Investigation of the Recycling of Geopolymer Cement Wastes as Fine Aggregates in Mortar Mixes

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Abstract—Fly ash-slag based Geopolymer Cement (GPC) is presenting mechanical properties and environmental advantages that make it the predominant "green" alternative to Portland Cement (PC). Although numerous life-cycle analyses praising its environmental advantages, disposal after the end of its life remains as an issue that has been barely explored. The present study is investigating the recyclability of fly ash-slag GPC as aggregate in mortars. The purpose of the study was to evaluate the effect of GPC fine Recycled Aggregates (RA), at replacement levels of 25% and 50%, on the main mechanical properties of PC and GPC mortar mixes. The results were compared with those obtained by corresponding mixes incorporating natural and PC-RA. The main physical properties of GPC-RA were examined and proven to be comparable to those of PC-RA and slightly inferior to those of natural sand. A negligible effect was observed at 28-day compressive and flexural strength of PC mortars with GPC aggregates having a milder effect than PC. As far as GPC mortars are concerned, the influence of GPC aggregates was enhancing for the investigated mechanical properties. Additionally, a screening test showed that recycled geopolymer aggregates are not prone of inducing alkali silica reaction.

Keywords—Concrete recycling, geopolymer cement, recycled concrete aggregates, sustainable concrete technology.

I. INTRODUCTION

THE cement industry accounts for around 5% of global carbon dioxide (CO_2) emissions [1], [2] and the production of 1 tonne of cement results to approximately 1 tonne of CO_2 [3], [4]. The CO_2 emitted during concrete production is due to both fuel combustion for the calcination process of limestone and its actual decomposition into calcium oxide and carbon dioxide. These figures are not high compared with other materials such as steel [4], but it is the ubiquity and growing demand of concrete that makes its environmental footprint so high. Considering these numbers, it becomes quite obvious why sustainability targets have been set and the cement industry, as well as the related research community, is investigating ways to meet these. Some of the options of reducing CO_2 investigated so far include; production of blended or low carbon cement mixes, energy efficient kilns, use of biomass fuels and methods for carbon capture [3], [4]. Some of these technologies have led to reductions in CO_2 but others are too niche and expensive to be applied on a wide scale. Considering this, it is obvious that the

change from the use of PC-based concrete to one with significantly lower embodied carbon dioxide would have a marked effect on the global carbon emissions. Although there was some earlier research, geopolymer binders were named and popularised by Davidovits in the 1970s and their production relies on minimally processed natural materials or industrial by-products. There are various categories of geopolymer binders (depending on the raw materials). Given that room temperature, hardening of geopolymers relies on the addition of calcium cations, typically iron blast furnace slag, the most appropriate type for high volume construction applications is considered slag/fly ash based geopolymer [5]. GPCs' manufacturing does do require extreme high temperature kilns with large expenditure of fuel whereas CO_2 emissions by the decomposition of calcium carbonate are avoided. Therefore, it is estimated that their adoption in a wide scale could lead to a 40-80% reduction of carbon emissions [6]. Before proceeding to their wider adoption though, the engineering community is conducting research to ensure tangible environmental benefits.

An aspect of GPCs' sustainability which has barely been investigated is the end of life impact. Most published environmental and life cycle assessments have explored cradle-to-gate stages but barely any research investigating disposal or reuse scenarios has been conducted [7]-[9]. The aim of the present research is to investigate the potential of recycling GPC concrete in a way identical to ordinary PC concrete. Fine RAs were produced and basic aggregate properties such as water absorption (WA) and particle density were investigated. Additionally, a screening test for alkali silica reactivity was conducted. The potential of recycling GPC in mortar mixes with PC and GPC matrices was investigated and the final mixes were subjected to compressive and flexural strength testing.

II. EXPERIMENTAL METHODOLOGY

A. Materials

For the GPC precursor ground granulated blast furnace slag (GGBS) and fly ash (FA) were used. The GGBS was provided by the Hanson Heidelberg cement group from the Port Talbot works, while the FA was CEMEX 450-S (BS EN 450 - 1 Fineness Category S; LOI Category B). The chemical analysis of the precursor materials is presented in Table I. The activating solution was produced by mixing sodium hydroxide pellets (NaOH, 98-100.5%) and sodium silicate solution ($Na_2O(SiO_2)$ x · xH_2O, Na_2O, ~10.6%, SiO_2, ~26.5%) with distilled water. The chemicals were both supplied by Sigma-Aldrich and the mixing took place 24 hours prior to casting.

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The same materials were used in both the RA and the RA mortars production. For the PC elements, General Purpose Portland fly ash cement/Sulfacrete EN 197-1 –CEMII/BV 32,5R supplied by Tarmac Cement & Lime Ltd was used. The natural aggregate incorporated in the mortars was standard sand complying with BS EN196-1 specifications.

TABLE I
CHEMICAL ANALYSIS OF PRECURSORS BY X-RAY FLUORESCENCE

Constituents (wt %)	GGBS	FA
SiO_2	35.15	49
Al_2O_3	13.07	23.5
Fe_2O3	0.28	8.7
CaO	39.6	2.4
MgO	8.47	1.4
SO_3	0.17	0.8
Na_2O	0.14	3.06
K_2O	0.51	0.87
TiO_2	0.66	-
MnO	0.44	-
P_2O_5	-	1.1
LOI	0.97	4.4

B. Production of RA

The GPC recycled aggregates (GPC-RA) used were produced in the laboratory by casting 100mm cubes of pure Geopolymer paste. PC recycled aggregates (PC-RA) were produced in an identical way using pure PC paste. Limiting the number of parameters such as original aggregate influence was considered as the optimum way to investigate the behaviour of GPC binder when used as RA, as well as its' effect on the final mixes during this first evaluation.

TABLE II
AMOUNT OF MATERIALS FOR ORIGINAL GPC & PC PASTE

Material	Quantity(kg)
Fly ash	5.00
GGBS	5.00
Water	2.375
Sodium hydroxide	0.033
Sodium Silicate	0.906
PC	10.00
Water	4.00

For the GPC, the proportion of FA/GGBS was 1/1, water/GPC solids was 0.4, the Na_2O/SiO_2 ratio 1 and the percentage of Na_2O in the activating solution was 5% of the precursor mass. The water/GPC solids ratio was used and indicates that the products deriving from the dissolution of sodium silicate and sodium hydroxide of the activating solution were taken into consideration for the calculation of the free water proportion for the mix. For the PC binder, the water/cement ratio was 0.4. The material proportions for the binders' casting are presented in Table II. It is noted that although fly ash PC was used the term "PC" will be used when referring to the specimens for convenience.

The cubes were demoulded 1 day after casting and then stored in sealed plastic bags at 20±1 ºC for a period of 28 days. After the end of the curing period the 100mm cubes were tested for compressive strength, with PC and GPC binder demonstrating mean values of 47.82 MPa and 45.54 MPa correspondingly.

TABLE III
PARTICLE SIZE DISTRIBUTION OF THE CEN REFERENCE SAND

Square mesh size(mm)	Cumulative sieve residue (%)
2.00	0
1.60	7±5
1.00	33±5
0.50	67±5
0.16	87±5

The fineness of the RA was a controversial issue since it has been proven that grain size has an effect on the RA properties. Given the consensus in literature that the replacement of fine particles is a more unfavourable case compared to coarse aggregate replacement [10], the aggregates in the present study were crushed to fineness similar to that of BS EN196-1 (Table III) standard natural sand. It was also considered possible that successive crushing could made available reactive constituents lying within the hardened GPC paste and provide a better indication of the material's behaviour. Therefore, the crushed cubes were put in a rock crusher repeatedly until passing through the jaws with the opening set at 0.50mm was possible.

TABLE IV
PARTICLE SIZE PERCENTAGES OF RA

Particle size(mm)	Percentage (%)
≥1	33±3
≤1 & ≥0.5	33±3
≤0.5	33±3

The resulting RA was sieved in such way that the particle size distribution presented in Table III was achieved. The aggregates were stored in sealed plastic bags for the whole duration of the project.

C. RA mortars

In total, 10 different mortar mixes were produced. Mortars with PC and GPC matrices were produced, while corresponding mixes incorporating PC-RA were casted in an identical way. The replacement percentages of natural sand were 25% (low replacement level) and 50% (high replacement level), while the reference mixes (0% replacement) incorporated standard sand only. For the GPC mortars the mix design was the same as for the original binder with the difference that the water/GPC solids ratio was raised to 0.47 and the percentage of Na_2O was lowered to 3%. For the PC binder, the water/cement ratio was 0.47. For all mortars the proportions were 2.25 parts of sand to 1 part of binder per mass. All the mix combinations along with the material proportions are presented in detail in Table V. The labelling of the specimens follows the rational: Matrix type_replacement percentage-aggregate type.

TABLE V
MIX PROPORTIONS FOR RA MORTARS

| Mix label | Amount of materials(kg/m^3) | | | | | | | | |
| | Binder | | | Aggregates | | | Activating solution | | |
	OPC	FA	GGBS	NA	OPC-RA	GPC-RA	NaOH	Na$_2$SiO$_3$	Water*
OPC_REF	960	-	-	2160	-	-	-	-	451
OPC_25%OPC	960	-	-	1620	540	-	-	-	451
OPC_50%OPC	960	-	-	1080	1080	-	-	-	451
OPC_25%GPC	960	-	-	1620	-	540	-	-	451
OPC_50%GPC	960	-	-	1080	-	1080	-	-	451
GPC_REF	-	480	480	2160	-	-	19	53	464
GPC_25%OPC	-	480	480	1620	540	-	19	53	464
GPC_50%OPC	-	480	480	1080	1080	-	19	53	464
GPC_25%GPC	-	480	480	1620	-	540	19	53	464
GPC_50%GPC	-	480	480	1080	-	1080	19	53	464

No water compensation or pre-saturation of aggregates prior to mixing took place as a fixed water/cement ratio was chosen over constant workability. It was considered that the mortars should be cured in conditions that would provide maximum enhancement for strength development depending on the matrix type. Therefore, PC matrix mortars were moist cured by immersion in water at 20±1 ºC, while for the GPC matrix mortars the equivalent moist curing conditions was storage in sealed plastic bags at 20±1 ºC. After the end of a 28day curing period the mortars were tested for compressive and flexural strength.

D. Testing

All the tests were conducted according to relevant British standards (BS). The RA were subjected to WA and particle density testing according to BS EN 1097-6:2013 Test for mechanical and physical properties of aggregates. Additionally, the alkali-silica reactivity of RA was investigated using an accelerated screening method described in DD 249:1999 Testing Aggregates-Method for the assessment of alkali silica Reactivity-Potential accelerated mortar-bar method. The standard imposes curing of 4 mortar bar specimens (25 mm x 25 mm x 250 mm) by submersion in water at 80 ºC for 24hours and then in 1 M NaOH solution at 80 ºC for 14 days. The length change of the bars is recorded at 1, 7 and 14 days after immersion in NaOH. Depending on whether the mean % expansion after 14day immersion in NaOH solution for is less than 0.10%, between 0.10% and 0.20% or more than 0.20%, the aggregates are classified as innocuous, inconclusive or potentially expansive. The results for GPC- RA and PC- RA were compared to those obtained by standard natural sand testing.

The test in flexure and compression was conducted in accordance with BS EN 196-1:2005 *Methods of testing cement*.

III. RESULTS AND DISCUSSION

A. RAs

As expected, the apparent particle density of both types of RA is lower than that of NA, with GPC-RA and PC-RA demonstrating values 22.5% and 14.5% lower (Table VI). It appeared that GPC-RA was less affected by oven drying and water immersion than PC-RA. Specifically, the difference between oven-dried and apparent particle density for GPC-RA was 43% while for PC-RA the same value was 60%. The corresponding differences between values for saturated-surface dried and apparent particle density are 22% and 33%. This indicates that although porosity of GPC-RA is significantly higher than that of NA, it is still lower than that of PC-RA. This is further supported by the results of the 24hour WA test. Both types of RA exhibited very high WA percentages, with the value of PC-RA being almost 10 times that of NA and double that of GPC-RA.

TABLE VI
PARTICLE DENSITY OF AGGREGATES

Particle density (kg/m^3) \ Aggregate type	NA	GPC RA	OPC RA
Apparent ρ_a	2,631.2	2,041.7	2,247.6
Oven-dried ρ_{rd}	2,202.1	1,151.3	895.9
Saturated-Surface dried ρ_{ssd}	2,367.3	1,588.3	1,497.9

The obtained values for density were slightly lower than those reported in literature, while WA was significantly higher. Generally, ρ_{rd} and ρ_{ssd} at the range of 1,970-2,140 kg/m^3 and 2,190-2,320kg/m^3 correspondingly were reported [10]-[13]. The general trend for WA of RA is to be 3-6 times higher than that of NA with fine RA presenting values at the range of 8-12% [10], [14]. There is a consensus that both particle density and WA of RA depend strongly on the particle size and the amount of hardened binder adhered on the RA. The original material tested in the present study did not incorporate any natural aggregate and was crushed to size corresponding to that of the finest particles of sand; therefore, the obtained results are justifiable, even though the WA value exceeded the usual ranges. The most significant observation is that although GPC-RA did demonstrate poorer performance than NA, they actually had better performance than PC-RA which are already used in practice. Especially, the effect of high WA was apparent during mixing as mortars with 50% replacement exhibited significantly low workability, with PC-50%PC and GPC-50%PC being almost impossible to mix.

In total, 4 mortar bars of each mix were tested for alkali silica reactivity. None of the mortar specimens demonstrated expansion indicating formation of alkali silica gel, while some

mixes exhibited shrinkage. The potential of alkali silica reaction was considered very likely, given the fact that the use of activator results in relatively high alkali percentages in the GPC binders which are possible to leach in the new mix when GPC-RA is used. This could result to an increase of the overall alkalinity of the mortar beyond the recommended limit for preventing ASR. Besides these encouraging results it should be noted that the testing of RA for ASR reactivity, especially using accelerated methods, presents many defects which are analysed extensively in specific studies. Accelerated methods are likely to provide false negatives and positives, [15] due to their aggressiveness and the high dependence of results on the alkali content of the used cement, while methods formulated for NA testing might be unsuitable for RA and they should preferably be used as screening tests [16]. Although no definite conclusion can be drawn for the ASR reactivity of GPC-RCA at this stage, the results of this screening test indicate that GPC-RA do not present high risk of inducing alkali silica reaction when incorporated as RA in mortar mixes.

B. Mortars

The results for the 28day compressive strength of mortar prisms are presented in Table VII. It appeared that the replacement of NA by RA did not have a significant effect on the 28day compressive strength of PC mortar prisms.

TABLE VII
COMPRESSIVE STRENGTH OF MORTAR PRISMS AT 28 DAYS

Mix Label	Compressive strength at 28days(MPa)	Standard deviation
OPC_REF	35.9	3.10
OPC_25%OPC	41.9	3.28
OPC_50%OPC	37.0	0.06
OPC_25%GPC	38.9	2.13
OPC_50%GPC	35.2	2.02
GPC_REF	24.2	2.50
GPC_25%OPC	12.3	0.53
GPC_50%OPC	11.6	0.60
GPC_25%GPC	35.3	1.38
GPC_50%GPC	37.4	4.10

All obtained values did not present significant divergence from that of the reference mix. Specifically, the incorporation of PC-RA led at an increase of strength of 14.3% and 2.8% for low and high replacement level correspondingly. The mixes with 25% and 50% GPC-RA presented 7.7% and 2.1% higher mean strength compared to the reference. For the same replacement levels, the aggregate type did not lead to remarkable differences between the strength values (Fig 1). For 25% replacement the mean strengths present a difference of about 10 MPa with PC-RA having a more enhancing effect. For high replacement level the effect of aggregate type becomes even less prominent with the difference dropping to about 4MPa. Although the replacement level appears to have some effect, the increase of RA percentage for the same aggregate type led to strength decrease of about 4 MPa in both cases which, considering that the standard deviation of the test results is at the range of 2MPa to 3 MPa, 4MPa could be

considered negligible. In the case of GPC mortars (Fig.2) the effect of RA type is more prominent. It is apparent that the replacement by PC-RA leads to a decline of the 28day mean compressive strength while GPC-RA incorporation resulted to increased values in relation to reference mix. The mixes with 25% and 50% PC-RA demonstrated 49% and 52% lower compressive strength than the reference. On the other hand, those with 25% and 50% GPC-RA presented a strength increase of 31.5% and 35.4% respectively. The correlation between compressive strength and aggregate type becomes apparent considering that for 25% replacement GPC-RA results to value 65% higher than that of PC-RA, while the difference increases to 69% for 50% replacement. The replacement level does not appear to have a significant effect for a given aggregate type. It is observed the strength difference obtained for increasing replacement level is less than 1MPa for PC-RA and at about 2 MPa for GPC-RA. Considering that the standard deviation ranges between 0.50 MPa and 4MPa, these differences are considered negligible.
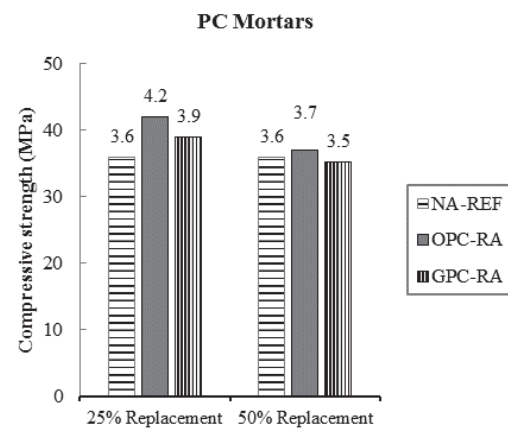

Fig. 1 Compressive strength of PC mortar prisms at 28days

According to literature a gradual decrease of strength with increasing replacement percentage usually occurs [10], [17], [18]. For low replacement levels several studies report minor effects on PC mortar mechanical properties [19]-[21], while the production of high strength concrete was proven possible with the appropriate mix design [22]. Apart from this, there are cases when RA were reported to have a favourable effect on mixes leading to strength gain [23], [24]. The strength enhancement on some of these cases was correlated with the higher strength of the original material of the aggregates compared to that of the finally produced concrete [10]. In this current research, as the original materials had similar strength, this is unlikely to influence results.

It is considered that the action of RA and their distinct effect on the two matrix types is mainly related to their physical and mechanical characteristics and secondarily to their intrinsic nature. The absence of original aggregates and the fineness to which the original binders were crushed to, is possible to have led the finer particles to act as filler resulting

to an overall increase of the binder material in the mortar mixes. Additionally, high WA of RA reduces the amount of free water in the mix. Finally, GPC binders have high alkali percentage and even after the end of geopolymerisation reaction it is possible that unreacted material still exists in the paste.
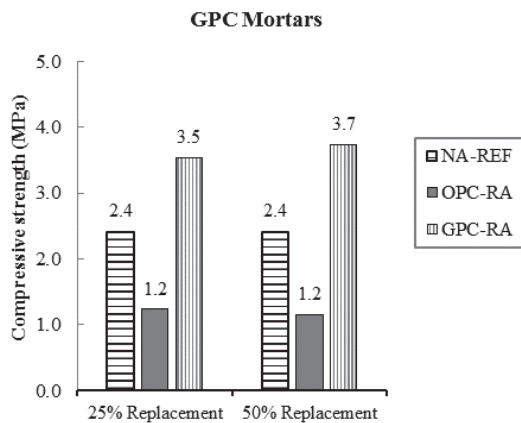

Fig. 2 Compressive strength of GPC mortar prisms at 28days

Based on the above remarks, it is reasonable to assume that in PC mortars with PC-RA the strength gain, in comparison with the reference mix, is related to the increased overall cementitious materials and the reduction of free water, which consequently led to a lower water/cement ratio. A similar process can be assumed for PC mortar and GPC-RA. Given that these mixes exhibited a thixotropic behavior and improved workability, usually related to addition of FA and GGBS, this assumption is further supported. For high replacement percentages this enhancement was probably counterbalanced by the dryness of the mixes resulting to the observed relative decrease. For GPC mortars the assumption of the reduction of free water in the mix and increase of binder material is plausible as well. Given that during the formation process of the GPC matrix, water plays the role of a reaction medium between the activating solution and the precursor [6], [25]-[27], reduced free water could have resulted to a lower degree of completion of the reaction and hence to strength decrease. Furthermore, degradation effects similar to those observed, have been reported in other studies investigating the use of geopolymeric matrices for PC recycling. The negative effect was attributed to the fact that PC-RA could not make a positive effect in the chemical reaction process determining the strength of GPC [23], [28], [29]. An interpretation of the completely opposite effect of GPC-RA addition could be based on the fact that GPC-RA derived from a binder with strength and alkalinity higher than that of the final mix. The alkalis deriving from the original binder could have led to an increase of the overall alkalinity of the final mortar which combined with the lower WA of GPC-RA possibly resulted to a stronger binder compared to the reference mix.

The obtained values for flexural strength are presented in Table VIII. For PC mortars it appears that GPC-RA had a slightly more detrimental effect compared to PC-RA. A 20% reduction of mean flexural strength is observed with 50% replacement by PC-RA, while for GPC-RA the reduction is 14.6% and 22% for low and high replacement level respectively.

The results for GPC mortars flexural strength follow the same trend as those for compressive strength, with PC-RA leading to a significant reduction of mean values. The 25% and 50% PC-RA result to values 50.5% and 56.6% lower than that of the reference mix. On the contrary, 25% replacement level of GPC-RA does not seem to have an effect, while when the RA percentage increases to 50% a 30% reduction is observed. It could be stated that with the exception of GPC mortars incorporating PC-RA, the effect of NA replacement on flexural strength is minor since all values are at the same order of magnitude. The replacement level seems to play a role on mortars with same type of matrix and RA. Specifically, a difference of around 2MPa is observed for increasing replacement percentage in PC mortar incorporating PC-RA and GPC mortar Incorporating GPC-RA. This difference is of small magnitude but still bigger compared to that exhibited by the other combinations which is lower than 0.5 MPa. In PC mortars the aggregate type, for given replacement levels, does not influence the resulting strength as the difference between the obtained values are at about 1MPa or less.

TABLE VIII
FLEXURAL STRENGTH OF MORTAR PRISMS AT 28 DAYS

Mix Label	Flexural strength (MPa)	Standard deviation
OPC_REF	7.8	0.25
OPC_25%OPC	7.8	0.80
OPC_50%OPC	6.3	0.39
OPC_25%GPC	6.7	0.79
OPC_50%GPC	6.1	0.31
GPC_REF	6.4	0.35
GPC_25%OPC	3.2	0.38
GPC_50%OPC	2.8	0.24
GPC_25%GPC	6.6	0.39
GPC_50%GPC	4.7	0.46

TABLE IX
FLEXURAL STRENGTH IN RELATION WITH REPLACEMENT PERCENTAGE FOR MORTARS WITH SATURATED FRCA [18]

Replacement percentage	W/C= 0.5	W/C=0.6
0%	12.14	10.63
10%	13.06	10.55
20%	12.05	10.21
30%	10.86	10.00
50%	10.53	9.16
100%	8.54	8.00

For GPC mortars and given replacement percentage, the effect of aggregate type is remarkable. Specifically, the values obtained by the incorporation of GPC-RA are double or larger than those resulting by the incorporation of PC-RA. An observation made during flexural strength testing is that PC

mortars presented brittle type of failure, while GPC specimens were suppler.

The trend followed by the PC mixes is similar to that identified in [18] (Table IX) for same size specimens. The results of both studies present similarities as far as the downward trend with increasing replacement levels and the negligible influence of low fine RA levels are concerned.

As far as GPC specimens are concerned, the mechanisms affecting flexural strength have not been intensively investigated therefore it is hard make any assumptions for the effect of RA other than those analysed in the "Compressive Strength" section. Additionally, it appears that in GPC mortars the relationship between compressive and flexural strength is different to that of PC mortars.

IV. Conclusions

The general conclusion that can be drawn is that GPC-RA the conducted tests did not indicate any risk or potential jeopardizing of mechanical properties of mortar mixes by the incorporation of GPC-RA. In summary, fine GPC-RA presented lower WA than same grading PC-RA. The exhibited value was almost 5 times that of Standard natural sand. The apparent, oven dried and saturated-surface dried particle density was lower than that of NA but within acceptable limits for RAs and PC-RA for about 22.4% and 9.2% correspondingly. Compared to PC-RA, oven dried and saturated surface dried particle density was higher. Generally, the WA and density results indicated that GPC-RA has a less porous structure than PC-RA which makes less water absorbent and less susceptible to temperature and humidity changes. A preliminary Alkali silica reactivity test indicated that GPC-RA do not present any high risk of inducing ASR, but this is a subject requiring further and in-depth investigation. By evaluating the results of the mean 28day compressive strength of mortar prisms it can be concluded that GPC-RA have a similar or more favorable effect compared to PC-RA. Their performance is inferior to natural sand but to levels acceptable for the case of RAs. Finally, it was observed that the effect of GPC-RA on flexural strength was comparable to that of PC-RA. No significant effect was observed at any of the PC mixes while in GPC mixes their effect was enhancing. Overall the effect of GPC-RA on mechanical properties for low replacement levels was negligible.

The results of a preliminary investigation proved to be satisfying and the potential of recycling fly ash/slag based GPC in the same way as ordinary PC seems to be feasible. Further investigation of the influence of GPC-RA on other mechanical properties of mortar and eventually concrete mixes is required in other to obtain a full image of their action. Furthermore, microscopic investigation of the internal structure and reaction products of these mixes has to be conducted.

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