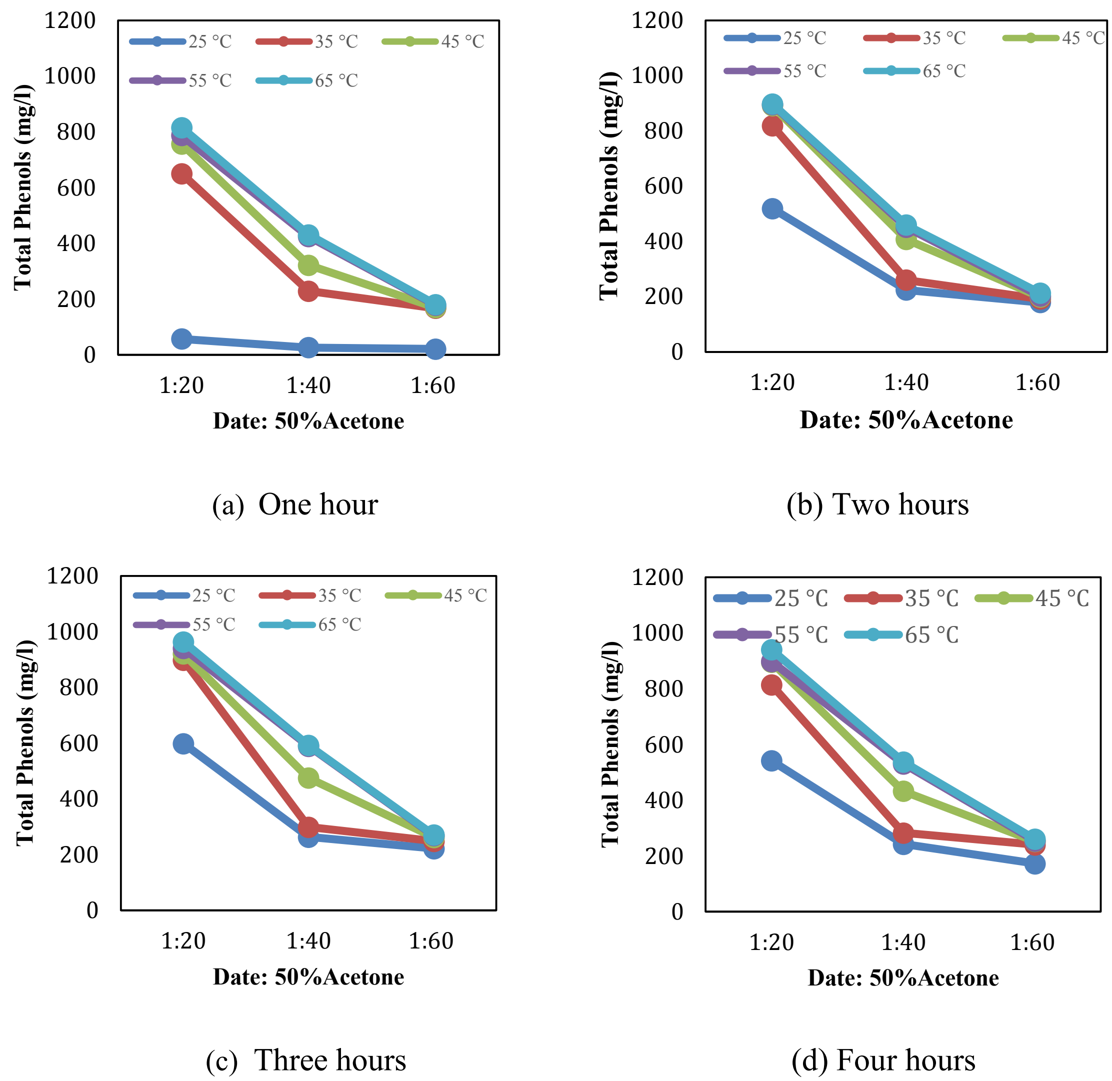
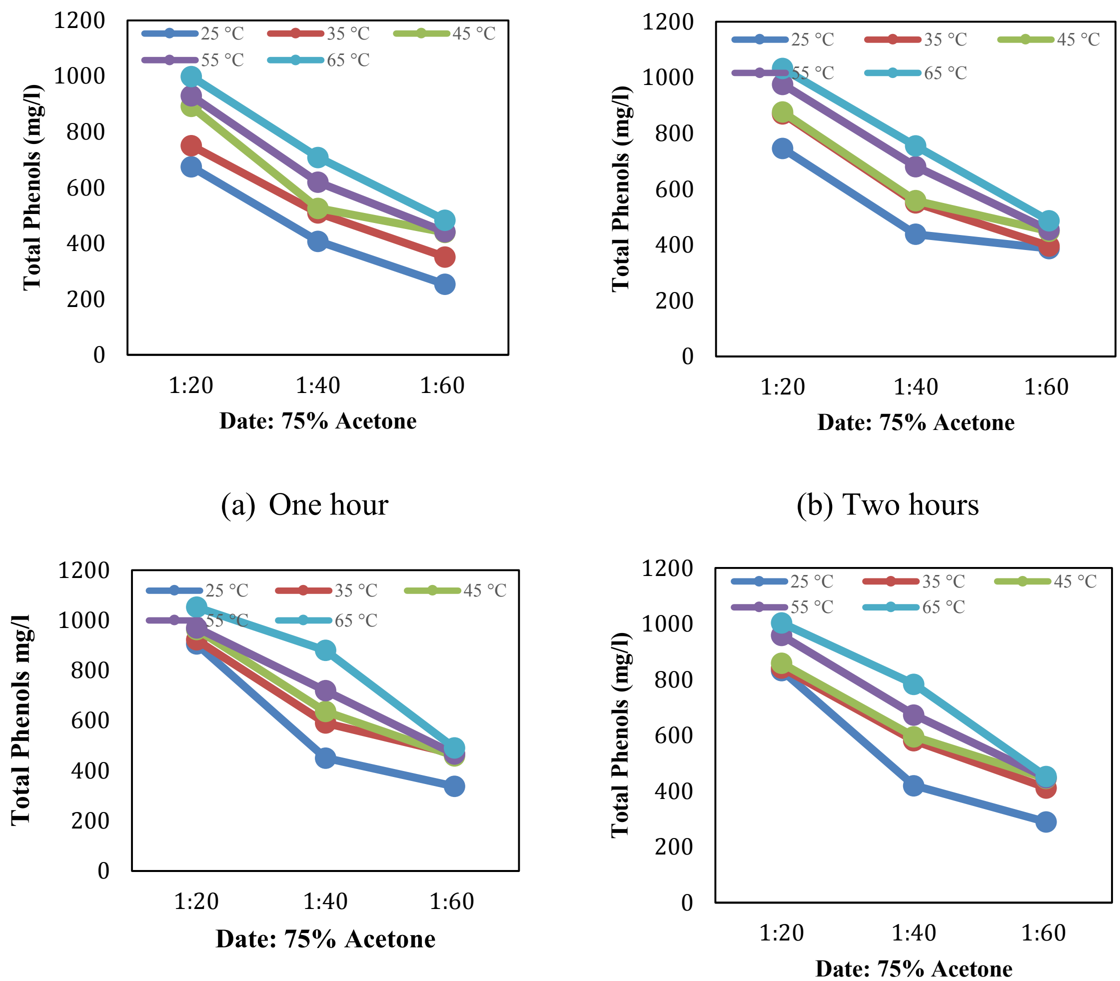


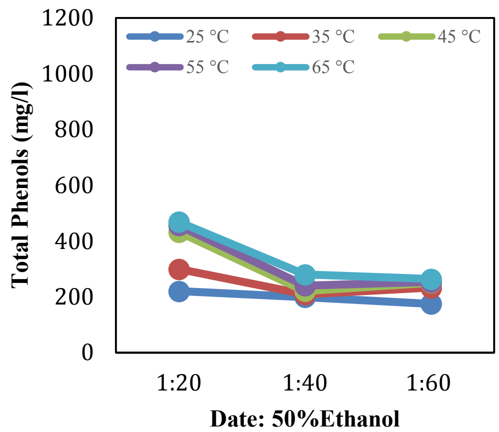
Figure 5.34. The effect of sample: solvent ratio on the total phenols yield from ajwa date fruit using 50% acetone at different reaction temperatures.



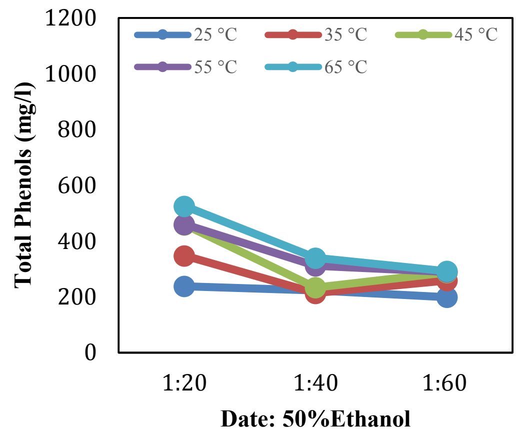
(a) One hour (b) Two hours

(c) Three hours (d) Four hours

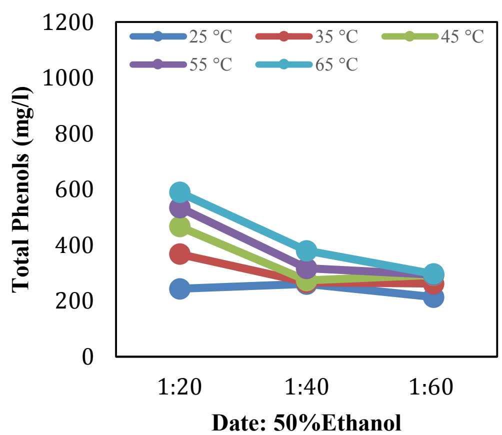
Figure 5.35. The effect of sample: solvent ratio on the total phenols yield from ajwa date fruit using 75% acetone at different reaction temperatures.



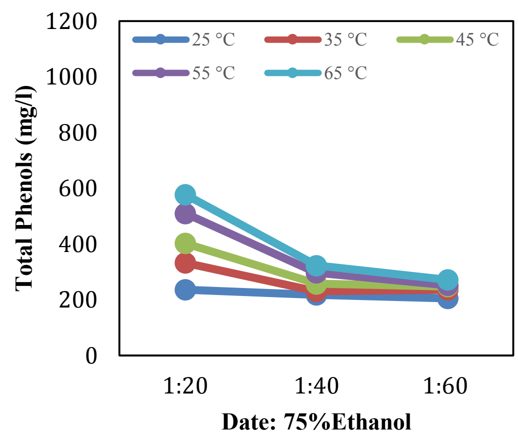
(a) One hour



(b) Two hours

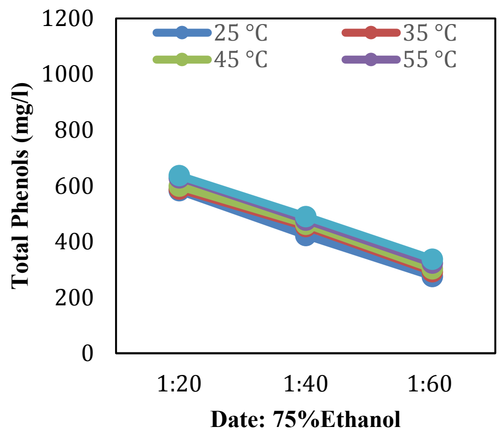


(c) Three hours

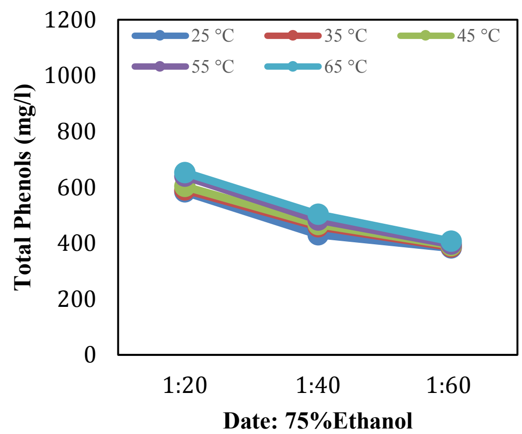


(d) Four hours

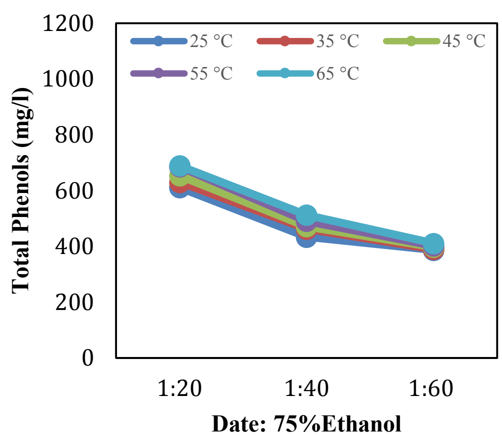
Figure 5.36. The effect of sample: solvent ratio on the total phenols yield from ajwa date fruit using 50% ethanol at different reaction temperatures.



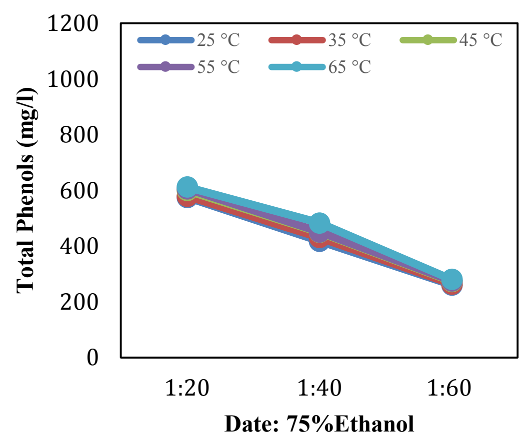
(a) One hour



(b) Two hours



(c) Three hours



(d) Four hours

Figure 5.37. The effect of sample: solvent ratio on the total phenols yield from ajwa date fruit using 75% ethanol at different reaction temperatures.

188.15 mg/l (60.81%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

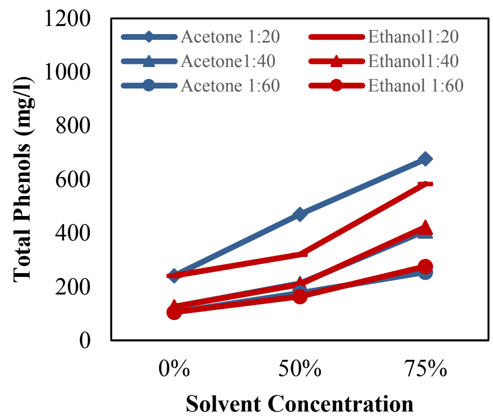
When acetone was used as solvent at the 50 % concentration, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the phenols yield from 470.46 to 176.00 mg/l (62.59%), from 650.08 to 232.31 mg/l (64.26%), from 756.62 to 250.88 mg/l (66.84%), from 787.00 to 253.54 mg/l (67.78%) and from 815.46 to 263.92 mg/l (67.64%) at the reaction time of 1 h, from 518.54 to 198.53 mg/l (61.71%), from 818.53 to 258.61 mg/l (68.41%), from 891.23 to 287.53 mg/l (67.74%), from 895.15 to 289.61 mg/l (67.65%) and from 897.69 to 291.30 mg/l (67.55%) at the reaction time of 2 h, from 598.92 to 222.00 mg/l (62.93%), from 898.46 to 261.53 mg/l (70.89%), from 920.38 to 293.61 mg/l (68.10%), from 940.07 to 293.38 mg/l (68.79%) and from 963.92 to 296.15 mg/l (69.28%) at the reaction time of 3 h and from 542.00 to 205.15 mg/l (62.15%), from 814.30 to 240.46 mg/l (70.47%), from 895.53 to 253.00 mg/l (71.75%), from 898.07 to 254.30 mg/l (71.68%) and from 940.53 to 271.07 mg/ml (71.18%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

When acetone was used as solvent at the 75% concentration, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the phenols yield from 675.08 to 253.50 mg/l (62.45%), from 750.85 to 351.62 mg/l (53.17%), from 891.62 to 439.69 mg/l (50.69%), from 930.31 to 439.69 mg/l (52.50%) and from 998.92 to 483.53 mg/l (51.59%) at the reaction time of 1 h, from 745.46 to 386.61 mg/l (48.14%), from 870.07 to 395.84 mg/l (54.50%), from 876.15 to 448.53 mg/l (48.81%), from 975.53 to 453.69 mg/l (53.49%) and from 1032.61 to 486.53 mg/l (52.88%) at the reaction time of 2 h, from 905.46 to 337.38 mg/l (62.74%), from 922.34 to 466.23 mg/l (49.45%), from 963.53 to 458.921 mg/l (52.37%), from 970.07 to 466.15 mg/l (51.95%) and from 1052.39 to 490.30 mg/l (53.41%) at the reaction time of 3 h and from 833.31 to 290.07 mg/ l (65.19%), from 842.76 to 412.38 mg/l (51.07%), from 859.30 to 445.85 mg/l (48.11%), from 959.30 to 448.61 mg/l (53.24%) and from 1003.92 to 453.00 mg/l (54.88%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

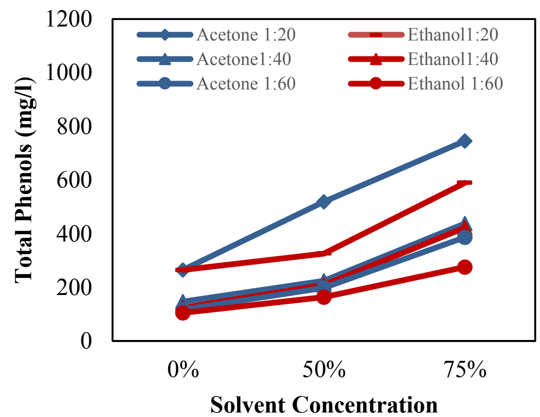
When ethanol was used as solvent at the 50% concentration, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the phenols yield from 320.08 to 162.69 mg/l (49.17%), from 328.69 to 167.77 mg/l (48.96%), from 432.76 to 169.46 mg/l (60.84%), from 456.38 to 178.45 mg/l (60.90%) and from 486.54 to 180.62 mg/l (62.88%) at the reaction time of 1 h, from 325.46 to 178.92 mg/l (45.03%), from 364.38 to 191.92 mg/l (47.33%), from 458.92 to 198.38 mg/l (56.77%), from 460.15 to 268.84 mg/l (56.17%) and from 525.46 to 212.84 mg/l (59.49%) at the reaction time of 2 h, from 343.53 to 213.53 mg/l (37.84%), from 443.15 to 248.61 mg/l (43.90%), from 476.62 to 262.15 mg/l (45.00%), from 534.69 to 268.84 mg/l (49.72%) and from 589.69 to 270.84 mg/l (54.07%) at the reaction time of 3 h and from 336.61 to 173.46 mg/l (48.47%), from 397.76 to 236.53 mg/l (40.53%), from 414.15 to 248.23 mg/l (40.06%), from 510.07 to 252.84 mg/l (50.43%) and from 578.07 to 261.07 mg/l (54.84%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65ºC, respectively.

When ethanol was used as solvent at the 75% concentration, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the phenols yield from 583.23 to 275.85 mg/l (52.70%), from 588.92 to 293.23 mg/l (50.21%), from 598.92 to 302.61 mg/l (49.47%), from 629.31 to 323.54 mg/l (48.59%) and from 637.00 to 338.15 mg/l (46.92%) at the reaction time of 1 h, from 589.53 to 382.76 mg/l (35.07%), from 591.30 to 386.30 mg/l (34.67%), from 603.00 to 391.23 mg/l (35.12%), from 640.07 to 402.15 mg/l (38.05%) and from 653.07 to 406.76 mg/l (37.72%) at the reaction time of 2 h, from 610.84 to 386.61 mg/l (36.71%), from 629.69 to 389.30 mg/l (38.18%), from 653.15 to 397.30 mg/l (39.17%), from 685.38 to 402.15 mg/l (41.32%) and from 688.92 to 410.92 mg/l (40.35%) at the reaction time of 3 h and from 575.07 to 260.69 mg/l (54.67%), from 579.69 to 264.30 mg/l (54.41%), from 599.69 to 273.23 mg/l (54.44%), from 606.53 to 275.69 mg/l (54.55%) and from 612.53 to 281.53 mg/ml (54.03%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65ºC, respectively.

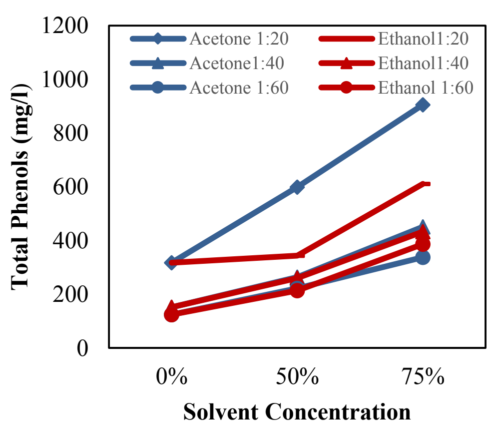
5.1.2.4. Effect of Solvent Concentration: Figures 5.38-5.42 show the effect of solvent concentration on the total phenols yield from ajwa date fruit at different sample: solvent ratios (1:20, 1:40 and 1:60), reaction times (1, 2, 3 and 4 h) and the reaction temperatures of (25, 35, 45, 55 and 65ºC) for acetone and ethanol. Generally, there was an increase in the total phenols yield when solvent concentration was increased from 0 to 75% for all reaction temperatures, sample: solvent ratios and reaction times for both solvents.



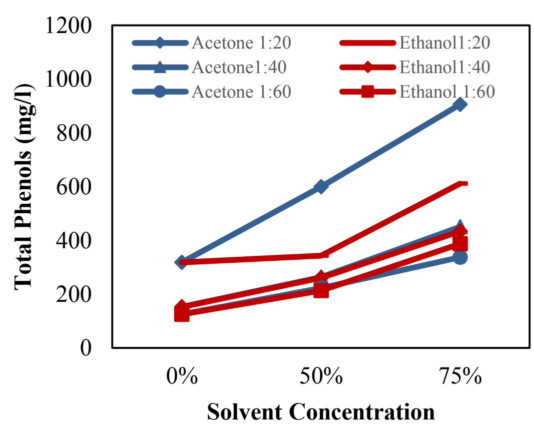
(a) One hour



(b) Two hours

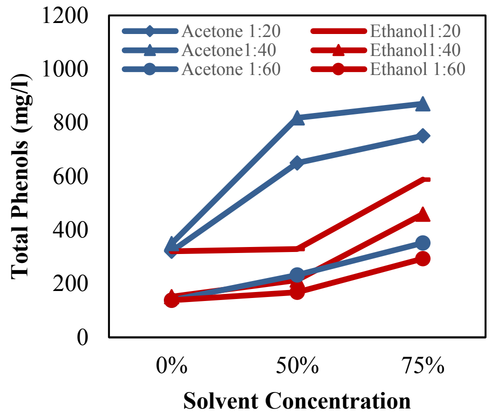


(c) Three hours

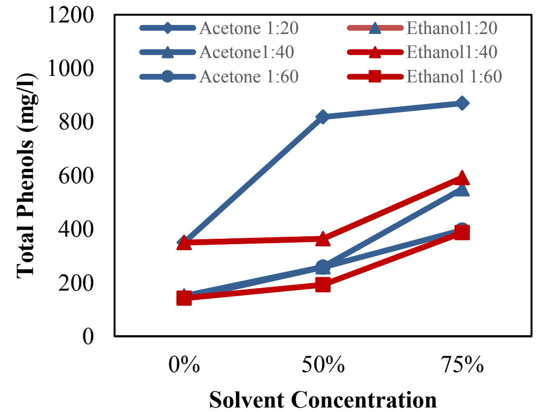


(d) Four hours

Figure 5.38. The effect of solvent (acetone and ethanol) concentration on the total phenols yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 25ºC.



(a) One hour



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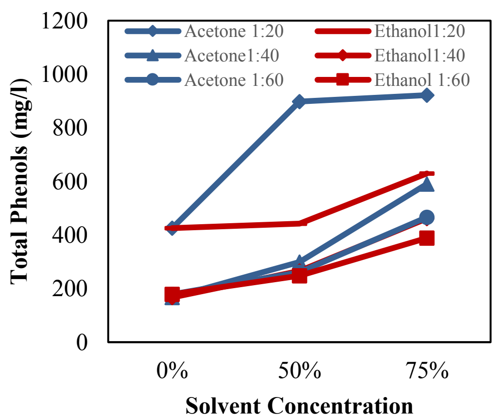
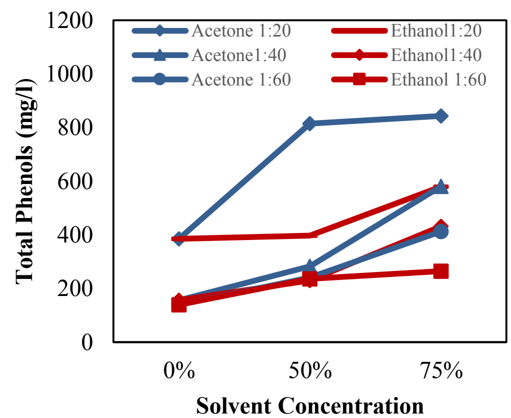
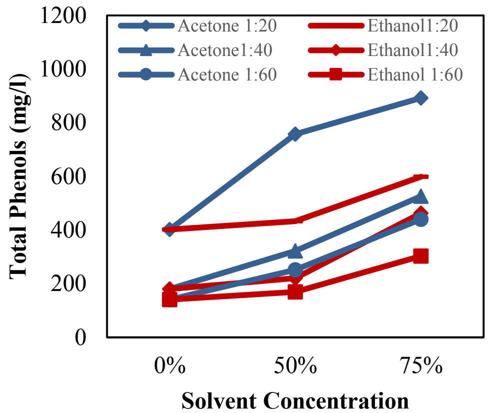


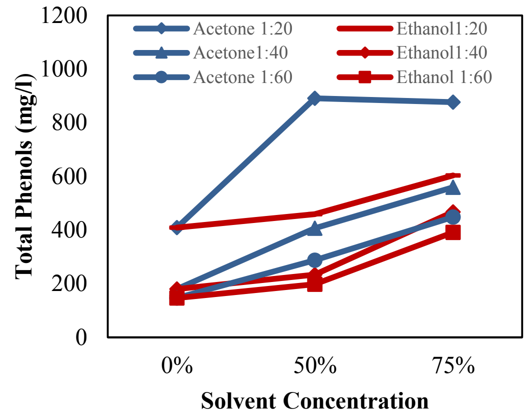
(c) Three hours



(d) Four hours

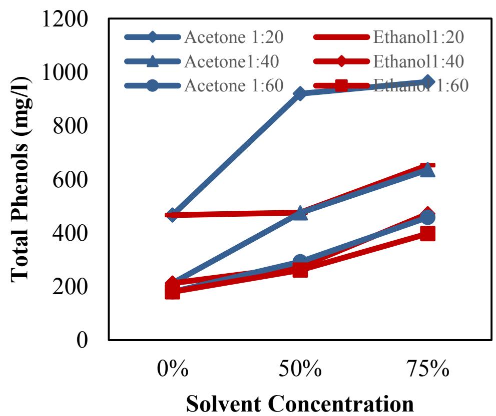
Figure 5.39. The effect of solvent (acetone and ethanol) concentration on the total phenols yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 35^{o}C.

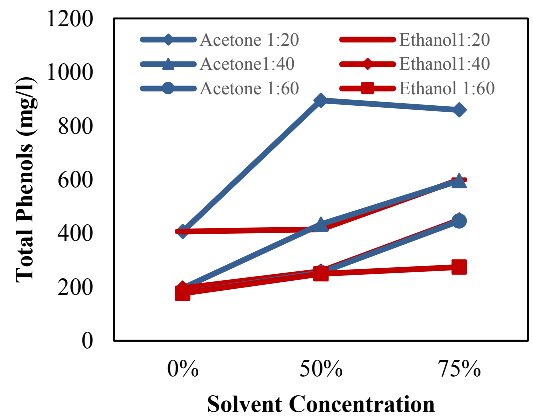




(a) One hour

(b) Two hours

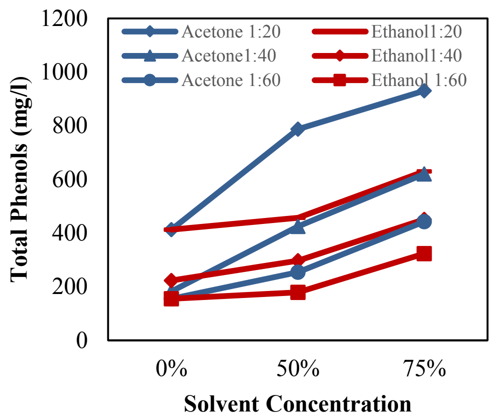




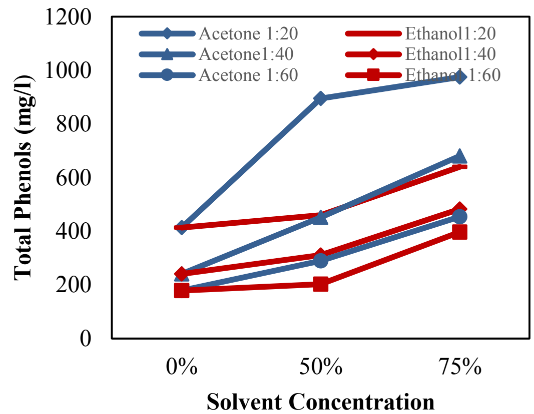
(c) Three hours

(d) Four hours

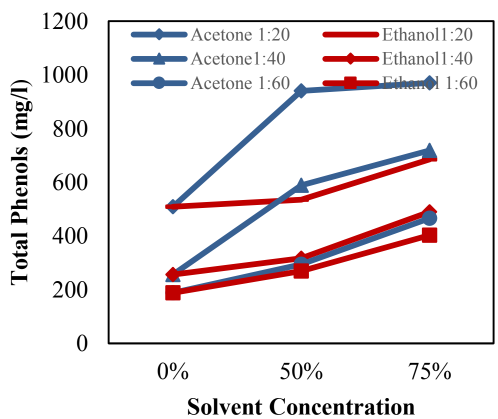
Figure 5.40. The effect of solvent (acetone and ethanol) concentration on the total phenols yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 45°C.



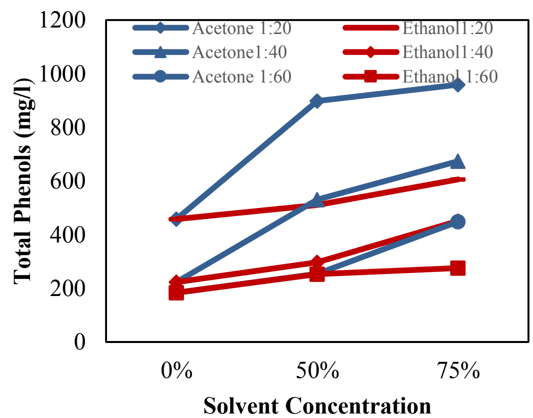
(a) One hour



(b) Two hours

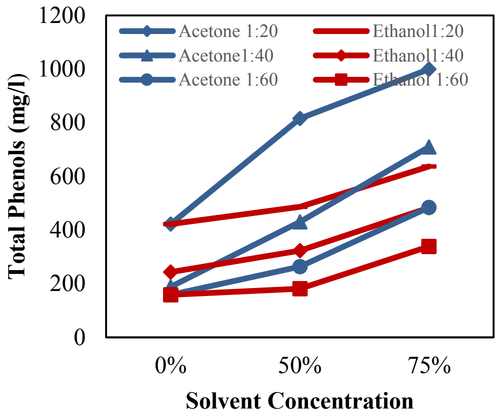


(c) Three hours

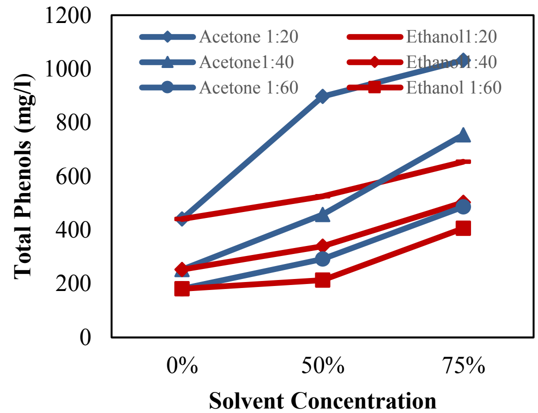


(d) Four hours

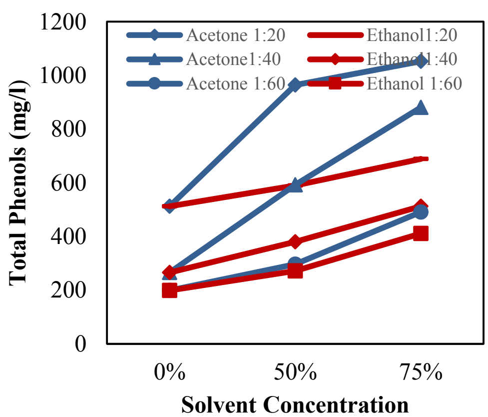
Figure 5.41. The effect of solvent (acetone and ethanol) concentration on the total phenols yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 55^{o}C.



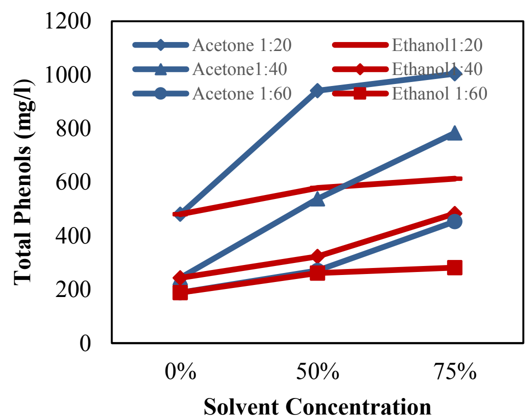
(a) One hour



(b) Two hours



(c) Three hour



(d) Four hours

Figure 5.42. The effect of solvent (acetone and ethanol) concentration on the total phenols yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 65^{o}C.

Increasing the concentration of acetone from 0 to 75% at the temperature of 25°C increased the total phenols yield from 241.62 to 675.08 mg/l (179.40%), from 124.69 to 407.77 mg/l (227.03%) and from 105.07 to 253.50 mg/l (141.27%) at the reaction time of 1 h, from 264.69 to 745.46 mg/l (181.64%), from 147.38 to 438.07 mg/l (181.64%) and from 116.61 to 386.61 mg/l (231.54%) at the reaction time of 2 h, from 317.46 to 905.46 mg/l (185.22%), from 151.00 to 450.69 mg/l (198.47%) and from 124.30 to 337.38 mg/l (171.42%) at the reaction time of 3 h and from 283.07 to 833.31 mg/l (194.38%), from 122.84 to 420.07 mg/l (241.97%) and from 117.76 to 290.07 mg/l (146.32%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 35°C increased the total phenols yield from 321.23 to 750.85 mg/l (133.74%), from 144.30 to 509.31 mg/l (252.95%) and from 138.53 to 351.62 mg/l (153.82%) at the reaction time of 1 h, from 349.30 to 870.07 mg/l (149.09%), from 150.76 to 550.46 mg/l (265.12%) and from 142.76 to 395.84 mg/l (177.28%) at the reaction time of 2 h, from 425.76 to 922.34 mg/l (116.63%), from 167.23 to 590.15 mg/l (252.90%) and from 178.92 to 466.23 mg/l (160.58%) at the reaction time of 3 h and from 385.15 to 842.76 mg/l (118.81%), from 155.76 to 581.23 mg/l (273.16%) and from 139.30 to 412.38 mg/ml (196.04%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 45°C increased the total phenols yield from 401.23 to 891.62 mg/l (122.22%), from 179.69 to 525.85 mg/l (192.64%) and from 140.53 to 439.69 mg/l (212.88%) at the reaction time of 1 h, from 408.92 to 876.15 mg/l (114.26%), from 180.46 to 558.61 mg/l (209.55%) and from 146.23 to 448.53 mg/l (206.73%) at the reaction time of 2 h, from 466.23 to 963.53 mg/l (106.66%), from 213.15 to 636.23 mg/l (198.49%) and from 180.46 to 458.921 mg/l (154.31%) at the reaction time of 3 h and from 405.69 to 859.30 mg/l (111.81%), from 195.46 to 595.84 mg/l (204.84%) and from 176.46 to 445.85 mg/l (152.66%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 55°C increased the total phenols yield from 412.00 to 930.31 mg/l (125.80%), from 182.76 to 619.69 mg/l (239.07%) and from 155.23 to 441.92 mg/l (184.69%) at the reaction time of 1 h, from 413.53 to 975.53 mg/l (135.90%), from 240.00 to 680.84 mg/l (183.68%) and from 178.84 to 453.69 mg/l (153.68%) at the reaction time of 2 h, from 509.30 to 970.07 mg/l (90.47%), from 255.53 to 719.30 mg/l (181.49%) and from 188.38 to 466.15 mg/l (147.45%) at the reaction time of 3 h and from 457.76 to 959.30 mg/l (109.56%), from 223.38 to 673.84 mg/l (201.66%) and from 183.46 to 448.61 mg/l (144.53%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 65^{o}C increased the total phenols yield from 421.61 to 998.92 mg/l (136.93%), from 188.53 to 708.92 mg/l (276.03%) and from 158.61 to 483.53 mg/l (204.85%) at the reaction time of 1 h, from 440.84 to 1032.61 mg/l (134.24%), from 252.76 to 754.46 mg/l (198.49%) and from 180.38 to 486.53 mg/l (169.73%) at the reaction time of 2 h, from 512.38 to 1052.39 mg/l (105.39%), from 265.00 to 880.00 mg/l (232.08%) and from 198.00 to 490.30 mg/l (147.63%) at the reaction time of 3 h and from 480.07 to 1003.92 mg/l (109.12%), from 243.53 to 783.84 mg/l (221.87%) and from 188.15 to 453.00 mg/l (140.73%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 25^{o}C increased the total phenols yield from 241.62 to 583.23 mg/l (141.38%), from 124.69 to 422.77 mg/l (239.06%) and from 105.07 to 275.85 mg/l (162.54%) at the reaction time of 1 h, from 264.69 to 589.53 mg/l (122.72%), from 147.38 to 430.15 mg/l (191.86%) and from 116.61 to 382.76 mg/l (228.24%) at the reaction time of 2 h, from 317.46 to 610.84 mg/l (92.41%), from 151.00 to 433.38 mg/l (187.01%) and from 124.30 to 386.61 mg/l (211.03%) at the reaction time of 3 h and from 283.07 to 575.07 mg/l (103.15%), from 122.84 to 418.30 mg/l (240.52%) and from 117.76 to 260.69 mg/l (121.37%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 35^{o}C increased the total phenols yield from 321.23 to 588.92 mg/l (83.33%), from 144.30 to 454.31 mg/l (214.84%) and from 138.53 to 293.23 mg/l (111.67%) at the reaction time of 1 h, from 349.30 to 591.30 mg/l (69.28%), from 150.76 to 459.30 mg/l (204.66%) and from 142.76 to 386.30 mg/l (170.59%) at the reaction time of 2 h, from 425.76 to 629.69 mg/l (47.90%), from 167.23 to 462.23 mg/l (176.40%) and from 178.92 to 389.30 mg/l (117.58%) at the reaction time of 3 h and from 385.15 to 579.69 mg/l (50.51%), from

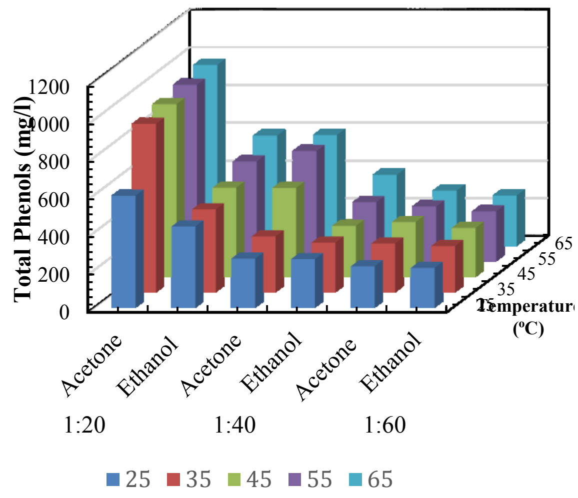
155.76 to 431.46 mg/l (177%) and from 139.30 to 264.30 mg/ml (89.73%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 45°C increased the total phenols yield from 401.23 to 598.92 mg/l (49.27%), from 179.69 to 462.77 mg/l (157.54%) and from 140.53 to 302.61 mg/l (115.33%) at the reaction time of 1 h, from 408.92 to 603.00 mg/l (47.46%), from 180.46 to 467.15 mg/l (158.87%) and from 146.23 to 391.23 mg/l (167.54%) at the reaction time of 2 h, from 466.23 to 653.15 mg/l (40.09%), from 213.15 to 471.07 mg/l (121%) and from 180.46 to 397.30 mg/l (120.16%) at the reaction time of 3 h and from 405.69 to 599.69 mg/l (47.82%), from 195.46 to 449.46 mg/l (129.95%) and from 176.46 to 273.23 mg/l (54.84%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

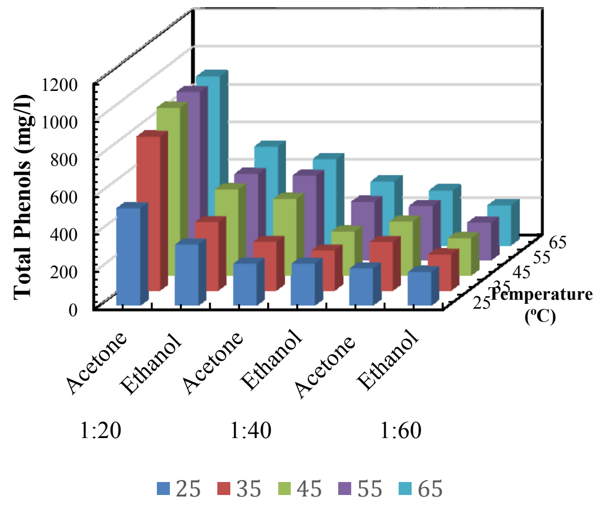
Increasing the concentration of ethanol from 0 to 75% at the temperature of 55°C increased the total phenols yield from 412.00 to 629.31 mg/l (52.75%), from 182.76 to 483.00 mg/l (161.59%) and from 155.23 to 323.54 mg/l (108.43%) at the reaction time of 1 h, from 413.53 to 640.07 mg/l (54.78%), from 240.00 to 483.00 mg/l (101.25%) and from 178.84 to 396.53 mg/l (121.72%) at the reaction time of 2 h, from 509.30 to 685.38 mg/l (34.57%), from 255.53 to 490.15 mg/l (91.82%) and from 188.38 to 402.15 mg/l (113.48%) at the reaction time of 3 h and from 457.76 to 606.53 mg/l (32.50%), from 223.38 to 451.23 mg/l (102%) and from 183.46 to 275.69 mg/l (50.27%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 65°C increased the total phenols yield from 421.61 to 637.00 mg/l (51.09%), from 188.53 to 490.38 mg/l (160.11%) and from 158.61 to 338.15 mg/l (113.20%) at the reaction time of 1 h, from 440.84 to 653.07 mg/l (48.14%), from 252.76 to 503.38 mg/l (99.15%) and from 180.38 to 406.76 mg/l (125.50%) at the reaction time of 2 h, from 512.38 to 688.92 mg/l (34.45%), from 265.00 to 512.07 mg/l (93.23%) and from 198.00 to 410.92 mg/l (107.54%) at the reaction time of 3 h and from 480.07 to 612.46 mg/l (27.58%), from 243.53 to 483.38 mg/l (98.49%) and from 188.15 to 281.53 mg/l (49.63%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

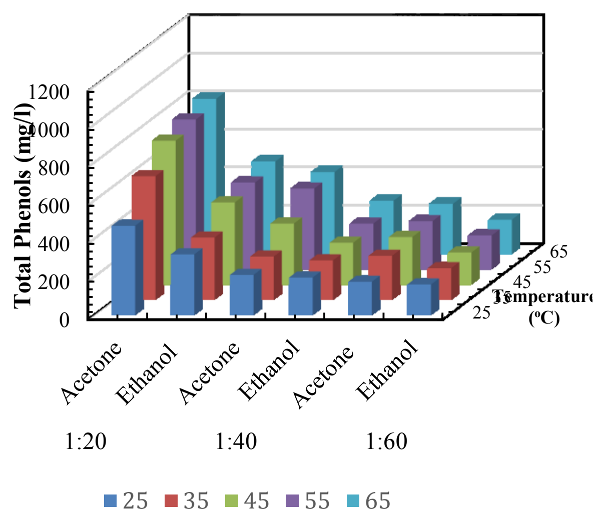
5.1.2.5. Effect of Solvent Type: Figures 5. 43 and 5.44 show the effect of solvent type on the total phenols yield from ajwa date fruit at different sample: solvent ratios (1:20, 1:40



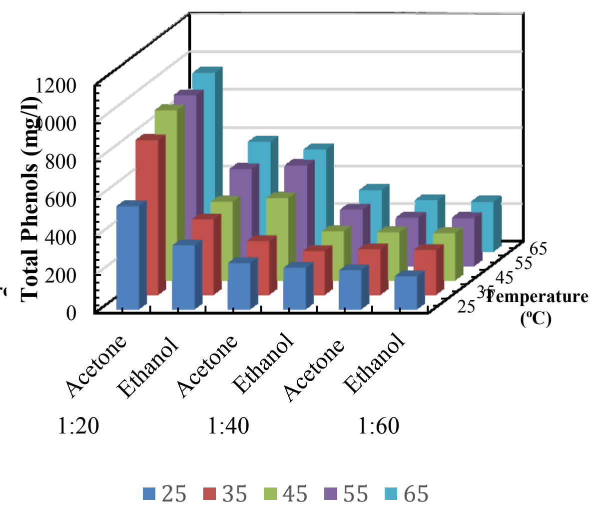
(a) One hour



(b) Two hours

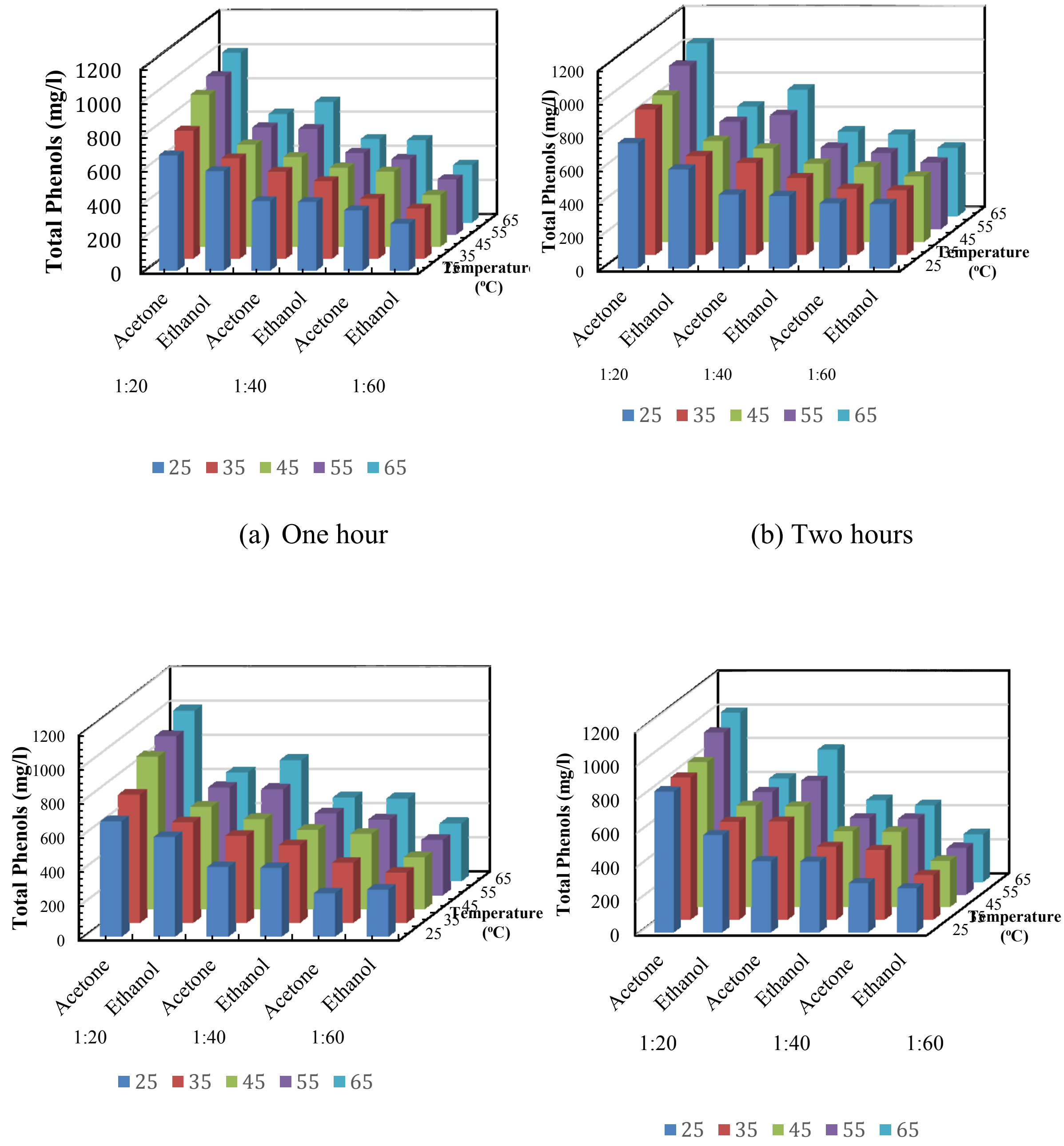


(c) Three hours



(d) Four hours

Figure 5.43. The effect of solvent (acetone and ethanol) at the concentration of 50% on the total phenols yield from ajwa date fruit at different sample: solvent ratios and different reaction temperatures.



(a) One hour

(b) Two hours

(c) Three hours

(d) Four hours

Figure 5.43. The effect of solvent (acetone and ethanol) at the concentration of 75% on the total phenols yield from ajwa date fruit at different sample: solvent ratios and different reaction temperatures.

and 1:60), reaction times (1, 2, 3 and 4 h) and reaction temperatures (25, 35, 45, 55 and 65^{o}C). Generally, when acetone was used as a solvent, higher total phenols yield was obtained compared to that obtained with ethanol at the same concentration at all reaction temperatures, sample: solvent ratios and reaction times with 50 and 75% concentrations.

Using ethanol at the 50% concentration with the sample: solvent ratio of 1:20decreased the total phenols yield over that obtained with acetone from 470.46 to 320.08 mg/l (31.96%), from 650.08 to 328.69 mg/l (49.44%), from 756.62 to 432.76 mg/l (42.80%), from 787.00 to 456.38 mg/l (42.01%) and 815.46 to 486.54 mg/l (40.34%) at the reaction time of 1 h, from 518.54 to 325.46 mg/l (37.24%), 818.53 to 364.38 mg/l (55.48%), 891.23 to 458.92 mg/l (48.51%), 895.15 to 460.15 mg/l (48.60%) and 897.69 to 525.46 mg/l (41.47%) at the reaction time of 2 h, from 598.92 to 343.53 mg/l (42.64%), from 898.46 to 443.15 mg/l (50.68%), from 920.38 to 476.62 mg/l (48.21%), 940.07 to 534.69 mg/l (43.12%) and from 963.92 to 589.69 mg/l (38.82%) at the reaction time of 3 h, from 542.00 to 336.61 mg/l (37.89%), from 814.30 to 397.76 mg/l (51.15%), from 895.53 to 414.15 mg/l (53.75%), from 898.07 to 510.07 mg/l (43.20%) and from 940.53 to 578.07 mg/l (38.54%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

Using ethanol at the 50% concentration with acetone with the sample: solvent ratio of 1:40 decreased the total phenols yield over that obtained from 212.38 to 198.92 mg/l (6.34%), from 228.84 to 208.46 mg/l (8.91%), from 321.61 to 220.83 mg/l (31.34%), from 424.30 to 240.46 mg/l (43.33%) and 430.92 to 280.15 mg/l (34.99%) at the reaction time of 1 h, from 223.54 to 223.30 mg/l (0.11%), 259.69 to 213.53 mg/l (17.78%), 407.00 to 233.53 mg/l (42.62%), 451.23 to 311.23 mg/l (31.03%) and 458.92 to 339.38 mg/l (26.05%) at the reaction time of 2 h, from 263.53 to 260.46 mg/l (1.16%), from 299.30 to 266.15 mg/l (11.08%), from 475.46 to 273.08 mg/l (42.58%), 589.30 to 317.00 mg/l (46.21%) and from 592.61 to 380.00 mg/l (35.88%) at the reaction time of 3 h, from 243.00 to 218.23 mg/l (10.19%), from 283.15 to 230.84 mg/l (18.47%), from 433.15 to 258.07 mg/l (40.42%), from 580.92 to 297.38 mg/l (48.81%) and from 538.15 to 323.15 mg/l (39.95%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

Using ethanol at the 50% concentration with acetone with the sample: solvent ratio of 1:60 decreased the total phenols yield over that obtained from 176.00 to 162.69 mg/l (7.56%), from 232.31 to 167.77 mg/l (27.78%), from 250.88 to 169.46 mg/l (32.45%), from 253.54 to 178.45 mg/l (29.62%) and from 263.92 to 180.62 mg/l (31.56%) at the reaction time of 1 h, from 198.53 to 178.92 mg/l (9.64%), from 258.61 to 191.92 mg/l (25.79%), from 287.53 to 198.38 mg/l (31.01%), from 289.61 to 201.69 mg/l (30.36%) and from 291.30 to 212.84 mg/l (26.93%) at the reaction time of 2 h, from 222.00 to 213.53 mg/l (3.82%), from 261.53 to 248.61 mg/l (4.94%), from 293.61 to 262.15 mg/l (10.71%), from 293.38 to 268.84 mg/l (8.36%) and from 296.15 to 270.84 mg/l (8.55%) at the reaction time of 3 h and from 205.15 to 173.46 mg/l (15.45%), from 240.46 to 236.53 mg/l (1.63%), from 253.00 to 248.23 mg/l (1.89%), from 254.30 to 252.84 mg/l (0.57%) and from 271.07 to 261.07 mg/l (3.69%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

Using ethanol at the 75% concentration with the sample: solvent ratio of 1:20 decreased the total phenols yield over that obtained with acetone from 675.08 to 583.23 mg/l (13.61%), from 750.85 to 588.92 mg/l (21.57%), from 891.62 to 598.92 mg/l (32.83%), from 930.31 to 629.31 mg/l (32.35%) and from 998.92 to 637.00 mg/l (36.23%) at the reaction time of 1 h, from 745.46 to 589.53 mg/l (20.92%), from 870.07 to 591.30 mg/l (32.04%), from 876.15 to 603.00 mg/l (31.18%), from 975.53 to 640.07 mg/l (34.39%) and 1032.61 to 653.07 mg/l (36.76%) at the reaction time of 2 h, from 905.46 to 610.84 mg/l (32.54%), from 922.34 to 629.69 mg/l (31.73%), 963.53 to 653.15 mg/l (32.21%), from 970.07 to 685.38 mg/l (29.35%) and from 1052.39 to 688.92 mg/l (34.54%) at the reaction time of 3 h and from 833.31 to 575.07 mg/l (30.99%), from 842.76 to 579.69 mg/l (31.22%), from 859.30 to 599.69 mg/l (30.21%), from 959.30 to 606.53 mg/l (36.77%) and from 1003.92 to 612.46 mg/l (38.99%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

Using ethanol at the 75% concentration with the sample: solvent ratio of 1:40 decreased the total phenols yield over that obtained with acetone from 407.77 to 402.77 mg/l (1.23%), from 509.31 to 454.31 mg/l (10.80%), from 525.85 to 462.77 mg/l (12%), from 619.69 to 478.08 mg/l (25.32%) and from 708.92 to 490.38 mg/l (30.83%) at the reaction time of 1 h, from 438.07 to 430.15 mg/l (1.81%), from 550.46 to 459.30 mg/l

(16.56%), from 558.61 to 467.15 mg/l (16.37%), from 680.84 to 483.00 mg/l (29.66%) and from 754.46 to 503.38 mg/l (33.28%) at the reaction time of 2 h, from 450.69 to 433.38 mg/l (3.84%), from 590.15 to 462.23 mg/l (21.68%), from 636.23 to 471.07 mg/l (25.96%), from 719.30 to 490.15 mg/l (31.86%) and from 880.00 to 512.07 mg/l (41.81%) at the reaction time of 3 h, from 420.07 to 418.30 mg/l (0.42%), from 581.23 to 431.46 mg/l (25.77%), from 595.84 to 449.46 mg/l (24.57%), from 673.84 to 451.23 mg/l (33.04%) and from 783.84 to 483.38 mg/l (38.33%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

Using ethanol at the 75% concentration with the sample: solvent ratio 1:60 decreased the total phenols yield over that obtained with acetone from 353.50 to 275.85 mg/l (8.82%), from 351.62 to 293.23 mg/l (16.61%), from 439.69 to 302.61 mg/l (31.18%), from 441.92 to 323.54 mg/l (26.76%) and from 483.53 to 338.15 mg/l (21.97%) at the reaction time of 1 h, from 386.61 to 382.76 mg/l (1%), from 395.84 to 386.30 mg/l (2.41%), from 448.53 to 391.23 mg/l (12.78%), from 453.69 to 396.53 mg/l (12.60%) and from 486.53 to 406.76 mg/l (16.40%) at the reaction time of 2 h, from 390.38 to 386.61 mg/l (0.97%), from 466.23 to 389.30 mg/l (16.50%), from 458.92 to 397.30 mg/l (13.43%), from 466.15 to 402.15 mg/l (13.73%) and from 490.30 to 410.92 mg/l (16.19%) at the reaction time of 3 h and from 290.07 to 260.69 mg/l (10.13%), from 412.38 to 264.30 mg/l (35.91%), from 445.85 to 273.23 mg/l (38.72%), from 448.61 to 275.69 mg/l (38.55%) and from 453.00 to 281.53 mg/l (37.85%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

5.1.3. Total Flavonoids

The flavonoids results are shown in Tables 5.15-5.19. Analysis of the variance (ANOVA) was performed on the total flavonoids data using Minitab Software (Minitab® 17.1.0., Minitab Inc., Canada).The results are shown in Table 5.20. The results obtained from Tukey's grouping are shown in Table 5.21.

The main effects of reaction time (Ti), sample: solvents ratio (R), reaction temperature (Te) and solvent concentration (C) on the total flavonoids yield were significant at the 0.001 level. The solvent type (s) was not significant at the 0.001 level. The two way interactions between reaction temperature and sample: solvent ratio, between the reaction temperature and solvent concentration, between sample: solvent ratio and

Table 5.15. Average flavonoids yield from ajwa date fruit using water at different sample: solvent ratio, reaction temperature and reaction time.

Sample: Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Flavonoids Yield* (mg/l)
1:20	25	1	22.68 ± 4.53
		2	24.68 ± 3.53
		3	28.27 ± 0.88
		4	27.33 ± 1.22
	35	1	26.68 ± 1.32
		2	29.50 ± 3.53
		3	31.37 ± 3.53
		4	30.35 ± 0.88
	45	1	33.43 ± 4.86
		2	38.12 ± 3.53
		3	40.87 ± 0.88
		4	32.21 ± 0.88
	55	1	40.12 ± 3.53
		2	43.25 ± 0.88
		3	45.12 ± 0.88
		4	43.56 ± 0.44
	65	1	44.56 ± 0.44
		2	46.12 ± 3.53
		3	50.32 ± 1.76
		4	46.87 ± 11.49
1:40	25	1	10.43 ± 1.76
		2	11.96 ± 1.32
		3	13.31 ± 0.44
		4	11.01 ± 0.44
	35	1	12.55 ± 0.88
		2	24.18 ± 1.32
		3	28.25 ± 1.76
		4	22.31 ± 3.09
	45	1	20.75 ± 1.76
		2	25.82 ± 3.09
		3	29.95 ± 0.88
		4	23.22 ± 0.44
	55	1	22.31 ± 1.76
		2	27.92 ± 1.32
		3	31.32 ± 2.20
		4	25.34 ± 0.44
	65	1	23.98 ± 3.97
		2	29.21 ± 0.88
		3	33.12 ± 1.32
		4	26.32 ± 0.44
1:60	25	1	8.25 ± 0.88
		2	9.56 ± 0.44
		3	10.02 ± 1.32
		4	8.31 ± 0.44
	35	1	9.83 ± 0.88
		2	10.22 ± 1.32
		3	11.32 ± 0.44
		4	9.03 ± 0.44
	45	1	11.31± 0.44
		2	14.81 ± 0.88
		3	17.00 ± 0.88
		4	10.21 ± 2.20
	55	1	12.62 ± 0.88
		2	16.06 ± 0.44
		3	17.93 ± 0.88
		4	12.33 ± 1.32
	65	1	19.68 ± 2.65
		2	20.75 ± 1.32
		3	21.06 ± 3.09
		4	14.32 ± 3.97

*average of two replicates

Table 5.16. Average flavonoids yield from ajwa date fruit using 50% acetone at different sample: solvent ratio, reaction temperature and reaction time.

Sample: Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Flavonoids Yield* (mg/l)
1:20	25	1	44.18 ± 4.86
		2	57.31 ± 2.20
		3	76.68 ± 6.18
		4	73.87 ± 11.04
	35	1	56.06 ± 11.93
		2	67.00 ± 14.14
		3	87.00 ± 2.65
		4	77.62 ± 15.90
	45	1	66.18 ± 30.49
		2	72.31 ± 27.40
		3	92.12 ± 31.81
		4	81.30 ± 6.62
	55	1	68.36 ± 2.65
		2	74.50 ± 5.30
		3	95.50 ± 7.51
		4	87.93 ± 6.62
	65	1	72.25 ± 6.62
		2	80.43 ± 13.25
		3	98.68 ± 7.51
		4	93.25 ± 3.53
1:40	25	1	21.68 ± 6.62
		2	26.06 ± 0.44
		3	31.43 ± 1.32
		4	30.06 ± 3.09
	35	1	33.56 ± 3.09
		2	30.68 ± 3.09
		3	52.31 ± 2.20
		4	50.62 ± 7.07
	45	1	37.31 ± 5.74
		2	42.62 ± 8.39
		3	54.70 ± 0.88
		4	53.01 ± 2.65
	55	1	52.31 ± 1.76
		2	58.25 ± 3.97
		3	69.81 ± 3.09
		4	56.06 ± 6.62
	65	1	65.81 ± 8.39
		2	69.18 ± 9.28
		3	71.06 ± 5.74
		4	63.31 ± 30.49
1:60	25	1	21.00 ± 1.32
		2	23.25 ± 4.41
		3	24.56 ± 1.32
		4	27.62 ± 0.88
	35	1	25.12 ± 1.76
		2	27.93 ± 8.83
		3	30.12 ± 1.32
		4	28.31 ± 1.76
	45	1	32.93 ± 0.44
		2	37.00 ± 0.88
		3	44.50 ± 0.88
		4	32.06 ± 0.44
	55	1	33.50 ± 1.76
		2	38.93 ± 1.76
		3	45.60 ± 3.97
		4	35.81 ± 0.44
	65	1	35.87 ± 1.76
		2	49.50 ± 6.18
		3	52.37 ± 0.44
		4	45.30 ± 0.88

*average of two replicates

Table 5.17. Average flavonoids yield from ajwa date fruit using 50% ethanol at different sample: solvent ratio, reaction temperature and reaction time.

Sample: Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Flavonoids Yield* (mg/l)
1:20	25	1	29.18 ± 3.09
		2	34.18 ± 2.20
		3	37.93 ± 4.86
		4	31.68 ± 3.97
	35	1	39.81 ± 4.86
		2	45.12 ± 4.41
		3	46.37 ± 3.53
		4	44.50 ± 12.37
	45	1	44.18 ± 6.18
		2	52.00 ± 6.62
		3	53.25 ± 10.60
		4	50.31 ± 4.41
	55	1	56.37 ± 1.32
		2	60.43 ± 5.74
		3	73.56 ± 1.32
		4	56.87 ± 7.95
	65	1	63.56 ± 4.86
		2	73.50 ± 6.18
		3	83.87 ± 2.65
		4	63.37 ± 28.28
1:40	25	1	17.31 ± 7.07
		2	20.87 ± 3.09
		3	23.21 ± 4.41
		4	19.33 ± 3.53
	35	1	20.43 ± 1.32
		2	29.18 ± 0.44
		3	31.37 ± 0.88
		4	24.18 ± 3.09
	45	1	29.50 ± 0.44
		2	31.99 ± 0.44
		3	33.68 ± 0.44
		4	26.31 ± 0.88
	55	1	36.87 ± 4.41
		2	38.63 ± 2.20
		3	46.68 ± 7.51
		4	30.81 ± 5.74
	65	1	40.33 ± 9.72
		2	43.12 ± 16.35
		3	49.36 ± 33.58
		4	33.06 ± 20.77
1:60	25	1	17.33 ± 2.20
		2	19.32 ± 1.76
		3	21.03 ± 2.65
		4	18.38 ± 3.09
	35	1	19.06 ± 5.74
		2	21.23 ± 7.07
		3	23.12 ± 3.97
		4	20.03 ± 1.30
	45	1	21.92 ± 7.50
		2	25.15 ± 11.04
		3	29.06 ± 16.79
		4	22.19 ± 14.58
	55	1	24.82 ± 3.97
		2	28.30 ± 19.44
		3	32.98 ± 17.23
		4	25.68 ± 26.07
	65	1	28.30 ± 18.11
		2	34.63 ± 108.11
		3	40.12 ± 26.51
		4	29.31 ± 22.53

*average of two replicates

Table 5.18. Average flavonoids yield from ajwa date fruit using 75% acetone at different sample: solvent ratio, reaction temperature and reaction time.

Sample: Solvent Ratio	Reaction Temperature (°C)	Reaction Time (h)	Total Flavonoids Yield* (mg/l)
1:20	25	1	52.00 ± 0.88
		2	66.37 ± 3.53
		3	79.18 ± 6.62
		4	75.18 ± 4.86
	35	1	73.87 ± 15.02
		2	75.12 ± 14.14
		3	92.93 ± 16.35
		4	83.25 ± 5.30
	45	1	78.25 ± 0.88
		2	79.81 ± 2.20
		3	103.25 ± 2.65
		4	96.37 ± 8.83
	55	1	118.56 ± 3.97
		2	161.06 ± 8.39
		3	169.18 ± 4.86
		4	159.81 ± 9.28
	65	1	120.43 ± 59.66
		2	168.25 ± 14.14
		3	173.93 ± 12.81
		4	163.43 ± 13.25
1:40	25	1	42.00 ± 2.65
		2	48.87 ± 5.30
		3	49.50 ± 3.53
		4	45.75 ± 0.88
	35	1	45.75 ± 6.18
		2	52.62 ± 2.65
		3	55.43 ± 2.65
		4	51.25 ± 1.32
	45	1	48.81 ± 7.51
		2	57.62 ± 0.88
		3	60.43 ± 5.30
		4	54.25 ± 2.20
	55	1	57.56 ± 5.74
		2	60.43 ± 2.20
		3	80.12 ± 4.86
		4	59.93 ± 23.86
	65	1	105.43 ± 5.30
		2	110.75 ± 6.62
		3	115.75 ± 23.86
		4	106.32 ± 35.79
1:60	25	1	34.68 ± 2.20
		2	39.75 ± 12.37
		3	41.93 ± 0.44
		4	37.21 ± 2.20
	35	1	35.12 ± 5.30
		2	40.23 ± 13.25
		3	42.93 ± 2.20
		4	39.62 ± 7.07
	45	1	39.50 ± 5.30
		2	49.81 ± 5.74
		3	61.06 ± 3.977
		4	57.31 ± 1.32
	55	1	44.87 ± 4.41
		2	51.25 ± 4.41
		3	73.56 ± 4.86
		4	58.23 ± 6.62
	65	1	49.18 ± 3.09
		2	53.37 ± 3.53
		3	77.12 ± 5.30
		4	60.33 ± 7.95

*average of two replicates

Table 5.19. Average flavonoids yield from ajwa date fruit using 75% ethanol at different sample: solvent ratio, reaction temperature and reaction time.

Sample: Solvent Ratio	Reaction Temperature (ºC)	Reaction Time (h)	Total Flavonoids Yield* (mg/l)
1:20	25	1	38.12 ± 3.09
		2	40.06 ± 2.20
		3	44.21 ± 4.86
		4	39.40 ± 3.97
	35	1	42.03 ± 4.86
		2	48.38 ± 4.41
		3	56.12 ± 3.53
		4	49.78 ± 12.37
	45	1	46.73 ± 6.18
		2	53.21 ± 6.62
		3	68.90 ± 10.60
		4	54.38 ± 4.41
	55	1	56.12 ± 1.32
		2	62.33 ± 5.74
		3	78.02 ± 1.32
		4	59.18 ± 7.95
	65	1	65.08 ± 4.86
		2	75.21 ± 6.18
		3	98.12 ± 2.65
		4	72.33 ± 28.28
1:40	25	1	28.68 ± 7.07
		2	38.12 ± 3.09
		3	39.50 ± 4.41
		4	32.78 ± 3.53
	35	1	30.12 ± 1.32
		2	43.28 ± 0.44
		3	45.66 ± 0.88
		4	38.12 ± 3.09
	45	1	38.73 ± 0.44
		2	49.23 ± 0.44
		3	52.31 ± 0.44
		4	41.06 ± 0.88
	55	1	42.12 ± 4.41
		2	53.36 ± 2.20
		3	59.22 ± 7.51
		4	49.31 ± 5.74
	65	1	53.86 ± 9.72
		2	58.21 ± 16.35
		3	60.33 ± 33.58
		4	53.38 ± 20.77
1:60	25	1	22.31 ± 2.20
		2	26.81 ± 1.76
		3	29.30 ± 2.65
		4	24.12 ± 3.09
	35	1	26.38 ± 5.74
		2	31.68 ± 7.07
		3	36.92 ± 3.977
		4	26.68 ± 1.32
	45	1	32.14 ± 3.977
		2	40.92 ± 11.04
		3	48.31 ± 16.79
		4	30.68 ± 14.58
	55	1	38.12 ± 3.97
		2	46.12 ± 19.44
		3	52.13 ± 17.23
		4	36.38 ± 26.07
	65	1	42.21 ± 18.11
		2	48.36 ± 18.11
		3	56.06 ± 26.51
		4	40.98 ± 22.53

*average of two replicates

Table 5.20. Analysis of the variance for total flavonoids capacity.

Source	DF	SS	MS	F	P
Total	719	1138099			
Model					
Ti	3	22190	7397	12.08	0.001
R	2	141331	7066	115.36	0.001
Te	4	63850	15962	26.06	0.001
S	1	161	161	0.26	0.609
C	2	284137	142069	231.93	0.001
Ti*R	6	1272	212	0.35	0.912
Ti*Te	12	3712	309	0.50	0.911
Ti*S	3	265	88	0.14	0.933
Ti*C	6	8727	1455	2.37	0.029
Te*R	8	43738	5467	8.93	0.001
Te*S	4	2033	508	0.83	0.507
Te*C	8	90435	11304	18.45	0.001
R*S	2	16887	8443	13.78	0.001
R*C	4	10434	2609	4.26	0.001
S*C	2	604	302	0.49	0.611
Ti*Te*R	24	6003	250	0.41	0.995
Ti*R*S	6	479	80	0.13	0.992
Ti*R*C	12	1355	113	0.18	0.999
Ti*Te*S	12	2485	207	0.34	0.982
Ti*S*C	6	454	76	0.12	0.993
Te*R*S	8	13006	1626	2.65	0.008
Te*R*C	16	88343	5521	9.01	0.001
R*C*S	4	11103	2776	4.53	0.001
Te*S*C	8	15439	1930	3.15	0.002
Ti*Te*C	24	11048	460	0.75	0.797
Ti*Te*R*S	24	4177	174	0.28	1.001
Ti*Te*R*C	48	9265	193	0.32	1.001
Ti*Te*S*C	24	4829	201	0.33	0.999
Ti*R*S*C	12	830	69	0.11	0.001
Te*R*S*C	16	49639	3102	5.06	0.001
Ti*Te*R*S*C	48	9353	195	0.32	0.001
Error	360	220514	613		

Reaction Time (Ti)
Reaction Temperature (Te)
Solvent: Sample Ratio (R)
Solvent Type (S)
Solvent Concentration (C)
R^2= 80.62%

Table 5.21. Tukey's grouping of total flavonoids yield.

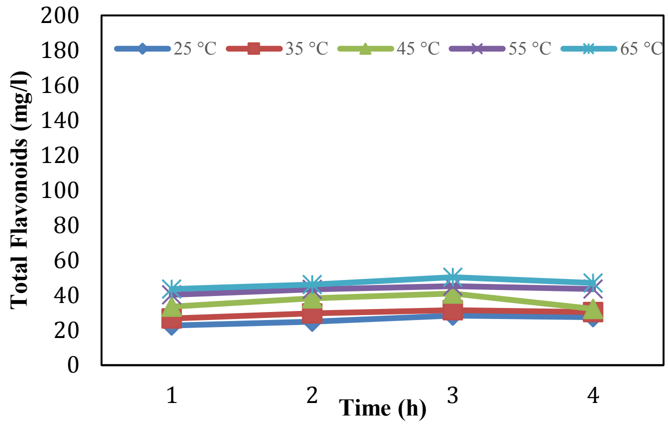
Factors	level	N	Mean	Tukey's Grouping
Sample: Solvent Ratio	20	240	72.369.	A
	40	240	47.497	B
	60	240	39.455	C
Reaction Time	1	180	45.149	A
	2	180	51.378	A B
	3	180	60.191	B C
	4	180	55.711	C
Reaction Temperature	25˚C	144	48.896	A
	35˚C	144	41.600	A
	45˚C	144	47.816	A
	55˚C	144	58.892	B
	65˚C	144	68.332	C
Solvent Concentration	0%	240	29.010	A
	50%	240	52.648	B
	75%	240	77.664	C
Solvent Type	Acetone	360	53.579	A
	Ethanol	360	52.635	A

Groups with the same letter are not significantly different from each other at the alpha significance level of 0.05.

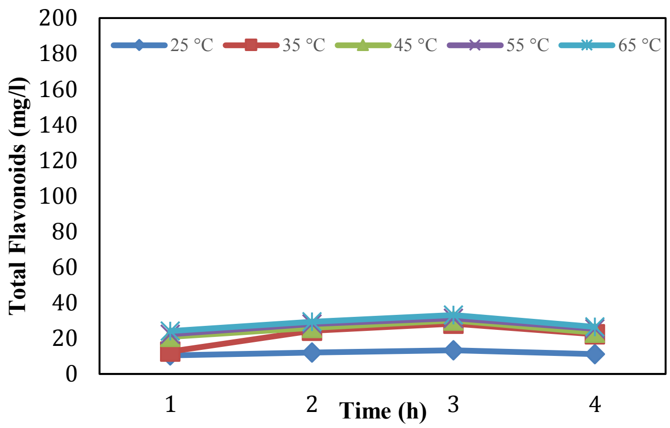
solvent type and between sample: solvent ratio and solvent concentration were significant at the 0.001 level. The three-way interactions between the reaction temperature and sample: solvent ratio and solvent concentration and between the sample: solvent ratio, solvent concentration and solvent type were significant at the 0.001 level. The four-way interactions between the reaction time, reaction temperature, sample: solvent ratio and solvent type, between the reaction temperature, sample: solvent ratio and solvent concentration, between the reaction time, sample: solvent ratio, solvent type and solvent concentration and between the reaction temperature, sample: solvent ratio, solvent type and solvent concentration were significant at the 0.001 level. The five-way interactions were significant at the 0.001 level.

All sample: solvent ratios (1:20, 1:40 and 1:60) were significantly different from one another at the 0.05 level. The highest average yield of total flavonoids (72.36 mg/l) was obtained at the sample: solvent ratio of 1:20. The reaction times 1 and 2, 2 h and 3 and 4 h and 3 and 4 h were not significantly different from each another at the 0.05 level. The highest average yield of total flavonoids (60.19 mg/l) was obtained after 4 hours. The reaction temperatures (25, 35 and 45˚C) were not significantly different from one another but were significantly different from the reaction temperature 55 and 65 ˚C at the 0.05 level. The highest average yield for total flavonoids (68.33 mg/l) was obtained a temperature of 65 ˚C. The solvent concentrations (0, 50 and 75%) were significantly different from one another at the 0.05 level. The highest average yield of total flavonoids (77.66 mg/l) was obtained at a concentration of 75%. The solvent types (acetone and ethanol) were not significantly different from one another at the 0.05 level. The highest average yield of total flavonoids (53.57 mg/l) was obtained with acetone.

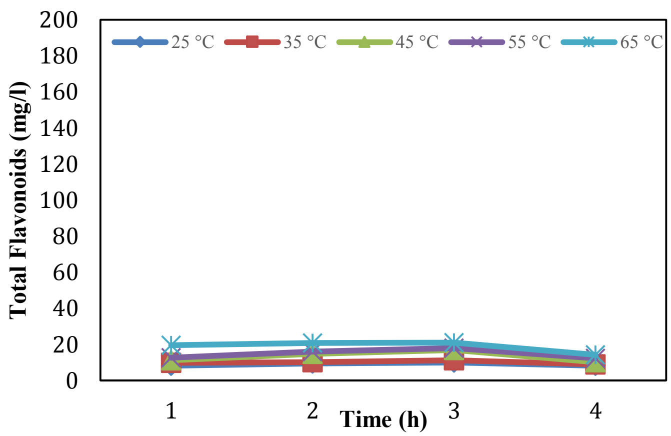
5.1.3.1. Effect of Reaction Time: Figures 5.44-5.48 show the effect of reaction time on the total flavonoids yield from ajwa date fruit at different sample: solvent ratios (1:20, 1:40 and 1:60) and reaction temperatures (25, 35, 45, 55 and 65 ^{o}C) using acetone and ethanol at different concentrations (0, 50 and 75%). Generally, there was an increase in the total flavonoids yield when the reaction time was increased from 1 to 3 h at all reaction temperatures, sample: solvent ratios and solvent concentration for both solvents. This was followed by a decrease in the flavonoids yield with a further increase in reaction time from 3 to 4 h.



(a) Sample: solvent ratio 1:20

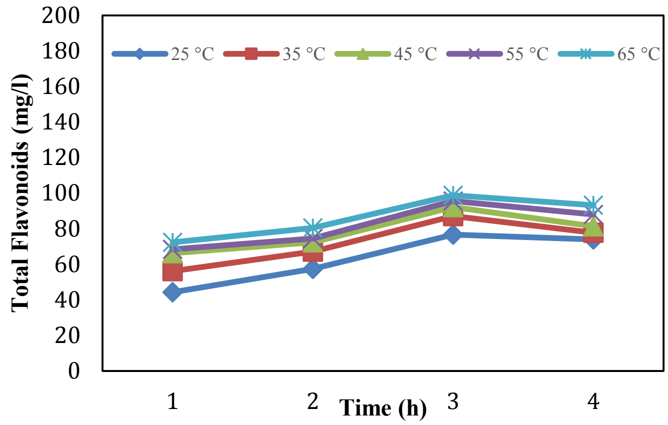


(b) Sample: solvent ratio 1:40

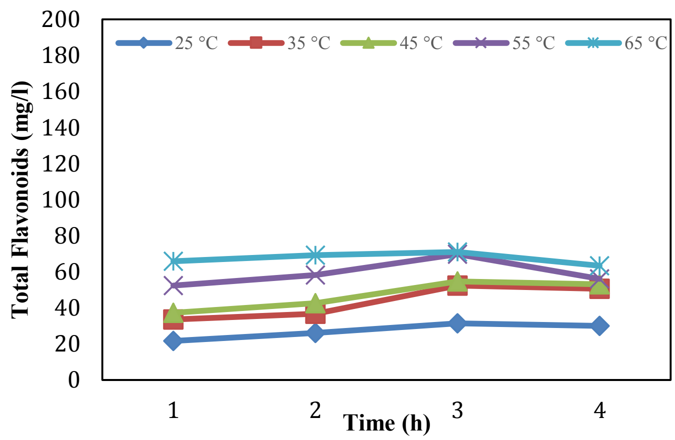


(c) Sample: solvent ratio 1:60

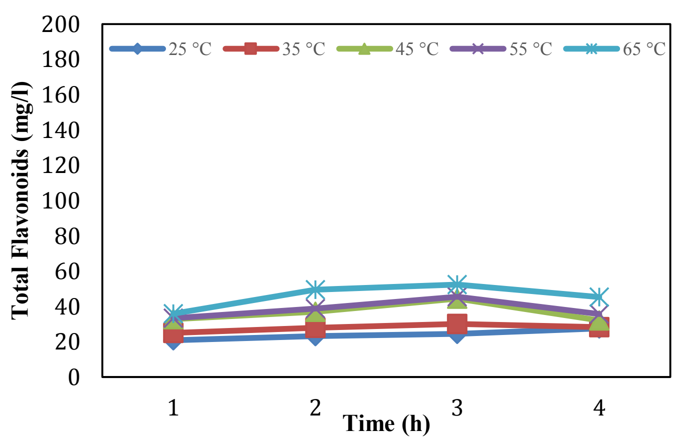
Figure 5.45. The effect of reaction time on the total flavonoids yield from ajwa date fruit using water at different temperatures.



(a) Sample: solvent ratio 1:20

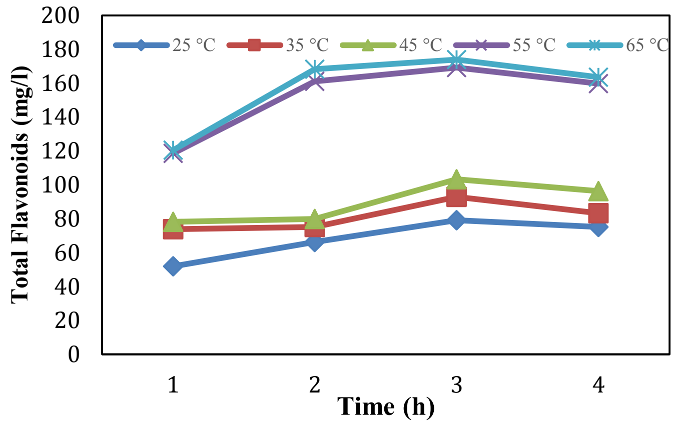


(b) Sample: solvent ratio 1:40

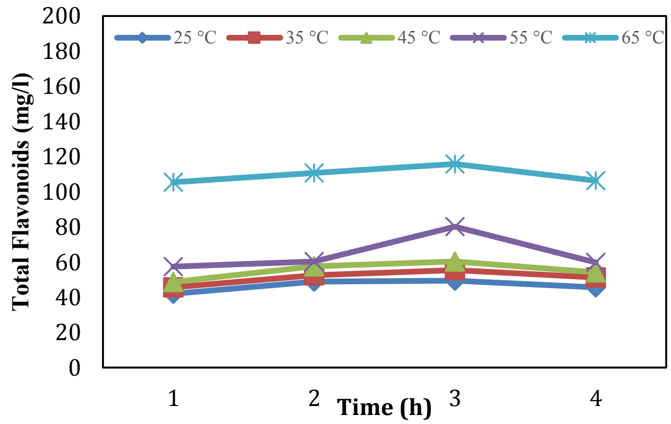


(c) Sample: solvent ratio 1:20

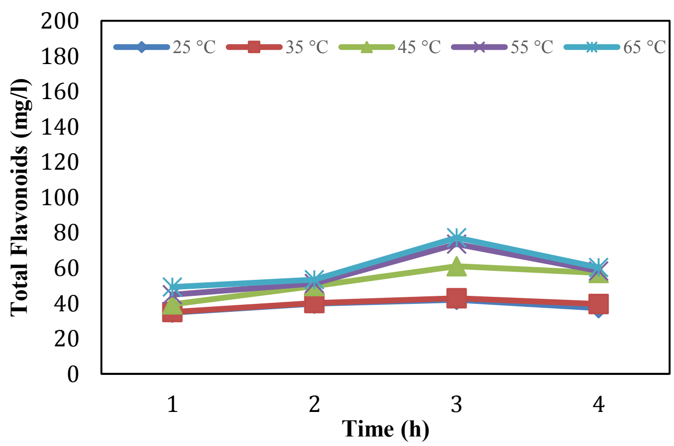
Figure 5.46. The effect of reaction time on the total flavonoids yield from ajwa date fruit using 50% acetone at different temperatures.



(a) Sample: solvent ratio 1:20

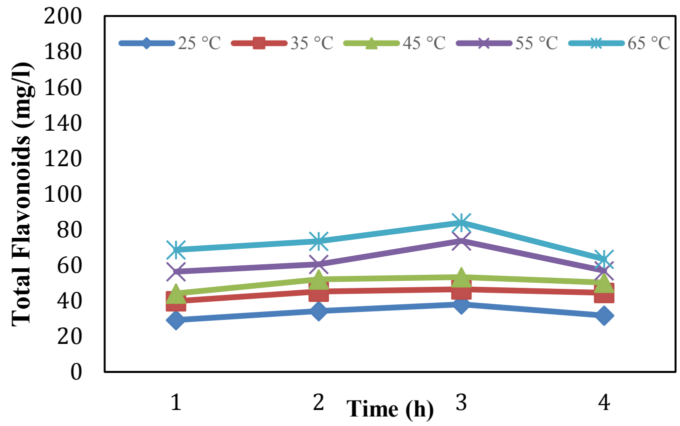


(a) Sample: solvent ratio 1:40

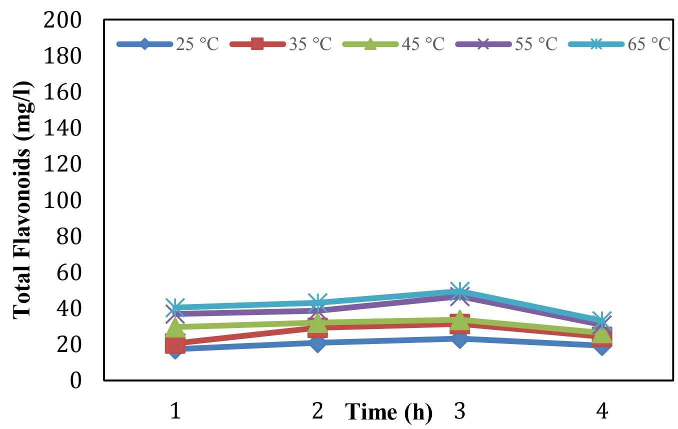


(c) Sample: solvent ratio 1:60

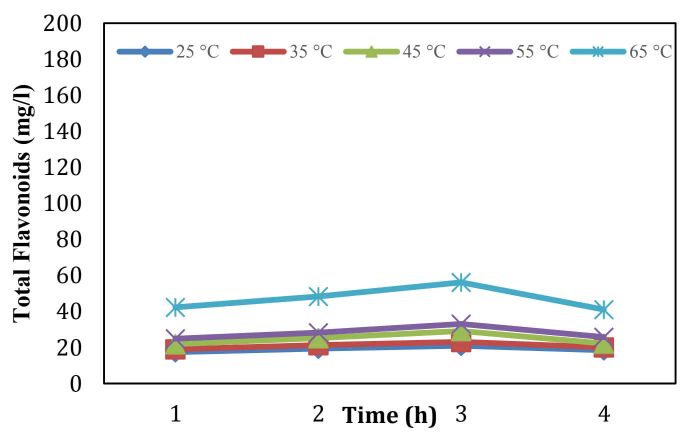
Figure 5.47. The effect of reaction time on the total flavonoids yield from ajwa date fruit using 75% acetone at different temperatures.



(b) Sample: solvent ratio 1:20

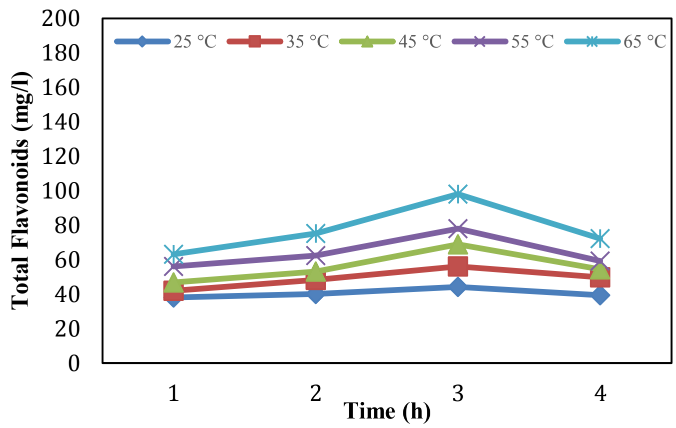


(b) Sample: solvent ratio 1:40

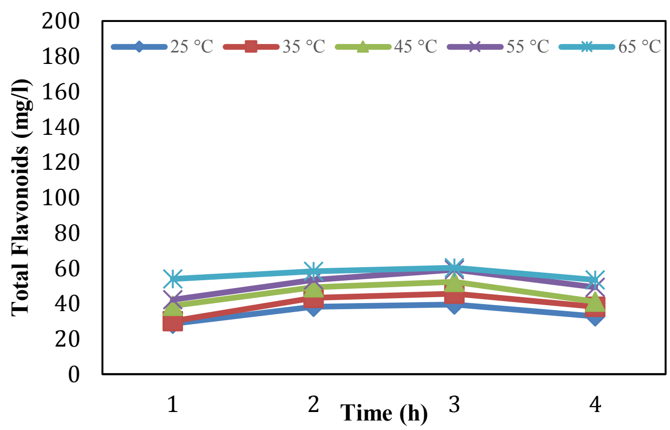


(c) Sample: solvent ratio 1:20

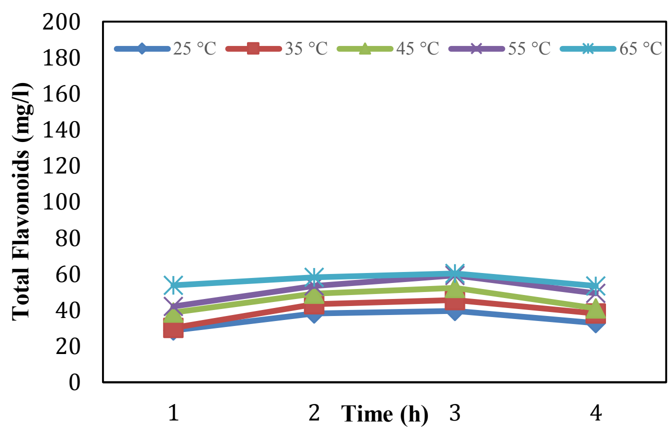
Figure 5.48. The effect of reaction time on the total flavonoids yield from ajwa date fruit using 50% ethanol at different temperatures.



(a) Sample: solvent ratio 1:20



(b) Sample: solvent ratio 1:40



(d) Sample: solvent ratio 1:60

Figure 5.49. The effect of reaction time on the total flavonoids yield from ajwa date fruit using 75% ethanol at different temperatures.

When water was used as a solvent, increasing the reaction time from 1 to 3 h, increased the flavonoids yield from 22.68 to 28.27 mg/l (24.65%), from 26.68 to 31.37 mg/l (17.58%), from 33.43 to 40.87 mg/l (22.26%), from 40.12 to 45.12 mg/l (5%) and from 44.56 to 50.32mg/l (12.93%) at the sample: solvent ratio of 1:20, from 10.43 to 13.31 mg/l (25.70%), from 12.55 to 28.25 mg/l (125.10%), from 20.75 to 29.95 mg/l (44.34%), from 22.31 to 31.32 mg/l (40.39%) and from 23.98 to 33.12 mg/l (38.12%) at the sample: solvent ratio of :40 and from 8.25 to 10.02 mg/l (21.45%), from 9.83 to 11.32 mg/l (15.16%), from 11.31 to 17.00 mg/l (50.31%), from 12.62 to 17.93 mg/l (42.08%) and from 19.68 to 21.06 mg/l (7.01%) at the sample: solve ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively. A further increase the reaction time from 3 to 4 h decreased the flavonoids yield from 28.27 to 27.33 mg/l (3.33%), from 31.37 to 30.35 mg/l (3.25%), from 40.87 to 32.21 mg/l (21.19%), from 45.12 to 43.56 mg/l (3.46%) and from 50.32 to 46.87 mg/l (6.86%) at the sample: solvent ratio of 1:20, from 13.31 to 11.01 mg/l (16.02%), from 28.25 to 22.31 mg/l (21.03%), from 29.95 to 23.22 mg/l (22.47%), from 31.32 to 25.34 mg/l (19.09%) and from 33.12 to 26.32 mg/l (20.53%) at the sample: solvent ratio of 1:40 and from 10.02 to 8.31 mg/l (17.07%), from 11.32 to 9.03 mg/l (20.23%), from 17.00 to 10.21 mg/l (39.94%), from 17.93 to 12.33 mg/l (31.23%) and from 21.06 to 14.32 mg/l (32%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

when acetone was used as solvent at the 50% concentration, increasing the reaction time from 1 to 3 h, increased the flavonoids yield from 44.18 to 76.68 mg/l (73.56%), from 56.06 to 87.00 mg/l (55.19%), from 66.18 to 92.12 mg/l (39.20%), from 68.36 to 95.50 mg/l (39.70%) and from 72.25 to 98.68 (36.58%) at the sample: solvent ratio of 1:20, from 21.68 to 31.43 mg/l (44.97%), from 33.56 to 52.31 mg/l (55.87%), from 37.31 to 54.70 mg/l (46.61%), from 52.31 to 69.81 mg/l (33.45%) and from 65.81 to 71.06 mg/l (7.98%) at the sample: solvent ratio of 1:40 and from 21.00 to 24.56 mg/l (16.95%), from 25.12 to 30.12 mg/l (19.90%), from 32.93 to 44.50 mg/l (35.14%), from 33.50 to 45.60 mg/l (36.12%) and from 35.87 to 52.37 mg/l (46%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively. A further increase in reaction time from 3 to 4 h decreased the flavonoids yield from 76.68 to 73.87 mg/l (3.66%), from 87.00 to 77.62 mg/l (10.78%), from 92.12 to 81.30 mg/l (11.75%), from

95.50 to 87.93 mg/l (7.93%) and from 98.68 to 93.25 mg/l (5.5%) at the sample: solvent ratio of 1:20, from 31.43 to 30.06 mg/l (4.36%), from 52.31 to 50.62 mg/l (3.23%), from 54.70 to 53.01 mg/l (3.09%), from 69.81 to 56.06 mg/l (19.70%) and from 71.06 to 63.31 mg/l (10.91%) at the sample: solvent ratio of 1:40 and from 24.56 to 27.62 mg/l (11.08%), from 30.12 to 28.31 mg/l (6.01%), from 44.50 to 32.06 mg/l (27.96%), from 45.60 to 35.81 mg/l (21.47%) and from 52.37 to 45.30 mg/l (13.50%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

When acetone was used as solvent at the 75% concentration, increasing the reaction time from 1 to 3 h, increased the flavonoids yield from 52.00 to 79.18 mg/l (52.27%), from 73.87 to 92.93 mg/l (25.80%), from 78.25 to 103.25 mg/l (31.95%), from 118.56 to 169.18 mg/l (42.70%) and from 120.43 to 173.93 mg/l (44.42%) at the sample: solvent ratio of 1:20, from 42.00 to 49.50 mg/l (17.86%), from 45.75 to 55.43 mg/l (21.16%), from 48.81 to 60.43 mg/l (23.81%), from 57.56 to 80.12 mg/l (39.19%) and from 105.43 to 115.75 mg/l (9.79%) at the sample: solvent ratio of 1:40 and from 34.68 to 41.93 mg/l (20.91%), from 35.12 to 42.93 mg/l (22.24%), from 39.50 to 61.06 mg/l (54.58%), from 44.87 to 73.56 mg/l (63.94%) and from 49.18 to 77.12 mg/l (56.81%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively. A further increase in reaction time from 3 to 4 h decreased the flavonoids yield from 79.18 to 75.18 mg/l (5.05%), from 92.93 to 83.25 mg/l (10.42%), from 103.25 to 96.37 mg/l (6.66%), from 169.18 to 159.81 mg/l (5.54%) and from 173.93 to 163.43 mg/l (6.04%) at the sample: solvent ratio of 1:20, from 49.50 to 45.75 mg/l (7.58%), from 55.43 to 51.25 mg/l (7.54%), from 60.43 to 54.25 mg/l (10.23%), from 80.12 to 59.93 mg/l (25.20%) and from 115.75 to 106.32 mg/l (8.15%) at the sample: solvent ratio of 1:40 and from 41.93 to 37.21 mg/l (11.26%), from 42.93 to 39.62 mg/l (7.71%), from 61.06 to 57.31 mg/l (6.14%), from 73.56 to 58.23 mg/l (20.84%) and from 77.12 to 60.33 mg/l (21.77%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

When ethanol was used as a solvent at the50% concentration, increasing in the reaction time from 1 to 3 h, increased the flavonoids yield from 29.18 to 37.93 mg/l (29.99%), from 39.81 to 46.37 mg/l (16.48%), from 44.18 to 53.25 mg/l (20.53%), from 73.56 to 56.87 mg/l (30.49%) and from 68.56 to 83.87 mg/l (22.33%) at the sample: solvent ratio of 1:20, from 17.31 to 23.21 mg/l (34.08%), from 20.43 to 31.37 mg/l (53.55%), from

29.50 to 33.68 mg/l (14.17%), from 36.87 to 46.68 mg/l (26.61%) and from 40.33 to 49.36 mg/l (22.39%) at the sample: solvent ratio of 1:40 and from 17.33 to 21.03 mg/l (21.35%), from 19.06 to 23.12 mg/l (21.30%), from 21.92 to 29.06 mg/l (32.57%), from 24.82 to 32.98 mg/l (32.88%) and from 28.30 to 40.12 mg/l (41.77%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively. A further increase in the reaction time from 3 to 4 h decreased the flavonoids yield from 37.93 to 31.68 mg/l (16.48%), from 46.37 to 44.50 mg/l (4.03%), from 53.25 to 50.31 mg/l (5.52%), from 73.56 to 56.87 mg/l (22.69%) and from 83.87 to 63.37 mg/l (24.44%) at the sample: solvent ratio of 1:20, from 23.21 to 19.33 mg/l (16.72%), from 31.37 to 24.18 mg/l (22.92%), from 33.68 to 26.31 mg/l (10.81%), from 46.68 to 30.81 mg/l (16.44%) and from 49.36 to 33.06 mg/l (18.03%) at the sample: solvent ratio of 1:40 and from 21.03 to 18.38 mg/l (12.60%), from 23.12 to 20.03 mg/l (13.37%), from 29.06 to 22.19 mg/l (23.64%), from 32.98 to 25.68 mg/l (22.13%) and from 40.12 to 29.31 mg/l (26.94%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

When ethanol was used as a solvent at the 75% concentration, increasing the reaction time from 1 to 3 h, increased the flavonoids yield from 38.12 to 44.21 mg/l (15.98%), from 42.03 to 56.12 mg/l (33.52%), from 46.73 to 68.90 mg/l (47.44%), from 56.12 to 78.02 mg/l (39.02%) and from 63.08 to 98.12 mg/l (55.55%) at the sample: solvent ratio of 1:20, from 28.68 to 39.50 mg/l (37.73%), from 30.12 to 45.66 mg/l (51.59%), from 38.73 to 52.31 mg/l (35.06%), from 42.12 to 59.22 mg/l (40.60%) and from 53.86 to 60.33 mg/l (12.01%) at the sample: solvent ratio of 1:40 and from 22.31 to 29.30 mg/l (31.33%), from 26.38 to 36.92 mg/l (39.95%), from 32.14 to 48.31 mg/l (50.31%), from 38.12 to 52.13 mg/l (36.75%) and from 42.21 to 56.06 mg/l (32.81%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively. A further increase in the reaction time from 3 to 4 h decreased the flavonoids yield from 44.21 to 39.40 mg/l (10.88%), from 56.12 to 49.78 mg/l (11.30%), from 68.90 to 54.38 mg/l (21.07%), from 78.02 to 59.18 mg/l (24.15%) and from 98.12 to 72.33 mg/l (26.28%) at the sample: solvent ratio of 1:20, from 39.50 to 32.78 mg/l (17.01%), from 45.66 to 38.12 mg/l (16.51%), from 52.31 to 41.06 mg/l (21.51%), from 59.22 to 49.31 mg/l (16.73%) and from 60.33 to 53.38 mg/l (11.52%) at the sample: solvent ratio of 1:40 and from 29.30 to 24.12 mg/l (17.68%), from 36.92 to 26.68 mg/l (27.74%), from 48.31 to 30.68 mg/l

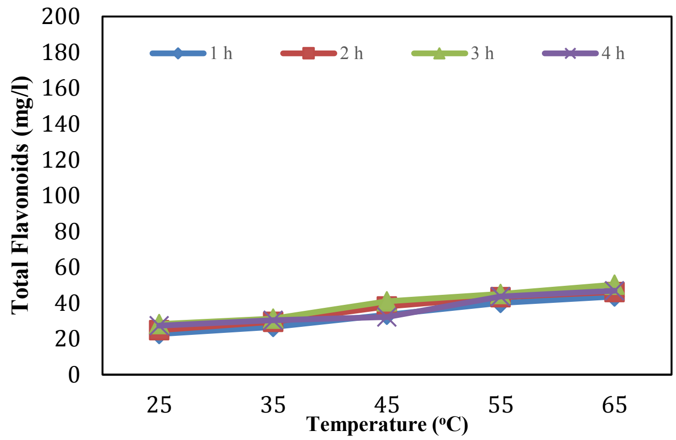
(36.49%), from 52.13 to 36.38 mg/l (30.21%) and from 56.06 to 40.98 mg/l (26.90%) at the sample: solvent ratio of 1:60 for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C respectively.

5.1.3.2. Effect of Reaction Temperature: Figures 5.49 -5.53 show the effect of reaction temperature on the total flavonoids yield from ajwa date fruit at different reaction times (1, 2, 3 and 4 h) and sample: solvent ratios (1:20, 1:40 and 1:60) using acetone and ethanol at different concentrations (0, 50 and 75%). Generally, there was an increase in the total flavonoids yield when the reaction temperature was increased from 25 to 65^{o}C at all sample: solvent ratio at all sample: solvent ratios and solvent concentrations for both solvents.

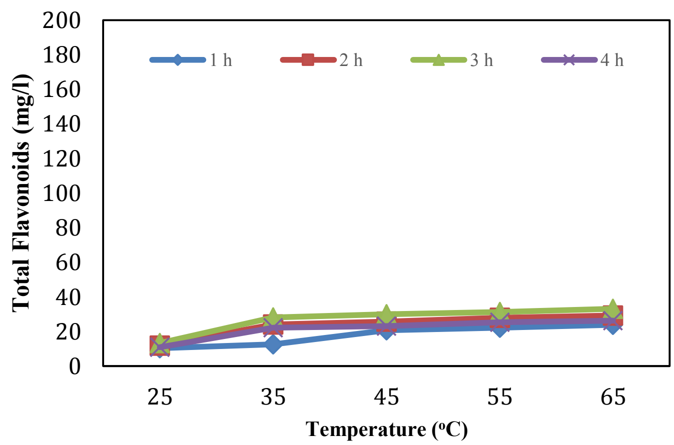
When water was used as a solvent, increasing the reaction temperature from 25 to 65^{o}C, increased the flavonoids yield from 22.68 to 44.56 mg/l (96.47%), from 24.68 to 46.12 mg/l (86.87%), from 28.27 to 50.32 mg/l (78%) and from 27.33 to 46.87 mg/l (71.50%) at the sample: solvent ratio of 1:20, from 10.43 to 23.98 mg/l (129.91%), from 11.96 to 29.21 mg/l (144.23%), from 13.31 to 33.12 mg/l (148.84%) and from 11.01 to 26.32 mg/l (139.06%) at the sample: solvent ratio of 1:40 and from 8.25 to 19.68 mg/l (138.55%), from 9.56 to 20.75 mg/l (117.05%), from 10.02 to 21.06 mg/l (110.18%) and from 8.31 to 14.32 mg/l (72.32%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

When acetone was used as solvent at the 50 % concentration, increasing the reaction temperature from 25 to 65^{o}C increased the flavonoids yield from 44.18 to 72.25 mg/l (63.54%), from 57.31 to 80.43 mg/l (40.34%), from 76.68 to 98.68 mg/l (28.69%) and from 73.87 to 93.25 mg/l (26.24%) at the sample: solvent ratio of 1:20, from 21.68 to 65.81 mg/l (203.55%), from 26.06 to 69.18 mg/l (165.46%), from 31.43 to 71.06 mg/l (126.09%) and from 30.06 to 63.31 mg/l (110.61%) at the sample: solvent ratio of 1:40 and from 21.00 to 35.87 mg/l (70.81%), from 23.25 to 49.50 mg/l (112.90%), from 24.56 to 52.37 mg/l (113.23%) and from 27.62 to 45.30 mg/l (64.01%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

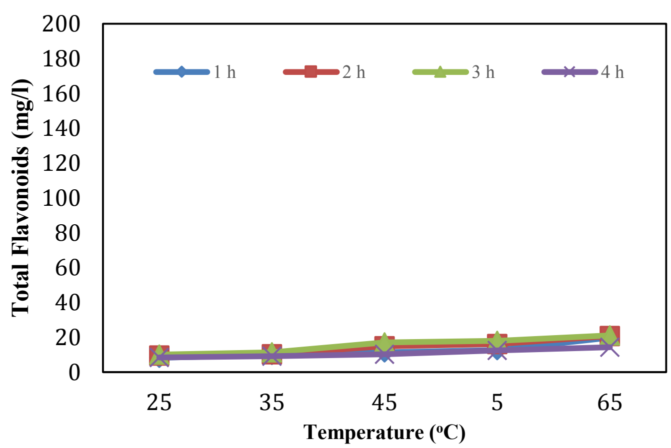
When acetone was used as solvent at the 75 % concentration, increasing the reaction temperature from 25 to 65^{o}C increased the flavonoids yield from 52.00 to 120.43 mg/l (131.60%), from 66.37 to 168.25 mg/l (153.50%), from 79.18 to 173.93 mg/l (119.66%)



(a) Sample: solvent ratio 1:20

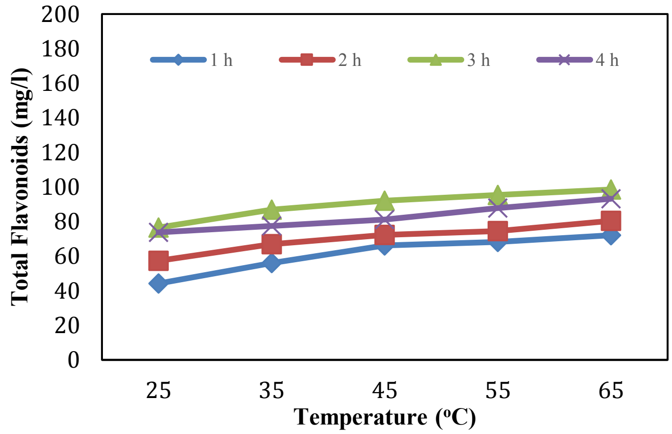


(b) Sample: solvent ratio 1:40

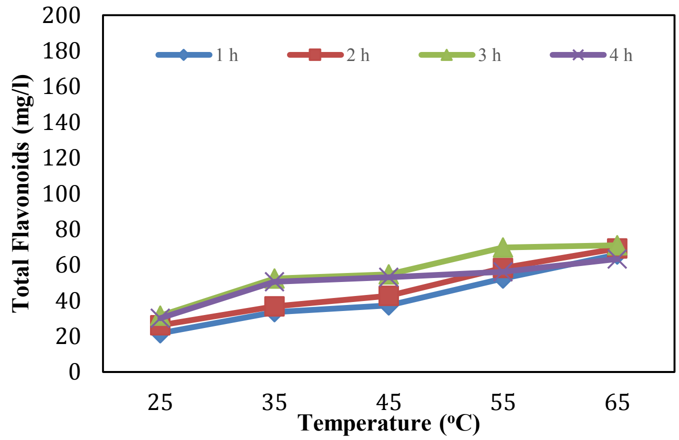


(c) Sample: solvent ratio 1:60

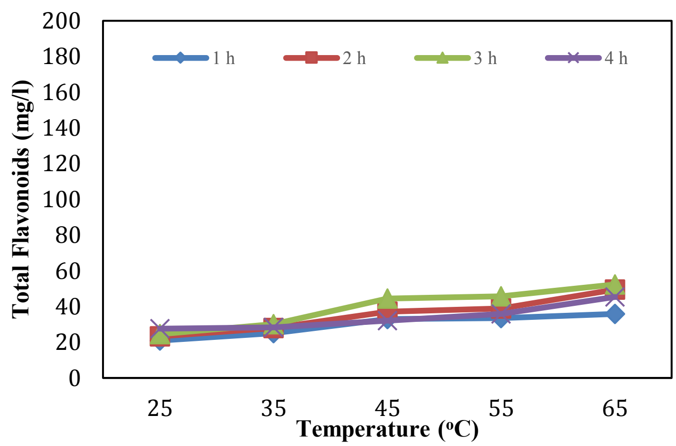
Figure 5.50. The effect of reaction temperatures on the total flavonoids yield from ajwa date fruit using water at different reaction times.



(a) Sample: solvent ratio 1:20

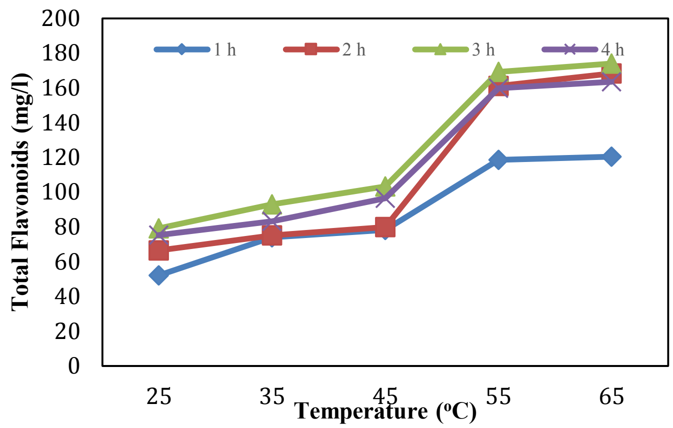


(b) Sample: solvent ratio 1:40

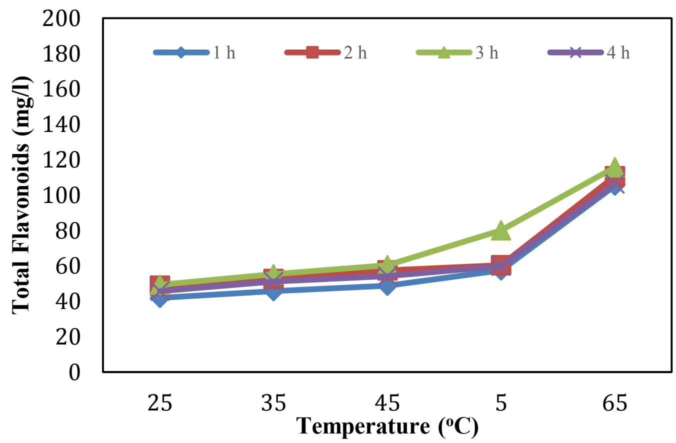


(c) Sample: solvent ratio 1:60

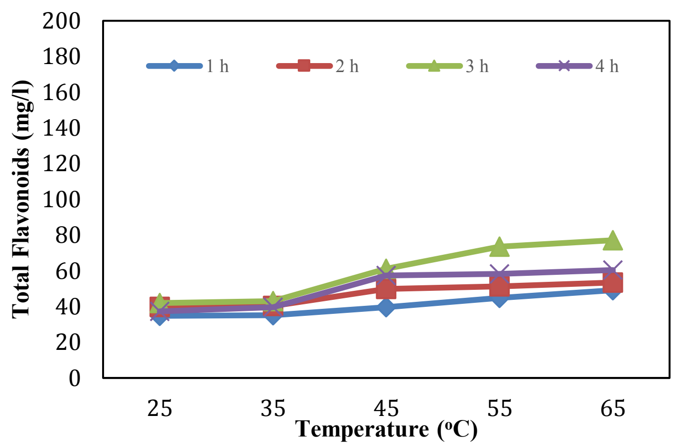
Figure 5.51. The effect of reaction temperatures on the total flavonoids yield from ajwa date fruit using 50% acetone at different reaction times.



(a) Sample: solvent ratio 1:20

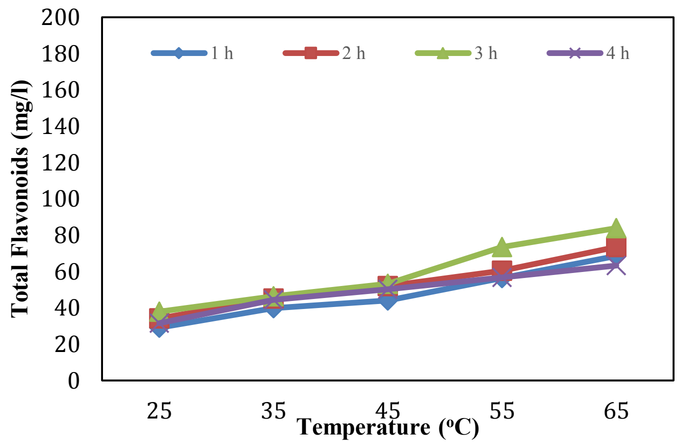


(b) Sample: solvent ratio 1:40

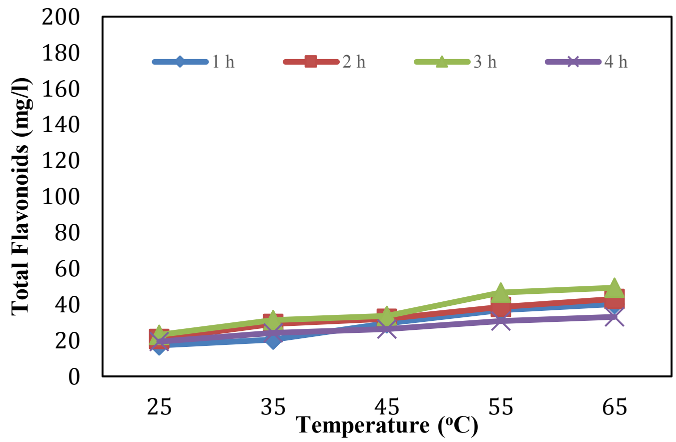


(c) Sample: solvent ratio 1:60

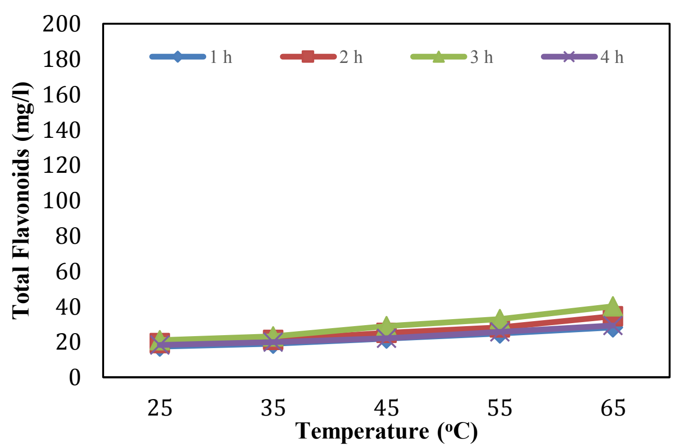
Figure 5.52. The effect of reaction temperatures on the total flavonoids yield from ajwa date fruit using 75% acetone at different reaction times.



(a) Sample: solvent ratio 1:20

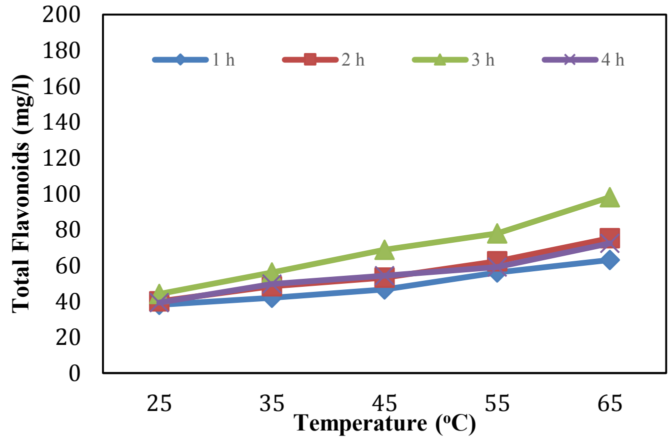


(b) Sample: solvent ratio 1:40

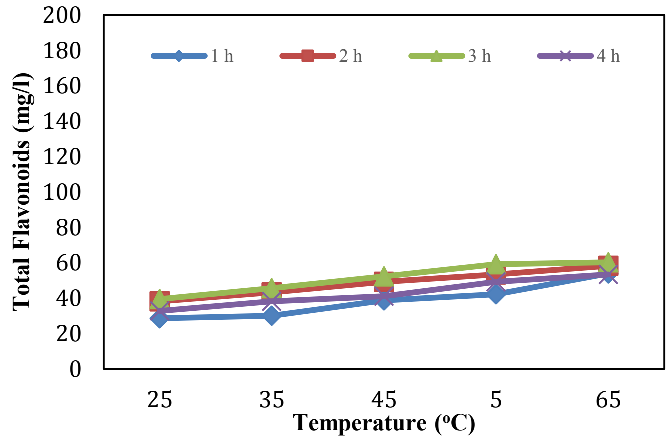


(c) Sample: solvent ratio 1:60

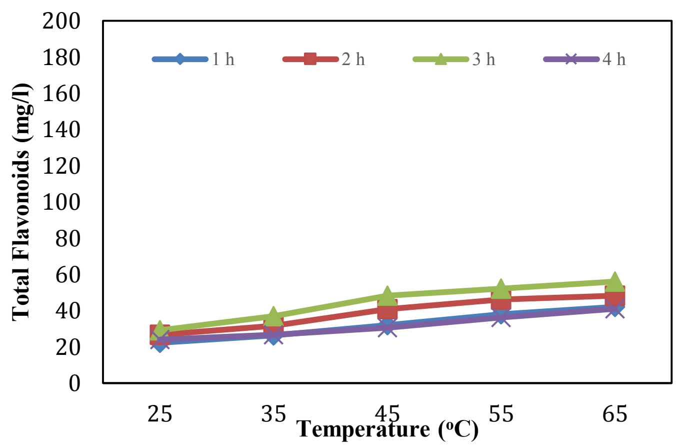
Figure 5.53. The effect of reaction temperatures on the total flavonoids yield from ajwa date fruit using 50% ethanol at different reaction times.



(a) Sample: solvent ratio 1:20



(b) Sample: solvent ratio 1:20



(c) Sample: solvent ratio 1:60

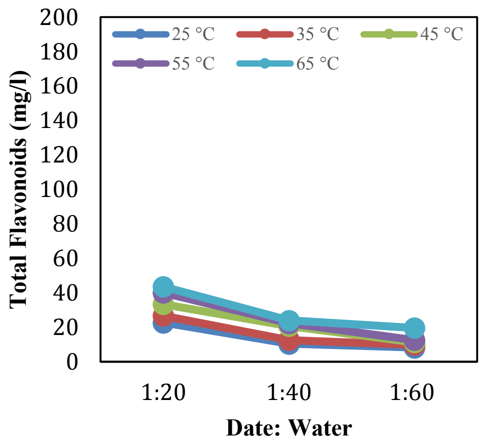
Figure 5.54. The effect of reaction temperatures on the total flavonoids yield from ajwa date fruit using 75% ethanol at different reaction times.

and from 75.18 to 163.43 mg/l (117.38%) at the sample: solvent ratio of 1:20, from 42.00 to 105.43 mg/ml (151.02%), from 48.87 to 110.75 mg/l (126.62%), from 49.50 to 115.75 mg/l (133.84%) and from 45.75 to 106.32 mg/l (132.39%) at the sample: solvent ratio of 1:40 and from 34.68 to 49.18 mg/l (41.81%), from 39.75 to 53.37 mg/l (34.26%), from 41.93 to 77.12 mg/l (86.33%) and from 37.21 to 60.33 mg/l (62.13%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

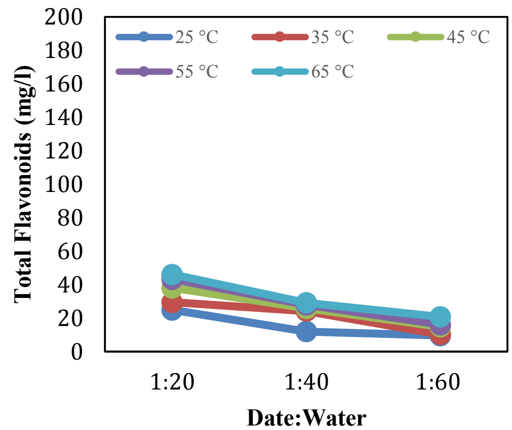
When ethanol was used as solvent at the 50 % concentration, increasing the reaction temperature from 25 to 65^{o}C increased the flavonoids yield from 29.18 to 68.56 mg/l (134.96%), from 34.18 to 73.50 mg/l (115.04%), from 37.93 to 83.87 mg/l (121.20%) and from 31.68 to 63.37 mg/l (100.03%) at the sample: solvent ratio of 1:20, from 17.31 to 40.33 mg/l (132.99%), from 20.87 to 43.12 mg/l (106.61%), from 23.21 to 49.36 mg/l (112.67%) and from 19.33 to 33.06 mg/l (71.03%) at the sample: solvent ratio of 1:40 and from 17.33 to 28.30 mg/l (63.30%), from 19.32 to 34.63 mg/l (79.24%), from 21.03 to 40.12 mg/l (90.78%) and from 18.38 to 29.31 mg/l (59.47%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

When ethanol was used as solvent at the 75 % concentration, increasing the reaction temperature from 25 to 65^{o}C increased the flavonoids yield from 38.12 to 63.08 mg/l (65.48%), from 40.06 to 75.21 mg/l (87.74%), from 44.21 to 98.12 mg/l (121.94%) and from 39.40 to 72.33 mg/l (83.58%) at the sample: solvent ratio of 1:20, from 28.68 to 53.86 mg/l (87.80%), from 38.12 to 58.21 mg/l (52.70%), from 39.50 to 60.33 mg/l (52.73%) and from 32.78 to 53.38 mg/l (62.84%) at the sample: solvent ratio of 1:40 and from 22.31 to 42.21 mg/l (89.20%), from 26.81 to 48.36 mg/l (80.38%), from 29.30 to 56.06 mg/l (91.33%) and from 24.12 to 40.98 mg/l (69.90%) at the sample: solvent ratio of 1:60 for the reaction times of 1, 2, 3 and 4 h, respectively.

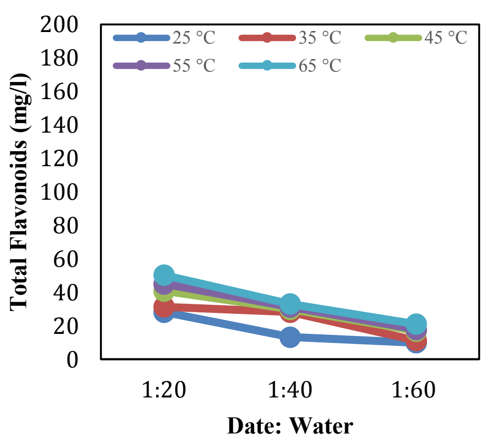
5.1.3.3. Effect of Sample: Solvent Ratio: Figures 5.54-5.58 shown the effect of sample: solvent ratio on the total flavonoids yield from ajwa date fruit at different reaction times (1, 2, 3 and 4 h) and reaction temperatures (25, 35, 45, 55 and 65^{o}C) using acetone and ethanol at different concentrations (0, 50 and 75%). Generally, there was a decrease in the total flavonoids yield when sample: solvent ratio was increased from 1:20 to 1:60 at all reaction temperatures, solvent concentration and reaction times for both solvents.



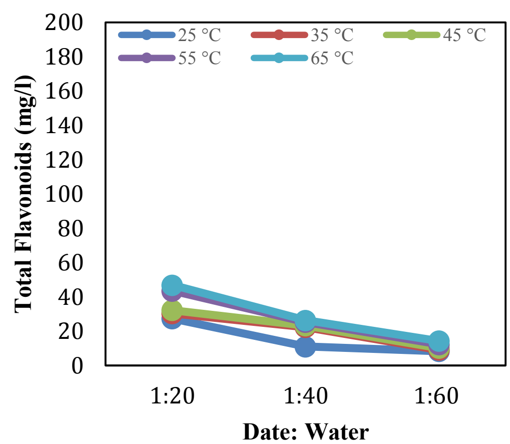
(a) One hour



(b) Two hours

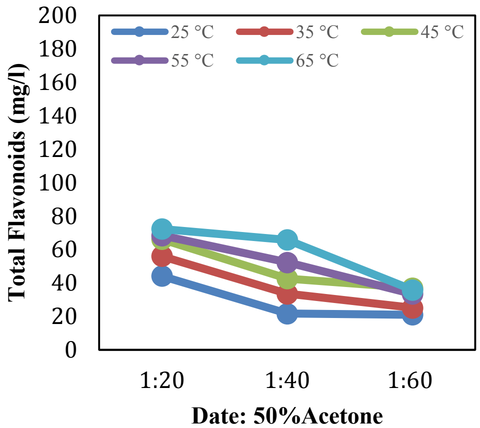


(c) Three hours

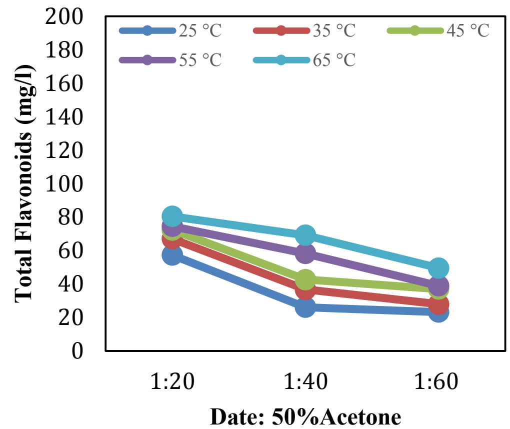


(d) Four hours

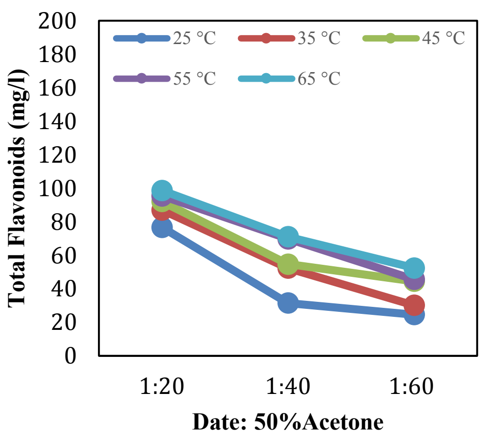
Figure 5.55. The effect of sample: solvent ratio on the total flavonoids yield from ajwa date fruit using water at different reaction temperatures.



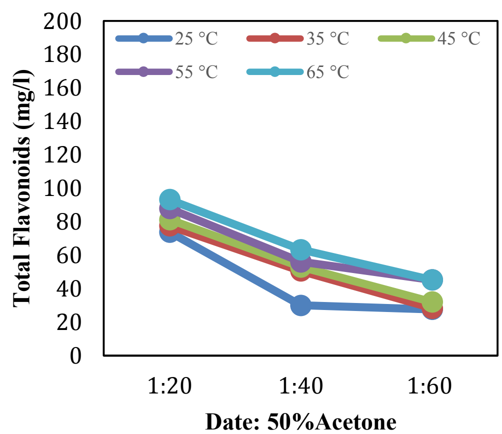
(a) One hour



(b) Two hours

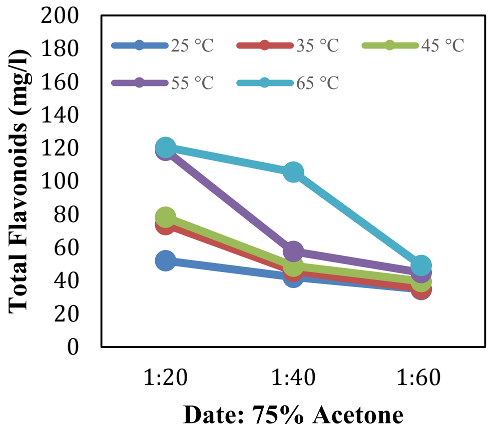


(c) Three hours

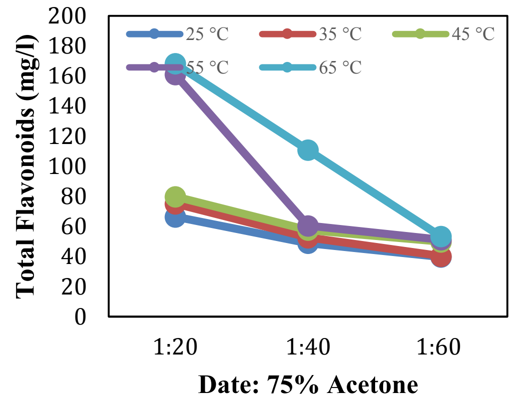


(d) Four hours

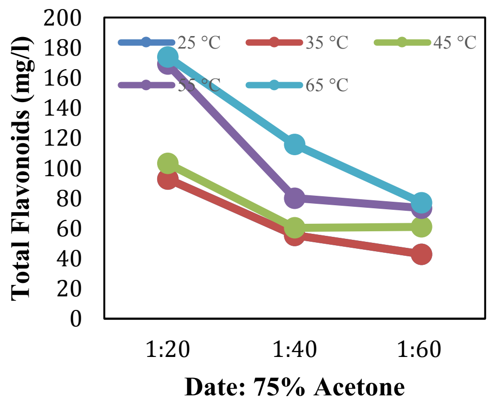
Figure 5.56. The effect of sample: solvent ratio on the total flavonoids yield from ajwa date fruit using 50% acetone at different reaction temperatures.



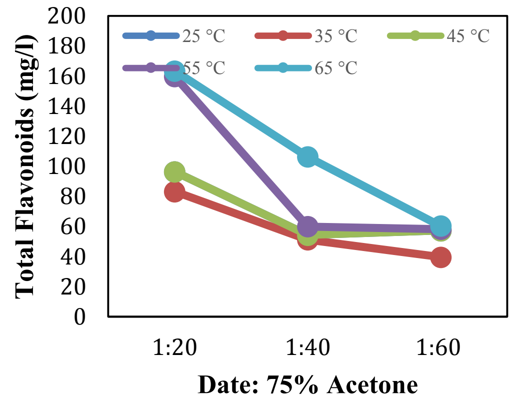
(a) One hour



(b) Two hours

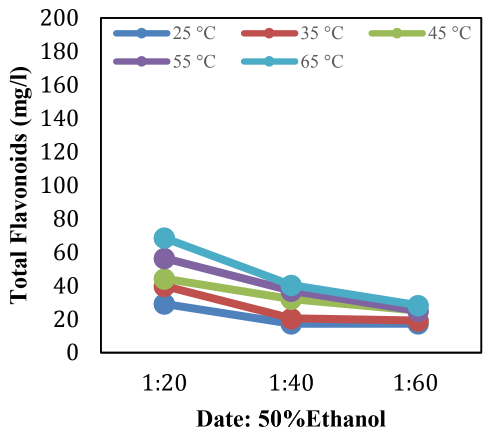


(c) Three hours

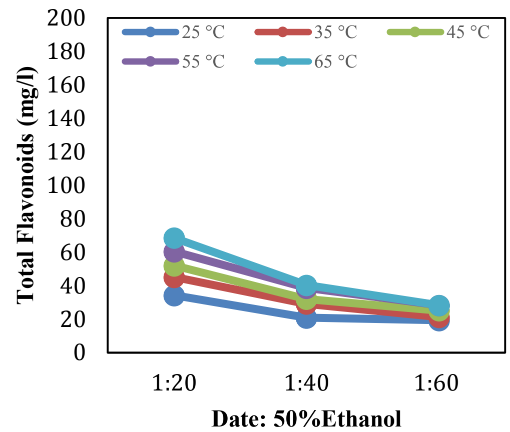


(d) Four hours

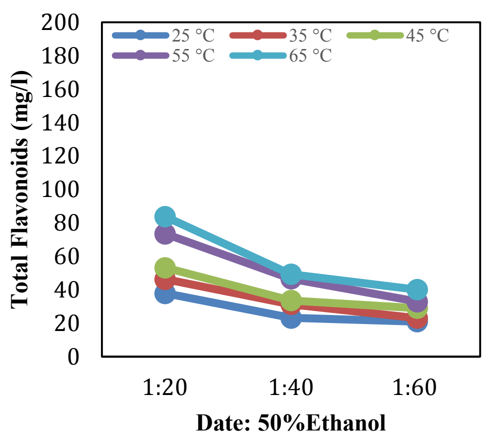
Figure 5.57. The effect of sample: solvent ratio on the total flavonoids yield from ajwa date fruit using 75% acetone at different reaction temperatures.



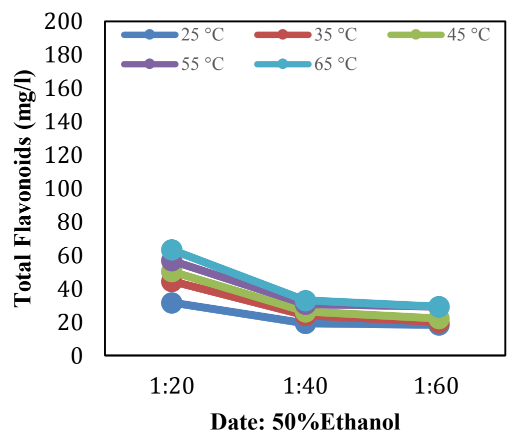
(a) One hour



(b) Two hours

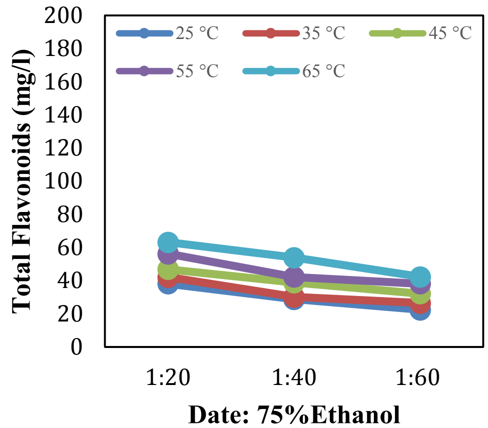


(c) Three hours

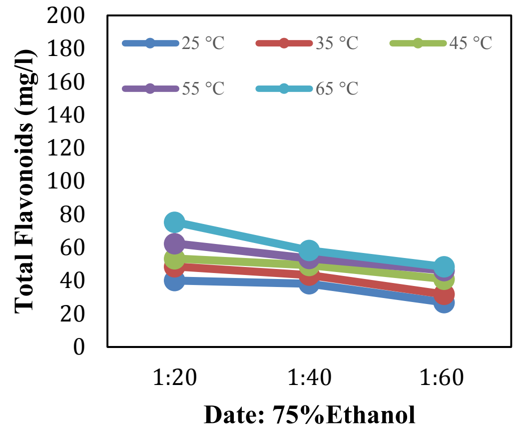


(d) Four hours

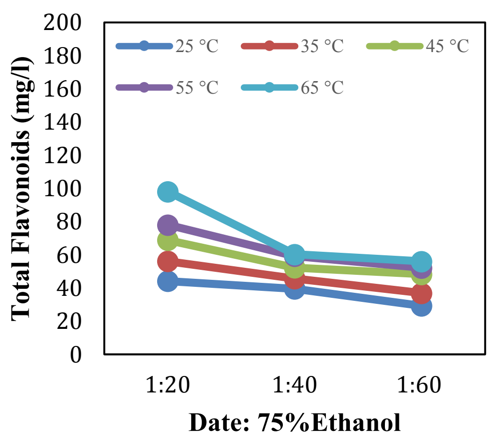
Figure 5.58. The effect of sample: solvent ratio on the total flavonoids yield from ajwa date fruit using 50% ethanol at different reaction temperatures.



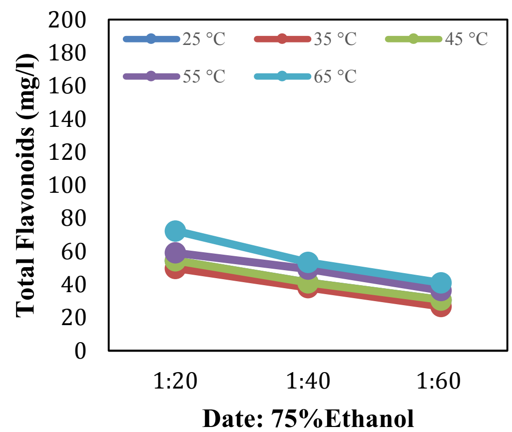
(a) One hour



(b) Two hours



(c) Three hours



(d) Four hours

Figure 5.59. The effect of sample: solvent ratio on the total flavonoids yield from ajwa date fruit using 75% ethanol at different reaction temperatures.

When water was used as solvent, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the flavonoids yield from 22.68 to 8.25 mg/l (63.62%), from 26.68 to 9.83 mg/l (63.16%), from 33.43 to 11.31 mg/l (66.17%), from 40.12 to 12.62 mg/l (68.54%) and from 44.56 to 19.68 mg/ml (55.83%) at the reaction time of 1 h, from 24.68 to 9.56 mg/l (61.26%), from 29.50 to 10.22 mg/l (65.36%), from 38.12 to 14.81 mg/l (61.15%), from 43.25 to 16.06 mg/l (62.87%) and from 46.12 to 20.75 mg/l (55.01%) at the reaction time of 2 h, from 28.27 to 10.02 mg/l (64.56%), from 31.37 to 11.32 mg/l (63.91%), from 40.87 to 17.00 mg/l (58.40%), from 45.12. to 17.93 mg/l (60.26%) and from 50.32 to 21.06 mg/l (58.15 %) at the reaction time of 3 h and from 27.33 to 8.31 mg/l (69.59%), from 30.35 to 9.03 mg/l (70.25%), from 32.21 to 10.21 mg/l (63.30%), from 43.56 to 12.33 mg/l (71.69%) and from 46.87 to 14.32 mg/l (69.45%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

When acetone was used as solvent at the 50 % concentration, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the flavonoids yield from 44.18 to 21.00 mg/l (52.47%), from 56.06 to 25.12 mg/l (55.19%), from 66.18 to 32.93 mg/l (50.24%), from 68.36 to 33.50 mg/l (50.99%) and from 72.25 to 35.87 mg/l (50.35%) at the reaction time of 1 h, from 57.31 to 23.25 mg/l (34.06%), from 67.00 to 27.93 mg/l (58.31%), from 72.31 to 37.00 mg/l (48.83%), from 74.50 to 38.93 mg/l (47.74%) and from 80.43 to 49.50 mg/l (38.46%) at the reaction time of 2 h, from 76.68 to 24.56 mg/l (67.97%), from 87.00 to 30.12 mg/l (65.38%), from 92.12 to 44.50 mg/l (51.69%), from 95.50 to 45.60 mg/l (52.25%) and from 98.68 to 52.37 mg/l (46.93%) at the reaction time of 3 h and from 73.87 to 27.62 mg/l (62.61%), from 77.62 to 28.31 mg/l (63.53%), from 81.30 to 32.06 mg/l (60.57%), from 87.93 to 35.81 mg/l (59.27%) and from 93.25 to 45.30 mg/ml (51.42%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

When acetone was used as solvent at the 75% concentration, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the flavonoids yield from 52.00 to 34.68 mg/l (33.31%), from 73.87 to 35.12 mg/l (52.46%), from 78.56 to 39.50 mg/l (49.52%), from 118.56 to 44.87 mg/l (62.15%) and from 120.43 to 49.18 mg/l (59.16%) at the reaction time of 1 h, from 66.37 to 39.75 mg/l (40.11%), from 75.12 to 40.23 mg/l (46.45%), from 79.81 to 49.81 mg/l (37.59%), from 161.06 to 51.25 mg/l (68.18%) and from 168.25 to

53.37 mg/l (68.28%) at the reaction time of 2 h, from 79.18 to 41.93 mg/l (47.04%), from 92.93 to 42.93 mg/l (53.80%), from 103.25 to 61.06 mg/l (40.86%), from 169.18 to 73.56 mg/l (56.52%) and from 173.93 to 77.12 mg/l (55.66%) at the reaction time of 3 h and from 75.18 to 37.21 mg/l (50.51%), from 83.25 to 39.62 mg/l (52.41%), from 96.37 to 57.31 mg/l (40.53%), from 159.81 to 58.23 mg/l (63.56%) and from 163.43 to 60.33 mg/l (63.09%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

When ethanol was used as solvent at the 50% concentration, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the flavonoids yield from 29.18 to 17.33 mg/l (40.61%), from 39.81 to 19.06 mg/l (52.12%), from 44.18 to 21.92 mg/l (50.38%), from 56.37 to 24.82 mg/l (55.97%) and from 68.56 to 28.30 mg/l (58.72%) at the reaction time of 1 h, from 34.18 to 19.32 mg/l (43.48%), from 45.12 to 21.23 mg/l (52.95%), from 52.00 to 25.15 mg/l (51.63%), from 60.43 to 28.30 mg/l (53.17%) and from 73.50 to 34.63 mg/l (52.88%) at the reaction time of 2 h, from 37.93 to 21.03 mg/l (44.56%), from 46.37 to 23.12 mg/l (50.14%), from 53.25 to 29.06 mg/l (45.43%), from 73.56 to 32.98 mg/l (55.17%) and from 83.87 to 40.12 mg/l (52.16%) at the reaction time of 3 h and from 31.68 to 18.38 mg/l (41.98%), from 44.50 to 20.03 mg/l (54.99%), from 50.31 to 22. 19 mg/l (55.89%), from 56.87 to 25.68 mg/l (54.84%) and from 63.37 to 29.31 mg/l (53.75%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

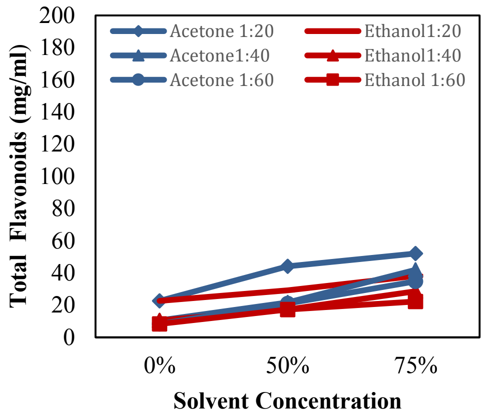
When ethanol was used as solvent at the 75% concentration, increasing the sample: solvent ratio from 1:20 to 1:60 decreased the flavonoids yield from 38.12 to 22.31 mg/l (41.47%), from 42.03 to 26.38 mg/l (37.24%), from 46.37 to 32.14 mg/l (31.22%), from 56.12 to 38.12 mg/l (32.07%) and from 63.08 to 42.21 mg/l (33.08%) at the reaction time of 1 h, from 40.06 to 26.81 mg/l (33.08%), from 48.38 to 31.68 mg/l (34.52%), from 53.21 to 40.92 mg/l (23.10%), from 62.33 to 46.12 mg/l (26.01%) and from 75.21 to 48.36 mg/l (35.70%) at the reaction time of 2 h, from 44.21 to 29.30 mg/l (33.73%), from 56.12 to 36.92 mg/l (34.21%), from 68.90 to 48.31 mg/l (29.88%), from 78.02 to 52.13 mg/l (33.18%) and from 72.33 to 56.06 mg/l (42.87%) at the reaction time of 3 h and from 39.40 to 24.12 mg/l (38.78%), from 49.78 to 26.68 mg/l (46.40%), from 54.38 to 30.68 mg/l (43.58%), from 59.18 to 36.38 mg/l (38.53%) and from 72.33 to 40.98 mg/ml (43.34%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65ºC, respectively.

5.1.3.4. Effect of Solvent Concentration: Figures 5.59 -5.63 show the effect of solvent concentration on the total flavonoids yield from ajwa date fruit at different sample: solvent ratios (1:20, 1:40 and 1:60), reaction times (1, 2, 3 and 4 h) and the reaction temperatures of (25, 35, 45, 55 and 65ºC) for acetone and ethanol. Generally, there was an increase in the total flavonoids yield when solvent concentration was increased from 0 to 75% for all reaction temperatures, sample: solvent ratios and reaction times for both solvents.

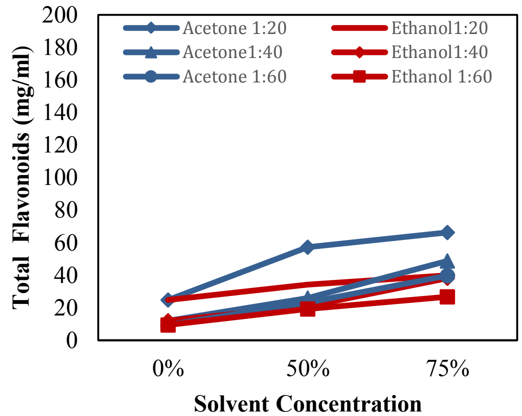
Increasing the concentration of acetone from 0 to 75% at the temperature of 25ºC increased the total flavonoids yield from 22.68 to 52.00 mg/l (129.28%), from 10.43 to 42.00 mg/l (302.68%) and from 8.25 to 34.68 mg/l (320.36%) at the reaction time of 1 h, from 24.68 to 66.37 mg/l (168.92%), from 11.96 to 48.87 mg/l (308.61%) and from 9.56 to 39.75 mg/l (315.79%) at the reaction time of 2 h, from 28.27 to 79.18 mg/l (180.08%), from 49.50 to 13.31 mg/l (271.90%) and from 10.02 to 41.93 mg/l (318.46%) at the reaction time of 3 h and from 27.33 to 75.18 mg/l (175.08%), from 11.01 to 45.75 mg/l (315.53%) and from 8.31 to 37.21 mg/l (347.77%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 35ºC increased the total flavonoids yield from 26.68 to 73.87 mg/l (176.87%), from 12.55 to 45.75 mg/l (264.54%) and from 9.83 to 35.12 mg/l (257.27%) at the reaction time of 1 h, from 29.50 to 75.12 mg/l (154.64%), from 24.18 to 52.62 mg/l (117.62%) and from 10.22 to 40.23 mg/l (293.64%) at the reaction time of 2 h, from 31.37 to 92.93 mg/l (196.24%), from 28.25 to 55.43 mg/l (96.21%) and from 11.32 to 42.93 mg/l (279.24%) at the reaction time of 3 h and from 30.35 to 83.25 mg/l (174.30%), from 22.31 to 51.25 mg/l (129.72%) and from 9.03 to 39.62 mg/ml (338.76%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

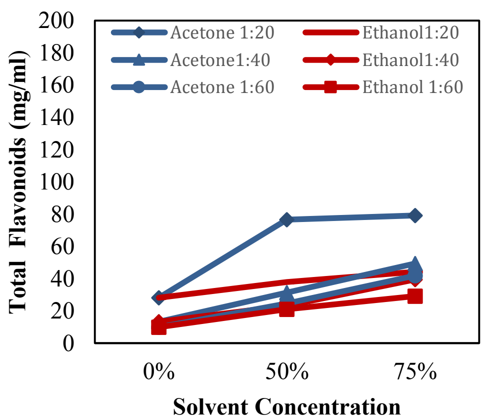
Increasing the concentration of acetone from 0 to 75% at the temperature of 45ºC increased the total flavonoids yield from 33.43 to 78.25 mg/l (134.07%), from 20.75 to 48.81 mg/l (135.23%) and from 11.31 to 39.50 mg/l (249.25%) at the reaction time of 1 h, from 38.12 to 79.81 mg/l (109.37%), from 25.82 to 57.62 mg/l (123.16%) and from 14.81 to 49.81 mg/l (236.33%) at the reaction time of 2 h, from 40.87 to 103.25 mg/l (152.63%),



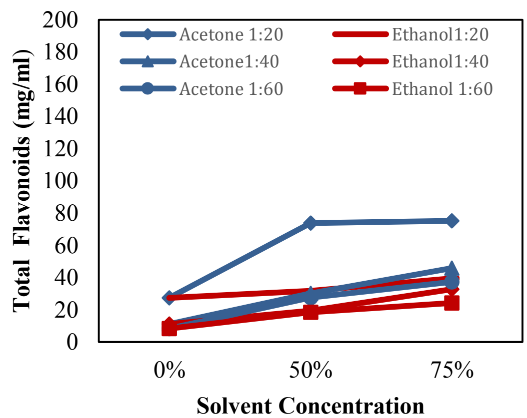
(a) One hour



(b) Two hours

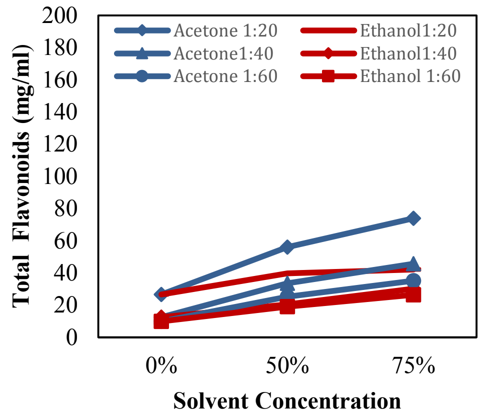


(c) Three hour

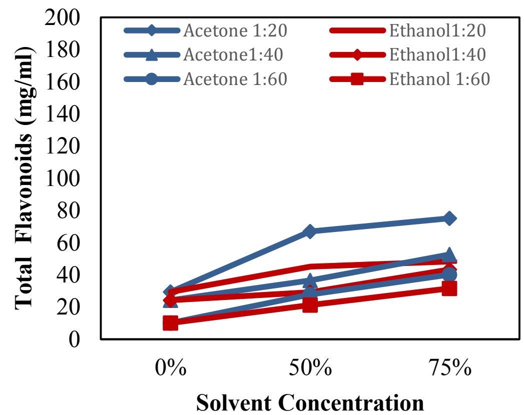


(d) Four hours

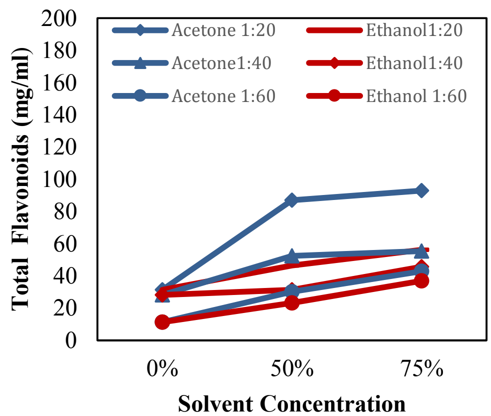
Figure 5.60. The effect of solvent (acetone and ethanol) concentration on the total flavoinds yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 25ºC.



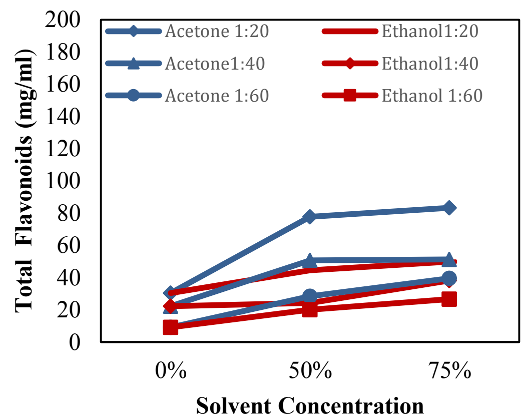
(a) One hour



(b) Two hours

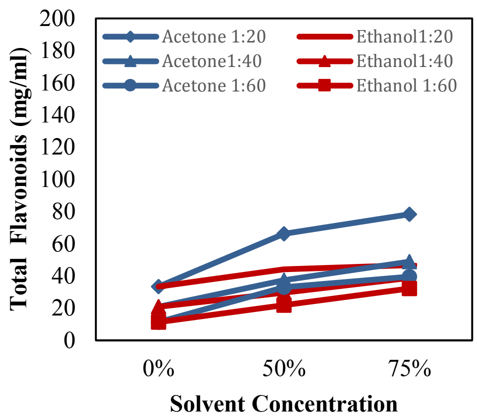


(c) Three hour

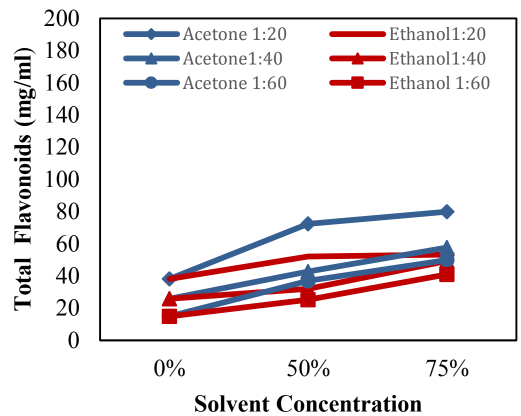


(d) Four hours

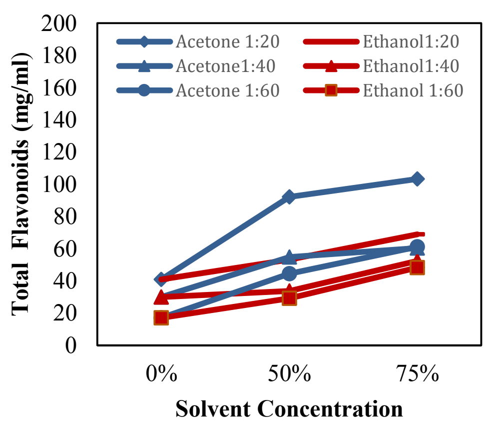
Figure 5.61. The effect of solvent (acetone and ethanol) concentration on the total flavoinds yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 35^{o}C.



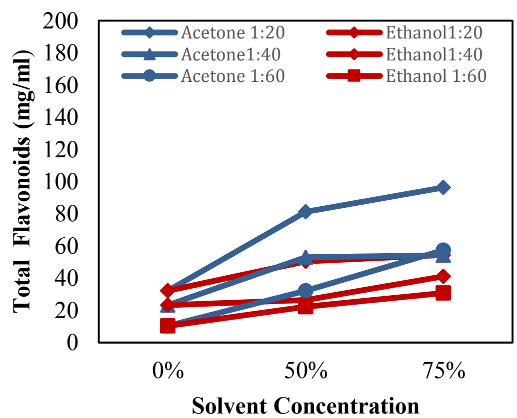
(a) One hour



(b) Two hours

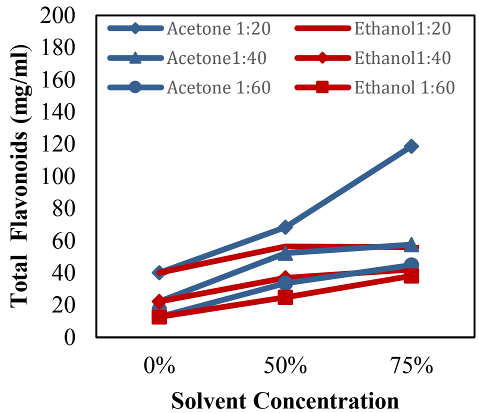


(c) Three hour

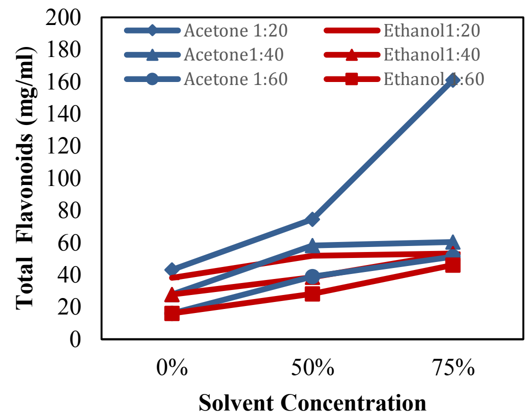


(d) Four hours

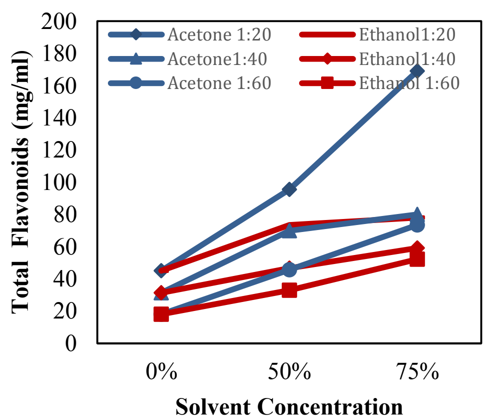
Figure 5.62. The effect of solvent (acetone and ethanol) concentration on the total flavoinds yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 45^{o}C.



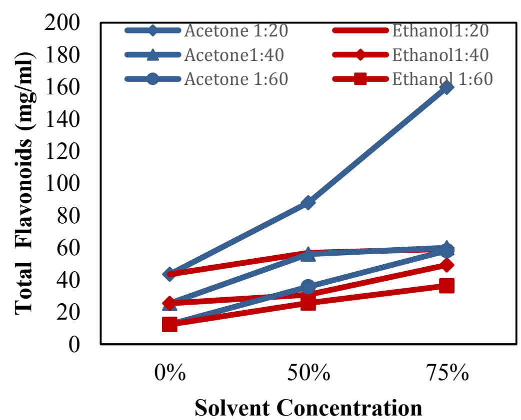
(a) One hour



(b) Two hours

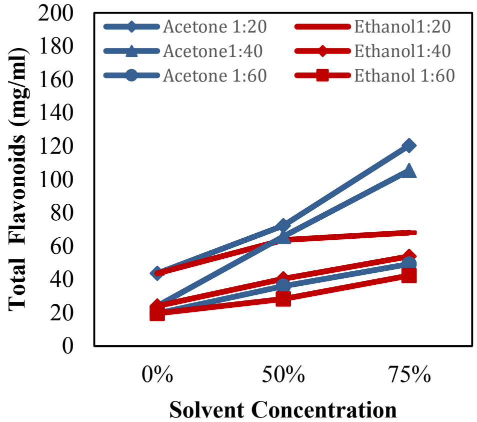


(c) Three hour

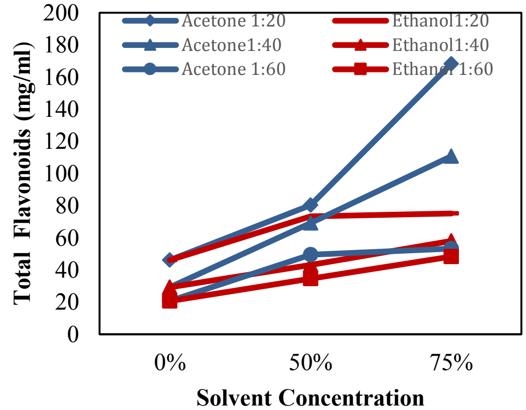


(d) Four hours

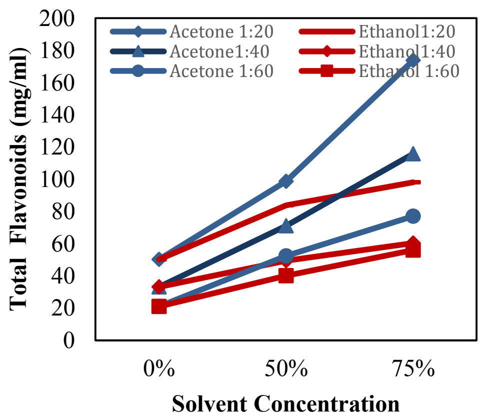
Figure 5.63. The effect of solvent (acetone and ethanol) concentration on the total flavoinds yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 55°C.



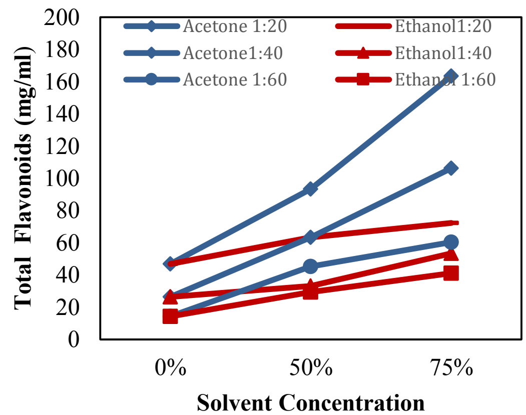
(a) One hour



(b) Two hours



(c) Three hour



(d) Four hours

Figure 5.64. The effect of solvent (acetone and ethanol) concentration on the total flavoinds yield from ajwa date fruit at different sample: solvent ratios and the reaction temperature of 56ºC.

from 29.95 to 60.43 mg/l (101.77%) and from 17.00 to 61.06 mg/l (259.18%) at the reaction time of 3 h and from 32.21 to 96.37 mg/l (199.19%), from 23.22 to 54.25 mg/l (133.63%) and from 10.21 to 57.31 mg/l (461.31%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 55^{o}C increased the total flavonoids yield from 40.12 to 118.56 mg/l (195.51%), from 22.31 to 57.56 mg/l (158%) and from 12.62 to 44.87 mg/l (255.55%) at the reaction time of 1 h, from 43.25 to 161.06 mg/l (272.39%), from 27.92 to 60.43 mg/l (116.44%) and from 20.75 to 53.37 mg/l (219.12%) at the reaction time of 2 h, from 45.12 to 169.18 mg/l (274.96%), from 31.32 to 80.12 mg/l (155.81%) and from 17.93 to 73.56 mg/l (310.26%) at the reaction time of 3 h and from 43.56 to 159.81 mg/l (266.87%), from 25.34 to 59.93 mg/l (136.50%) and from 12.33 to 58.23 mg/l (372.26%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of acetone from 0 to 75% at the temperature of 65^{o}C increased the total flavonoids yield from 44.56 to 120.43 mg/l (170.26%), from 23.98 to 105.43 mg/l (339.66%) and from 19.68 to 49.18 mg/l (149.90%) at the reaction time of 1 h, from 46.12 to 168.25 mg/l (264.81%), from 29.21 to 110.75 mg/l (279.15%) and from 20.75 to 53.37 mg/l (157.20%) at the reaction time of 2 h, from 50.32 to 173.93 mg/l (245.65%), from 33.12 to 115.75 mg/l (249.49%) and from 21.06 to 77.12 mg/l (266.19%) at the reaction time of 3 h and from 46.87 to 163.43 mg/l (248.69%), from 26.32 to 106.32 mg/l (303.95%) and from 14.32 to 60.33 mg/l (321.30%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 25^{o}C increased the total flavonoids yield from 22.68 to 38.12 mg/l (68.08%), from 10.43 to 28.68 mg/l (174.98%) and from 8.25 to 22.31 mg/l (170.42%) at the reaction time of 1 h, from 24.68 to 40.06 mg/l (62.32%), from 11.96 to 38.12 mg/l (218.73%) and from 9.56 to 26.81 mg/l (180.44%) at the reaction time of 2 h, from 28.27 to 44.21 mg/l (56.38%), from 49.50 to 39.50 mg/l (196.77%) and from 10.02 to 29.30 mg/l (192.42%) at the reaction time of 3 h and from 27.33 to 39.40 mg/l (44.16%), from 11.01 to 32.78 mg/l (197.73%) and from 8.31 to 24.12 mg/l (190.25%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 35°C increased the total flavonoids yield from 26.68 to 42.03 mg/l (57.53%), from 12.55 to 30.12 mg/l (140%) and from 9.83 to 26.38 mg/l (168.36%) at the reaction time of 1 h, from 29.50 to 48.38 mg/l (64%), from 24.18 to 43.28 mg/l (78.99%) and from 10.22 to 31.68 mg/l (209.98%) at the reaction time of 2 h, from 31.37 to 56.12 mg/l (78.90%), from 28.25 to 45.66 mg/l (61.63%) and from 11.32 to 36.92 mg/l (226.15%) at the reaction time of 3 h and from 30.35 to 49.78 mg/l (64.02%), from 22.31 to 38.12 mg/l (70.87%) and from 9.03 to 26.68 mg/ml (195.46%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 45°C increased the total flavonoids yield from 33.43 to 46.73 mg/l (39.78%), from 20.75 to 38.73 mg/l (86.65%) and from 11.31 to 32.14 mg/l (184.17%) at the reaction time of 1 h, from 38.12 to 53.21 mg/l (39.59%), from 25.82 to 49.23 mg/l (90.67%) and from 14.81 to 40.92 mg/l (176.30%) at the reaction time of 2 h, from 40.87 to 68.90 mg/l (68.58%), from 29.95 to 52.31 mg/l (74.66%) and from 17.00 to 48.31 mg/l (184.18%) at the reaction time of 3 h and from 32.21 to 54.38 mg/l (68.83%), from 23.22 to 41.06 mg/l (76.83%) and from 10.21 to 30.68 mg/l (200.49%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

Increasing the concentration of ethanol from 0 to 75% at the temperature of 55°C increased the total flavonoids yield from 40.12 to 56.12 mg/l (39.88%), from 22.31 to 42.12 mg/l (88.79%) and from 12.62 to 38.12 mg/l (202.06%) at the reaction time of 1 h, from 43.25 to 62.33 mg/l (44.12%), from 27.92 to 53.36 mg/l (91.12%) and from 20.75 to 46.12 mg/l (187.17%) at the reaction time of 2 h, from 45.12 to 78.02 mg/l (72.92%), from 31.32 to 59.22 mg/l (89.08%) and from 17.93 to 52.13 mg/l (190.74%) at the reaction time of 3 h and from 43.56 to 59.18 mg/l (35.86%), from 25.34 to 49.31 mg/l (94.59%) and from 12.33 to 36.38 mg/l (195.05%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

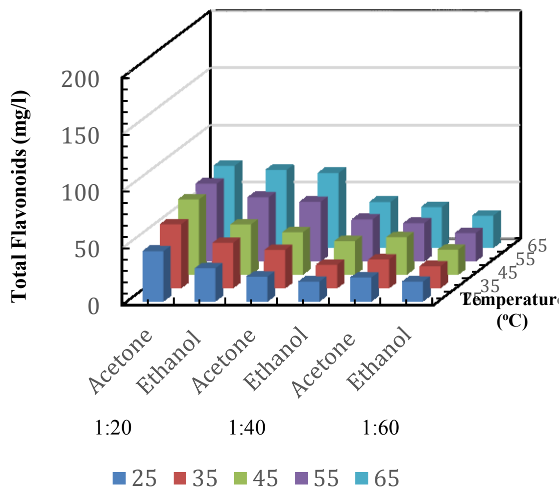
Increasing the concentration of ethanol from 0 to 75% at the temperature of 65°C increased the total flavonoids yield from 44.56 to 63.08 mg/l (41.56%), from 23.98 to 53.86 mg/l (124.60%) and from 19.68 to 42.21 mg/l (114.48%) at the reaction time of 1 h, from 46.12 to 75.21 mg/l (63.07%), from 29.21 to 58.21 mg/l (99.28%) and from 20.75 to 48.36

mg/l (133.06%) at the reaction time of 2 h, from 50.32 to 98.12 mg/l (94.99%), from 33.12 to 60.33 mg/l (82.16%) and from 21.06 to 56.06 mg/l (166.19%) at the reaction time of 3 h and from 46.87 to 72.33 mg/l (54.32%), from 26.32 to 53.38 mg/l (102.81%) and from 14.32 to 40.98 mg/l (186.17%) at the reaction time of 4 h for the sample: solvent ratios of 1:20, 1:40 and 1:60, respectively.

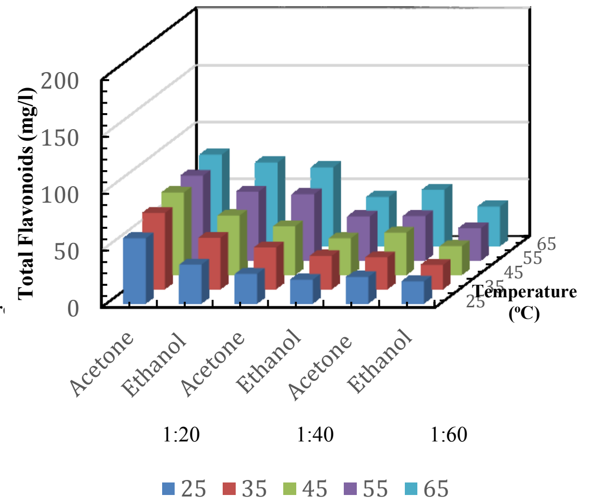
5.1.3.5. Effect of Solvent Type: Figures 5. 62 and 5.63 show the effect of solvent type on the total flavonoids yield from ajwa date fruit at different sample: solvent ratios (1:20, 1:40 and 1:60), reaction times (1, 2, 3 and 4 h) and reaction temperatures (25, 35, 45, 55 and 65^{o}C). Generally, when acetone was used as a solvent, higher total flavonoids yield was obtained compared to that obtained with ethanol at the same concentration at all reaction temperatures, sample: solvent ratios and reaction times with 50 and 75% concentrations.

Using ethanol at the 50% concentration with the sample: solvent ratio of 1:20 decreased the total flavonoids yield over that obtained with acetone from 44.18 to 29.18 mg/l (33.95%), from 56.06 to 39.81 mg/l (28.99%), from 66.18 to 44.18 mg/l (33.24%), from 68.36 to 56.37 mg/l (17.54%) and 72.25 to 68.56 mg/l (5.11%) at the reaction time of 1 h, from 57.31 to 34.18 mg/l (40.36%), from 67.00 to 45.12 mg/l (32.66%), from 72.31 to 52.00 mg/l (29.07%), from 68.36 to 56.37 mg/l (18.89%) and from 72.25 to 68.56 mg/l (8.62%) at the reaction time of 2 h, from 76.68 to 37.93 mg/l (50.53%), from 87.00 to 46.37 mg/l (46.70%), from 92.12 to 53.25 mg/l (42.19%), from 95.50 to 73.56 mg/l (22.97%) and from 98.68 to 83.87 mg/l (15.01%) at the reaction time of 3 h, from 73.87 to 31.68 mg/l (57.11%), from 77.62 to 44.50 mg/l (42.67%), from 81.30 to 50.31 mg/l (38.12%), from 87.93 to 56.87 mg/l (38.12%) and from 93.25 to 63.37 mg/l (32.04%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

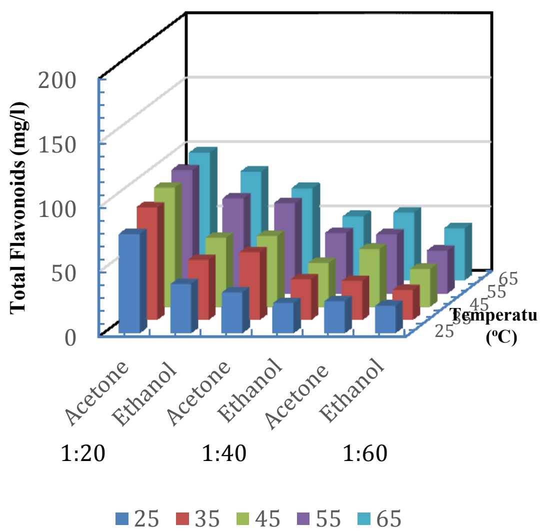
Using ethanol at the 50% concentration with the sample: solvent ratio of 1:40 decreased the total flavonoids yield over that obtained with acetone from 21.68 to 17.31 mg/l (20.16%), from 33.56 to 20.43 mg/l (39.12%), from 37.31 to 29.50 mg/l (20.93%), from 52.31 to 36.87 mg/l (29.52%) and from 65.81 to 40.33 mg/l (38.72%) at the reaction time of 1 h, from 26.06 to 20.87 mg/l (19.92%), from 36.68 to 29.18 mg/l (4.89%), from 42.62 to 31.99 mg/l (24.94%), from 58.25 to 38.63 mg/l (33.68%) and from 69.18 to 43.12 mg/l (37.67%) at the reaction time of 2 h, from 31.43 to 23.21 mg/l (26.15%), from 52.31



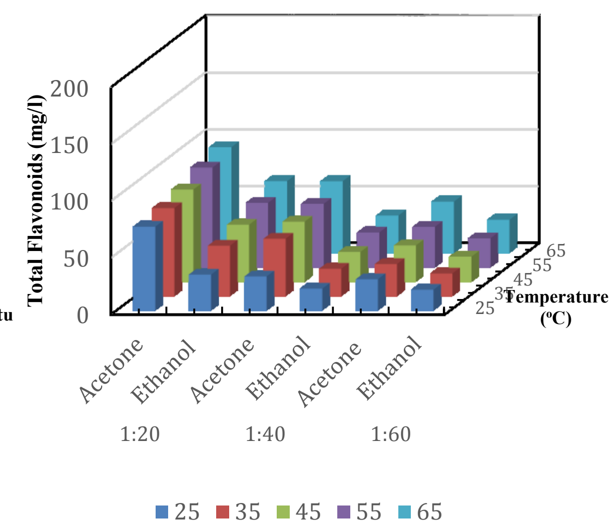
(a) One hour



(b) Two hours

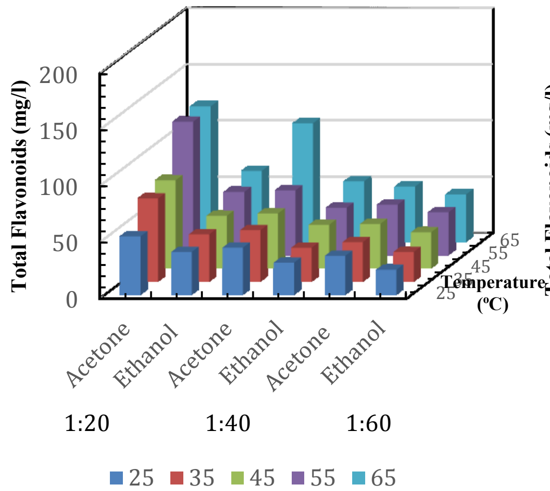


(c) Three hours

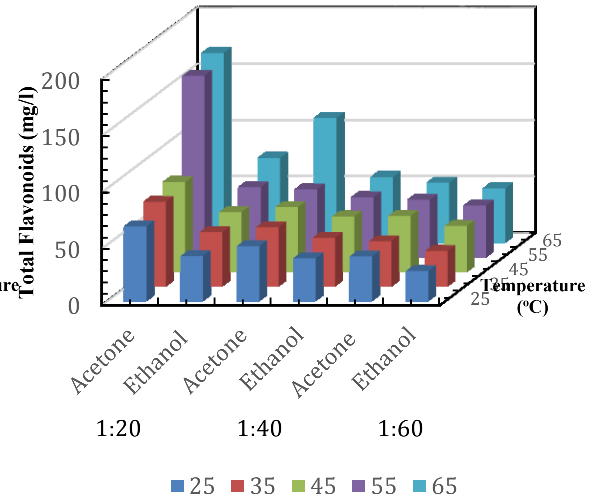


(d) Four hours

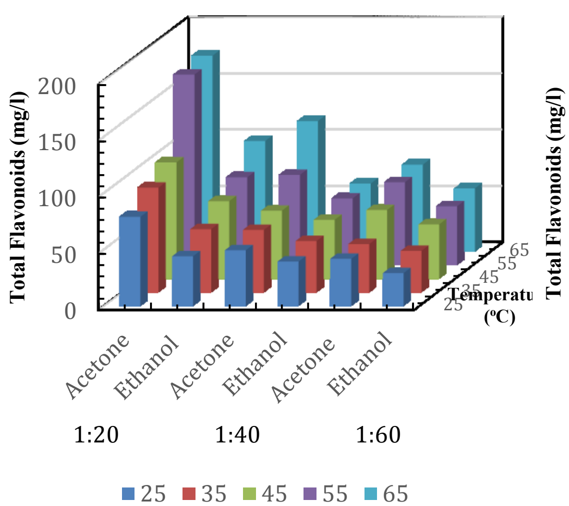
Figure 5.65. The effect of solvent (acetone and ethanol) at the concentration of 50% on the total flavonoids yield from ajwa date fruit at different sample: solvent ratios and different reaction temperatures.



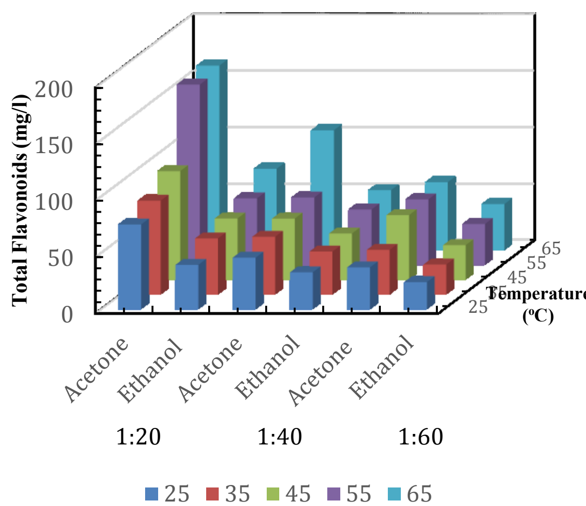
(a) One hour



(b) Two hours



(c) Three hours



(d) Four hours

Figure 5.66. The effect of solvent (acetone and ethanol) at the concentration of 75% on the total flavonoids yield from ajwa date fruit at different sample: solvent ratios and different reaction temperatures.

to 31.37 mg/l (40.03%), from 54.70 to 33.68 mg/l (38.43%), from 69.81 to 46.68 mg/l (33.13%) and from 71.06 to 49.36 mg/l (30.54%) at the reaction time of 3 h, from 30.06 to 19.33 mg/l (35.70%), from 50.62 to 24.118 mg/l (52.23%), from 53.01 to 26.31 mg/l (50.37%), from 56.06 to 30.81 mg/l (45.04%) and from 63.31 to 33.06 mg/l (47.78%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

Using ethanol at the 50% concentration with the sample: solvent ratio of 1:60 decreased the total flavonoids yield over that obtained with acetone from 21.00 to 17.33 mg/l (17.48%), from 25.12 to 19.06 mg/l (24.12%), from 32.93 to 21.92 mg/l (33.43%), from 33.50 to 24.82 mg/l (25.91%) and from 35.87 to 28.30 mg/l (21.10%) at the reaction time of 1 h, from 23.25 to 19.32 mg/l (16.90%), from 27.93 to 21.23 mg/l (23.99%), from 37.00 to 25.15 mg/l (32.03%), from 38.93 to 28.30 mg/l (27.31%) and from 49.50 to 34.63 mg/l (30.04%) at the reaction time of 2 h, from 24.56 to 21.03 mg/l (14.37%), from 30.12 to 23.12 mg/l (23.24%), from 44.50 to 29.06 mg/l (34.70%), from 45.60 to 32.98 mg/l (27.68%) and from 52.37 to 40.12 mg/l (23.39%) at the reaction time of 3 h and from 27.62 to 18.38 mg/l (33.45%), from 28.31 to 20.03 mg/l (29.25%), from 32.06 to 22.19 mg/l (30.79%), from 35.81 to 25.68 mg/l (28.29%) and from 45.30 to 29.31 mg/l (35.30%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65^{o}C, respectively.

Using ethanol at the 75% concentration with the sample: solvent ratio of 1:20 decreased the total flavonoids yield over that obtained with acetone from 52.00 to 38.12 mg/l (26.69%), from 73.87 to 42.03 mg/l (43.10%), from 78.25 to 46.73 mg/l (40.28%), from 118.56 to 56.12 mg/l (52.67%) and from 120.43 to 63.08 mg/l (47.62%) at the reaction time of 1 h, from 66.37 to 40.06 mg/l (39.64%), from 75.12 to 48.38 mg/l (35.60%), from 79.81 to 53.21 mg/l (33.33%), from 161.06 to 62.33 mg/l (61.30%) and 168.25 to 75.21 mg/l (55.30%) at the reaction time of 2 h, from 79.18 to 44.21 mg/l (44.17%), from 92.93 to 56.12 mg/l (39.61%), 103.25 to 68.90 mg/l (33.27%), from 169.18 to 78.02 mg/l (53.88%) and from 173.93 to 98.12 mg/l (43.59%) at the reaction time of 3 h and from 75.18 to 39.40 mg/l (47.59%), from 83.25 to 49.78 mg/l (40.20%), from 96.37 to 54.38 mg/l (43.57%), from 159.81 to 59.18 mg/l (62.97%) and from 163.43 to 72.33

mg/l (55.74%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

Using ethanol at the75% concentration with the sample: solvent ratio of 1:40 decreased the total flavonoids yield over that obtained with acetone from 42.00 to 28.68 mg/l (31.71%), from 45.75 to 30.12 mg/l (34.16%), from 48.81 to 38.73 mg/l (20.65%), from 57.56 to 42.12 mg/l (26.82%) and from 105.43 to 53.86 mg/l (48.91%) at the reaction time of 1 h, from 48.87 to 38.12 mg/l (22%), from 52.62 to 43.28 mg/l (17.75%), from 57.62 to 49.23 mg/l (14.56%), from 60.43 to 53.36 mg/l (11.70%) and from 110.75 to 58.21 mg/l (47.44%) at the reaction time of 2 h, from 49.50 to 39.50 mg/l (20.20%), from 55.43 to 45.66 mg/l (17.63%), from 60.43 to 52.31 mg/l (13.44%), from 80.12 to 59.22 mg/l (26.09%) and from 115.75 to 60.33 mg/l (47.88%) at the reaction time of 3 h, from 45.75 to 32.78 mg/l (28.35%), from 51.25 to 38.12 mg/l (25.62%), from 54.25 to 41.06 mg/l (24.31%), from 59.93 to 49.31 mg/l (17.72%) and from 106.32 to 53.38 mg/l (49.79%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

Using ethanol at the 75% concentration with the sample: solvent ratio 1:60 decreased the total flavonoids yield over that obtained with acetone from 34.68 to 22.31 mg/l (35.67%), from 35.12 to 26.38 mg/l (24.89%), from 39.50 to 32.14 mg/l (18.63%), from 44.87 to 38.12 mg/l (15.04%) and from 49.18 to 42.21 mg/l (14.17%) at the reaction time of 1 h, from 39.75 to 26.81 mg/l (32.55%), from 40.23 to 31.68 mg/l (21.25%), from 49.81 to 40.92 mg/l (17.85%), from 51.25 to 46.12 mg/l (10.01%) and from 53.37 to 48.36 mg/l (9.39%) at the reaction time of 2 h, from 41.93 to 29.30 mg/l (30.12%), from 42.93 to 36.92 mg/l (14%), from 61.06 to 48.31 mg/l (20.88%), from 73.56 to 52.13 mg/l (29.13%) and from 77.12 to 56.06 mg/l (27.31%) at the reaction time of 3 h and from 37.21 to 24.12 mg/l (35.18%), from 39.62 to 26.68 mg/l (32.66%), from 57.31 to 30.68 mg/l (46.47%), from 58.23 to 36.38 mg/l (37.52%) and from 60.33 to 40.98 mg/l (32.07%) at the reaction time of 4 h for the reaction temperatures of 25, 35, 45, 55 and 65°C, respectively.

5.2. Optimal Extraction Conditions using Fresh and Freeze dried Ajwa

The result of the total antioxidants, total phenols and total flavonoids obtained from fresh and freeze dried ajwa fruit at the optimum sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 55˚C for antioxidants and 65˚C for phenols and flavonoids using ethanol at 75% concentration for antioxidants and acetone at 75% concentration for phenols and flavonoids are shown in Table 5.22. Freeze drying increased the antioxidants, phenols and flavonoids yields over that obtained from fresh fruit. The antioxidants yield increased from 198 to 365.52 mg/ml (84.61%), the phenols yield increased from 1003.92 to 1563.12 mg/l (76.93%) and the flavonoids yield increased from 173.93 to 226.06 mg/l (65.37%).

5.3.Total Antioxidants, Phenols and Flavonoids from Different Date Varieties

The result of total antioxidants, phenols and flavonoids determined from five date varieties (ajwa, sukkari, red sukkari, khalas and sofry) at the optimum extraction conditions (sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 55˚C for antioxidants and 65˚C for phenols and flavonoids using ethanol at 75% concentration for antioxidants and acetone at 75% concentration for phenols and flavonoids) are shown in Table5.23. Ajwa dates had the highest antioxidants, phenol and flavonoids followed by khalas, sukkari, red sukkari and sofry.

Table 5.22. The total antioxidants, phenols and flavonoids obtained from fresh and freezr dried ajwa fruit.

Methods	Total Antioxidants (mg/ml)	Total Phenols (mg/l)	Total Flavonoids (mg/l)
Fresh Ajwa	198.00	1003.92	173.93
Freeze dried Ajwa	365.52	1776.231	287.625

Sample: Solvent Ratio = 1:20
Reaction Time = 3 h
Reaction Temperature = (55˚C) for antioxidants and (65˚C) for phenols and flavonoids
Solvent Type = ethanol for antioxidants and acetone for phenols and flavonoids
Solvent Concentration = 75%

Table 5.23. Total antioxidants, phenols and flavonoids from different date varieties.

Date Varieties	**Total Antioxidants (mg/ml)**	**Total Phenols (mg/l)**	**Total Flavonoids (mg/l)**
Ajwa	365.52	1776.231	287.625
Khalas	314.25	1260.06	262.25
Sukkari	312.25	1023.25	215.32
Red Sukkari	305.51	1002.95	208.25
Sofry	301.96	920.25	206.14

Sample: Solvent Ratio = 1:20
Reaction Time = 3 h
Reaction Temperature = (55˚C) for antioxidants and (65˚C) for phenols and flavonoids
Solvent Type = ethanol for antioxidants and acetone for phenols and flavonoids
Solvent Concentration = 75%

CHAPTER 6. DISCUSSION

6.1. Total Antioxidants, Phenols and Flavonoids Yields from Ajwa Date

The extraction yields of antioxidants, phenols and flavonoids were affected by the reaction time, reaction temperature, sample: solvent ratio, solvent concentration and the solvent type. There were also significant interactions among these parameters.

6.1.1. Effect of Reaction Time

In this study, increasing the reaction time from 1 to 3 h, increased the yields of the total antioxidants, phenols and flavonoids for both solvents (ethanol and acetone) at all reaction temperatures, sample: solvent ratios and solvent concentrations. This was followed by decreases in the yields of these compounds with a further increase in reaction time from 3 to 4 h. Durling et al. (2007), Al-Farsi and Lee (2008) and Uma et al. (2010) obtained similar results and stated that increasing the extraction time above 3 h will have a negative impact on the quality of the extracted compounds due to the long exposure to temperature and the evaporation of solvent which in turn affects the sample: solvent ratio. However, some researchers (Yalmaz and Toledo, 2006; Zhao et al., 2006) reported shorter reaction times for the extraction of these compounds, while others reported longer reaction times (Pinelo et al., 2005 and Spigno and De Faveri, 2007). The variation in the reaction time among different studies are the result of the relationship between the extraction time and the extraction temperature. Generally, increasing the reaction time with high temperatures will result in decreased yields of the total antioxidants, phenols and flavonoids. Spigno et al. (2007) and Durling et al. (2007) stated that the reaction time plays an important role in the extraction of total antioxidants, phenols and flavonoids and can have a significant effect on the cost of extraction and production processes.

6.1.1.1.Antioxidants: Komes et al. (2011) reported that increasing extraction time from 5 to 15 min increased the antioxidants yield from lemon palm from 0.10187 to 0.16169 mg/g. Bonilla et al. (1999) found that the optimum extraction time for extracting 50.20 mg/l of antioxidants from crushed grape marc was 20 min. Thoo et al. (2010) used extraction times in the range of 20 - 180 min and found the 80 min was the optimum reaction time for extracting 1.977 mg/g and 4.730 mg/g antioxidants from mengkudu (*Morinda citrifolia*) using ABTS and DPPH methods, respectfully. La et al. (2013) found the 2 h was the optimum reaction time for extracting 88.7 mg/g of antioxidants from rambutan peel using the DPPH method. Spigno et al. (2007) found the 5 h reaction time was the optimum for extracting antioxidants from grape marc. Uma et al. (2010) reported that the optimum reaction time for extracting antioxidants from henna (*Lawsonia inermis*) leaves was 3 h. Lapornik et al. (2005) extracted antioxidants from red grape pressed marc after vinification and found that the reaction time had a significant effect on the antioxidant activity and increasing the reaction time from 1 to 24 h increased the antioxidant yield from 1.237 to 1.661 mg/g.

In this study, the total antioxidants obtained from ajwa date fruit at the optimum reaction time of 3 h was 20.266 mg/g. The antioxidants results are within the range of 0.10187 - 88.7 mg/g reported in the literatures. The extraction time depends on factors such as reaction temperature, extraction methods, type of solvent, sample type, and preservation methods. The reaction time reported in the literature for the extraction of antioxidants ranged from 5 min to 24 h. The differences can be attributed to the different materials and extraction procedures used. However, the optimum reaction time of 3 h observed in this study is relatively short and will have a positive impact on the economics of antioxidants extraction. According to Uma et al. (2010), increasing the reaction time beyond the optimum value will not result in more antioxidants.

6.1.1.2. Phenols: Bonilla et al. (1999) studied the effect of reaction time (5 - 30 min) on the extraction of phenols from red grape marc and found that the 5 min reaction time gave the best yield. Goli et al. (2005) found that the 5 min was the optimum reaction time for extracting phenols (32.0–34.0 mg/g) from pistachio over the tested range of 15- 40 min. Uma et al. (2010) reported that the total phenols yield increased from 66.49 to 59.10 mg/g when the reaction time was increased from 30 to 90 min. Khiari et al. (2009) found that increasing the reaction time to 6 h with a low reaction temperature resulted in higher phenol yields. Spigno and De Faveri (2007) extracted 0.0265 mg/g phenols from red grape marc and reported that predrying powdered red grape marc (after vinification) before the extraction of phenols had an impact on the total phenols yield.

In this study, the total phenols obtained from ajwa date fruit was 97.2018 mg/g at the optimum reaction time of 3 h. These results are higher than those reported in the literature (0.0265 mg/g to 66.49 mg/g). The reaction times reported in the literature for the extraction of phenols from the different sources ranged from 5 min to 6 h. These difference can be attributed to the different type of samples and extraction procedures used. Spigno et al. (2007) and Pinelo et al. (2005) stated that the reaction time depends on the temperature and higher temperature will improve the solubility of solute and the diffusion coefficient. Thoo et al. (2010) and Silva et al. (2007) found that the optimum extraction time for total phenols depended on the degree of phenolic polymerisation, solubility of phenolic compounds and presence of other food constituents. Chew et al. (2011) stated that reaction time is an important factor in solvent extraction system of phenolic compounds and can result in cost saving. The reaction time achieved in this study was relatively short and will have a positive effect on the extraction and production costs of phenols.

6.1.1.3. Flavonoids: Hertog et al. (1992) reported that some fruits (cranberry, black currant, crowberry and date) are good sources of flavonoids (containing about 100-263 mg/kg). Xiao et al. (2008) used short irradiation times (5 - 10 min) to extract 1.234 mg/g flavonoids from *Radix Astragali* and observed improved yields with increased microwave power along with increased reaction time. Al-Farsi and Lee (2007) found that the best reaction time to extract flavonoids (0.054mg/g) from date seed was 1 h. Oomah and Mazza (1996) obtained an average of 3.87 mg/g of flavonoid from Buckwheat using a reaction time of 2 h. Thoo et al. (2010) reported that 80 min was the optimum reaction time for extracting flavonoids (3.7 mg/g) from mengkudu (Morinda citrifolia). Chew et al. (2011) extracted a total flavonoids of 1230 mgCE/100g from *Orthosiphon stamineus* at the reaction time of 2 h.

In this study, the total flavonoids obtained from ajwa date fruit was 11.142 mg/g at the optimum reaction time of 3 h. These results are comparable to those reported in the literature (1.234 - 12.30 mg/g). The reaction time reported in the literature for the extraction of flavonoids ranged from 5 min to 2 h. The extraction time of 3 h observed in this study was longer than those reported in the literature. This could be due to the use of different materials and procedures as well as factors such as reaction temperature, type of solvent and preservation methods.

6.1.2. Effect of Reaction Temperature

In this study, the total antioxidants yield increased as the reaction temperature was increased from 25 to 55°C whereas the yields of the total phenols and flavonoids increased as the temperature was increased from 25 to 65°C for both solvents (acetone and ethanol) at all reaction times, sample: solvent ratios and solvent concentrations. A decrease in the antioxidants yield was observed with a further increase in reaction temperature from 55 to 65°C. Several researches (Pinelo et al. 2005; Cacace and Mazza, 2006; Chew et al., 2011) obtained similar results. Spigno et al. (2007) and Durling et al. (2007) stated that the reaction temperature plays an important role in extraction of the total antioxidants, phenols and flavonoids and can have a significant effect on the cost of the process.

6.1.2.1. Antioxidants: La et al. (2013) found that the 25 °C reaction tempreature was the best for extracting 55.2 mg/g of antioxidants from mangosteen peel. Durling et al. (2007) found that the 40°C was the optimum reaction temperature for extracting antioxidants from dried sage (*Salvia officinalis)*. Bucić-Kojić et al. (2011) reported that the highest antioxidant yield (0.015 mg/g) was obtained from fig fruits at a reaction temperature of 50 °C. La et al. (2013) reported that the 50 °C was the best reaction temperature for extracting antioxidants from rambutan peel using the DPPH method. Moure et al. (2001) reported that high extraction temperatures (over 70°C) have a negative impact on the stability of desired compounds due to degradation and losses of some chemicals. Uma et al. (2010) reported that high extraction temperatures increased the chance of solvent loss and decreased the solvent concentration and polarity in the extract which in turn reduced the yield of extracted compounds. Chew et al. (2011) reported that antioxidants are sensitive to high temperature and the yield was decreased when the temperature was increased above 65°C. Spigno and De Faveri (2007) found that the temperature (over the range of 28 - 60°C) has an impact on the total antioxidants extracted from the dried powder of red grape marc. Perva-Uzanalic et al. (2006) suggested that short extraction times must be used with high temperature.

In this study, the total antioxidants obtained from ajwa date fruit was 22.7616 mg/g at the optimum reaction temperature of 55 °C. These results are within the range reported in the literature (1.5 - 55.20 mg/g). The reaction temperatures reported in the literatures for the extraction antioxidants from different sources are in the range from 25 - 65 °C. The differences can be attributed to the use of different extraction procedures. Also, the extraction temperature depends on factors such as reaction time, type of solvent, sample type and preservation methods. The optimum reaction temperature for the extraction of antioxidants reported in this study of 55 ºC is within the range reported in the literature.

6.1.2.2. Phenols: La et al. (2013) reported that the phenols yield obtained from mangosteen peel was 218.39 mg/g at a reaction temperature of 25 ºC. Akowuah et al. (2009) obtained the highest yield of total phenols at a reaction temperature of 40ºC. Wang et al. (2008) found that the 60ºC reaction temperature was the best for extracting phenols from wheat bran and reported a yield of 0.0279 mg/g. Chew et al. (2001) reported an optimum reaction temperature of 65 ºC for extracting phenolic compounds from *Orthosiphon stamineus*. Bucić-Kojić et al. (2011) reported total phenols yield of 2.4 - 3.7 mg/g from fig fruits at a reaction temperature under 80 ºC. Li et al. (2006) studied the effect of temperature on enzymatic extraction of phenols from banana juice and found that endogenous enzymes worked well under warm temperature and were inactivated at high temperature.

In this study, the total phenols obtained from ajwa date fruit was 109.460 mg/g at the optimum reaction temperature of 65 ºC. These results are within the range reported in the literature (0.0279 - 218.39 mg/g). The reaction temperatures listed in the literature for the extraction of total phenols from different sources are within the range of 25 - 80 ºC. The differences in these reaction temperatures can be attributed to the different type of materials and extraction procedure used. The extraction temperature can also be affected by factors such as reaction time and type of solvent. The optimum reaction temperature of 65 ºC for extracting phenols is within the range reported in the literature.

6.1.2.3. Flavonoids: Al-Farsi and Lee (2008) state that 45ºC was the optimum reaction temperature for extracting 1.593 mg/g flavonoids from date seeds. Bucić-Kojić et al. (2011) reported flavonoids yield in the range 0.44 - 2.5 mg/g at a reaction temperature of 80 ºC. Xiao et al. (2008) found the 110 ºC to be the optimum reaction temperature for extracting flavonoids from Rasix Astragali. Chew et al. (2011) reported that the total flavonoids obtained from *Orthosiphon stamineus* at 65 ºC was 16.11 mg/g. Thoo et al. (2010) state that 65 ºC was the optimum reaction temperature for extracting 4.72 mg/g flavonoids from mengkudu (*Morinda citrifolia*). Xu and Chang (2007) reported that the total flavonoids extract from green pea at room temperature was 0.39 mg/g.

In this study, the total flavonoids obtained from ajwa date fruit was 13.66 mg/g at the optimum reaction temperature of 65 °C. These results are comparable to the higher values reported in the literature (0.44 - 15.93 g/g). The reaction temperatures reported in the literature for the extraction of total flavonoids from different sources are within the range of 45 - 110 °C. The differences can be attributed to the different types of material and extraction methods used. The reaction temperature for extracting flavonoids depends on factors such as reaction time, type of solvent and preservation methods. The optimum reaction temperature found in this study is within the lower values of this range and will have a significant impact on the economics of flavonoids production.

6.1.3. Effect of Sample: Solvent Ratio

In this study, there was a decrease in total antioxidant, phenol and flavonoid yields when the sample: solvent ratio was increased from 1:20 to 1:60 for both solvents at all reaction temperatures, solvent concentrations and reaction times. Perva-uzunalic (2006) found that using a lower sample: solvent ratio is favourable. Pompeu et al. (2009) stated that decreasing the sample: solvent ratio will increase the yield of antioxidants, phenols, and flavonoids. Pinelo et al. (2005) stated that the lower the value of solvent used, the higher the concentration of extract attained. Other studies concluded that the higher sample: solvent ratios resulted in higher yields, depending on the temperature, solvent type and solvent concentration used (Pinelo et al., 2005; Al-Farsi and Lee, 2008). Durling et al. (2007) stated that increasing the sample: solvent ratio will increase bioactive compounds as well as the ratio of bioactive compounds to the soluble solids. Pinelo et al. (2005) reported that the sample: solvent ratio has an impact on total phenols and antioxidant activities from dried red and white grape marc. Cacace and Mazza (2003) stated that the sample: solvent ratio and the solid solubility are affected by fluctuations in the activity coefficient, which depends also on the temperature and type of solution. However, the cost of solvent and energy connected with the solvent evaporation must be taken into consideration.

6.1.3.1. Antioxidants: Pompeu et al. (2009) reported that the total antioxidant yield of 1.1088 mg/g was obtined from *Euterpe oleracea* fruits at the 1:4 sample: solvent ratio. Wang et al. (2011) reported that increasing the sample: solvent ratio from 1:5 to 1:15 increased the antioxidants yield from pomegranate peels by 431.8 mg/g over the tested range of 1:15 - 1:50 but further increases in sample: solvent ratio above 1:15 reduced the yield. Al-Farsi et al. (2005) reported that the total antioxidants obtained from Omani date was 22.96 mg/g at the 1:40 sample: solvent ratio. Xu and Chang (2007) reported antioxidants yield of 0.00227 mg/g from green pea and 0.0420 mg/g from lentil at the sample: solvent ratio of 1:15. Anwar and Przybylski, (2012) used a sample: solvent ratio of 1:50 to extract antioxidants from flaxseed (*Linumusitatissimum L.*) and obtained 130.mg/g. Kähkönen et al. (1999) reported that the 1:20 sample: solvent ratio was good for extracting antioxidants (0.025 mg/g) from onion.

In this study, the total antioxidants obtained from ajwa date fruit was 24.232 mg/g at the optimum sample: solvent ratio of 1:20. These results are within the range reported in the literature (0.00227 - 431.80 mg/g). The sample: solvent ratio range reported in the literature for extracting antioxidants from various sources is 1:15 - 1:50. This could be due to variations in the reaction time, extraction methods, type of solvent and sample type. The optimum sample: solvent ratio of 1:20 reported in this study is within the range reported in the literature.

6.1.3.2. Phenols: Pompeu et al. (2009) reported that the total phenols yield from of *Euterpe oleracea* fruits was 2.1538 mg/g of fruits at a 1:2 sample: solvent ratio. Thi Phuong Lien et al. (2015) found that increasing the sample: solvent ratio from 1:10 to 1:60 decreased the yields of phenols. Wang et al. (2011) reported that using a sample: solvent ratio of 1:15 increased the yield of phenols from pomegranate peels by 465.10 mg/g over that of 1:50. Al-Farsi and Lee (2008) reported that increasing the sample: solvent ratio from 1:20 to 1:60 decreased the total phenols yield from date seed from 5.05 to 2.05 mg/g. Kähkönen et al. (1999) reported that the 1:20 sample: solvent ratio was good for extracting phenols (3 mg/g) from red onion and maple leaf (12.1 mg/g). Xu and Chang (2007) reported that phenols yields of 1.81 mg/g from chickpea and 5 mg/g from black soybean. Anwar and Przybylski (2012) used a sample: solvent ratio of 1:50 to extract phenols from flaxseed (*Linumusitatissimum L.*) and obtained 27 mg/g. Al-Farsi et al. (2005) reported that the total phenols obtained from Omani date was 0.016 mg/g using a sample: solvent ratio of 1:40.

In this study, the total phenols obtained from ajwa date fruit was 123.534 mg/g using the optimum sample: solvent ratio of 1:20. These results are within the range reported in the literature (0.016 - 465.10 mg/g). The range of sample: solvent ratio reported in the literature for the extraction of flavonoids from different sources is 1:15 - 1:80. The differences can be attributed to the different types of materials and extraction methods used. The sample: solvent ratio also depends on the reaction time and type of solvent. The sample: solvent ratio of 1:20 reported in this study is within that range.

6.1.3.3. Flavonoids: Wang et al. (2011) obtained a flavonoids yield of 39.20 mg/g from the pomegranate peel extract using a sample: solvent ratio of 1:15. Tan et al. (2011) reported that the 1:20 sample: solvent ratio was the best ratio for the extraction of 9.30 mg/g flavonoids from pomegranate peels. Xiao et al. (2008) found that the 1:25 sample: solvent ratio was the optimum ratio for the extraction of 1.019 mg/g flavonoids from *Radix Astragali* over the tested range of 1:15 to 1:35. Al-Farsi and Lee (2008) obtained 54 mg/g of flavonoids from date seed at the 1:60 sample: solvent ratio. Chew et al. (2011) reported that the total flavonoids from *Orthosiphon stamineus* was 116.119 mg/g at the sample: solvent ratio of 1:33. Anwar and Przybylski (2012) used a 1:50 sample: solvent ratio to extract flavonoids from flaxseed (*Linumusitatissimum L.*) and obtained 240 mg/g. Xu and Chang (2007) reported a flavonoids yield of 0.50 mg/g from yellow soybean and 2.49 mg/g from black bean at the sample: solvent ratio of 1: 10.

In this study, the total flavonoids obtained from ajwa date fruit was 14.473 mg/g at the optimum sample: solvent ratio of 1:20. These results are within the lower range reported in the literature (0.5 – 116.119 mg/g). The sample: solvent ratio range reported in the literature for flavonoids extraction from different sources is 1:15 - 1:80. The differences can be attributed to the different types of materials and the extraction methods used as well as reaction time and type of solvent. The sample: solvent ratio of 1:20 reported in this study is with that range.

6.1.4. Effect of Solvent Concentration

In this study, there was an increase in the total antioxidant, phenol and flavonoid yields when the solvent concentration was increased from 0 to 75% for both solvents at all reaction temperatures, sample-to-solvent ratios, and reaction times. Acetone at 75% concentration was the best solvent for the extraction of phenols and flavonoids, while ethanol at 75% concentration was the best solvent for the extraction of antioxidants. Karacabey and Mazza, (2008) reported that increasing ethanol concentration resulted in increases in the antioxidant, phenol and flavonoid yields. Stanojević et al. (2009) found that the antioxidant yield increased with increases in the solvent concentration. However, in terms of safety and cost, this study showed that acetone - water mixture is better solvent for extracting these compounds.

6.1.4.1. Antioxidants: Chew et al. (2011) obtained a total antioxidant yield of 2.024 mg/g from *Orthosiphonstamineus* using 40% ethanol. Stanojević et al. (2009) reported a total antioxidant yield of 440 mg/g from *Hieraciumpilosella* using 50% ethanol. Alothman et al. (2009) obtained a total antioxidants of 0.441 mg/ g fresh weight from pineapple using 70% acetone while using 70% ethanol resulted in a yield of 190.43 mg/g fresh weight. Alothman et al. (2009) obtained a total antioxidants of 107.780 mg/g fresh weight from *Banana pisang mas* using 70% acetone while using 70% ethanol resulted in a yield of 163.067 mg/g fresh weight. Al-Farsi and Lee (2008) reported that using 50% acetone resulted in 10.10mg/g of antioxidants from date seed. Xu and Chang (2007) reported a total antioxidants yield of 0.26 mg/g from green pea using 50% acetone which increased to 1.71 mg/g when using 70% acetone. Chew et al. (2011) reported a total antioxidants yield of 2.067 mg/g from *Orthosiphonstamineus* using 40% ethanol.

In this study, the total antioxidant yield obtained from ajwa date fruit was 28.394 mg/g using ethanol as solvent at the optimum concentration of 75%. These results within the range reported in the literature (0.26 - 440 mg/g). The solvent concentration range reported in the literature for extracting antioxidants is 50 - 75%. The difference in solvent concentration depends on various factors such as reaction time, extraction methods used, type of solvent and materials. The 75% concentration of ethanol is within that reported range of solvent concentration.

6.1.4.2. Phenols: Kahkonenet et al. (2001) reported that extractions of phenols from different fruits using 100% water at room temperature (low temperature) resulted in a low phenol yield, while using a mixture of water and organic solvent (methanol and acetone) at the same temperature increased the phenols yield. Zhao et al. (2006) stated that higher phenols yield were obtained from Barley (*Hordeum vulgare L*) using 80% acetone compared to 80% ethanol and 80% methanol. Chew et al. (2011) reported that the total phenol yield from *Orthosiphonstamineus* was 20.034 mg/g using 40% ethanol. Xu and

Chang (2007) extracted phenols from yellow pea, green pea and yellow soybean using 50% acetone and found that 70 % acetone was good for black beans, lentils, black soybeans and red kidney beans. Al-Farsi and Lee (2008) noticed that using 50% acetone increased the total phenols yield from 65.10– 88 mg/g compared to 0.112 – 0.181 mg/g when using water. Do et al. (2014) reported a total phenols yield of 29.60 mg/g from *Limnophila aromatic* using 50% acetone and 39.10 mg/g using75% acetone while using 50% ethanol resulted in 30.30 mg/g and 75% ethanol resulted in 30.60 mg/g. Uma et al. (2013) reported that the total phenols obtained from henna (*Lawsonia inermis*) was 47.96 mg/g using 40% acetone. Sultana et al. (2009) reported that using 80% ethanol resulted in a total phenols yield 0.116 mg/g from *Moringa oleifera* leaves.

In this study, the total phenols yield obtained from ajwa date fruit was 127.765 mg/g using acetone as solvent at a concentration of 75%. These results are higher than those reported in the literature (0.1121 – 88.000 mg/g). The solvent concentrations found in the literature for the extraction of phenols from date fruit are in the range of 0 - 80%. The differences in the solvent concentration depend on factors such as reaction time, extraction method, type of solvent and type of material. The acetone concentration of 75% is within the range of solvent concentration reported in the literature.

6.1.4.3. Flavonoids: Chew et al. (2011) reported that the total flavonoids yield from *Orthosiphonstamineus* was 16.119 mg/g using 40% ethanol. Xu and Chang (2007) reported that 50% acetone was favourable for extracting flavonoids from yellow peas (0.18 - 0.32 mg/g) and green peas (0.08 - 0.39 mg/g). Alothman et al. (2009) obtained a total flavonoids of 0.0258 mg/g of fresh weight from pineapple using 70% acetone whereas when using 70% ethanol, the yield was 0.0417 mg/g of fresh weight. Bucić-Kojić et al. (2011) reprted the total flavonoids obtained from *Banana pisang mas* was 0.115 mg/g of fresh weight using 70 % acetone and 0.0935 mg/g of fresh weight using 70% ethanol. Al-Farsi and Lee (2008) reported that using 0 % solvent (water) resulted in 81.30 mg/g of phenolic compounds while using 50% acetone resulted in 159.30 mg/g of phenolic compounds. Do et al. (2014) reported that the total flavonoids yield from *Limnophila aromatic* was 19.22 mg/g using 50% acetone and 29.34 mg/g using 75% acetone while using 50% ethanol resulted in 17.19 mg/g and 75% ethanol resulted in 19.47 mg/g. Sultana et al. (2009)

reported that using 80% ethanol obtained a total flavonoids yield 0.0314 mg/g from *Azadirachta indica* bark.

In this study, the total flavonoids obtained from ajwa date fruit was 15.53 mg/g using acetone as a solvent at a concentration of 75%. These results are within the range reported in the literature (0.0258 - 159.30 mg/g). The range of solvent concentrations reported in the literature for the extraction flavonoids from date fruit is 0 – 80%. The differences among the solvent concentrations used are due to factors such as reaction time, extraction method, type of solvent and type of material. The acetone concentration of 75% is within the range of solvent concentration reported in the literature.

6.1.5. Effect of Solvent Type

Acetone and ethanol were used as extraction solvents in this study as they are safer compared to other solvents. Acetone is grouped with class three solvents which means that it has the lowest toxicity. When acetone was used as a solvent, higher total phenols and flavonoids yields were obtained compared to these obtained with ethanol at the same extraction conditions (solvent concentrations, reaction temperature, sample: solvent ratio and reaction time). However, a higher antioxidant yield was obtained when ethanol was used compared to that obtained with acetone. Al-Farsi and Lee (2008) and Wypych (2001) reported that phenols and flavonoids have similar extraction conditions and acetone was the best solvent for extraction. Chirinos et al. (2007) and Tabart et al. (2007) found that acetone is the appropriate solvent for proanthocyanidins and tannins extraction. Chew et al. (2011) found ethanol to be the best solvent for the extracting antioxidants from *Orthosiphonstamineus*. Do et al. (2014) reported that using ethanol at high concentration resulted in more antioxidants from *Limnophila aromatic*.

It is evident from the results that the recovery of phenolic compounds was dependent on the solvent used and its polarity. Siddhuraju and Becker et al. (2003) and Sultana et al. (2007) reported that the efficiency of extraction may be affected by the solubility of endogenous compounds in the sample. Alothman et al. (2009) and Naczkand Shahidi (2006) reported that the yield of antioxidants, phenols and flavonoids from plant materials is affected by the solubility of the compounds in the solvent used for the extraction. Tan et al. (2011) stated that the extraction yield and the activities of the extracted compounds depend on the solvent used in the extraction procedure.

6.1.5.1. Antioxidants: Lu and Foo (2000) found that a mixture of acetone and water is a good solvent for polar antioxidants. Anwar and Przybylski, (2012) reported that using 80% ethanol to extract antioxidants from flaxseed (*Linumusitatissimum L.*) resulted in a yield of 109 mg/g while using 100% ethanol yielded 96 mg/g. Abozed et al. (2014) reported that using 50% acetone resulted in an antioxidant yield of 226.2 mg/g from Delta Egypt whole wheat (Gemiza-9)while using 70% ethanol yielded 239 mg/g. Xu and Chang (2007) reported antioxidants yield of 0.0049 mg/g from lentil and 0.0484 mg/g from red kideny using acetone. Kahkonen et al. (1999) extract 4.3 mg/g antioxidants from cucumber leaf using acetone. Chew et al. (2011) reported that the total antioxidants from *Orthosiphon stamineus* was 2.039 mg/g using ethanol. Anwar and Przybylski, (2012) reported that ethanol was the best solvent to extract antioxidants from flaxseed (*Linumusitatissimum L.*) and obtained 96 mg/g.

In this study, the total antioxidants obtained from the ajwa date fruit using ethanol as a solvent was 20.379 mg/g. These antioxidant results are within the range reported in the literature (0.0049 - 239 mg/g). The solvents reported in the literature for the extraction of antioxidants included ethanol and acetone at different concentrations. The optimum solvent type observed in this study is ethanol at 75% concentration. The effectiveness of a solvent is influenced by the reaction temperature, extraction methods, solvent concentration and materials.

6.1.5.2. Phenols: Zhao et al. (2006) stated that acetone and ethanol were good solvent for extracting phenols. Alothman et al. (2009) reported that acetone at 70% concentration resulted in the highest yield of phenols (72.20 mg/g) from *pisang mas* extracts. Tabart et al. (2007) found that 50% acetone was favoured over methanol for extracting phenols from black currant leaves and buds. Anwar and Przybylski (2012) and Sultana et al. (2007) reported that using 80% ethanol resulted in higher phenols from the bark of some plants and flaxseed (*Linumusitatissimum L.*) than using pure ethanol (100%). Abozed et al. (2014) reported that using 50% acetone resulted 2.57 mg/g of phenols from Delta Egypt whole wheat (Gemiza-9) while 70% ethanol resulted in 1.11 mg/g. Kähkönen et al. (1999) reported that using acetone to extract phenols from potato peel resulted in 4.3 mg/g. Anwar and Przybylski (2012) used ethanol to extract phenols from flaxseed (*Linumusitatissimum L.*) and obtained 32.6 mg/g. Xu and Chang (2007) reported that phenols yield from black bean was 5.54 mg/g using acetone and 3.20 mg/g using ethanol.

In this study, the total phenols obtained from ajwa date fruit was 94.803 mg/g using acetone as a solvent at a concentration of 75%. The results are higher than those reported in the literature (1.11 - 72.20 mg/g). The type of solvent and solvent concentration are affected by the reaction temperature, extraction methods and materials.

6.1.5.3. Flavonoids: Zhao et al. (2006) reported higher flavonoids yield with 80% of acetone compared to 80% ethanol. Anwar and Przybylski (2012) reported that using 80% ethanol to extract flavonoids from flaxseed (*Linumusitatissimum L.*) resulted in 3.9 mg/g, while using 100% ethanol resulted in 1.90 mg/g. Abozed et al. (2014) used 50% acetone to extract flavonoids from the Upper Egypt whole wheat variety (Beni-suef-3) and obtained 3.305 mg/g. Chew et al. (2011) reported that the total flavonoids from *Orthosiphon stamineus* was 11 mg/g using ethanol. Xu and Chang (2007) used acetone to extract flavonoids from lentil and obtained 2.01 mg/g while using ethanol resulted in 1.65 mg/g. Anwar and Przybylski, (2012) used ethanol to extract flavonoids from flaxseed (*Linumusitatissimum L.*) and obtained 3.90 mg/g. These differences can be attributed to the different types of samples and extraction procedures used.

In this study, the total amount of flavonoids obtained from ajwa date fruit was 10.71 mg/g using acetone as the solvent at a concentration of 75%. The results obtained in this study are within the range reported in the literature (0.18 - 11 mg/g). The solvent types reported in the literature for the extraction of flavonoids were acetone and ethanol in different concentrations.

6.2. Freeze Drying of Ajwa Date

Freeze drying is a process that works to remove frozen liquid from the sample by sublimation. The quality of the sample after undergoing freeze drying is high, since drying takes place under low temperatures and the frozen liquid is removed under vacuum without disturbing the cellular components (Ratti, 2001). Krokida and Philippopoulos (2006) and Peruck and Materska (2007) stated that freeze drying is considered as a modern dehydration method and its most important benefit is using low temperature which is suitable for the heat sensitive compounds. Genin and René. (1995) and Irzyniec et al. (1995) reported that the best method to remove water with high quality products is vacuum freeze drying. Gerge and Datta (2002) reported that the best dehydration methods to preserve product quality is freeze drying. Ratti (2001) reported that the quality of freeze-dried foods are 4–6 times higher than air-dried foods. Michalczyk et al. (2009) reported that the freeze drying sample contains higher amount of antioxidants than raw samples even with exposed to atmospheric oxygen. Spigno et al. (2006) stated that freeze drying do not have influence on phenols and antioxidants activity. Perez-Gregorio et al. (2011) found that freeze drying increased the flavonoids in onions due to structure changes of the tissue. Similar result were reported by Chang et al. (2006), Lee et al. (2008) and Wojdylo et al. (2009).

6.2.1. Antioxidants

Schauss et al. (2006) reported that the total antioxidant yield from freeze- dried Acai was 26.19 mg/g. Soong and Barlow (2004) reported a total antioxidants yield of 3.49 mg/g from freeze dried mango seed and 1.81 mg/g from freeze dried avocado seed. Mahn et al. (2014) reported that the total antioxidants obtained from broccoli was 399 mg/g using freeze drying. Chan et al. (2009) reported that using freeze drying to dry *A.zerumbet* resulted in 25.3 mg/g antioxidants yield. Ceballos et al. (2012) reported that the total antioxidants obtained from apple using freeze drying was 67.5 mg/g. Chang et al. (2006) reported that freeze drying recovered slightly higher yield of antioxidants (0.87 mg/g) from tomato than fresh tomato (0.85 mg/g). Sablani et al. (2011) reported that a total antioxidants of 0.0199 mg/g from fresh red raspberries and 0.223 mg/g from freeze dried raspberries. They also obtained 0.048 mg/g from fresh blueberries and 0.0231 mg/g from freeze dried blueberries. Sablani et al. (2011) reported that freeze drying recovered higher antioxidant than that obtained from fresh sample. Tomsone and Kruma (2014) reported that the total antioxidants obtained from fresh horseradish leaves (*Armoracia rusticana L.)* was 95.73 mg/g while that obtained from the freeze drying sample was 159.94 mg/g.

In this study, the total antioxidants extracted from freeze dried ajwa fruit at the optimum extraction conditions (sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 55˚C and ethanol at 75% concentration) was 73.10 mg/g. Freeze drying increased the antioxidants yield over that obtained from fresh fruit by 84.60 % (39.60 mg/g for fresh sample to 73.10 mg/g for freeze dried sample).

6.2.2. Phenols

Alonzo-Macías et al. (2013) reported a total phenols obtained of 18.41 mg/g from freeze dried strawberry. Schauss et al. (2006) reported a total phenols yield of 13.9 mg/g from Acai using freeze drying. Chan et al. (2009) reported that using freeze drying to dry *A.zerumbet* resulted in a 25.5 mg/g phenols yield. Soong and Barlow (2004) reported that the total phenols obtained using freeze drying was 1.17 mg/g for mango seed and 0.882 mg/g for avocado seed. Vuthijumnok et al. (2013) reported that the total phenols yield from freeze dried berries was 9.52 mg/g. Tomsone and Kruma (2014) reported that the total phenols yield obtained from fresh horseradish leaves (*Armoracia rusticana L.)* was 23.68 mg/g while that obtained from freeze dried sample was 27.22 mg/g. Sablani et al. (2011) reported a total phenols yield of 18.00 mg/g from the fresh Blueberries and 18.30 mg/g from freeze dried Blueberries. Chang et al. (2006) reported that using freeze drying resulted in slightly more phenols than that obtained from fresh tomato (0.39 mg/g using freeze drying and 0.38 mg/g using fresh tomato).

In this study, the total phenols extracted from freeze dried ajwa date fruit at the optimum extraction condition (sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 65˚C and acetone at 75% concentration) was 312.624 mg/g. Freeze drying increased the phenols yield over that obtained from fresh fruit by 55.70 % (from 200.784 mg/g for fresh fruit sample to 312.624 mg/g for freeze dried sample).

6.2.3. Flavonoids

Vuthijumnok et al. (2013) reported that the total flavonoids yield from freeze dried berries was 2.98 mg/g. Pérez-Gregorio et al. (2011) reported that the total flavonoids yield obtained from onion using freeze drying was 0.402 mg/g. Alonzo-Macías et al. (2013) reported that the total flavonoids yield obtained from freeze dried strawberry was 3.99 mg/g. Schauss et al. (2006) reported that the flavonoids yield from Acai was 3.19 mg/g using freeze drying. Tomsone and Kruma (2014) reported that the total flavonoids obtained from fresh lovage leaves (*Levesticum officinale L.*) was 29.65 mg/g while that obtained from freeze dried sample was 61.78 mg/g, and that obtained from horseradish leaves

(*Armoracia rusticana L.)* was 58.89 mg/g while that obtained from freeze dried sample was 61.780 mg/g. Chang et al. (2006) reported that freeze drying recovered higher yield of flavonoids (0.081 mg/g) from tomato than fresh tomato (0.047 mg/g).

In this study, the total flavonoids extracted from freeze dried ajwa date fruit at the optimum extraction conditions (sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 65˚C and acetone at 75% concentration) was 45.212 mg/g. Freeze drying increased the flavonoids yield over that obtained by 29.97 % (from 34.786 mg/g for fresh sample to 45.212 mg/g for freeze dried sample).

6.3. Total Antioxidants, Phenols and Flavonoids from Different Date Varieties

The date sample obtained from Saudi Arabia which is most popular and consumed dates (ajwa, sukkari, red sukkari, Khalas and sofry).

6.3.1. Antioxidants

Ghiaba et al. (2014) reported that the total antioxidants from date fruit in Algerian ranged from 0.0331 mg/g to 0.0852 mg/g with the lowest value obtained from Taf variety and the highest value was obtained from Dn variety. Mansouri et al. (2005) studied seven Algerian date fruit varieties and reported total antioxidants ranging from 0.08 to 0.22 mg/g. Al-Farsi et al. (2005) studied three Omani date fruit and reported total antioxidants ranging from 0.020 to 0.031 mg/g. Vinson et al. (2005) found that American date fruits had significantly greater contents of ascorbic acid, b-carotene and vitamin E than other date fruits. Biglari et al. (2007) reported that the jiroft date cultivated from Iran had 0.057 mg/g antioxidants while kharak date had 1.25 mg/g antioxidants. Allaith (2008) found that no differences in antioxidants among tamer date locally grown in Bahrain and those imported from Saudi Arabia and Tunisi.

In this study, the total antioxidants yield from different date varieties extracted at the optimum extraction condition (sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 55˚C, ethanol at 75% concentration and freeze drying sample) were in following order: Ajwa (73.10 mg/g), khalas (62.85 mg/g), sukkari (62.45 mg/g), red sukkari (61.10 g/mg) and sofry (60.39 mg/g).

6.3.2. Phenols

Allaith (2008) reported that the amount of total phenols vary between date cultivars and the ripening stages of fruit. The author reported a total phenols of 2.263 mg/g for fresh wet Mawaji date at rutab stage and 1.141 mg/g for fresh wet Berhi date. Myhara et al. (1999) studied the relationship between the tannin contents of Omani date fruits and their astringency in Khala satge and found that the khalal stage and tamer stage produced the highest tannin. Al-Farsi et al. (2005) reported a total phenols for several Omani tamer varieties ranged between 2.17 and 3.43 mg/g. Biglari et al. (2007) reported 0.0741 mg/g total phenols for the date fruit Bam variety and 0.0085 mg/g total phenols for the kabkab variety. Ghiaba et al. (2014) reported that the total phenols from date fruit ranged from 0.095 mg/g to 0.230 mg/g and the lowest was obtained from the Taf variety and the highest was obtained from the Tam variety.

In this study, the total phenols yield from date varieties obtained from Saudi Arabia at optimum extraction condition (sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 65˚C, acetone at 75% concentration and freeze drying sample) were in order following order: Ajwa (355.24mg/g), khalas (252.01 mg/g), sukkari (204.65 mg/g), red sukkari (200.59 mg/g) and sofry (184.05 mg/g).

6.3.3. Flavonoids

Al-farsi and Lee (2008) obtained a total flavonoids of 54 mg/g from date fruit seed. Biglari et al. (2007) obtained a total flavonoids of 0.0279 mg/g from Bam variety and 0.0162 mg/g from kabkab. Kchaou et al. (2014) reported that the total flavonoids from Tunisia date were 2.13 mg/g for allig, 0.58 mg/g for deglet nour and 1.50 mg/g for bejo. Manickavasagan et al. (2012) reported a total flavonoids from date fruit of 0.33 mg/g for fardh and 0.2729 mg/g for kassab. Louaileche et al. (2015) obtained a total flavonoids of 0.952 mg/g from ourrous and 0.286 mg/g from outkbala, both cultivated in Algerian. Chibane et al. (2007) reported a total flavonoids of 0.226 mg/g for frezza and 0.692 mg/g for mesh degla.

In this study, the total flavonoids yield from date varieties obtained from Saudi Arabia at optimum extraction condition (sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 65˚C, acetone at 75% concentration and freeze drying sample) were in order following order: Ajwa (57.52mg/g), khalas (52.47 mg/g), sukkari (43 mg/g), red sukkari (41.65 g/mg) and sofry (41.22 mg/g).

CHAPTER 7. RECOMMENDATIONS

1. The yields of antioxidants, phenols and flavonoids in dates change during the ripping period. Therefore, the yield of these compounds should be determine during the ripping stage in order to determine the appropriate time for harvesting the dates used for extraction of these compounds.
2. A large percentage of dates (23%) are not suitable for human consumption because of their low quality and insect contamination, it is not clear whether the dates that are not suitable for human consumption have different yields of antioxidants, phenols and flavonoids. Therefore, the antioxidants, phenols and flavonoids yield in dates not suitable for human consumption (waste date) should be investigated and compared to those used for human consumption.
3. The effects of long reaction times and high temperatures on the activities and stability of the antioxidants, phenol and flavonoids should be investigated.

CHAPTER 8. CONCLUSIONS

The effect of reaction times (1, 2, 3 and 4h), reaction temperatures (25, 35, 45, 55 and 65°C), sample: solvent ratio (1:20, 1:40 and 1:60), solvent concentration (0, 50, 75%) and solvent type (acetone and ethanol) on the antioxidants, phenols and flavonoids yield were evaluated. A comparison between fresh ajwa date and freeze dried ajwa date was carried out at the optimum extraction condition. The antioxidants, phenols and flavonoids contents of five date verities obtained from Saudi Arabia (ajwa, sukkari, red sukkari, khalas and sofry) were studied. The following are the conclusions drawn from the study.

1. The reaction times, reaction temperatures, sample: solvent ratio, solvent concentrations and solvent type had significant effects on the antioxidants yield at the 0.001 level. There appeared to be significant interaction among these parameters.
    - (a) Increasing the reaction time from 1 to 3 h increased the antioxidants yield (from 18.26 mg/g to 20.26 mg/g) and a further increase in the reaction time decreased antioxidants yield (from 20.26 mg/g to 20.20 mg/g).
    - (b) Increasing the reaction temperature from 25 to 55 °C increased the antioxidants yield (from 17.12 mg/g to 22.76 mg/g) and a further increase in the reaction temperature decreased antioxidants yield (from 22.76 mg/g to 21.36 mg/g).
    - (c) Increasing the sample: solvent ratio from 1:20 to 1:60 decreased the antioxidants yield (from 24.23 mg/g to 15.23 mg/g).
    - (d) Increasing the solvent concentration from 0 to 75% increased the antioxidants yield (from 12.17 mg/g to 28.39 mg/g).
    - (e) Using ethanol as a solvent produced higher antioxidants yield compared to acetone (20.37 mg/g for acetone and 18.70 mg/g for ethanol).
    - (f) The optimum extraction conditions were sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 55˚C and ethanol at 75% concentration.

2. The reaction times, reaction temperatures, sample: solvent ratio, solvent concentrations and solvent type had significant effects on the phenols yield at the 0.001 level. There appeared to be significant interaction among these parameters.
    (a) Increasing the reaction time from 1 to 3 h increased the phenols yield (from 8.08 mg/g to 9.72 mg/g) and a further increase in the reaction time decreased phenols yield (from 9.72 mg/g to 9.46 mg/g).
    (b) Increasing the reaction temperature from 25 to 65 °C increased the phenols yield (from 6.58 mg/g to 10.94 mg/g).
    (c) Increasing the sample: solvent ratio from 1:20 to 1:60 decreased the phenols yield (from 12.35 mg/g to 8.96 mg/g).
    (d) Increasing the solvent concentration from 0 to 75% increased the phenols yield (from 6.09 mg/g to 12.77 mg/g).
    (e) Using acetone as a solvent produced higher antioxidants yield compared to ethanol (9.48 mg/g for acetone and 8.46 mg/g for ethanol).
    (f) The optimum extraction conditions were sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 65˚C and acetone at 75% concentration.
3. The reaction times, reaction temperatures, sample: solvent ratio and solvent concentrations had significant effects on the flavonoids yield at the 0.001 level. There appeared to be significant interaction among these parameters.
    (a) Increasing the reaction time from 1 to 3 h increased the flavonoids yield (from 0.90 mg/g to 1.20 mg/g) and a further increase in the reaction time decreased flavonoids yield (from 1.20 mg/g to 1.11 mg/g).
    (b) Increasing the reaction temperature from 25 to 65 °C increased the flavonoids yield. (from 0.977 mg/g to 1.36 mg/g).
    (c) Increasing the sample: solvent ratio from 1:20 to 1:60 decreased the flavonoids yield (from 1.44 mg/g to 0.78 mg/g).
    (d) Increasing the solvent concentration from 0 to 75% increased the flavonoids yield (from 0.58 mg/g to 1.55 mg/g).
    (e) Using acetone as a solvent produced higher antioxidants yield compared to ethanol (1.07 mg/g for acetone and 1.05 mg/g for ethanol).

(f) The optimum extraction conditions were sample: solvent ratio of 1: 20, reaction time of 3 h, reaction temperature of 65˚C and acetone at 75% concentration.

4. Freeze drying resulted in higher antioxidant, phenol and flavonoids yields.
   (a) The antioxidants yield increased from 39.60 mg/g to 73.10 mg/g.
   (b) The phenols yield increased from 200.784 mg/g to 312.624 mg/g.
   (c) The flavonoids yield increased from 34.786 mg/g to 45.212 mg/g.
5. Ajwa dates had the highest antioxidants, phenol and flavonoids followed by khalas, sukkari, red sukkari and sofry.
   (a) Ajwa had 73.10 mg/g antioxidants, 355.24mg/g phenols and 57.52mg/g flavonoids.
   (b) Khalas had 62.85 mg/g antioxidants, 252.01 mg/g phenols and 52.47 mg/g flavonoids.
   (c) Sukkari had 62.45 mg/g antioxidants, 204.65 mg/g phenols and 43 mg/g flavonoids.
   (d) Red sukkari had 61.10 g/mg antioxidants, 200.59 mg/g phenols and 41.65 g/mg flavonoids.
   (e) Sofry had 60.39 mg/g antioxidants, 184.05 mg/g phenols and 41.22 mg/g flavonoids.

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