Synthesis and characterization of fly ash and slag based geopolymer concrete

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Synthesis and characterization of fly ash and slag based geopolymer concrete

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Abstract. This paper presents the formulation and characterization of composite fly ash and slag based geopolymer concrete. Potassium hydroxide (KOH) and potassium silicate (K_2SiO_3) liquid alkaline activators were used in the preparation of the geopolymer concrete. The mechanical response of the best recipe was investigated through compressive, tensile and flexural strength tests after 3, 7 and 28 days of curing. One batch was tested for compressive strength after 16 and 32 weeks of curing. The microstructure of the concretes was characterized using scanning electron microscopy (SEM). The testing results are compared with a reference ordinary portland cement-based concrete. After 28 days of curing, test results showed that the geopolymer concretes can approach the compressive strength of the reference ordinary concrete. Moreover, the tensile strength and flexural strength attained reached up to 68% and 80% of the reference cement, respectively. The results indicated the potential application of geopolymer that can satisfy the standard strength requirement. After longer time of curing, 32 weeks, some of the geopolymer preparations exhibited 111% compressive strength, compared to the ordinary cement reference.

1. Introduction

Concrete is found nearly everywhere. Among others, concrete is used in many areas of civil engineering works such as for bridges, foundations, dams, commercial and residential buildings, tunnels, pipes, offshore structures etc. Mechanically, concrete can resist high compressive forces, also tensile forces when reinforced. It is resistant to rough weather conditions and moisture, it has a medium to high fire-resistance and can be cast in almost any shape or form. However, it has some drawbacks linked to its relative mechanical properties like resistance to deformation, efflorescence, extremely high energy intensive (clinker formation and grinding) and high CO_2-emitting production processes, etc. Especially to address the environmental issues that are getting more and more considerations, it is critical to find an alternative with solutions to the problems associated with this most important and the top commercial product in the world after water.

A geopolymer concrete is an emerging inorganic polymer produced by alkali activation/alkalination of silicate, aluminate and aluminosilicate materials [1, 2]. These groups of materials are one of the most abundant on earth, besides being components of the enormously produced by-products released in hundreds of millions of tons per year, like fly ash from thermal power stations and slag materials from metal productions. It is a promising endeavor that can pave way for utilization of solid waste and by-products with the ensuing multiple benefits.

The formation of geopolymers involves different stages: release of silicates and aluminates, gelation, polymerization and hardening [3]. They involve the formation of macromolecules after polymerization involving the aluminate, silicate and aluminosilicate groups. Geopolymers have similarities with zeolites with regards to their chemical compositions, but are amorphous in their microstructures [4-6].

Besides its low carbon footprint and minimized energy utilization, geopolymer concrete has a number of advantages. They have attractive mechanical, thermal and physicochemical properties such as low density, micro-porosity, high mechanical strength, good surface hardness, thermal stability, and resistance to chemical exposures, and weathering like freezing and thawing [7-9]. Geopolymerization is considered an easy, cheap way of producing concrete with attractive characteristics that can, thus, be utilized for the handling of diverse ground granulated or powdered inorganic waste materials for a cleaner and sustainable environment [10]. As a consequence, it has grabbed increasing interest in the past couple of decades academically and commercially, leading even to large scale productions and regulatory standardization [11]. Geopolymer concrete is, thus, an interesting direction to explore that can potentially yield cementitious products capable of solving some of the issues raised with Ordinary Portland Cement based concrete (OPC).

This paper is not intended as a contribution towards the general climate change debate, although it is difficult to mention geopolymer concrete without touching the subject of CO_2 and the impacts of replacing OPC with geopolymer binders could have. During the cement production process, a significant amount of CO_2 is released into the atmosphere. If these anthropogenic CO_2-emissions are a driving factor towards a long-term climate change on a global scale, or whether the anthropogenic greenhouse gases are negligible compared to nature's own cyclic activities can be discussed. However, it is estimated that OPC production alone is responsible for approximately 5% of the world's anthropogenic CO_2-emissions [12]. Thus, if reduction of CO_2-emissions is a proclaimed goal the world has decided to work towards, curbing the emissions from the cement industry seems like a good place to start.

In the recent years, geopolymer research gained increased interest by both industry and the academic world with many new discoveries and patents. The concept and usage of geopolymer concrete or synthesized rocks is, however, not recent discoveries. Alkali-activation have been utilized sporadically in the past hundred years, but gained more recognition recently due to the environmental pollution concerns [11]. Even much earlier, in the Roman time, hydraulic concrete materials were produced based on naturally occurring volcanic fly ash and limestone capitalizing on the pozzolanic characteristics [13]. Portland cement, the main binding ingredient in modern concrete (OPC), was invented in 1824 [14]. Geopolymer concrete has already been used for load bearing structures in some large-scale projects, and some of the precursors of alkali-activated concrete (blast furnace slag and fly-ash) are increasingly being commercially utilized as additives to regular Portland cement in concrete. However, as the building industry tends to be a conservative one, geopolymer is not yet widely used as a substitute for regular cement. Geopolymer concrete can be made using many naturally occurring materials. Some materials have natural pozzolanic properties, such as volcanic ash, while other materials such as kaolin clay can be calcined into metakaolin (at much lower temperatures than Portland Cement, thus requiring less energy for calcination process). Metakaolin is a well tried out precursor for geopolymer concrete, but the use of such material as a geopolymer precursor can be a source of conflict with other industries, as kaolin clay is also used to make for instance ceramics, and it is not an abundantly available resource.

Diverse materials were used for the preparation of geopolymer concretes at ambient and elevated temperatures along with sand and other different aggregates. Among the recent attempts in such preparations are the use of copper slags [7], fly ash [15], fly ash and furnace slag [16-17], waste olivine and silica sands [10], construction and demolition wastes [18], volcanic ash [19], geothermal nanosilica waste [20] and natural pozzolan feldspars [21] to mention few. There are also recent attempts to modify the properties of the geopolymer concretes through the use of additives like fibers, carbon nanotube and graphene [22]. Along with the experiment with diverse materials, optimization of the experimental parameters and elucidation of the mechanisms of the geopolymerization processes are current areas of research.

This paper presents the formulation and testing of alkali-activation of Ground Granulated Blast Furnace Slag (GGBFS) and Fly Ash (FA) as main binders in geopolymer concrete. Both ingredients are industrial waste products, and require no extra calcination, and very little extra processing (GGBFS is milled into fine powder, FA is used as-is). There is an estimated yearly production of 367 million tons of fly ash alone, and only about 55% are utilized in other ways than ending up in landfills [23].

Both FA and GGBFS are being commercially used by the construction industry today, but mostly mixed with regular Portland cement in various percentages. The recipes applied in this paper are however completely without Portland cement (except for the reference batches), thus indicating that the potential of these materials is not only in the use as additives or partial substitutes for Portland cement, but also as completely stand-alone binders in concrete.

Other advantages to geopolymer concrete are that the hydration process seems to continue much longer and at a steeper rate after a few weeks, than that of OPC. When the hydration process of OPC slows down, the geopolymer concrete can still hydrate and gets harder over time. It can also have a much higher fire resistance [24] and a higher sulfur and acid resistance than OPC [25].

In this work, several recipes of geopolymer concrete have been formulated and characterized. However, the paper presents only some of the formations that led to good results. Moreover, the formulated geopolymer will also be compared with the ordinary Portland cement. Destructive mechanical testing and SEM analysis were carried out to evaluate the strength and the internal structure of the samples.

2. Theory and Methods

The mechanical strength of the geopolymer and OPC-reference cubes were investigated through Compressive strength testing, tensile splitting test and flexural strength tests.

2.1 Tensile split test

Concrete in general is weak in tensile strength and its strength determined indirectly by a split test (Brazilian test). The specimen is prepared in 32mm diameter by 66mm length cylindrical cup. Tensile testing was carried out according to standard NS-EN 12390-6:2009 [26]. During testing, the specimen is loaded between two parallel plates and loaded along the axial direction. The load continuously increases until failure. The tensile strength is given as:

$$\sigma_t = \frac{2F}{\pi DL} \tag{1}$$

where F is the maximum load, D is the diameter and L is the length of the specimen. The unit of the tensile strength is in N/mm2 or MPa.

2.*2 Compressive tests*

The compressive strength of the specimen describes the measure of the maximum load carrying capacity for the applied compressive load using Zwick Z/020 test machine. Unlike the tensile testing, the specimen is loaded between parallel plate and the loading is on the surface of the cube. The size of the cubes was (LxWxH) 50x50x50mm, where the contact surface that the uniaxial loading applied is 2500mm^2. The testing procedures is according to NS-EN 196-1 standard [27]. The compressive strength is given by:

$$\sigma_c = \frac{F}{L*W} = \frac{F}{2500} \tag{2}$$

where F is maximum compressive load in N, LxW is the contact surface area of the cube.

2.3 Flexural strength

The flexural strength of the beam specimen is the measure of the beam's ability to withstand bending loading. The measurement is a three-point testing frame and conducted by inserting in the compression chamber. This three-point test frame directs the forces to a middle point of a specimen of 40x40x200mm, which is supported by to supports distanced 100mm apart. The reference standard is: NS-EN 12390-5:2009– single point load [28]. Flexural strength is calculated by:

$$F_{cf} = \frac{3FL}{2d_1 d_2^2} \quad (3)$$

F_{cf} (N) is the flexural strength in N/mm^2 or MPa, L is the distance between the supports (100 mm in this case) d_1 is the cross-sectional width of the beam, and d_2 is the cross-sectional height of the beam.

2.4 Scanning Electron Microscopy

Scanning Electron Microscopy with Backscatter Electron Detector (SEM-BSED) was used to obtain high-resolution micrographs of compositional maps that reveal the internal structure of the cured geopolymer components. Fragments from batches 14 and 18 were coated with palladium and were analysed in a Zeiss Supra 35VP instrument. TEAM™ Software Suite was used for data processing.

3. **Experimental works**

The Eurocode Part 2, NS-EN 1992-1-1:2004 (EC2) [29], is the governing standard for concrete design work. The EC2 defines the standard strength classes and overview of their class parameter values and limitations. Eurocode Part 2 (EC2) defines all use of concrete, as from material, to design ULS/SLS and ULS, to execution.

3.1 Materials

A total of 21 different geopolymer mix designs have been formulated. In this paper, only a few selected recipes are presented. The slurry formulation was according to mix design, which is according to the procedure described in NS-EN 196-1:2016 – Methods of testing cement [27].

Table 1A presents batch 14 recipe formulations, while Table 1B presents some calculated properties of the same mix design. Table 2A and Table 2B present the same formulations and calculated properties for batch 18 while Table 3A and 3B show formulations and calculated properties for the reference batch made with OPC. The geopolymer concrete was prepared by mixing aggregate (quartz sand), binder (GGBFS and FA) and binder/hardener (KOH and K_2SiO_3). The ratio of aggregate and solid binder is constant; however, the ratios of the hardeners, water and other additives vary.

The composition proportion of geopolymer concrete is very different from OPC since the liquid hardener, also known as liquid binder/activators contains water – which needs to be deducted from the total amount of the required water. The solids that are blended in the liquid hardener are considered as parts of the binder and should be added to the binder calculations for an accurate proportioning. After mixing, the batches are cast in cubes of 50x50x50mm, and wrapped in plastic film to prevent evaporation of liquids.

The reference batch (OPC-based) was mixed with cement and aggregates proportionately the quantities of GGBFS+FA and quartz sand respectively. Furthermore, it was mixed to resemble the same workability/consistency as the geopolymer batches – as a mix with a w/b-ratio as low as batch 18 would have produced a non-workable mix. All batches were de-moulded next day and placed in a water bath keeping a consistent temperature of 40°C.

Table 1A. Batch 14 recipe formulation

Ingredients	Quantity (kg)	Solid (g)	Liquid (g)	Density (kg/m^3)	Recipe (kg/m^3)
FA	0.468			2300	337.31
GGBFS	0.468			2920	337.31
QS	1.8			2650	1297.36
KOH 4M	0.14528	28.28	117	1153	104.71
K2SiO3 3.4M	0.1655	66.1	99.5	1315.26	119.29
H20	0.0926			1000	66.74
Sx-N				1050	0.00
Sugar				849	0.00
Sum, ex addit.	3.14				2262.73

Table 1B. Batch 14 calculated mix properties

Total water content (kg/m^3)	Total solids (kg/m^3)	Total binder (kg/m^3)	Total liquids (kg/m^3)	Water/ binder ratio	Solid/ liquid ratio	Modular ratio (K2SiO3/ KOH)	Matrix volume (l/m^3)
222.74	2039.99	742.62	290.74	0.30	7.02	2.34	510.43

Table 2A. Batch 18 recipe formulation

Ingredients	Quantity (kg)	Solid (g)	Liquid (g)	Density (kg/m^3)	Recipe (kg/m^3)
FA	0.468			2300	343.69
GGBFS	0.468			2920	343.69
QS	1.8			2650	1321.89
KOH 4M	0.14528	28.28	117	1153	106.69
K2SiO3 3.4M	0.23002	91.8	138.37	1315.26	168.92
H20	0.0178			1000	13.07
Sx-N	0.0211			1050	15.50
Sugar	0.002			849	1.47
Sum, ex addit.	3.129				2297.96

Table 2B. Batch 18 calculated mix properties.

Total water content (kg/m^3)	Total solids (kg/m^3)	Total binder (kg/m^3)	Total liquids (kg/m^3)	Water/ binder ratio	Solid/ liquid ratio	Modular ratio [K2SiO3/ KOH]	Matrix volume (l/m^3)
200.55	2097.42	775.52	288.69	0.26	7.27	3.24	501.17

Table 3A. Reference OPC batch recipe formulation.

Ingredients	Quantity (kg)	Solid (g)	Liquid (g)	Density (kg/m^3)	Recipe (kg/m^3)
OPC (IND.)	0.936			3130	716.75
QS	1.8			2650	1378.37
H20	0.3276			1000	250.86
Sx-N	0.01866			1050	14.29
Sum, ex addit.	3.06				2345.99

Table 3B. Reference OPC batch calculated mix properties.

Total water content (kg/m^3)	Total solids (kg/m^3)	Total binder (kg/m^3)	Total liquids (kg/m^3)	Water/ binder ratio	Solid/ liquid ratio	Modular ratio [K2SiO3/ KOH]	Matrix volume (l/m^3)
250.86	2095.13	716.75	250.86	0.35	8.35	N/A	479.86

Aggregates: The aggregate used is a high purity, very well sorted medium quartz sand with grain sizes ranging between 200 μm and 500 μm. The sand used for these experiments is called "H 33" made by the company "Quartzwerke GmbH" [30].

Binders: Fly Ash (FA) is an industrial waste product that is precipitated from the flue gases from industrial furnaces burning any solid fuel. A waste product such as FA is an environmentally friendly alternative to regular Portland cement, which requires calcination of limestone at high temperatures and therefore, a high amount of energy is used. Additionally, the CO2 emissions from this process are formidable. As for FA, the calcination process has already taken place during the furnace burning, and therefore no extra time and energy is needed for neither calcination nor milling/grinding. ASTM C618 defines FA into class C (typically Ca content over 20%) and class F (Calcium content less than 7%) [31]. ASTM C618 Class F can roughly be translated into NS-EN 450-1 Class A. The fly ash used in this thesis is supplied by Norcem [32] and is classified as class A according to NS-EN 450-18 [33].

Ground Granulated Blast Furnace Slag (GGBFS): Ground Granulated Blast Furnace Slag (GGBFS) is an industrial waste product obtained from steel production. As opposed to FA, it needs to be ground into fine powder before being utilized as an alkali activated precursor. The slag used in this paper is a commercial product branded "Merit 5000" - supplied by SSAB Merox AB and is a by-product from the steel plant in Oxelösund. The slag is rapidly cooled in water, and thereafter dried and milled at a mill in Grängesberg [34].

Liquid activators: Two liquid activators were used. The first one is Potassium Hydroxide (KOH), colloquially called caustic potash. KOH is a strong base. The Potassium hydroxide-solutions were prepared, high purity KOH-pellets produced by "Riedel-de Hëin" [35] were dissolved in distilled water according to the desired Molar concentration. The second one is Potassium silicate (K2SiO3), which is an inorganic aqueous solution containing approximately 62% H2O (approx. 3.4 M-solution). The potassium silicate solution used is a commercial product from Univar AB in Malmö, Sweden [36].

Superplasticizer: Dynamon SX-N is a commercial superplasticizer made by the company Mapei [37] designed as a concrete additive to increase its workability. In other words, one can reduce the water/cement (water/binder)-ratio, and therefore increase the final strength of the concrete, while preserving the workability of the mix. It can be added in quantities of 0.2-2% (by weight of binder) without any significant effects on the concretes properties when set.

Retarder: There are several commercial retarders available, but regular table sugar is a known retarder in OPC-based concrete. Even very small amounts in the mix can retard the setting time significantly. Regular granulated table sugar was introduced as an attempted retarder in some of the geopolymer batches, to see if we can get any similar results as it would cause in OPC-based mix designs.

4. Results and Discussion

4.1 Mechanical testing

4.1.1 Compressive strength after 1, 7 and 28 days of curing: With reference to NS-EN 12390 testing of concrete, the geopolymers were tested after one day, seven days, and 28 days. Even though testing after 21 days is not a part of the testing standard, including the earlier mention days, the OPC were tested 21 days as well. Figure 1 shows the comparisons of the compressional strength of the geopolymer recipes to OPC reference batch. All datapoints represents the average value of three cubes.

In terms of strength development, the figure shows that the reference concrete curing rate is higher until seven days of curing and fully developed its strength gently after 28 days. On the other hand, the geopolymer batches strength development is relatively lower than the reference until seven days, but the strength increases with a higher rate and reaching to nearly the strength of the reference concrete after 28 days. This observation leads to the conclusion that it is possible to make FA- and GGBFS-based geopolymer concrete fulfilling most of the strength classes. The higher strength slope of the geopolymers after day seven suggests that at some point, the compressive strength of the geopolymer concrete could be equal, or even above, that of OPC. Based on these results the decision was made to leave more cubes of batch 18 and the OPC reference to cure in similar conditions over a long period of time to test the long-term strength development.

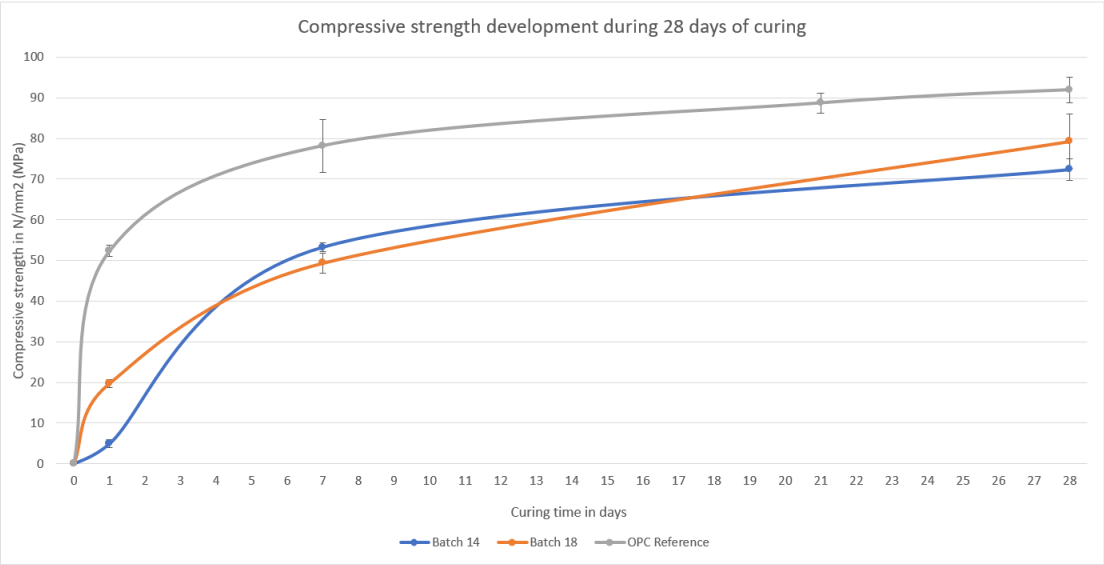

Figure 1. 28 days compressive strength development of batches 14 and 18, compared with the OPC-reference.

4.1.2 Flexural strength. Here, the best geopolymer recipe (Batch 18) was selected for testing of flexural strength. The result is compared with the reference OPC based concrete. A repeat test was carried out and the average value is reported. The test results in Table 4 shows that the geopolymer recipe obtained 80% of flexural strength relative to OPC after 28 days of curing.

Table 4. Comparison of flexural test results for OPC and Batch 18.

#	Concrete systems	Flexural strength, MPa
1	OPC Reference batch	7.8 ± 0.6
2	Geopolymer Batch #18	6.22 ± 0.06
	% Reduction	-20%

4.1.3 Tensile split strength. Table 5 shows the tensile strength of Batch 18 compared with the tensile strength of the reference ordinary Portland cement-Batch-R1. As shown in the table, the geopolymer recipe obtained 62% of tensile strength relative to OPC after 28 days of curing

Table 5. Comparison of tensile test results for OPC and Batch 18.

#	Concrete systems	Tensile strength, MPa
1	OPC Reference batch	4.0 ± 0.1
2	Geopolymer Batch #18	2.5 ± 0.3
	% Reduction	-38%

4.1.4 Compressive strength after long term curing (112 days/16weeks and 224 days/32 weeks). Exceeding the standards testing interval for what is considered final strength, we decided to see what kind of strength development we would get from long term curing. Cubes from both batch 18 and the reference batch was left to cure under similar conditions (40°C, immersed in water). As Figure 1 suggests, the curing rate of batch 18 has a steeper curve than the OPC-reference after seven days. Figure 2 starts with the same dataset as figure 1, but with a much longer timespan. It shows how the geopolymer mix design, batch 18, continues the hydration process long after the first 28 days. The three cube average values after 224 days were 106.6 MPa for batch 18 and 95.8 MPa for the OPC reference batch. The trend seems to be that the geopolymer concrete does indeed exceed the OPC reference but given the error margins further experiments should be conducted.

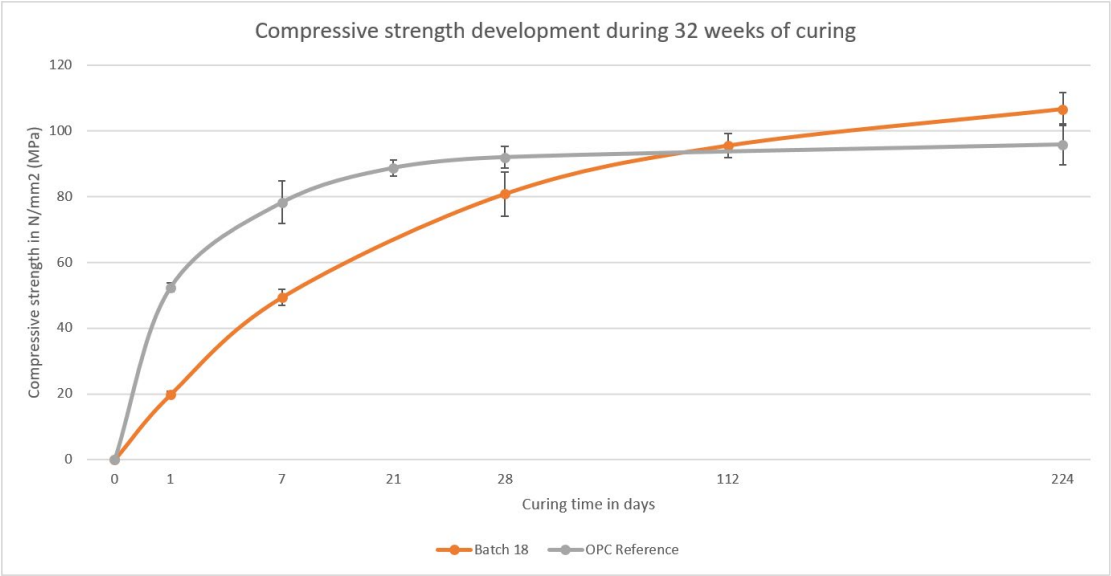

Figure 2. 224 days compressive strength development of batch 18, compared with the OPC-reference.

4.2 SEM analysis

Furthermore, the internal structure of the crushed specimen was analyzed through SEM. For this, it is wise to evaluate the best slurry (Batch 18) with respect to a relatively good one (Batch 14). Figure 2 shows the comparisons of the compressive strength. As shown after 28 days Batch #18's compressive strength is 10MPa higher than the Batch #14.

Figure 3 shows a SEM images on 1.5k x magnification of batches 14 (A) and batch #18 (B). The pictures clearly indicate the correlation between compressive strength and how the particles are bonded on a microscopic level. A higher degree of cohesion at microscopic level has led to a greater compressive strength. As shown in the figure 3A, batch 14 contains some micro cracks and fissures. However, one can observe a uniform and well-developed bonding between the particles. It is also interesting to see that the largest particles, of both FA and GGBFS have remained partially or completely

undissolved/unreacted. Figure 4 displays the same trends and the same batches, but at higher magnifications (500x and 10,000x).

Batch 18 (B) shows relatively a better degree of polymerization, filling of the pore spaces and good boding among the diverse components as well. It can also be observed that Batch 18 has some partially unreacted particles (Figure 4C and 4D, at low and high magnifications, respectively), but the surrounding matrix shows a very good and uniform bond. Even the bond to the larger aggregates looks intact and of very good quality.

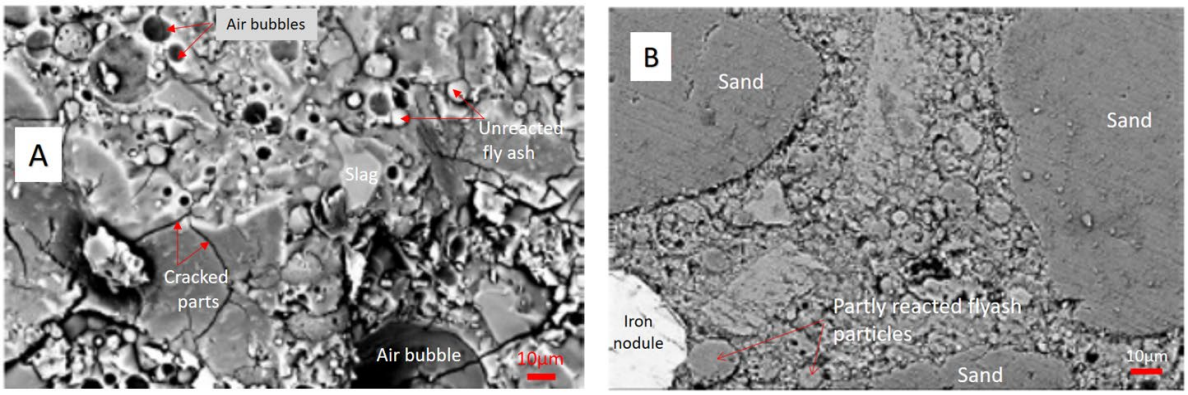

Figure 3. Back scatter electron (BSE) images showing internal structures of batch 14 (A) and Batch 18 (B) at 1.5k x magnification.

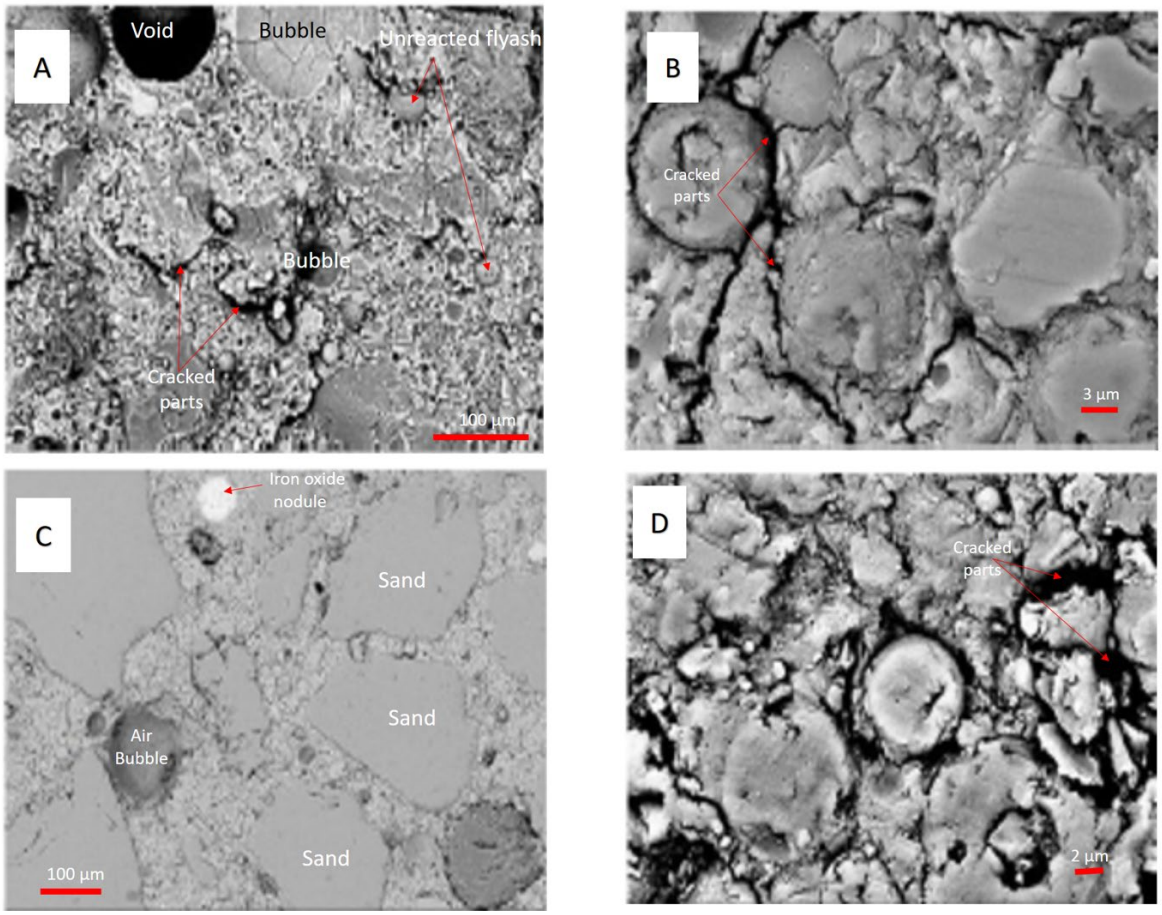

Figure 4. BSE images showing internal structures of batch 14 at 500x (A) and 10^4 x (B) magnification and Batch 18 seen at 500x (C) and 10^4 x (D) magnifications.

5. Conclusion

Several fly ash and slag- based geopolymer slurries were formulated and tested. The mechanical compressive strength is one of the primary properties of concrete. There are many other important properties of a successful mix design, but if a concrete mix does not perform within its designed strength class, all other properties are irrelevant. That does not mean that stronger is always better. In civil construction today, the most used strength classes are B20, B30 and B45, which would place characteristic cube compressive strength after 28 days test at 25, 37 and 55MPa respectively. The compositions and microstructures of the raw materials and geopolymer concretes produced were also characterized using light microscopy, electron microscopy and spectroscopic techniques.

Based on the prepared geopolymers and the reference OPC, the 28th day test results can be summarized as follows:

- The compressive strength of the geopolymer attained the strength of the OPC.
- The tensile and flexural strength of the geopolymer attained 68% and 80% of the OPC.
- Fly ash- GGBFS-based geopolymer has a potential to fulfill most of the strength classes, even the less used high-strength classes.
- The strength development of certain geopolymer mixes looks to be at a steeper rate than OPC-based concrete after day seven and up towards the standard defined final strength measured after 28 days.

Based on the long-term curing results, we can predict that the reactivity in this FA and GGBFS mix design (Batch 18) goes on unabated compared to the OPC reference, and ultimately producing a product that yields higher and higher compressive strengths than the reference. It has reached already 111% strength compared to the OPC reference after 32 weeks. This continuity in the strength gain is one of the interesting characteristics of the geopolymers.

The main drawback to the mix designs witnessed is the short setting time. Sugar was implemented as a retarder, but it resulted in little effect in improving the setting time. Other retarders should be investigated in the future. Through an intensive research, it is plausible to formulate and optimize a better and more economical production of geopolymer concretes that can fulfill standard requirements. With the materials selected for the test, it was possible to acquire a user and environmentally friendly geopolymer formulation. Further research should be conducted towards optimizing matrix volume, aggregate types and curing temperatures. Geopolymer concretes can potentially replace OPC in the future, with advancements in their formulations, characterizations and evaluations of their performances in practical settings outside the laboratory.

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