Ceramic Waste Sludge as a Partial Cement Replacement

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ABSTRACT: In this research study, waste ceramic sludge derived from orthopaedic implant mass finishing processes were employed as a supplementary cementitious material (SCM) in the partial replacement of ordinary Portland cement (OPC).The cement was replaced by dried ceramic sludge in powder form accordingly in ranges up to 50%wt. The results demonstrate that the addition of ceramic sludge powder as an active addition to OPC can enhance the mechanical strength of concrete when incorporated in the range of 15-20%. Furthermore, results show improved cohesiveness and a reduction of bleed waters in the concretes fresh phase. The results also show that the overall durability of the concrete was improved through a reduced water absorption capacity. The waste ceramic sludge employed constitutes a significant volume of the source factory's waste stream. This waste is traditionally disposed of in landfill, incurring significant economic and environmental costs. As such, there is demand for research into potential re-use of the material.

KEY WORDS: Waste ground ceramic; Pozzolan; Supplementary cementitious material; Concrete compressive strength.

1 INTRODUCTION

1.1 Cement production and the environment

The environmental impact of cement production has been well documented [1]. Large volumes of virgin raw materials such as limestone and clays are consumed along with the significant greenhouse gas emissions which can be attributed to the production of cement clinker. It has been estimated to take 1.7 tonnes of finite virgin material to produce 1 ton of cement clinker. Cement production facilities also consume enormous amounts of fossil fuels, making cement production an extremely carbon intensive industry [2].

The cement and concrete industry has recognized the environmental impact of cement production which has prompted much research into more sustainable cement production alternatives. The need for research has been further exacerbated by the necessity to comply with Kyoto Protocol driven emission targets [3].

1.2 Alternative materials for concrete and cement

There has been a great volume of studies concerning the replacement of virgin material aggregates for concrete and cement production with non-hazardous post consumer and industrial wastes [4]. Alternative concrete aggregate studies have utilized such materials as construction and demolition wastes [5,6], glass [7,8], plastics [9], rubber [10], municipal incinerator bottom ash [11] and ceramics [12], to mention but a few. Each of these alternative aggregates presents their own unique advantages/disadvantages to the properties of fresh and hardened concrete. The variety of properties affected along with issues relating to the consistency of supply, heightened quality control requirements and conformity related problems [13] have led to reluctance to implement wide scale production of concrete products containing many of these wastes.

Similarly there has been wide scale research into the use of end of life wastes as supplementary cementitious materials otherwise known as pozzolans, for the partial replacement of ordinary Portland cement as a binder for concrete. These studies have considered the replacement of OPC through either, incorporating wastes with virgin materials in the production of cement clinker prior to milling or through the blending of preprocessed wastes with OPC. The latter of these techniques is generally regarded as being the most efficient in terms of the environmental impact.

A wide range of waste materials have been considered, mainly due to their chemical composition being similar to that of known pozzolanic materials. The waste materials which have been considered through these studies vary greatly and include well known SCM's such as silica fume [14], fly ash [15] and granulated blast furnace slag [16]. Other studies have considered potential SCM's such as glass [17,18], rice husk ash [19], municipal sewage sludge ash [20] and fine ground ceramics [21-28].

1.3 Hydraulic reactions in cement

The hydration of cement is an exothermic chemical reaction between cementitious minerals and water which leads to setting and eventual hardening of concrete. In its anhydrous state, cement consists of four main minerals alite, belite, aluminate and ferrite. Of these, alite (C_3S or tricalcium silicate) and belite (C_2S or dicalcium silicate) are the main reactive minerals contributing to concrete strength. In the presence of moisture these minerals will react and produce calcium silicate hydrate C-S-H which is the main strength component phase of the cement reaction. The chemical formula for these reactions can be seen in equations 1 and 2 below.

$$2C_3S + 6H \rightarrow C_3S_2H_3 + 3CH \tag{1}$$

$$2C_2S + 4H \rightarrow C_3S_2H_3 + CH \tag{2}$$

Where;
C= CaO (calcium oxide); S= SiO_2 (silicon dioxide); H= H_2O
CH= $Ca(OH)_2$ (calcium hydroxide)

Calcium hydroxide or lime is a by-product of these reactions which has no cementing or strength giving properties and is prone to chemical attack and leaching. The strength of concrete is very much dependent upon the hydration reaction of which water plays a critical role. The hydration reaction itself consumes a specific amount of water. Typically more water is added to concrete than is needed for full cement hydration. This excess water will remain in the microstructure pore space, ultimately weakening the concrete [29].

1.4 Pozzolanic reactions of SCM's

Natural pozzolanic materials such as volcanic ash and pumicite have been used in hydraulic binders for thousands of years with examples found which date between 5000-4000B.C. [30]. Pozzolanic materials are best defined as siliceous or alumino-siliceous material that, in finely divided form and in the presence of moisture, chemically reacts at ordinary temperatures with calcium hydroxide released by the hydration of Portland cement to form compounds possessing cementing properties [31].

$$\text{Pozzolan} + \text{CH} + \text{H} \rightarrow \text{C-S-H} \tag{3}$$

The pozzolanic chemical reaction with calcium hydroxide can be seen in equation 3. This reaction is much slower than the reactions of alite and belite but has the benefit of replacing porous CH with C-S-H. A well designed cement blend incorporating pozzolanic materials will produce a denser and ultimately stronger and more durable concrete.

1.5 Waste ceramic as a SCM

This preliminary study into the possible pozzolanic characteristics of fine ground waste ceramics, utilizes spent ceramic media which is a waste stream product of mass finishing processes. Mass finishing is a term used to describe a group of abrasive industrial processes by which large lots of parts or components made from metal or other material are processed to achieve a variety of surfaces finishes such as deburring and surface smoothing [32].

Ceramic media is formed through the extrusion and vitrification of wet clay with the addition of abrasives such as aluminium oxide, silicon carbide, quartz or others usually ranging between 80-200um in diameter. In the finishing process the media becomes ground, spent grinding metals are then recovered from the media and the process ultimately produces a fine ceramic sludge waste product. This waste is traditionally then disposed of in landfill, incurring significant economic and environmental costs. Mass finishing processes are used throughout the world in industries such as automotive, aerospace, jewelery, medical and firearms to mention but a few. Instances of ceramic media waste in Ireland are mainly from the medical industry with in excess of 1000 tonnes/annum being produced between two of the larger orthopaedic implant manufacturers. As such, there is demand for research into potential re-use of the material.

Several authors have indicated the potential for the incorporation fine ground ceramic waste as an SCM, these studies are varied in terms of the type and origin of ceramic wastes utilized. One common theme throughout these studies is the indication that the chemical composition of various waste ceramics is favorable in terms of potential Pozzolanicity owing to high SiO_2 and Al_2O_3 content [21-28 and 33].

The waste from ceramic media used in mass finishing is highly controlled at source and therefore a homogenous waste product is produced. The economic benefits that would accrue from the utilization of ceramic media sludge waste as a SCM are listed below;

- Payment to receive this raw material at or above current landfill rates (exceeds the cost of cement per ton due to current landfill tax charges which can only increase in the future).
- Minimal reprocessing costs (Drying at low temperature and minimal milling required)
- Energy and emissions savings for the cement producer
- Reduced extraction and use of virgin materials

The aim of this study is to conduct initial testing to determine various fresh and hardened state concrete characteristics of concrete utilizing supplementary cementitious ceramics (SCC)/cement blends, which may indicate the potential for ceramic media sludge to be used as a SCM and justify further more extensive research. The use of the acronym SCC in this paper should not be confused with the acronym for self-compacting concrete.

2 EXPERIMENTAL PROCEDURE

2.1 Materials

2.1.1 Waste ceramic sludge

As previously mentioned, the pozzolanic material utilized in the study was ceramic waste sludge derived from mass finishing processes. Mass finishing is a wet process which produces a ground ceramic sludge with moisture content up to and above 50%.

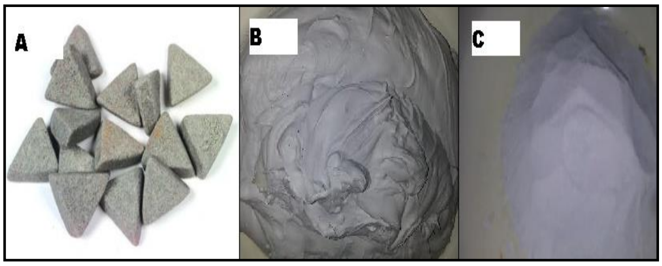

Figure 1: SCC production process (A) ceramic media prior to mass finishing process (B) ceramic waste sludge (C) dry milled SCC

In order to produce a dry powdered SCC, the ceramic sludge was dried at $T=105^0C$ for a period of 48hours.The dried sludge forms a cake like solid in static drying conditions. This ceramic cake was subsequently milled until a ceramic powder was created with a particle diameter passing a 150µm sieve. The production process of SCC from orthopedic mass finishing media can be seen in Figure 1.

2.1.2 Water

The water used in the making of concrete samples was of drinking quality with a pH of 7.8. The water cement ratio used in the design of the concrete reference mix was 0.5. The actual volume of mix water was maintained constant for all mixes despite reductions in OPC content. The decision to maintain the water content was taken in order to make direct comparison between the mixes in their fresh state, and to avoid the use of water reducing admixtures which may have influenced the results.

2.1.3 Aggregates

The sand and coarse aggregate used to make concrete samples were extracted from a limestone sand/gravel quarry. The aggregates consisted of a well graded limestone sand, 14mm and 10mm nominal sized aggregate. These aggregates had a sub angular to rounded particle shape and moderately smooth surface texture. The aggregate particle size distribution can be seen in Figure 2.

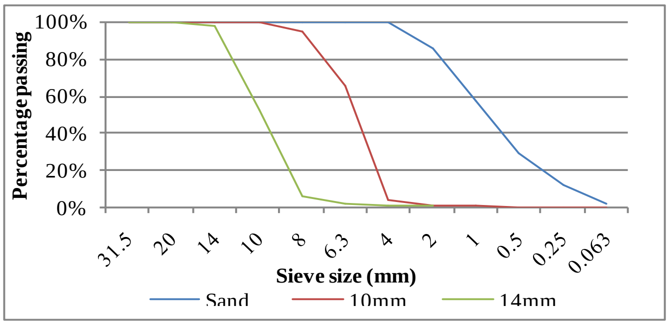

Figure 2: Aggregate grading used for concrete samples

2.1.4 Cement and SCC compositional properties

The reference cements used in the study were CEM I 42.5R and CEM II A/L 32.5N type cements according to EN 197- 1:2011 Standard [31]. The chemical composition of these cements and SCC powders used in this study are shown in Table 1.

Table 1: Chemical composition of cement binders and SCC powder

	Materials		
Chemical Constituent %	CEM I 42.5R	CEM II A/L 32.5N	SCC powder
SiO_2	19.6	18.63	68.8
Al_2O_3	4.41	5.41	18.4
Fe_2O_3	3.3	2.9	4.6
CaO	63.21	62.42	1.5
MgO	4.2	3.2	0.72
Na_2O	0.3	0.3	2
K_2O	0.5	0.4	1.62
SO_3	1.4	2.86	0.06
Loss on ignition	1.4	3.68	0.5

2.2 Sample preparation

The present investigation studied the partial replacement of cement with ground ceramic powder by adding several combinations of SCC powder. Cement blend binders were prepared by dry mixing the powdered SCC with CEM II A/L 32.5N in the range of 20, 35 and 50wt% and CEM I 42.5R in the range of 15, 20, 25, 30, 35, 40 and 50wt%. The percentage SCC was set on the basis of the typical compositional range for Portland composite cement blends as suggested in EN 197-1:2011 [31]. A cement type utilizing ceramic pozzolana would be considered a pozzolanic cement type with the notation CEM IV in EN 197-1 and would contain Portland cement clinker in the range of 45-90%.

Table 2: Concrete mix proportions using CEM II A/L 32.5

Mix Ref.	Kg		Litres	Kg			
	Cement	**SCC**	**Water**	**Sand**	**10mm**	**14mm**	**w/c ratio**
Control (OPC)	320	-	160	885	440	440	0.5
20% SCC	256	64	160	885	440	440	0.5
35% SCC	208	112	160	885	440	440	0.5
50% SCC	160	160	160	885	440	440	0.5

The cement blend mixing procedure was carried out in a mixer for 15 min to ensure repeatability of the blend. The reference binder was 100% OPC CEM II A/L 32.5N in the first instance and 100% OPC CEM I 42.5R in the second instance. Concrete samples were prepared by mixer for characteristic compressive strength f_c= 20N/mm^2 according to the Building Research Establishment (BRE) mix design procedure [34]. The mix proportions for concrete samples using cement blends with CEM II A/L 32.5N are shown in Table 2 and CEM I 42.5R blends in Table 3.

Table 3: Concrete mix proportions using CEM I 42.5R

Mix Ref.	Kg		Litres	Kg			
	Cement	**SCC**	**Water**	**Sand**	**10mm**	**14mm**	**w/c ratio**
Control (OPC)	280	-	140	885	440	440	0.50
15% SCC	238	42	140	885	440	440	0.50
20% SCC	224	56	140	885	440	440	0.50
25% SCC	210	70	140	885	440	440	0.50
30% SCC	196	84	140	885	440	440	0.50
35% SCC	182	98	140	885	440	440	0.50
40% SCC	168	112	140	885	440	440	0.50
50% SCC	140	140	140	885	440	440	0.50

In the case of compressive strength tests, prepared concrete samples were placed in 100x100x100mm cube molds in three layers using a vibrating table for compaction (vibration time ≈ 1min per layer). The concrete samples were de-molded after 24h and placed in a curing tank for (7, 28, 91 days) at T=20^{0}C and relative humidity of 65%.

2.3 Fresh state testing

In order to determine the fresh state characteristics of blended cement concrete using SCC, slump [35], degree of compactability [36] and sieve segregation tests [37] were conducted on samples produced using CEM I 42.5R and SCC as binders. The tests were conducted for the blended cement concrete samples in the range of 0%, 20%, 30%, 40% and 50%wt cement replacement with a water/cement ratio of 0.5. The addition of 15.7L/m^3 of Plasticement® 186 was added to mixes in order to increase the consistence prior to conducting sieve segregation tests.

2.4 Hardened state testing

2.4.1 Density and water absorption

The density [38] and water absorption [39] of SCC blended cement concrete samples were determined for all cement replacement ranges produced using CEM I 42.5R. The samples were cured in a curing tank for 28 days at T=20^{0}C and relative humidity of 65% prior to conducting the tests.

2.4.2 Compressive strength

For each mix using SCC and CEM II A/L 32.5N blends, samples were tested to determine the compressive strengths at 7, 28 and 91 days of curing. Mixes utilizing SCC and CEM I 42.5R were tested to determine compressive strength development at 7 and 28 days only. The compressive strength for each mixture was obtained from an average of five sample specimens for each curing time interval. A 2000-kN capacity uniaxial compressive testing machine was used to test the specimens at a constant loading rate of 0.6N/mm2.S in accordance with EN 12390-3:2009[40].

3 RESULTS AND DISCUSSION

3.1 *Fresh state characteristics*

The results of slump tests shown in Figure 3 show a decrease in concrete consistence as the percentage of SCC increases. Recent studies [22, 23, and 26] have shown that this is a common phenomenon with fine ground ceramic waste blended cements. This is most likely due to a higher water absorption rate of the ground ceramic particles. It is likely that low water/cement ratio concretes would require a water reducing agent or superplasticizor when incorporating SCC blended cements.

An increase in the percentage SCC also had a decreasing effect on the degree of compactability of the concrete which can be seen in Figure 3. The addition of SCC blended cement had a noticeable decreasing effect on the amount of bleed water which appeared on the concrete surface during the test. This effect can be attributed to both the water absorption rate of the SCC powder and to its particle packing effect. The specific gravity of the fine ground SCC is substantially less than that of the cement and therefore when added by %wt, a larger volume of fines are incorporated in the mix.

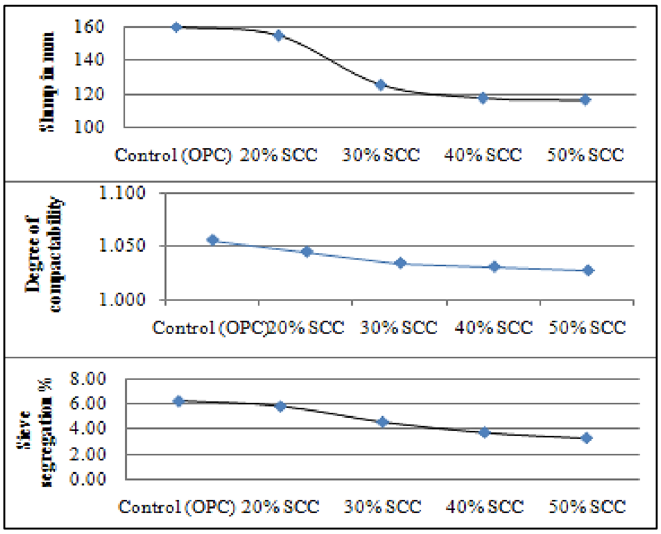

Figure 3: Fresh SCC concrete test results

Sieve segregation tests were conducted on the samples as a means to assess the cohesive nature of SCC concretes. The results shown in Figure 3 illustrate a decrease in segregation as the percentage of SCC increases. These results represent an increase in the cohesiveness of the concrete as SCC content is increased. This increase in cohesion would be of benefit to pumped or self-compacting concrete applications.

3.2 *Density and water absorption*

The density of SCC blended cement concretes were only slightly reduced from that of the reference concrete with the largest reduction in the order of 7%. Figure 4 shows that in terms of water absorption, SCC concretes have a lower water absorption capacity than the reference concrete even as early as 28 days. A low water absorption capacity is analogous to low permeability, which is a crucial factor in the overall durability of concrete. Less permeable concrete's are more resistant to the ingress of water and salts which can be deleterious to concrete and steel reinforcement.

The largest water absorption reduction was recorded for the 30% SCC concrete with an approximate 24% reduction, all other SCC concretes displayed a reduction when compared to the reference concrete, however, a gradual increase occurred in the SCC concretes above 30% cement replacement.

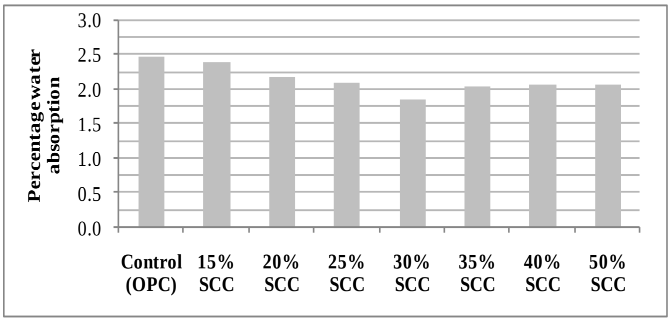

Figure 4: Water absorption

These results reflect those of a study carried out after 90 days using fine ground ceramic tiles, where all concretes using up to 40% cement replacement recorded lower absorption capacities [18]. It would be expected that as pozzolanic reactions continue, the water absorption of SCC concretes would decrease further due to the formation of void filling C-S-H.

3.3 *Aesthetics of SCC blended cement concrete*

Blended cements incorporating pozzolans as a cement replacement tend to have an effect on the color of the hardened concrete they produce. This color change is generally accepted as a positive aesthetic characteristic. Well known pozzolans such as GGBS and metakaolin generally produce concretes with a lighter surface finish than OPC concretes.

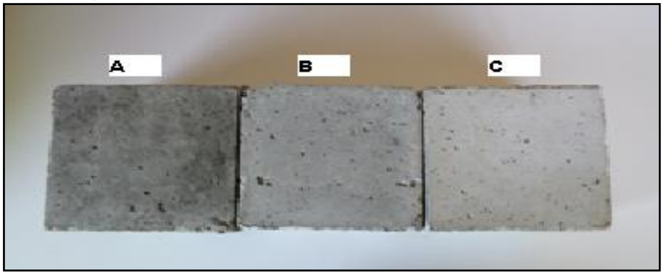

Figure 5: Concrete color variation (A)100% CEM I OPC (B) 20% SCC (C) 50% SCC

It is apparent from the concrete samples produced in this study that the color of hardened concrete produced from ground ceramic media, also produces a lighter surface finish. Figure 5 shows the gradual lightening of the concrete color with increased %wt cement replacement with SCC material.

3.4 *Compressive strength*

3.4.1 CEM II A/L 32.5N/SCC blended cement

The results obtained from compressive strength testing indicate significant strength development reductions ≈ 36% at early curing ages between the control sample and all of the SCC samples. This result was expected as all pozzolans with the exception of highly reactive pozzolans exhibit this mechanical strength behaviour. Early strength of both pastes and concretes decreases while ultimate strength is often found to exceed that of the reference Portland cement. If cements contain small amounts of very active pozzolana (silica fume, for example), both early and ultimate strengths may be higher than those of the substituted cement [30].

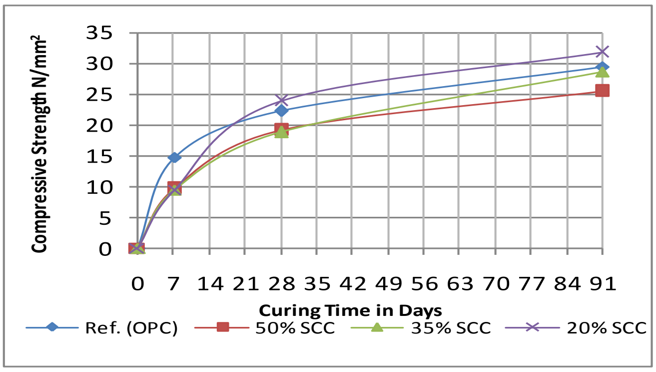

Figure 6: Compressive strength of CEM II A/L 32.5N/SCC samples

All three SCC sample mixes exhibited similar strength development characteristics up to 7 days which is most likely an indication of delayed pozzolanic reaction in the concrete. The reaction between alite and belite in the presence of a reduced cement content would limit the formation of calcium hydroxide ($Ca(OH)_2$) in the concrete matrix [29]. Therefore it is reasonable to assume that early compressive strength reductions are due to inhibited reactions between ceramic pozzolana and a limited early supply of $Ca(OH)_2$.

Results showed that strength decreased with an increase in SCC cement replacement with the lowest strength occurring for an SCC cement replacement of 50%. However, the strength reduction with 50% cement replacement was within 15% of the OPC reference mix at 28 and 91 days. Similarly with a SCC cement replacement of 35%, a 15% strength reduction was recorded at 28 days and a mere 3% at 90 days. Both of these mixes failed to meet the mix design characteristic strength of $20N/mm^2$ at 28 days, however, both were found to be well above $20N/mm^2$ at 90 days. The SCC 20% sample recorded a marginal strength increase of approximately 7.5% when compared to the OPC reference mix at 28 and 91 days.

In terms of emulating the characteristic compressive strength (f_c) performance of CEM II A/L 32.5N cements, the optimal range for SCC cement replacement would be in the order of 15-20%wt. However with careful design consideration it may be possible to incorporate SCC produced from ceramic mass finishing media at higher ratios.

3.4.2 CEM I 42.5R/SCC blended cements

The results obtained from CEM I 42.5R/SCC sample compression tests are shown in Figure 7. These results indicate a gradual and significant decrease in early compressive strength as the %SCC is increased. These findings are consistent with other research using fine ground ceramics [23, 25]. The early age strength is contrary to the results obtained from CEM II blends where 7 day strengths were approximately equal. The availability of free lime at an early age in CEM II A/L cements would have initiated pozzolanic reactions and therefore would influence early age strength.

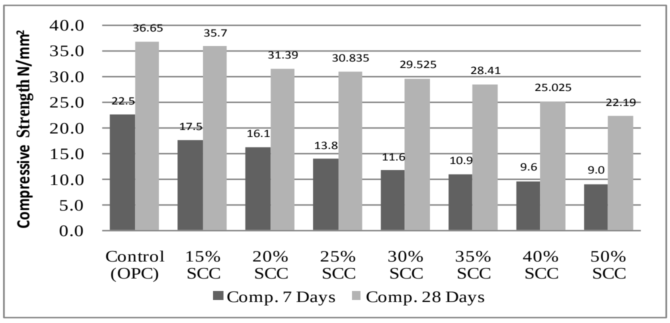

Figure7: Compressive strength of CEM I 42.5R samples

A gradual decrease in compressive strength was also recorded as SCC content increased for sample characteristic compressive strengths after 28 days. The lowest recorded strength was found in 50%SCC samples representing a 40% reduction.

The optimal cement replacement range indicated by CEM I/SCC samples correlates with the results from CEM II/SCC samples at 15-20%. Reductions in compressive strength in 20%SCC samples were 28% and 14% after 7 and 28 days of curing respectively. At a 15%SCC content, early age strength was reduced by 22% and only 3% after 28 days. Based on the results from CEM II/SCC samples it is expected that both 15%SCC and 20%SCC samples would exceed or at least match the compressive strength of the reference concrete after an extended period of curing.

4 CONCLUSIONS

The potential of using fine ground ceramic media sludge derived from mass finishing processes as a blended cement replacement has been investigated in this study. The results demonstrate this potential and the following conclusions can be drawn from the study:

I. The workability of SCC cement concretes reduce as the percentage SCC is increased, however, less energy would be required for compaction of the concrete when compared to OPC concrete.
II. SCC cement concretes display improved cohesion characteristics and are less prone to segregation.
III. The durability of SCC cement concretes would be improved in comparison to OPC concretes due to a reduction in water absorption capacity.
IV. SCC cement concretes lighten in color as the percentage SCC is increased which presents aesthetic benefits.

V. The optimal range for the incorporation of SCC has been found to be in the order of 15-20%. At this cement replacement rate SCC concretes display reduced early age strength, however, 28 day characteristic strengths are similar to those of OPC concretes. The long term strength of SCC concretes would be expected to exceed that of OPC concretes.

If sufficient volumes of waste ceramic media sludge were available, its use as a cement replacement would be potentially lucrative for re-processors.

ACKKNOWLEDGEMENTS

This research programme was realized through the funding and support of John Gannon Concrete Ltd.

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