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Manufactured Aggregate from Cement Kiln Dust

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Abstract

This paper presents the results of a laboratory study that evaluates the geotechnical and geo-environmental properties of a manufactured aggregate derived from a cement kiln dust (CKD). The aggregate manufacturing process involves accelerated carbonation technology (ACT), which has been used to treat contaminated soils at trial-scale. The process is operating at commercial scale in the United Kingdom, producing aggregate from thermal residues. The ACT process relies on the accelerated reaction of carbon dioxide with the calcium oxide in the CKD material in the presence of water. No additional binder is used in this study, instead relying solely on the formation of carbonate to form the aggregate.

In this paper, the aggregate manufacturing process is briefly described. To explore future potential construction applications of the aggregate, several geotechnical test results are used to assess strength and durability (i.e. individual particle strength, internal shear strength of the particle assemblage, wet/dry testing, freeze/thaw testing). Screening tests for the aggregate's geo-environmental characteristics are discussed (metal leaching, dissolved heavy metal adsorption and hydraulic conductivity) to further assess potential uses. It is shown that the aggregate studied has adequate properties for a variety of construction applications, but is unsuitable for use in freezing and thawing environments.

Keywords

Ash utilization, Contaminated material, Strength and testing of materials

List of Notation

ACT is Accelerated Carbonation Technology
CID is consolidated isotropic drained
C_C is coefficient of curvature
C_u is coefficient of uniformity
CKD is cement kiln dust
D is aggregate diameter
D_{10} is aggregate diameter at which 10 percent of sample is finer
D_{30} is aggregate diameter at which 30 percent of sample is finer
D_{60} is aggregate diameter at which 60 percent of sample is finer
SP is poorly graded sand
ICP-OES is inductively coupled plasma optical emission spectrometry
ICP-MS is inductively coupled plasma mass spectrometry
LOI is loss on ignition
S is compressive strength of aggregate
SSD is saturated surface dry
XRF is x-ray fluorescence
WP-XRF is wavelength dispersion Fluorescence
MIP is Mercury intrusion porosimetry
P is absolute pressure
T_s is surface tension
θ is contact angle between the mercury and the soil
ϕ is the drained internal angle of friction
LVDT is linear variable differential transformer
PC is failure load of the individual aggregate
σ'_{1f} is maximum effective major principal stress at failure
σ'_{3f} is maximum effective minor principal stress at failure

1. Introduction

Accelerated carbonation technology (ACT) utilises fine-grained mineral and waste materials in the production of engineering products, including lightweight and relatively low strength aggregates (Gunning et al., 2009). The process is patented for commercially use in the United Kingdom (UK) to produce aggregate from industrial waste for use in concrete block manufacturing (CIWM, 2014). In essence, ACT involves a chemical reaction with water and calcium-based minerals in the waste and added carbon dioxide (CO_2). The carbonate-based reaction products can cause rapid hardening of the waste, as cementation between grains forms agglomerations or monolithic materials.

In nature, carbonation is caused by the reaction between alkaline materials and atmospheric CO_2, however, due to the low CO_2 concentration in the atmosphere, carbonation generally proceeds slowly. ACT uses a higher concentration of CO_2 to complete this same reaction within hours (Costa et al., 2007; Huijgen and Comans, 2005), or minutes when applied at scale. Water is added in the ACT process to form a thin aqueous film around the particles of the material being carbonated. The water layer reacts with the calcium phases in the material to form calcium hydroxide and water. Carbon dioxide injected into the mixture then reacts with the aqueous film, forming carbonic acid. Calcium ions from the calcium hydroxide react with carbonate ions of the carbonic acid to form calcium carbonate. To produce lightweight aggregates, the process is performed via a pelleting process such that aggregates are formed with concentric layers of carbonate with a “hard outer shell”. The carbonation process is exothermic, releasing heat as well as free water (Fernández et al., 2004; Arandigoyen et al., 2006; Domingo et al., 2006).

With respect to waste management practices, the main advantages of the ACT process when applied to waste materials is that: 1) it can produce a useful lightweight aggregate from the waste material, 2) it can be used for sequestration of CO_2, and, 3) it can provide stabilization of some contaminants within a waste material (Fernández et al., 2004). Depending on the chemical composition of the waste material, binders such as cement may be added to promote the carbonation process and/or provide enhanced stabilization.

Waste materials such as cement kiln dust offer significant potential for manufacturing of sustainable aggregates due to its high calcium oxide (CaO) and potential to develop a stronger, more durable aggregate than that currently being developed from other industrial wastes. An additional advantage is the sequestration of CO_2 into a waste stream, resulting in a process that has a carbon ‘footprint’ much lower than sintered, kiln-fired or quarried and transported natural stone (see Table 1).

Global cement production in 2014 totaled 4.3 billion metric tonnes, with 82 million tonnes produced in the United States alone (CEMBUREAU, 2016). Cement Kiln Dust (CKD) is an

alkali-rich waste derived from cement manufacture which is normally sent to landfill if not used for other beneficial reuse products. The last decade has seen significant changes in the management of CKD, thus reducing the amount disposed to landfill (Adaska and Taubert, 2008). In the US, approximately 1 million tonnes of CKD is produced (14kg for every tonne of cement clinker), with 85% of this being landfilled (PCA, 2011). The US cement industry has agreed to reduce the amount of CKD landfilled by 60%, while also curbing CO_2 emissions by 10%, compared with 1990 levels (PCA, 2011). Amongst the largest cement producers, there is an overall drive towards resource and energy efficiency through the Cement Sustainability Initiative (CSI, 2016). Therefore, technologies that can make use of the gaseous and solid wastes from cement manufacture can potentially make a valuable contribution to achieving these goals. In this respect, accelerated carbonation has been demonstrated as a commercially viable means of recycling calcium-rich solid waste streams (such as CKD) as new construction products, whilst permanently sequestering carbon dioxide (Carbon8, 2016).

Throughout the world, the requirement of aggregates for construction purposes (i.e. roadways, concrete, asphalt, etc.) is increasing. Through 2017, global consumption of aggregates is forecast to expand by 5.8% to 53.2 billion metric tons (Freedonia Group, 2013). In the United States alone, demand rose from 809 million in 2011 to 931 million tonnes in 2015 (USGS, 2015). Unfortunately, in many geographical areas, aggregate supply is limited, while in developed countries, environmental permitting of mineral workings is becoming increasingly difficult. Sustainable aggregate manufacturing via ACT is one potential solution to these challenges. New technologies which are able to divert waste from landfills into carbon negative manufactured aggregates help reduce the reliance on natural resources and contribute to a circular economy.

The objective of this paper is to investigate CKD as a waste-based feed-stock for the manufacture of a lightweight carbonated aggregate. No other binders or cement, were added during the ACT process. The aggregate produced will be examined for its physical and chemical properties for suitability for engineering applications, such as concrete aggregate, roadway aggregate, drainage material, or reactive permeable barriers. However, the potential end-use of the manufactured aggregate will depend on the aggregate's particle size distribution, density, strength, durability and environmental performance. The following outlines the methods used to characterize the raw CKD waste and the resulting ACT aggregate produced. Based on the test results from these characterization techniques, potential beneficial re-use applications in construction or environmental applications are then discussed.

2. Experimental Materials and Methods

2.1 Cement Kiln Dust

The CKD utilized in this research was sampled from a Lafarge cement plant in Brookfield (Nova Scotia, Canada), in August 2014.

To gain some understanding of the chemical composition of the CKD material used in this study, two randomly selected samples (nominally 100 g of CKD-1 and CKD-2) were submitted to SGS Mineral Services Geochemical (Lakefield, Ontario, Canada) for x-ray fluorescence (XRF) analyses. Major element and rare earth oxides, and loss on ignition (LOI) were determined using a borate fusion method. Sample preparation involved taking a nominal 0.5 g of sample and forming a homogeneous glass disk by the fusion of the sample and a lithium tertraborate/lithium metaborate mixture. The LOI was determined separately and gravimetrically at 1000 ^{o}C. Disks were analyzed by wavelength dispersion fluorescence (WD-XRF). The test is accredited by the Standards Council of Canada, through the laboratory to which the samples were submitted.

As with the XRF analysis, two random samples (nominally 100g of CKD-3 and CKD-4) were submitted to Dalhousie University's Mineral Engineering Research Centre for total elemental analysis. Sample preparation involved oven-drying the samples at 60^{o}C for 24hr, and then grinding using a mortar and pestle to less than 40 µm. Approximately 2.5g of the sample was then mixed with 5ml of nitric acid (HNO_3), 5ml of hydrofluoric acid (HF) and 2 ml of chloric acid ($HClO_3$), and heated for 12 hours by gradually raising the initial temperature of 25^{o}C to a final temperature of 205^{o}C. When finished, 5ml of hydrochloric acid (HCl) was added to digest the sample for 15 minutes at 120^{o}C followed by the addition of 15ml of deionized water for 15 minutes at 120^{o}C. The digested sample was then diluted and elements analysed by inductively coupled plasma optical emission spectrometry (ICP-OES) using a Varian CCD simultaneous ICP-OES device.

Tables 2 and 3 show the results of the XRF and elemental analyses, respectively, for the CKD. The majority of the CKD consists of calcium, iron, silica, aluminum, magnesium and potassium oxides. Of particular note is the 42% CaO content. The high loss on ignition (LOI) for this sample indicates that the CKD has relatively low amounts of reactive free lime (Bhatty et al., 1996; Asha and Maria, 2006; Mackie et al., 2010). The approximately 10 percent of oxides unaccounted for are likely to be sulfur oxides (not measured by the method employed), as suggested by results in Table 3.

2.2 Pelletising Process Used To Produce The ACT Aggregates

The moisture content, premixing time and rotation speed can all affect the size and/or strength of the aggregate produced during the ACT process (Russell et al., 2001; Beruto et al., 2005; Gunning, 2011). In this research, ACT aggregate particle size was controlled by the premixing time and water content. Table 4 shows the proportions of CKD and water used in the mixing process (based on previous trial and error by the authors). Batching of aggregates consisted of pre-mixing 400g (dry mass) of CKD with 130g water using a laboratory paddle mixer. During this phase of the mixing process, carbon dioxide was added for 90 seconds (60 seconds at 50 rpm

speed mixing and 30 seconds at 120 rpm speed mixing). Subsequently, an additional 60g of CKD was added to the mixer and mixed for an additional 90 seconds at 50 rpm to produce more rounded aggregates. The mix was then placed in a rotating drum for 20 minutes at 50 rpm drum speed to make the aggregate (based on work reported by Gunning, 2011). Figure 1 shows a photo of the aggregate produced by this ACT process.

Unless otherwise noted, ACT aggregates in this study were cured under laboratory temperature (20°C±2°C) and relative humidity (~25%) conditions for at least 28 days prior to being subjected to any testing. This air curing is representative of likely field curing conditions that would occur (i.e. in a silo). Figure 2 shows the resulting grain size distribution of three subsamples taken from the ACT aggregate produced from the pelleting process described above. As shown in Figure 2, and quantified in Table 5, there was very little variability in the aggregate grain size distribution produced using this process. The poorly graded sand, as determined from ASTM (2011c), had a relative density and moisture absorption properties as reported in Table 5.

2.3. Aggregate Evaluation Tests

2.3.1 Internal Aggregate Porosity via Mercury Intrusion Porosimetry

Various factors such as porosity, pore size distribution and absorption can influence the durability properties of an aggregate (Richardson, 2009; Mindess et al., 2003). The ability of the pores in the aggregate to redistribute moisture during freeze/thaw or wet/dry events relate to many factors, but internal pore size distribution plays an important role. As discussed by Richardson (2009), aggregates with pore sizes ranging from 0.1 μm to 10 μm seem prone to freeze-thaw problems, as water distributed in these pore sizes have difficulty escaping during freezing events. It is suggested by Richardson (2009) that in aggregate with larger pores, water has the ability to leave the aggregate during freezing events and hence are less likely to be damaged.

To assess the internal pore size of the ACT aggregate, mercury intrusion porosimetry (MIP) was used. Simms and Yanful (2004) review the concepts of MIP testing for examining internal pore sizes of soils. MIP involves forcing mercury into the pores of a moisture-free material under increasing amounts of pressure. The pressure required to push the mercury (a non-wetting fluid), into cylindrical-shaped pores with a diameter, d, is given by the Washburn equation (Washburn, 1921):

$$P = \frac{4T_s cos\theta}{d} \quad [1]$$

Where:

P: absolute pressure

T_s = surface tension

θ = contact angle between the mercury and the soil

Prior to testing, samples were dried at a temperature of 150 °C for at least 24 hr to remove fluid from the pores. Several aggregate particles were weighed and placed in the sample holder prior to placement in the porosimeter (Poremaster 60, Quantachrome, USA). The sample cell was then filled with mercury and the pressure in the sample cell was increased incrementally and the volume of mercury intruded into the pores was recorded. Equation 1 was used in combination with these measurements by the Poremaster software to develop a relationship between intruded pore volume and pore diameter for the ACT aggregate.

2.3.2 Strength

2.3.2.1 Individual Particle Strength

To gain perspective on the strength of individual aggregate particles, a single pellet compressive strength test, specified in ASTM (2011a) for use with formed catalyst shapes, was used to determine strengths for single aggregate particles in this study. The particle strength was calculated using equation (2) as described by Arslan and Baykal (2006) and Li et al. (2000). Each aggregate diameter was measured at three different axes by Vernier caliper (precision 0.01mm) and the average diameter calculated. The single pellet strength was then measured using Wykeham Farrance loading frame and proving ring (capacity 4452 N, precision of 1N). The reported aggregate strength was calculated from the mean value of at least 10 aggregate particles. Aggregates were tested at 7 days, 14 days and 28 days of air curing.

$$S = (2.8\ P_C) \,/\, (\pi D^2) \qquad [2]$$

where

S : the aggregate computed compressive strength (MPa)

P_C : the failure load (MN)

D : the average aggregate diameter (m)

2.3.2.2 Particle Assemblage Shear Resistance via Drained Triaxial Testing

Any application in which an aggregate will be subjected to shear stresses (i.e. roadways, drainage on slopes, etc) will require knowledge of the drained internal angle of friction, ϕ', of the material. To estimate ϕ', consolidated isotropic drained (CID) triaxial testing was performed on the ACT aggregate assemblage under a relatively loose compaction state (i.e. a conservative approach) to establish its shear resistance under confining stress. To meet ASTM (2011b) requirements for particle size, the material described in Figure 2 was passed through a 4.75 mm sieve prior to testing. Sample preparation involved placing a sample membrane on the split mold and filling the mold and membrane partially with de-aired water. The ACT aggregates were then placed in equally spaced layers in the split mold, by allow the aggregate to free fall through the water. After the placement of a layer of ACT aggregate, light tamping of the aggregate was performed 10 times in a circular pattern using a wooden rod (49 g, 3 cm end

diameter) dropped from a height of 5 cm. This was repeated for each of the 6 layers such that the total compaction energy was 1.3 KJ/m^3. The final specimen diameter was 70 mm and the height 150 mm.

Triaxial testing used an effective confining pressure of 35 kPa followed by a saturation stage (ASTM, 2011b). After saturation was completed, the drainage lines were opened and the samples were then consolidated under the desired effective confining pressure (i.e.100 kPa, 150 kPa, 200 kPa or 250 kPa). Consolidation was deemed to be complete when sample volume change, as measured using a S-500 Triaxial/Permeability panel manufactured by Durham GeoSlope indicator (USA), ceased. Axial loading of the sample then began at a strain-controlled rate of 2 mm/min. This strain rate met ASTM (2011b) requirements for drainage during shear. Failure of the sample was either taken as the maximum value of the major principal stress, σ_1, or at 15% axial strain if maximum value was not reached. Axial displacement of the specimen during testing was measured with a linear variable differential transformer (LVDT) (0.01 – 10.00 mm range; MPE, United Kingdom) while axial load on the sample was measured with a 1 kg capacity load cell. LVDT and load cell measurements were recorded using a GDS data acquisition system and GDS software (GDS Instruments, UK). After the completion of the triaxial test, the specimen was air-dried and the physical appearance of the aggregate visually observed.

Failure stresses from triaxial tests were plotted in p'-q' space. The definition of p' and q' are as previously described by Lambe and Whitman (1969):

$$p' = \frac{\sigma'_{1f}+\sigma'_{3f}}{2} \quad [3]$$

$$q' = \frac{\sigma'_{1f}-\sigma'_{3f}}{2} \quad [4]$$

where:

σ'_{1f} = maximum effective major principal stress at failure

σ'_{3f} = maximum effective minor principal stress at failure

σ'_{1f} - σ'_{3f} = effective deviator stress at failure

2.3.3 Durability

2.3.3.1 Particle Breakage Evaluation

Natural carbonate sands have shown the propensity to be susceptible to particle breakage under high compression stresses (e.g. Altuhafi and Coop, 2011). Similar behaviour may be anticipated by the carbonated aggregates presented in this research. Researchers have used various types of tests to evaluate the particle breakage of aggregate assemblages (e.g. Lee and Farhoomand, 1967; Valsangkar and Holm, 1999), but testing usually involves some form of compression or shear test on the sample and monitoring changes in particle size before and

after testing. In this research, the change in grain size of the ACT aggregate after isotropic compression in a triaxial cell was used to evaluate particle breakage (see Lee and Farhoomand, 1967). Although not a standard method, it has been used by other researchers (e.g. Hardin, 1985) to demonstrate particle breakage of aggregates. Similar to the triaxial testing sample preparation mentioned previously, the material shown in Figure 2 was passed through a 4.75 mm sieve prior to testing, compacted in a split mold and subjected to saturation under an effective confining pressure of 35 kPa. The sample was then subjected to consolidation under an effective confining pressure of 300 kPa (a mid-range stress level that would represent a typical upper bound stress for construction applications). The grain size curves of the aggregate were compared before and after the triaxial consolidation to assess any particle breakage that may have occurred.

2.3.3.2 Wet/Dry Cycling

The slake durability test is useful in determining the durability of rocks or weak aggregate under wetting and drying conditions (ASTM, 2008). The testing in this research involved placing a known dry weight of the ACT aggregates (400 g) into the drum of the apparatus. The drum consists of a mesh with a nominal aperture size of 2.3 mm, with a length of 100 mm and a diameter of 140 mm. The drum was then rotated with the water level approximately 20 mm below the drum axis. The drum was rotated at a speed of 10 rpm for a period of 10 minutes. After slaking for 10 minutes, aggregates were then dried in an oven at a temperature of 105 ^{o}C for up to 6 hrs. The mass of dried samples was weighed to obtain the mass remaining after the first cycle. The test was repeated for another cycle and the dry mass again reported. The percent of sample remaining after these wet-dry cycling tests was then recorded.

2.3.3.3 Freeze Thaw

As noted by Edwards (2006), it is important to understand any changes in aggregates as they undergo freeze thaw. Disintegration of aggregates to finer grain size after freeze thaw could cause unwanted settlement or instability in roadway applications or clogging in drainage applications. Past studies have shown that resistance to freeze/thaw cycling can be influenced by the aggregate moisture content and strength (Edwards, 2006). In this study, the initial aggregate grainsize (400 g sample) as shown in Figure 2 was initially soaked in water for 4 hours. After soaking, the aggregates were allowed to surface dry for approximately 15 minutes. The test aggregate samples were then subjected to 10 or 20 freeze-thaw cycles which included freezing to -17.5 ± 2.5°C for 24 hours and then thawing in a water bath at room temperature (~ 20ºC ± 2°C) for 4 hours (see BSI, 2002a). The temperatures chosen reflect the British Standard BS EN 130055-1 (BSI, 2002a). After the end of the freeze-thaw cycles, the samples were subjected to grain size analysis to compare particle size distribution before and after freeze-thaw cycling. In addition, MIP testing as described in section 2.3.1 was performed on the

aggregate before and after freeze thaw to examine the potential for changes in pore size distribution after freeze thaw.

2.3.4 ACT Aggregate Geo-environmental Properties: Leachate Testing, Single Point, Batch Sorption Testing and Hydraulic Conductivity

Cement kiln dust is normally landfilled due to its high pH, particle size and soluble heavy metals. Thus, the potential for the ACT aggregate to be used in practice will ultimately rely on the management of risk, including leaching of any contaminants (i.e. metals) from the aggregate. To assess metals leaching, a modification of the British Standard for evaluating wastes was used (BSI, 2002b). The procedure involved taking a 20 g sample of the aggregate (not crushed to reflect actual conditions) and combined with 200 g of de-ionised water in glass bottles. The bottles were then rotated for 24 hours after which the resultant leachate was filtered through a 0.45 um filter. Samples were then subjected to metal analyses with inductively coupled plasma optical emission spectrometry (ICP-OES) using a Varian CCD simultaneous ICP-OES device.

If the aggregate were to be used in any type of permeable barrier application for metal retention (i.e. storm water, acid drainage, wastewater etc.), a knowledge of the capacity of the aggregate to sorb metals is required. To establish estimates of sorption properties of the ACT aggregate for potential metal removal applications, a modified version of ASTM C1733 was used. Since the goal of the testing was to quickly examine sorption properties for a range of different metal solutions, the modification involved a single point batch sorption test which was adopted with separate 500µg/l solutions of chromium, zinc, cadmium, arsenic, lead and copper, combined with 10g (dry weight) of the ACT aggregates. Each 200 ml solution and ACT aggregate was placed on a rotating shaker table for 48hr at 100 rpm. After 48hr, the samples were vacuum filtered through a fritted-glass crucible fitted with a filter paper to retain the precipitate. After filtering, 2-3 drops of HNO_3 acid was added to each of the filtered solutions and the samples were then submitted to Dalhousie University Clean Water Laboratory Centre for analysis. Each metal was quantified by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) analysis.

The potential to use the aggregate for drainage in roadway applications, septic system drainage, permeable reactive barriers, etc. requires an estimate of the hydraulic conductivity. The hydraulic conductivity of the aggregate was established via the constant head test method (ASTM, 2006). Hydraulic conductivity tests used ACT-derived CKD aggregates air-cured for 7 days. Samples were loosely tamped into the constant head permeameters using an approximate energy level of 7900 J/m^3.

3. Test Results

3.1 Internal Aggregate Porosity via Mercury Intrusion Porosimetry

Figure 3 shows the pore size distribution of the CKD aggregate, as measured by MIP. It can be seen that the majority of pores range from approximately 0.01 um to slightly less than 1 um. As noted by Richardson (2009), this pore size distribution may be an indication of freeze thaw susceptibility, although other factors such as particle size, particle strength, pore length, mineralogy, absorption, and specific gravity can also have an influence. Also shown on Figure 3 is the pore size distribution of the aggregate after 20 cycles of freeze thaw. It appears there was very little change in the pore structure of the aggregate after 20 cycles of freeze thaw. Rupturing of the pores due to freeze thaw damage would not be detected by this test method as this would result in particle breakage of the aggregate. Hence any major changes in pore size that could cause rupture would only be detected visually or through changes in grain size, and is discussed further in section 3.3.3.

3.2 Strength

3.2.1 Individual Particle Strength

As shown in Figure 4, average individual aggregate strengths reached 0.8 MPa after 7 days of curing, 1.1 MPa after 14 days of curing and 1.2 MPa after 28 days of curing. The variability of the test results are presented in Figure 4 via error bars, showing one standard deviation from the mean. This strength gain is due to carbonation/hydration continuing over 28 days. It should be noted that the strengths reported in Figure 4 are comparable to commercially available sintered light-weight aggregates (e.g. Lytag sintered aggregate strength: 0.4 MPa; see Gunning, 2011).

3.2.2 Particle Assemblage Shear Resistance via Drained Triaxial Testing

Isotropic drained triaxial test results are shown on Figure 5. As would be expected for a re-compacted soil (dry unit weights for the ACT samples tested ranged from 11.5 kN/m^3 to 11.7 kN/m^3), deviator stresses at failure exhibited slow, sustained increases to the maximum axial stress. The subsequent stress paths of the triaxial tests are plotted in p'-q' space (Figure 6). The results of the stress conditions at failure show a linear trend as the confining pressures increased. When fitting a regression line through the origin, the drained friction angle of the aggregate is established as 39^o. It is interesting to note that the shear strength envelope remains linear at the higher confining pressures which suggests that there is no significant breakage of the aggregate at these confining stresses resulting in a degradation of strength. No particle breakage was observed in post-test inspection of the aggregate.

3.3 Durability

3.3.1 Particle Breakage Evaluation

The particle size distributions for the ACT aggregate before and after isotropic consolidation in the triaxial cell at 300 kPa are shown in Figure 7. As can be seen from the figure, very little, if any, changes in the grain size of the sample occurred after the application of the 300 kPa effective stress on the sample. As well, no visual deterioration of the aggregate was observed.

The confining pressure of 300 kPa equates to burial at a depth of approximately 25 m depth (unit weight of 12 kN/m^3 and without a static water table) and preliminary results suggest that damage to the aggregate would be minimal at similar effective stresses and isotropic stress conditions. It should be noted that Lee and Farhoomand (1967) showed that anisotropic loading can influence the amount of particle breakage observed.

3.3.2 Wet/Dry Strength Test

The results of the slake durability test are presented in Table 6. Similar to the limited amount of particle breakage observed after the isotropic compression of the aggregate presented in the previous section, the wet-dry cycling, as performed using the slake durability test resulted in very little change in the ACT aggregate after 2 cycles. Although this is a fairly empirical test method designed for rock materials, according to the Gamble (1971) classification, the aggregate achieved a very high durability rating.

3.3.3 Freeze/Thaw Cycle Tests

As discussed in section 2.3.3.3, two subsamples of aggregates were subjected to 10 or 20 freeze thaw cycles. Grain size distributions before and after the freeze thaw cycling are shown on Figure 8. The grain size distribution before freeze thaw are presented in comparison to the range of grain size observed in Figure 2 (error bars on Figure 8). Unlike the two previous durability tests, freeze thaw cycling appeared to have an effect on the grain size of the aggregate. After 10 cycles of freeze thaw, the 2.5 mm aggregate fractions became noticeably finer, while less change at this fraction was observed at the end of the 20th freeze thaw cycle. Overall there was a general shift in the original grain size distribution curve, moving to a finer grain size. Photos of the ACT aggregate during the f/t cycling process are provided in Figure 9, and show an apparent reduction in the size of the larger aggregates, as opposed to the smaller aggregates. The breakage of the ~ 5 mm particles appeared to result from the flaking of the outer surface of the aggregate and this behavior is subject to further detailed work by the authors.

3.4 ACT Aggregate Geo-environmental Properties: Leachate Testing, Single Point, Batch Sorption Testing and Hydraulic Conductivity

The leaching test results for the ACT aggregate are shown in Table 7. The table shows the results of triplicate analyses of the aggregate (ACT-1,2,3) compared to the average total mass of the metals reported in the CKD sample in Table 3. Results are reported both in terms of mass of metal leached per dry mass of ACT (to allow comparison to Table 3) as well as leachate concentrations. Only the metals detected in Table 3 are reported in Table 7. As can be seen from Table 7, of the metals detected in the ACT aggregate from Table 3, many of these metals did not leach from the aggregate to any appreciable level; many were below the detection limit

of the method. Also shown in Table 7 for comparison to leachate values are groundwater standards that must be met in the province of Nova Scotia (NSE, 2013), Canada (the source of the CKD) for remediation. Ba, Cr, Sr, and Zn leaching was observed for the aggregate, but were below the groundwater guidelines provided in Table 7. As noted from Table 7 results, there was minimal leaching of the metals shown from the ACT aggregate and provides insight into the ability of the carbonation process to stabilise and solidify CKD.

The single point batch tests with the heavy metal ions and the ACT aggregate allowed for a preliminary assessment of the aggregate's adsorptive properties for potential reactive barrier applications. As noted in Table 8, very good removal rates were observed for all of the heavy metals tested. The pH in the flasks ranged from 11.3 to 11.5 after 48 hours of mixing with the ACT aggregate and metal solutions. The alkaline nature of the ACT aggregate likely contributed to the high removal rates as well as the high absorption ability of the aggregate (i.e. internal porosity).

The ACT derived CKD aggregate hydraulic conductivity varied little, as would be expected given the lack of grain size variability in the ACT derived CKD aggregate particle size. Measurement of the hydraulic conductivity on three different samples yielded hydraulic conductivities of 1.5×10^{-4} m/s, 1.5×10^{-4} m/s, and 1.4×10^{-4} m/s. The dry density of the tested samples was 1190 kg/m^3.

4 Discussion

The use of waste to generate new construction materials that are fit for purpose offers significant sustainability gains. A 'cold' process, such as used here, will have low energy requirements and result in an attractive carbon 'footprint'. Other benefits include: 1) a route for the valorisation of waste that would otherwise be disposed of in landfill, 2) a feedstock that can substitute for virgin stone and, 3) carbon dioxide, otherwise released to the atmosphere, is permanently sequestered into the aggregate as a carbonate cement. The objective of this paper was to explore the carbonation potential of CKD to produce an aggregate that has physical and chemical properties that enable beneficial re-use in, for example, concrete, roadway engineering, drainage materials or reactive permeable barriers.

One of the attractive properties resulting from cementation by carbonation is the low density compared to most natural aggregates (a bulk density of 1100 kg/m^{3}compared to >2000 kg/m^3, respectively). As aggregates produced in the geosphere are normally formed at great depth and at elevated temperature, they tend to be dense in nature. However, when using ACT to carbonate waste at or near normal atmospheric temperatures and pressures, the density of the carbonate cemented product is technically "lightweight", although not as lightweight as some sintered or bloated commercially manufactured aggregates (see Table 9 for a comparison). Thus, the CKD-based aggregate is lighter than natural aggregate, but denser (both particle and

bulk) than expanded clay, shale, and glass aggregates and sintered fly ash. The manufactured aggregates given in Table 9 (with the exception of the C8A aggregate) are produced using high energy intensive processes, primarily extreme heating in their production, and have a high 'carbon footprints'.

The individual particle strength of the CKD aggregate is within the range of the other aggregates shown in Table 9, and in recent work by the authors using a modified processing method of CKD aggregate, individual particle strengths greater than 4MPa have been achieved. This suggests improved durability properties with respect to freeze thaw may be realised.

In comparison with Nova Scotian quarried aggregate, with particle strengths ranging from 25 MPa to 300 MPa, the particle strength of carbonated CKD aggregate is low, and its freeze thaw resistance, particle breakage (under wet/dry), compression and shear conditions is not surprisingly inferior. However it would be suitable as fill material for roadway construction given its adequate performance in wet/dry testing and triaxial compression and shear.

In the province of Nova Scotia, the use of "borrow" material for fill is limited little by specification (see NSTIR, 2014) and more by its engineering performance properties and environmental characteristics (NSE, 2013) in determining that an aggregate is "fit for purpose" in roadway construction applications. Given its good drainage and lightweight characteristics, the aggregate would be beneficial as backfill for retaining structures. The low bulk density relative to natural aggregates and adequate internal angle of friction would serve to reduce loads on the retaining wall, resulting in more economical designs (i.e. smaller walls).

Further applications such as in permeable reactive barriers where the sorption of metals is important could be another application for investigation. For the specific metals examined here, over 90% were removed from solution in single point sorption tests on exposure to the CKD aggregate. Metals removal is likely due to the internal porosity available within the aggregate as well as the alkaline aggregate surface which promotes metal precipitation; as such the potential interaction of the aggregate and ground water passing through a permeable barrier would have to be assessed using long term testing/modelling studies.

It has been the authors' experience that the beneficial properties of manufactured aggregates are key to market development. When the technical advantages are combined with government carbon offset incentives (being discussed more frequently in Canada) and the drive to a circular economy (ECCC, 2016), low carbon solutions become attractive to regulators, specifiers and construction materials users alike.

5 Summary and Conclusions

To assess the geotechnical and geo-environmental properties of carbonated aggregate made from CKD, several performance tests were applied to gauge potential re-use options. The sand product was shown to have a mature individual aggregate strength of more than 1 MPa, and upon compaction a friction angle of 39^{o} when subjected to drained triaxial testing, under confining pressures ranging from 100 kPa to 250 kPa. The aggregate displayed a very high durability to wet-dry cycling using the slake durability test and no noticeable changes in grain size when subjected to an isotropic confining compression test. Freeze thaw cycling at -17.5 ±2.5°C for 20 cycles showed a reduction in grain size that warrants further investigation, and future improvement. One-point batch adsorption showed that the ACT aggregate removed 90% of the selected heavy metals from prepared solutions, indicating potential for geo-environmental applications such as permeable reactive barriers. The resulting grain size and hydraulic conductivity of the aggregate suggest it is a reasonably permeable material that may be used in drainage or permeable reactive barrier applications.

Future studies on the aggregate will focus more on its durability to determine its performance under environmental loading and on optimizing its particle size distribution and curing to improve its durability and performance for use in real-world applications.

Acknowledgements

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Figure 1

Click here to download Figure Figure 1 (wide 10 cm, 600dpi).tif

Figure 2

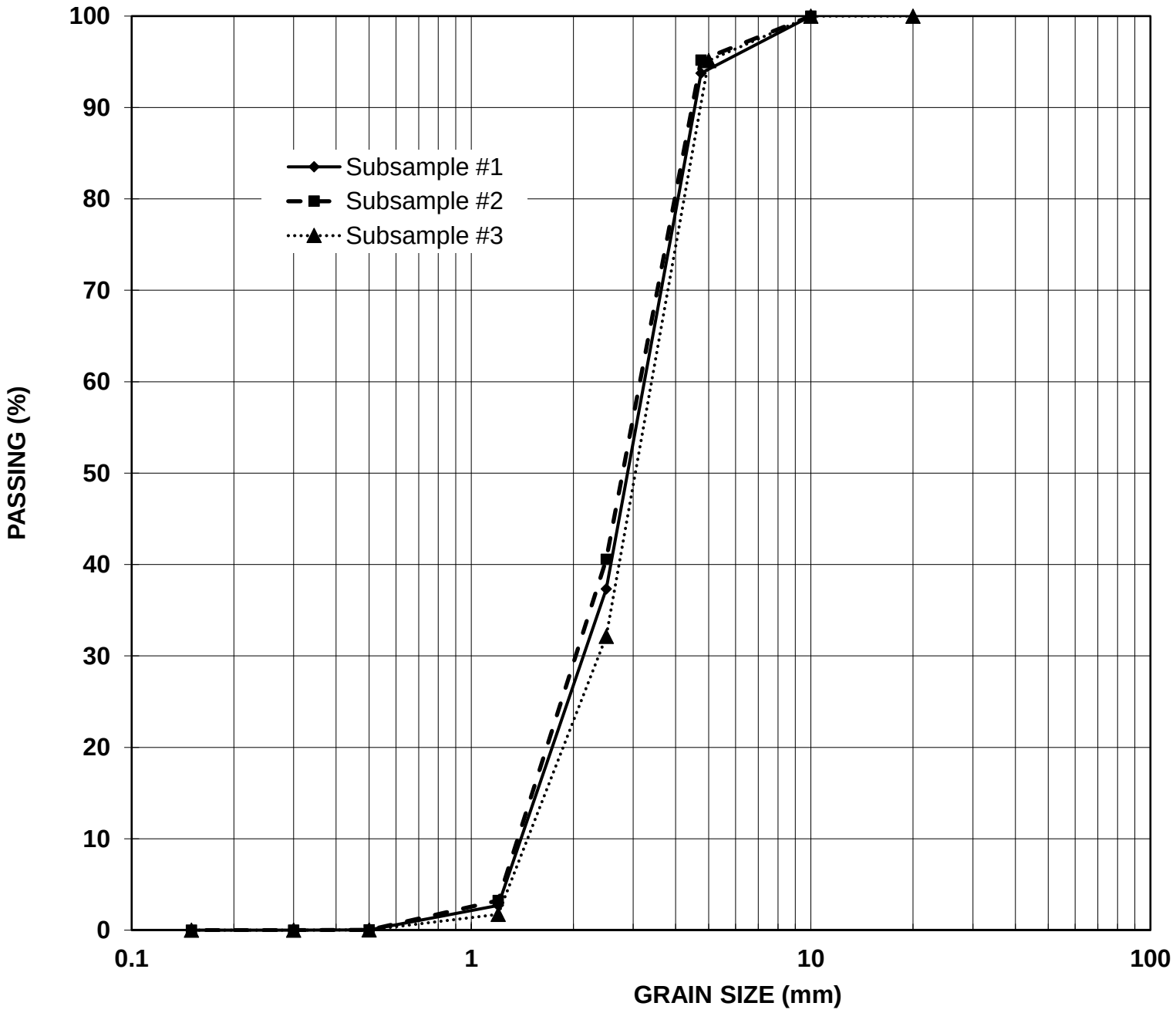

100
90
80
70
60
50
40
30
20
10
0
PASSING (%)
Subsample #1
Subsample #2
Subsample #3
0.1
1
10
100
GRAIN SIZE (mm)

Figure 3

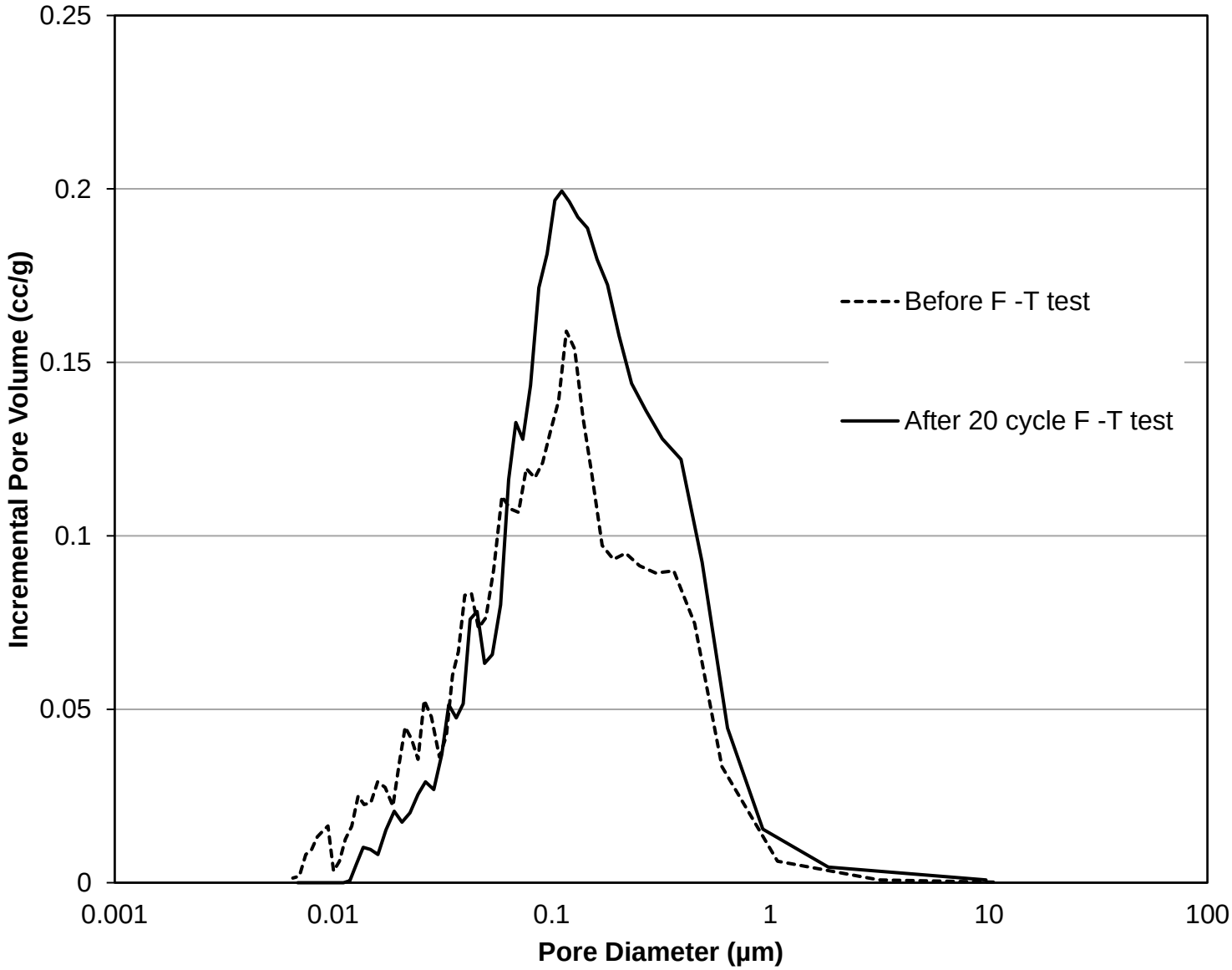

0.25
0.2
0.15
0.1
0.05
0
Incremental Pore Volume (cc/g)
Before F -T test
After 20 cycle F -T test
0.001
0.01
0.1
1
10
100
Pore Diameter (μm)

Figure 4

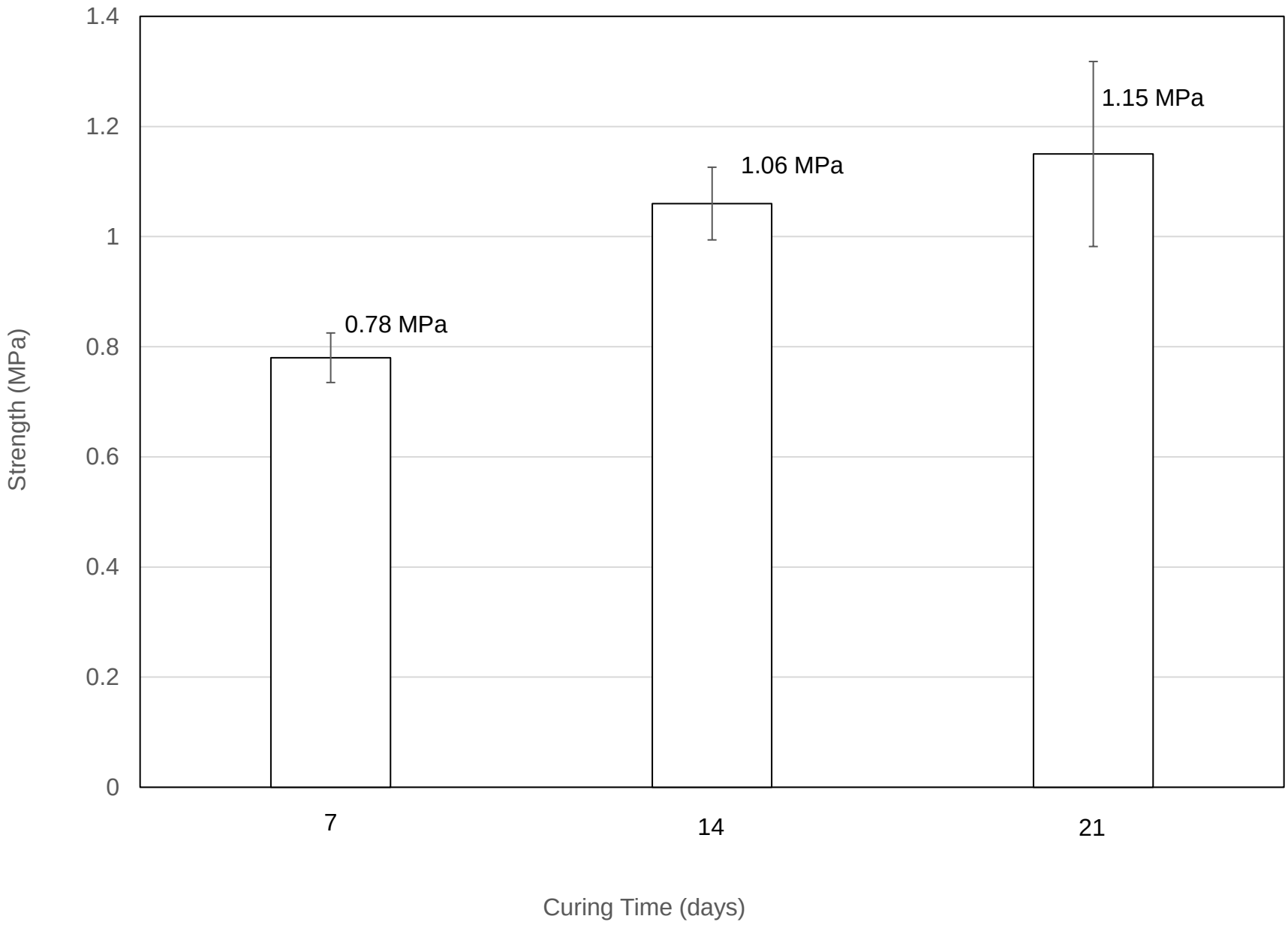

1.4
1.2
1
0.8
0.6
0.4
0.2
0
Strength (MPa)
0.78 MPa
1.06 MPa
1.15 MPa
7
14
21
Curing Time (days)

Figure 5

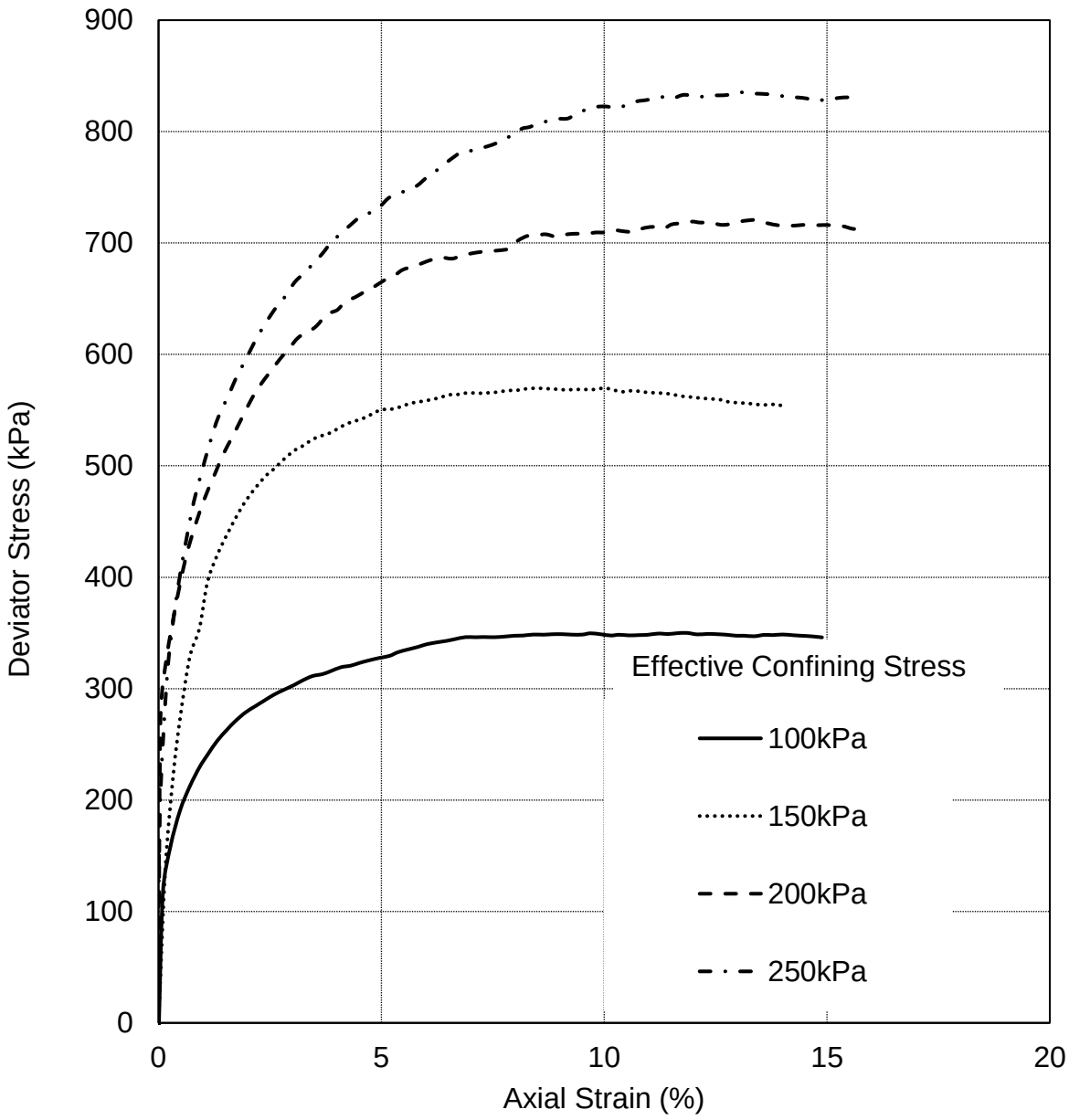

900
800
700
600
500
400
300
200
100
0
Deviator Stress (kPa)
0
5
10
15
20
Axial Strain (%)
Effective Confining Stress
100kPa
150kPa
200kPa
250kPa

Figure 6

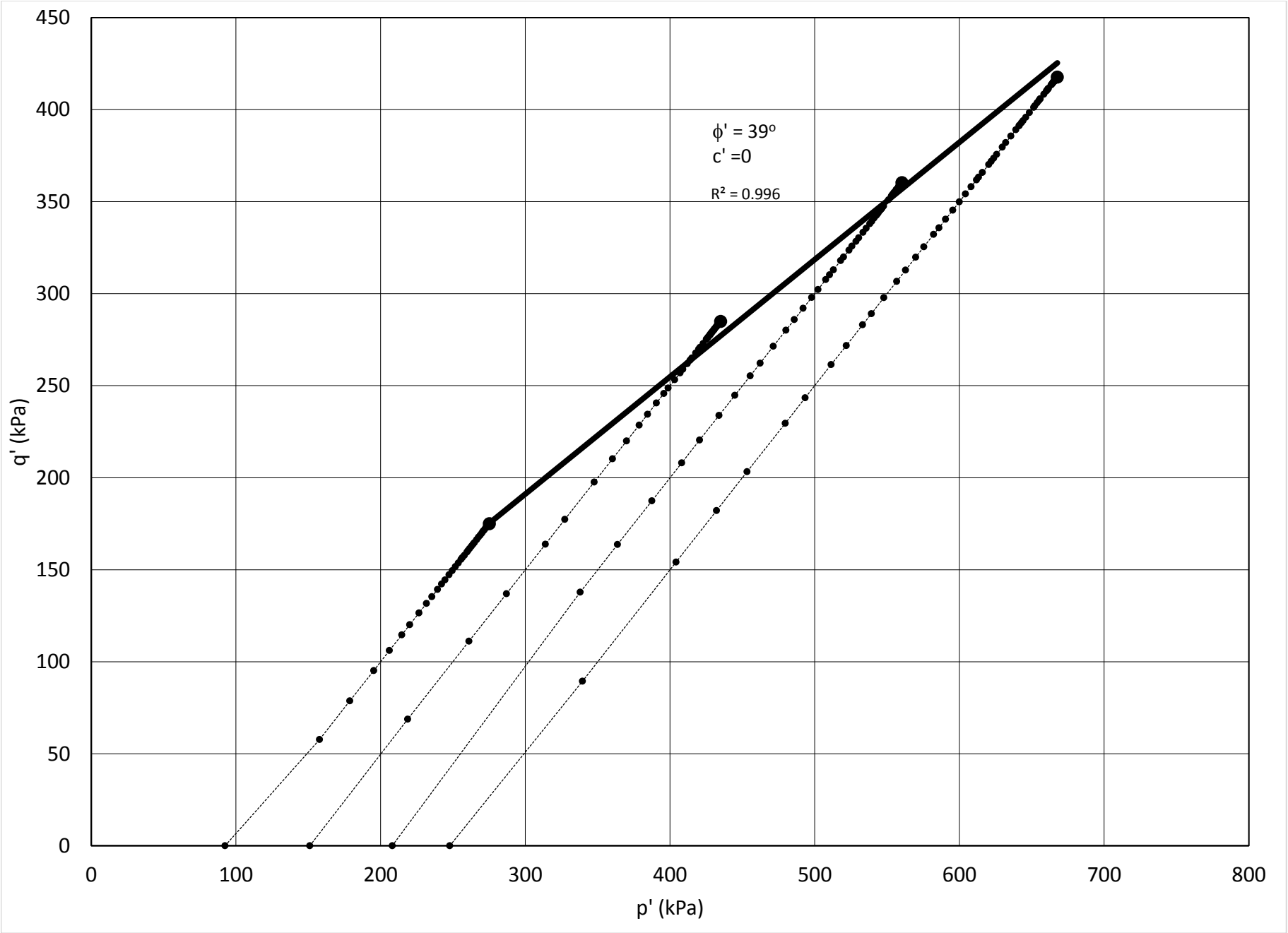

450
400
350
300
250
200
150
100
50
0
q' (kPa)
0
100
200
300
400
500
600
700
800
p' (kPa)
ϕ' = 39°
c' =0
R² = 0.996

FIgure 7

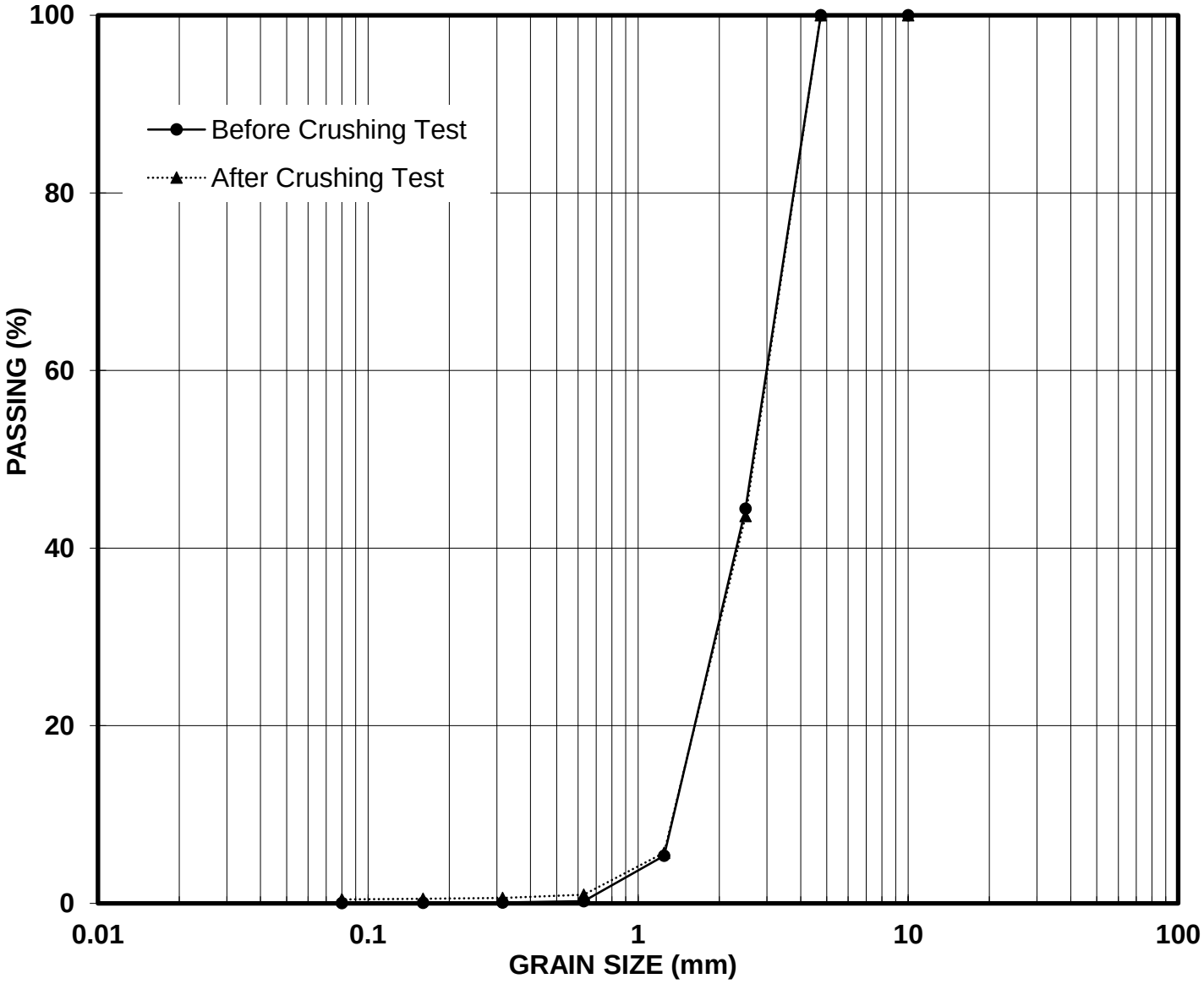

Before Crushing Test
After Crushing Test
PASSING (%)
100
80
60
40
20
0
0.01
0.1
1
10
100
GRAIN SIZE (mm)

Figure 8

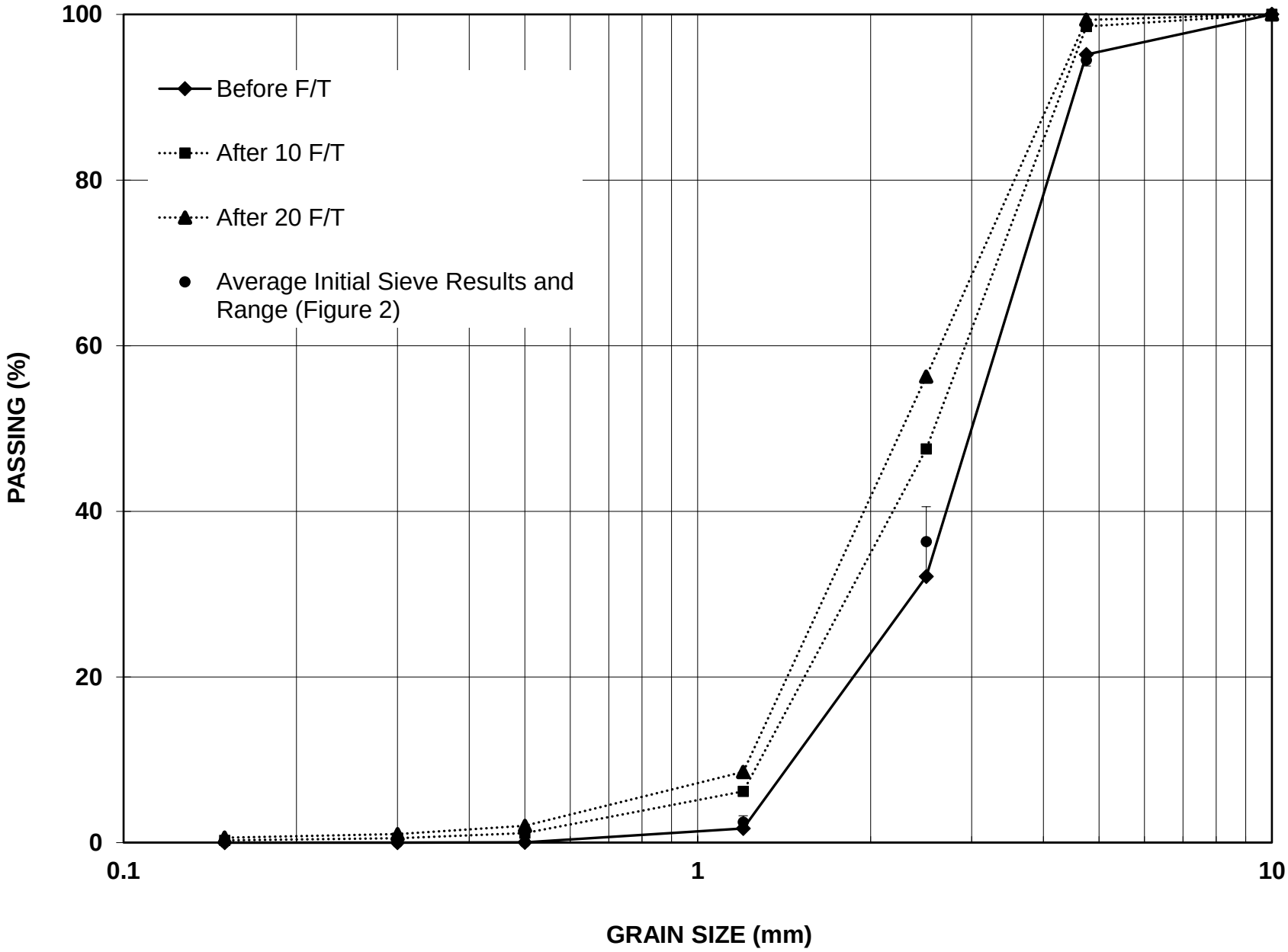
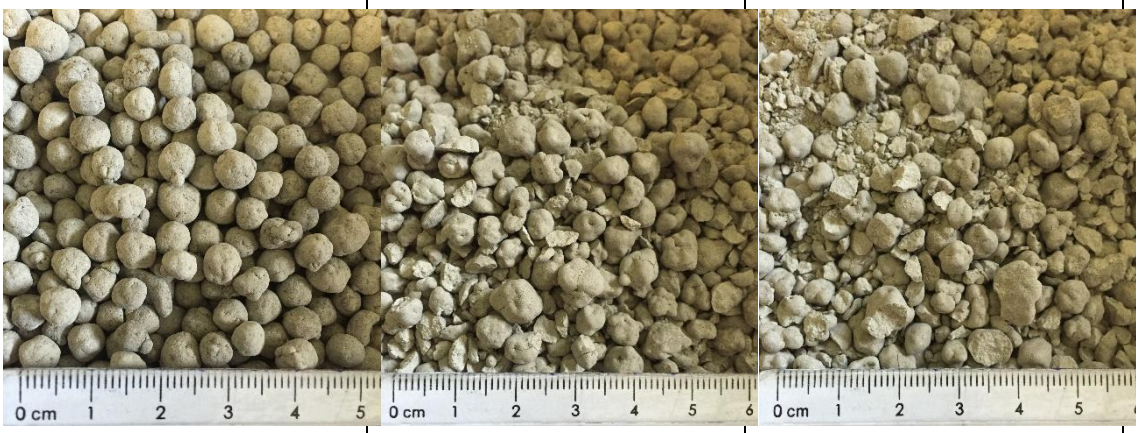

100
80
60
40
20
0
PASSING (%)
0.1
1
10
GRAIN SIZE (mm)
Before F/T
After 10 F/T
After 20 F/T
Average Initial Sieve Results and Range (Figure 2)

Figure 9



ACT aggregate	10 cycle	20 cycle

Table 1. Categories and examples of lightweight aggregate (modified from BS EN 13055: BSI, 2016)

Source Material	**Lightweight Aggregate Type**
Natural Lightweight Aggregate	Pumice, Scoria, Tuff
Manufactured lightweight aggregate from natural source materials	Expanded clay, expanded shale, expanded slate, expanded perlite, expanded vermiculite
Manufactured lightweight aggregate from by-products or recycled source materials	Sintered fly ash, cold-bonded fly ash, foamed blast furnace slag, expanded blast furnace slag, expanded glass, foamed glass
LWA as by-products of industrial processes	Furnace clinker, furnace bottom ash, fly ash

Table 2. Major oxide results for the CKD used in this study.

Oxide	CKD-1 (%)	CKD-2 (%)
CaO	42.2	42.3
SiO_2	13.1	12.9
K_2O	5.5	5.5
Al_2O_3	3.8	3.8
Fe_2O_3	1.3	1.3
MgO	1.1	1.1
Na_2O	0.3	0.3
TiO_2	0.2	0.2
P_2O_5	0.03	0.3
MnO	0.04	0.05
Cr_2O_3	0.01	0.01
V_2O_5	< 0.01	< 0.01
LOI	22.4	22.2
SUM	90.0	89.7

Table 3. Elemental analysis for the CKD used in this study*

Metal	Concentration (mg/kg)		Metal	Concentration (mg/kg)	
	CKD-3	CKD-4		CKD-3	CKD-4
Ag	0.8	0.7	Li	42	42
Al	20470	20800	Mg	6614	6750
Ba	315	310	Mn	329	326
Be	0.6	0.6	Na	2499	2554
Ca	283579	280539	Ni	15	13
Ce	13	13	P	149	150
Co	5	5	Pb	158	156
Cr	10	9	S	43643	43619
Cu	13	11	Sr	355	359
Fe	9275	9058	Ti	1046	1063
K	48887	49380	V	39	39
La	10	10	Zn	19	19
Li	42	42	Zr	27	27

*Note: As, Bi, Cd, Ga, In, Mo, Nb, Sb, Se, Sn, Ta were below detection limits of the instrument.

Table 4. ACT Derived CKD Aggregate Mix Design and Pelleting Production Process

CKD(g)	Water(g)	Pre-mixing (sec)			Pelleting Time in Drum (min)	Drum Rotation Speed (rpm)
460	130	60 @ 50 rpm	30 @ 120 rpm	90 @ 50 rpm	20	50

Table 5. Grain size, relative density and absorption of ACT derived CKD aggregates

Sub-sample No.	D_{10} (mm)	D_{30} (mm)	D_{60} (mm)	C_u	C_c	Group Classification	Relative density* (SSD)	Absorption* (%)
1	1.4	2.0	3.3	2.4	0.9	SP		
2	1.4	2.0	3.3	2.4	0.9	SP	2.1	25.4
3	1.4	2.1	3.3	2.4	1.0	SP		

*Determined from ASTM (2015).

Table 6. Slake durability test results

Before Test (g)	After First Cycle (g)	After Second Cycle (g)	Slake Durability Index (I_d) (%)	Slake Durability Classification (Gamble 1971)
400	397.2	395.3	98.8	Very high durability

Table 7. Leaching test results on ACT aggregate

Metal	Leached Mass per Mass of ACT Aggregate (mg/kg)			% of Average Values of Table 3	Average Leachate Concentrations (ug/L)	Nova Scotia Provincial Guidelines, Tier I Environmental Quality Standards For Groundwater (NSE, 2013)
	ACT-1	ACT-2	ACT-3			(ug/L)
Al	12.9	15.6	23.6	0.1%	1736	-
Ba	0.5	0.5	1.0	0.2%	66	1000
Cr	0.1	0.1	0.1	1.0%	12	25
Cu	0.2	0.3	0.6	3.0%	34	-
Sr	11.8	11.6	12.6	3.4%	1205	4400
Zn	0.2	0.3	0.6	2.0%	38	5000

*Note: Ag, As, Be, Cd, Co, Mn, Ni, Pb, Sb, Se, Sn, Ti, Tl, and V were below detection limits of the instrument.

Table 8. Single point heavy metal ion batch testing.

Heavy Metal Ions	Concentration of Heavy Metal Ion (μg/l)		Removal Rate (%)
	Initial Concentration	Final Concentration	
Chromium (Cr)	507.4	38.8	92.4
Zinc (Zn)	546.8	52.0	90.5
Cadmium(Cd)	534.6	1.3	99.8
Arsenic (As)	511.8	20.2	96.0
Lead (Pb)	480.2	4.6	99.0
Copper (Cu)	450.2	27.7	93.8

Table 9. Comparison of Physical Properties of Various Manufactured Aggregates.

	LECA[1]	Lytag[2]	C8A[3]	Poraver[4]	Solite[5]	This study
Description	Lightweight expanded clay	Sintered fly ash	Carbonation stabilised waste	Expanded glass	Expanded shale	ACT Aggregate from CKD
Bulk Density (kg/m^3)	215-285	750	950-1100	190-400	720-880	1200 (from hydraulic conductivity)
Dry Particle Density	0.4-0.6	1.45	1.94	0.3-0.9	Not specified	2.1 (SSD)
Water Absorption (% by weight)	28	17.5	18.8	14-35	Not specified	25.4
Individual Particle Strength (MPa)	0.89[6]	3.86[6]	3.13[6]	Not specified	Not specified	1.15[6]

Notes: [1]Weber Saint Gobain (2016); [2]Lytag (2016); [3]Carbon8 (2016); [4]Poraver (2016); [5]Nesolite (2016) [6]Evaluated as part of this study using the methods described in section 2.3.2.1