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Flexural strength of plain and fibre-reinforced boroaluminosilicate geopolymer

Ali Nazari ^{a,*}, Ali Maghsoudpour ^b, Jay G. Sanjayan ^c

a) Research Fellow, Centre for Sustainable Infrastructure, Faculty of Science, Engineering and Technology, Swinburne University of Technology, Victoria, 3122, Australia

b) Researcher, Department of Geopolymer and Concrete, WorldTech Scientific Research Center (WT-SRC), Tehran, Iran

c) Professor, Centre for Sustainable Infrastructure, Faculty of Science, Engineering and Technology, Swinburne University of Technology, Victoria, 3122, Australia

* Corresponding author, Tel: +61 3 92148370, Email: alinazari@swin.edu.au

ABSTRACT

In the present work, flexural strength of plain and fibre-reinforced boroaluminosilicate geopolymers is studied. Traditional aluminosilicate geopolymers are produced by alkali activation of an aluminosilicate source. Alkali activator is normally made by mixing a high alkali solution (such as sodium hydroxide) and a silica-rich source (such as sodium silicate). Alkali activation of fly ash in this study, to fabricate boroaluminosilicate binders, was performed by mixtures of anhydrous borax and sodium hydroxide. Flexural strength of the specimens in unreinforced and reinforced conditions was measured by three-point bending. Reinforced specimens were prepared by using 2, 3 and 5 wt.% of steel fibres, with length and diameter of 30 and 0.5 mm respectively. The highest flexural strength of unreinforced specimens was 9.5 ± 0.4 MPa, with borax to NaOH solution weight ratio of 0.912 and alkali activator to fly ash weight ratio of 0.9. Reinforcing of this mixture by 5 wt.%

of steel fibres resulted in the highest flexural strength, 11.8 ± 0.9 MPa. Maximum and minimum average increase of flexural strength of about 47 and 5 % were achieved by adding 5 and 2 wt.% of steel fibres to some mixtures respectively. Results indicated the ability of these new classes of construction materials for using in flexural load-bearing sections in both unreinforced and reinforced situations.

Keywords: boroaluminosilicate alkali-activated binders; steel fibres; adhesion; flexural strength

1. Introduction

Alkali activated binders (geopolymers) are increasingly developed nowadays and have attracted a great deal of attentions because of their ecofriendly nature [1, 2]. These cement-free constructional materials are normally made by alkali activation of an aluminosilicate source such as fly ash [3]. Normal alkali activators are combinations of a high alkali solution such as sodium hydroxide (NaOH) or potassium hydroxide (KOH), and a silica-rich source with high content of amorphous silica such as sodium silicate or potassium silicate solutions. Alkali activation of fly ash results in production of geopolymeric compounds, which is normally conducted at slightly above ambient temperatures (e.g. 60 °C) [4]. General formula of an aluminosilicate binder is $nM_2O \cdot Al_2O_3 \cdot xSiO_2 \cdot yH_2O$, where M is an alkali element such as potassium or sodium [5]. However, some attempts have been made to produce geopolymers by utilizing other sources, and evidence of production of geopolymers with reasonable performance can be found in Refs. [6-8]. The current research is inspired from Williams and van Riessen work [8], where borosilicate geopolymers were made by alkali activation of silica fume. Alkali activation of their silica source material was performed by a mixture of NaOH and anhydrous borax ($Na_2B_4O_7$), and compressive strength of about 57

MPa was reported to be achievable. In the present study, same alkali activator has been used for activation of fly ash and hence, it is anticipated that boroaluminosilicate geopolymers form. Formation of B–O bonds has been shown in the authors’ previous work [9]. In that work, possibility of achieving boroaluminosilicate geopolymers with higher strength than aluminosilicate one from a specific fly ash source was achieved. It was suggested that main aluminosilicate geopolymeric compounds change slightly to boroaluminosilicate compounds as a result of formation of B–O bonds. Additionally, microstructure of the considered specimens showed different nature with respect to aluminosilicate geopolymers. Therefore, boroaluminosilicate geopolymers seems to have potential for considering as a new class of construction materials for further studies.

Although reinforcing of geopolymers by different fibres has not been widely reported, it may be of interest and of many unknown features. Ohno and Li [10] studied compressive and tensile strength of randomly oriented short Poly-Vinyl-Alcohol (PVA) fibre-reinforced geopolymers. Tensile strain hardening behaviour with very high ductility of over 4% was one of the interesting findings of their reinforced composite. While Puertas et al. [11] reported that incorporation of polypropylene fibres causes improvement of compressive strength. Bernal et al. [12] showed that reinforcing geopolymer concrete by steel fibres leads to a reduction of about 25 % in compressive strength. Such a contradictory in a specific property is not observed in flexural strength of reinforced geopolymers, where Zhang et al. [13], Natali et al. [14], Li et al. [15] and Zhang et al. [16] all have reported improvement of flexural strength by fibre-reinforcing. A comprehensive review on short fibre-reinforced geopolymers can be found in Shaikh work [17] and other related papers [18-27] on fibre-reinforced geopolymer paste and concrete.

In this work, flexural strength of boroaluminosilicate geopolymer pastes in unreinforced and steel fibre-reinforced forms is studied. The effect of mixture proportions on

flexural strength of specimens is the key feature of the present work. Additionally, fracture surface of unreinforced specimens and adhesion of steel fibres to geopolymeric paste is studied through scanning electron microscopy (SEM).

2. Experimental procedure

Materials used in this study for production of unreinforced and steel fibre-reinforced boroaluminosilicate geopolymer pastes were class F fly ash, sodium hydroxide solution (NaOH), anhydrous borax ($\text{Na}_2\text{B}_4\text{O}_7$) and steel fibres. Class F fly ash was used as aluminosilicate source. NaOH and anhydrous borax were mixed by different weight ratios and used as alkali activator. Steel fibres with the length of 30 mm and diameter of 0.5 mm were used as reinforcements in steel fibre-reinforced boroaluminosilicate specimens.

Class F fly ash with average particle size of 9 μm , Blaine surface of 37.3 m^2/g and chemical composition (acquired by XRF) in accordance to Table 1 was used as aluminosilicate source.

Alkali activator was achieved by mixing NaOH solution and anhydrous borax ($\text{Na}_2\text{B}_4\text{O}_7$). NaOH solution with concentration of 8 M was prepared by mixing NaOH flakes and distilled water. Anhydrous borax was prepared by a two stage heating of borax decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$). First stage was done at 150 °C for 30 min to dry some of the water content and to achieve borax pentahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$). Subsequent heating was performed at 300 °C for 15 h to remove the remained intermolecular water. A detailed program on production of anhydrous borax can be found in Williams and van Riessen [8] work. After mixing NaOH solution and anhydrous borax, the mixtures were left in room temperature for 2 hour to cool down and then, were used for alkali activation of fly ash.

In total, two kinds of specimens namely G series (unreinforced) and RG series (reinforced) specimens were prepared. G series specimens comprising of nine different mixtures were prepared by only fly ash and alkali activator. Table 2 illustrates mixture proportions of these series of specimens. RG series specimens were those reinforced by steel fibres. In other words, G series specimens reinforced by 2, 3 and 5 wt.% of steel fibres produced 27 series of RG mixtures. The percentage of steel fibres in the mixture was in the form of "steel weight/total specimen weight". Mixture proportions of RG series specimens are given in Table 3.

Mixtures of fly ash and alkali activator were the only materials used for preparation of G series specimens. On the other hand, to produce RG series specimens, fly ash was first dry-mixed by steel fibres for 5 min and then, dry mixture was alkali activated. Specimens were prepared by pouring the mixtures into moulds in two layers and vibrating for 45 sec after pouring of each layer. The filled moulds were placed in plastic bag for 24 h to minimise carbonation and pre-curing. After that, demoulded specimens were cured at 70 °C for additional 24 h. The specimens then were left uncovered at ambient temperature for 27 days. Flexural strength of specimens was achieved at 28 days of the beginning of oven-curing.

Flexural strength of specimens was acquired in accordance to the ASTM C293-10 standard [28] by using samples with 20 cm length, 4 cm width and 4 cm thickness. Three specimen of each mixture was made and tested, and the average value was reported as the flexural strength of that mixture. SEM images of fracture surface of specimens were achieved by a VEGA TESCAN by a voltage of 15 KV and secondary electron mode.

3. Results and discussion

3.1. Unreinforced geopolymer pastes

Fig. 1 illustrates flexural strength of unreinforced geopolymeric pastes. The minimum flexural strength is 5 ± 0.1 MPa for the specimen with borax to NaOH solution weight ratio of 0.700 and alkali activator to fly ash weight ratio of 0.75. The maximum flexural strength is 9.5 ± 0.4 MPa for the specimen with borax to NaOH solution weight ratio of 0.912 and alkali activator to fly ash weight ratio of 0.9. When alkali activator to fly ash weight ratio is 0.75, flexural strengths at any borax to NaOH solution weight ratios are very close. It is while for the ratio of 0.9, the difference between flexural strength of specimens is high. For the specimens with borax to NaOH solution weight ratio of 0.593, variation in flexural strength due to changes in alkali activator to fly ash weight ratio is low. On the other hand, this variation for specimens with borax to NaOH solution weight ratio of 0.912 is high. For the specimens with borax to NaOH solution weight ratio of 0.912, an increase of about 86 % in flexural strength is observed by changing alkali activator to fly ash weight ratio from 0.75 to 0.9. Both of borax to NaOH and alkali activator to fly ash weight ratios seems to be key factors determining flexural strength of unreinforced specimens.

In Fig. 1, borax to NaOH solution weight ratios of 0.593 and 0.912 have the similar shape of curve, but this is not the case for the ratio of 0.700. As indicated in the previous work [9], the behaviour of boroaluminosilicate geopolymers requires further studies in terms of their chemical characteristics. There are two challenging effect to determine strength of these types of geopolymers: formation of aluminosilicate or boroaluminosilicate compounds. While in low ratios of borax to NaOH solution aluminosilicate compounds are most likely to form, in higher ratios boroaluminosilicate ones are expected to appear. Both of these phenomena results in strength evolution especially when the amount of alkali activator increases. For borax to NaOH solution weight ratio of 0.700, complex mechanisms occur causing irregular pattern of strength evolution that the other ratio. Further works are suggested to be conducted on the effect of various factors on this ratio.

Fig. 2 shows SEM images of fracture surface of some unreinforced specimens. Figs. 2a and 2b illustrate fracture surface of two of the weakest specimens. In both of them, a brittle fracture is occurred and large crystals are appeared after fracture. Formation of these crystals is observed in the authors' previous work [9]. Although large crystals in some cases may indicate the possibility of high strength paste, here, sudden crack growth occurs, causing low strength. Fig. 2c shows SEM image of a low strength specimen as well, but by a different mechanism. Some cracks are visible in the fracture surface. Propagation of cracks along the weak paste seems to be the reason of low flexural strength. Fig. 2e shows a different fracture surface than the former three ones and is related to a medium strength specimen. Although flexural strength of this specimen is not much higher than the previous three ones, very few needle-like crystals are observed that are suggested to be the main reason of strength gain in boroaluminosilicate geopolymers [9]. This is more evident in Figs. 2d and 2f, where obvious visible needle-like crystals are available. It seems that in high strength boroaluminosilicate binders (G5 and G9 specimens), the required energy for crack propagation is divided between many fine needle-like crystals, with approximate diameter of 1 μm , and hence, lots of energy is required for fracture. These needle-like crystals are the characteristic feature of fracture surface of boroaluminosilicate geopolymers, which are formed during successful synthesis of these pastes, and are not observed in traditional aluminosilicate geopolymeric binders.

3.2. Reinforced geopolymer pastes

Fig. 3 illustrates flexural strength of geopolymeric pastes reinforced by different amounts of steel fibres. Alkali activator to fly ash weight ratio has not an obvious effect on flexural strength of specimens. For example, RG1-RG3, RG10-RG12 and RG19-RG21 specimens are three groups of specimens with borax to NaOH weigh ratios of 0.593 and

different alkali activator to fly ash weight ratios. The behaviour of these groups is similar. This type of behaviour is also observed for the other groups with same borax to NaOH but different alkali activator to fly ash weight ratios. Highest flexural strength is related to the specimens with borax to NaOH solution weight ratio of 0.912 and 5 wt.% of steel fibres. For specimens with borax to NaOH solution weight ratio of 0.593, steel fibres have not much effect on increasing flexural strength. In other words, weak paste is not able to make a strong bond by steel fibres, and fibres are easily pulled-out without any positive effect on increasing of flexural strength. For specimens with borax to NaOH solution weight ratio of 0.700, flexural strength of specimens with 3 and 5 wt.% of steel fibres are not much different, but are higher than those containing 2 wt.% of steel fibres. Borax to NaOH solution weight ratio of 0.912, incorporating of 5 wt.% of steel fibres, results in reasonable increase in flexural strength. This is while the difference between flexural strength of specimens with 2 and 3 wt.% of steel fibres is not high. To determine the effect of mixture proportions on flexural strength of specimens, contour plot examinations are presented as following.

Fig. 4a illustrates the effect of borax to NaOH solution weight ratio on flexural strength of steel fibre-reinforced specimens. It is obvious that higher strengths are achievable in higher borax to NaOH solution weight ratios and higher percentages of steel fibres. The higher borax to NaOH solution weight ratio means the higher amount of boron in the mixture. Therefore, production of more boroaluminosilicate binders causes higher bond strength between the paste and steel fibres. Increasing flexural strength at higher percentage of steel fibres can validate this assumption as well. As Fig. 4b shows, there is no regular pattern showing the effect of alkali activator to fly ash weight ratio on flexural strength of reinforced geopolymers. Fig. 4c illustrates the effect of strength of the paste on reinforcing by steel fibres. As obvious, when the strength of the paste is higher, steel fibres have better reinforcing effect. This is not strongly depends on the amount of steel fibres, and for a

specific flexural strength of the paste, by adding different amount of steel fibres, relatively same flexural strength are achieved, especially for pastes with lower strength.

Fig. 5 shows average percentage of increase in flexural strength of the pastes by adding different amount of steel fibres. As Fig. 5a illustrates, borax to NaOH weight ratio has an irregular effect on this factor. On the other hand, for specimens with low alkali activator to fly ash weight ratio (the first nine specimens in the left side of Fig. 5b), average percentage of increase in flexural strength of specimens due to adding steel fibres is higher than the other ratios. However, to have a more comprehensive understanding, contour plots of the effect of mixture proportions are provided as following.

Fig. 6a illustrates the effect of borax to NaOH solution weight ratio on average increase of flexural strength of geopolymers at different percentages of steel fibres. It is obvious that percentage of increase mainly depends on the percentage of steel fibres rather than this ratio. For all ratios, by increasing steel fibres, percentage of increase of flexural strength is higher as well. Figs. 6b and 6c illustrate the effect of alkali activator to fly ash weight ratio and flexural strength of the paste on percentage of increase of flexural strength of fibre-reinforced specimens respectively. Both factors have irregular patterns; however, it seems that the content of steel fibres has greater influence on percentage of increase of flexural strength. In other words, the amount of steel fibres mainly determines the percentage of increase of flexural strength of reinforced boroaluminosilicate geopolymeric pastes rather than the other characteristics of these specimens.

Finally, Fig. 7 shows an SEM image of a steel fibre in boroaluminosilicate paste. As obvious, there is a reasonable bond between this fibre and the paste. Therefore, higher strengths are achieved by reinforcing geopolymers with steel fibres. Fig. 7b illustrates that the paste around the steel fibre is uniform and hence, steel fibres may not have harmful effects

such as inducing stress concentration to the paste during shrinkage and hardening processes. Since a good bond was observed between steel fibres and all geopolymeric pastes, it is anticipated that higher amount of steel fibres cause higher strengths as indicated earlier.

4. Conclusions

From this study, following conclusion remarks were arisen:

- Produce boroaluminosilicate binders with high flexural strength in both unreinforced and reinforced conditions is achievable. Maximum flexural strength of 9.5 ± 0.4 and 11.8 ± 0.9 MPa was achieved for unreinforced and reinforced specimens.
- While increase in flexural strength of unreinforced specimens mainly depends on characteristics of the produced pastes, for reinforced specimens, it depends mainly on percentage of steel fibres.
- SEM images of unreinforced specimens showed that different mechanisms of crack propagation are supposed to occur. High strength boroaluminosilicate geopolymers had fine needle-like crystals while low strength ones showed large crystals through a brittle fracture.
- In reinforced specimens, there is a good adhesion between steel fibres and geopolymeric paste, which make it possible to successfully use boroaluminosilicate pastes as a flexural-load barrier in constructions.

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Table 1. Chemical composition of fly ash

Oxide	Wt.%
SiO ₂	65.5
Al ₂ O ₃	26.7
Fe ₂ O ₃	1.85
CaO	2.26
SO ₃	0.43
Na ₂ O	0.38
L.O.I.	0.9

Table 2. Mixture proportions of unreinforced geopolymer pastes

Sample designation	Borax to NaOH solution weight ratio	Alkali activator to fly ash weight ratio	Content of fly ash (kg/m ³)	Content of borax (kg/m ³)	Content of NaOH flakes (kg/m ³)	Content of water (kg/m ³)	Content of superplasticizer (kg/m ³)
G1	0.593	0.75	1312	366	198	416	4.20
G2	0.593	0.80	1276	380	205	431	4.35
G3	0.593	0.90	1208	405	219	459	4.64
G4	0.700	0.75	1312	405	185	390	3.94
G5	0.700	0.80	1276	420	192	404	4.08
G6	0.700	0.90	1208	448	205	431	4.35
G7	0.912	0.75	1312	469	165	347	3.5
G8	0.912	0.85	1241	503	177	371	3.75
G9	0.912	0.90	1208	519	182	383	3.87

1 Table 3. Mixture proportions of reinforced geopolymer pastes

Sample designation	Borax to NaOH solution weight ratio	Alkali activator to fly ash weight ratio	Percentage of steel fibres (wt.%)	Content of Materials (kg/m ³)					
				Fly ash	Anhydrous borax	NaOH flakes	Water	Superplasticizer	Steel fibres
RG1	0.593	0.75	2	1286	359	194	408	4.12	45.9
RG2	0.593	0.80	2	1250	372	201	422	4.26	45.9
RG3	0.593	0.90	2	1184	397	215	450	4.55	45.9
RG4	0.700	0.75	2	1286	397	181	382	3.86	45.9
RG5	0.700	0.80	2	1250	412	188	396	4.00	45.9
RG6	0.700	0.90	2	1184	439	201	422	4.26	45.9
RG7	0.912	0.75	2	1286	460	162	340	3.43	45.9
RG8	0.912	0.85	2	1216	493	173	364	3.68	45.9
RG9	0.912	0.90	2	1184	509	178	375	3.79	45.9
RG10	0.593	0.75	3	1273	355	192	404	4.07	68.9
RG11	0.593	0.80	3	1238	369	199	418	4.22	68.9
RG12	0.593	0.90	3	1172	393	212	445	4.50	68.9
RG13	0.700	0.75	3	1273	393	179	378	3.82	68.9
RG14	0.700	0.80	3	1238	407	186	392	3.96	68.9

RG15	0.700	0.90	3	1172	435	199	418	4.22	68.9
RG16	0.912	0.75	3	1273	455	160	337	3.40	68.9
RG17	0.912	0.85	3	1204	488	172	360	3.64	68.9
RG18	0.912	0.90	3	1172	503	177	372	3.75	68.9
RG19	0.593	0.75	5	1246	348	188	395	3.99	115
RG20	0.593	0.80	5	1212	361	195	409	4.13	115
RG21	0.593	0.90	5	1148	385	208	436	4.41	115
RG22	0.700	0.75	5	1246	385	176	371	3.74	115
RG23	0.700	0.80	5	1212	399	182	384	3.88	115
RG24	0.700	0.90	5	1148	426	195	409	4.13	115
RG25	0.912	0.75	5	1246	446	157	330	3.33	115
RG26	0.912	0.85	5	1179	478	168	352	3.56	115
RG27	0.912	0.90	5	1148	493	173	364	3.68	115

2

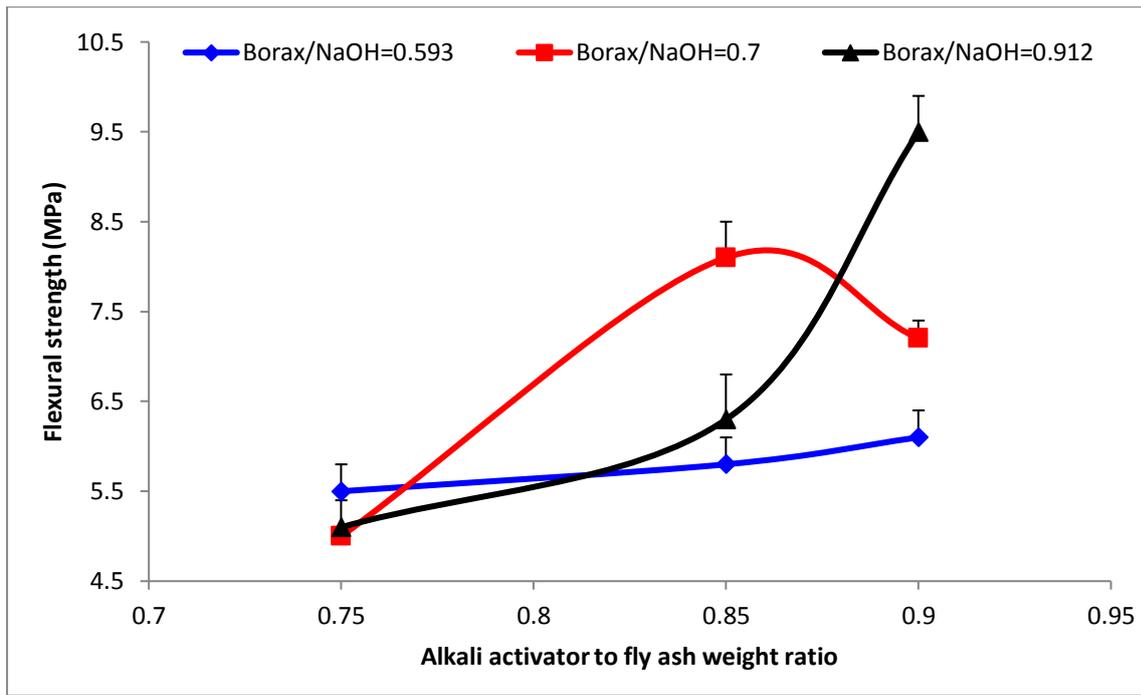
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9 Fig. 1. Flexural strength of G series (unreinforced) specimens

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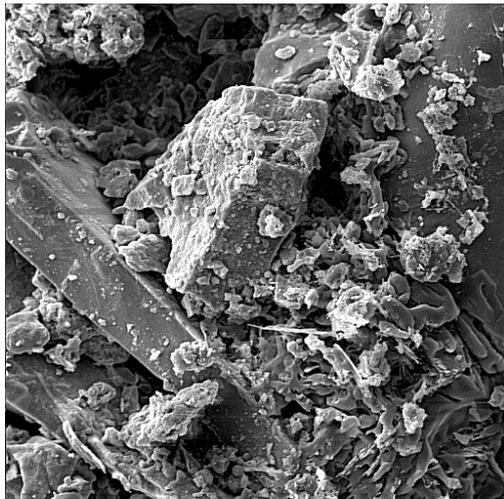
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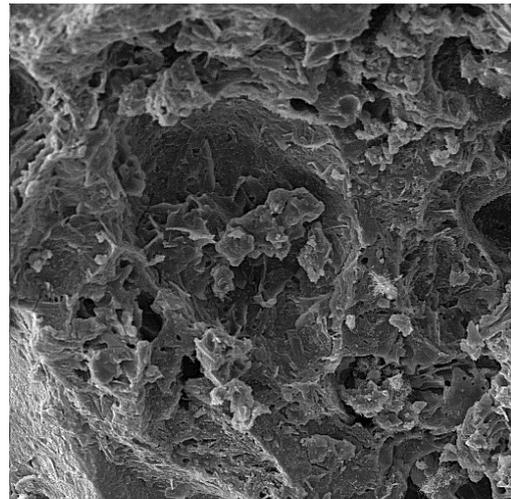
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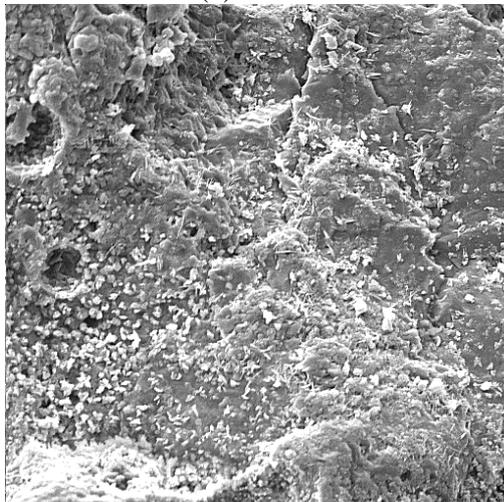


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(a)

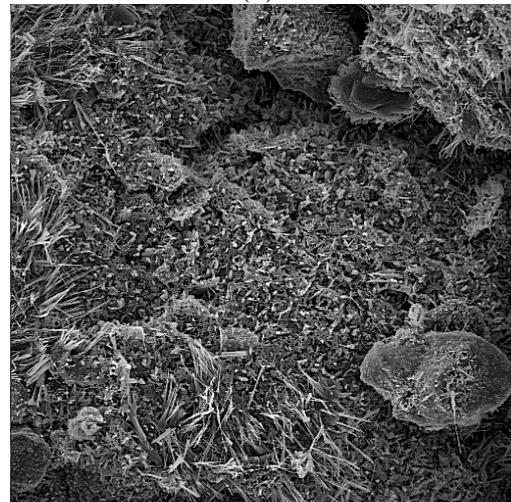


(b)

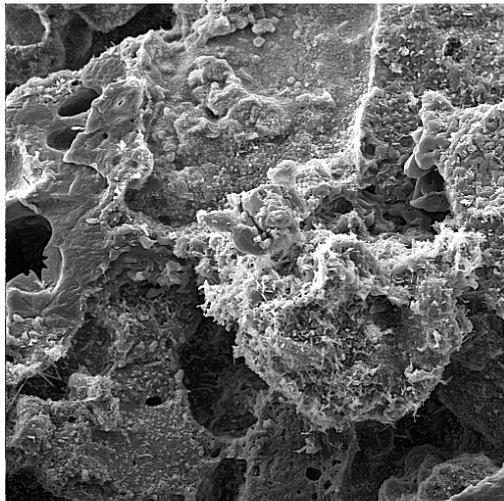


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(c)

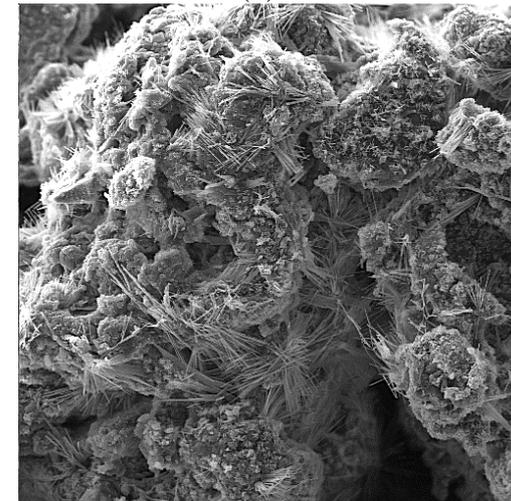


(d)



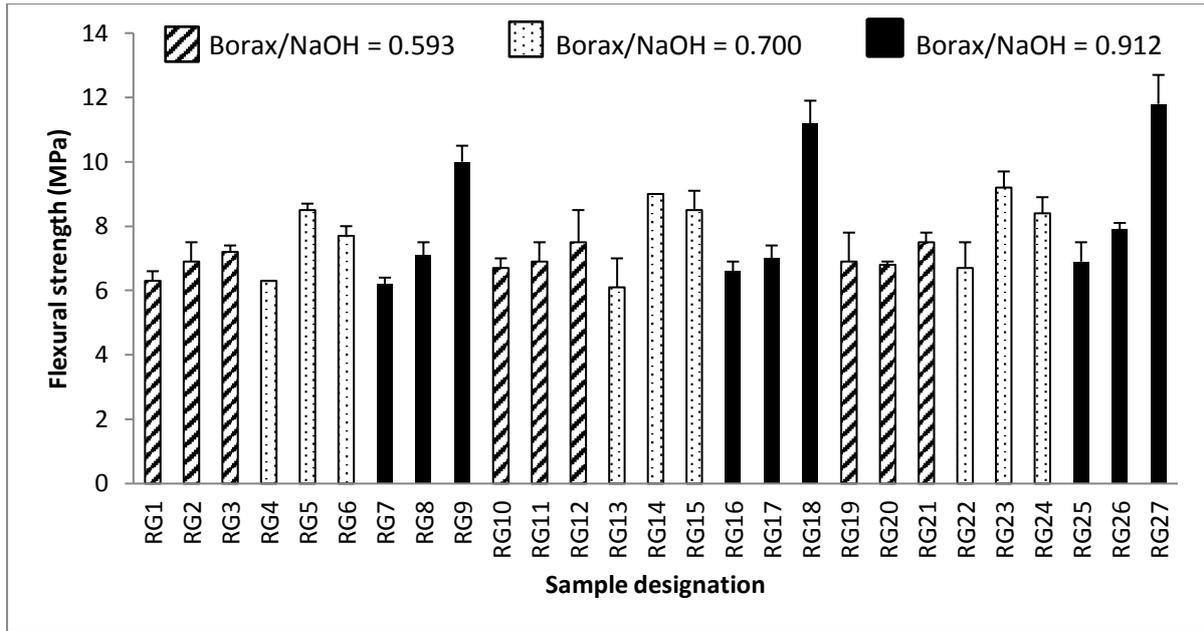
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(e)



(f)

26 Fig. 2. SEM image of a) G1, b) G4, c) G5, d) G6, e) G9 and f) G10 specimens



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28 Fig. 3. Flexural strength of RG series (reinforced) specimens

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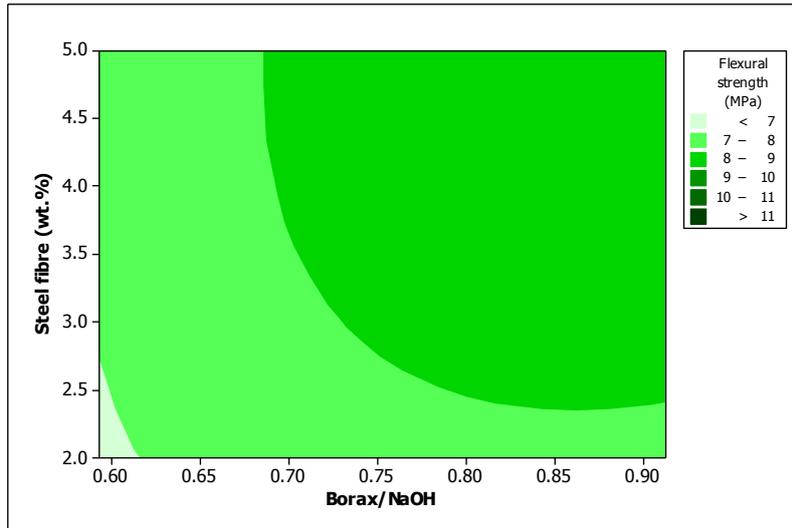
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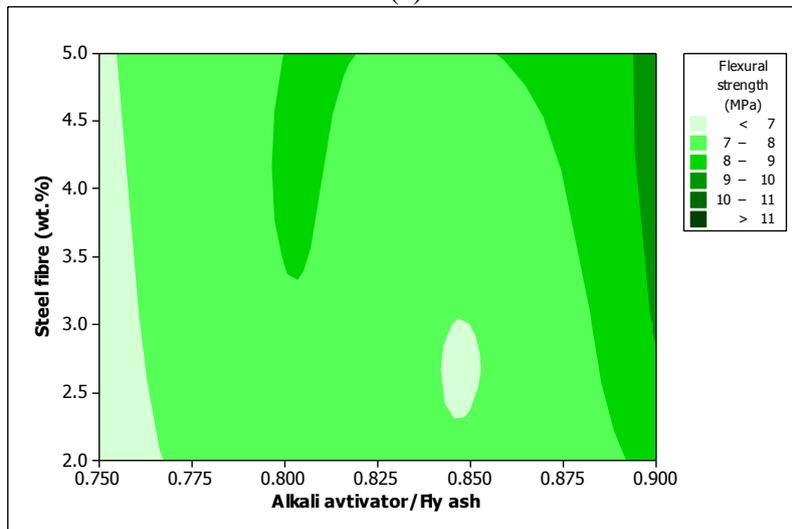
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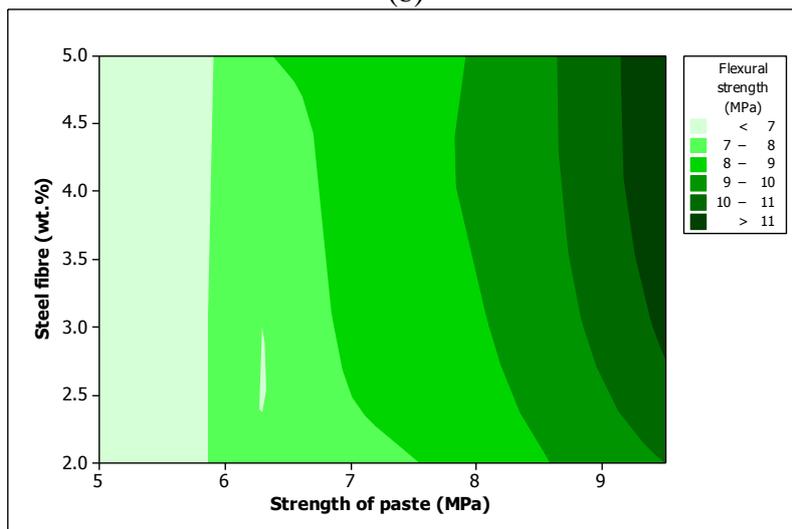
(a)

42
43



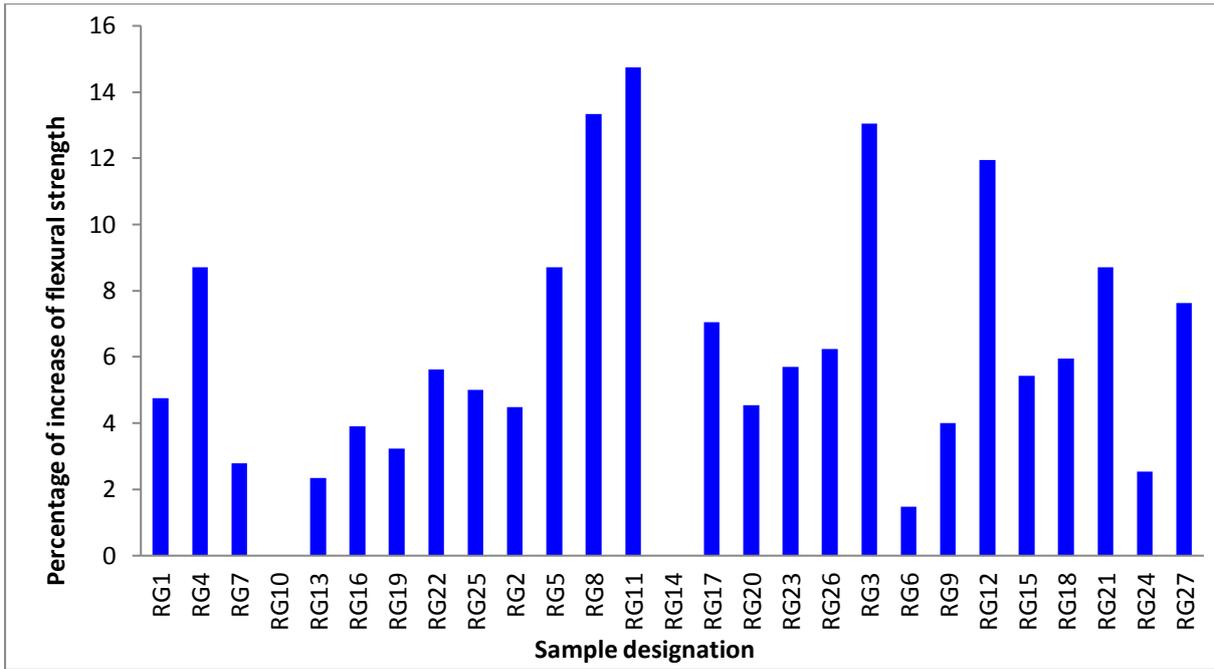
(b)

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(c)

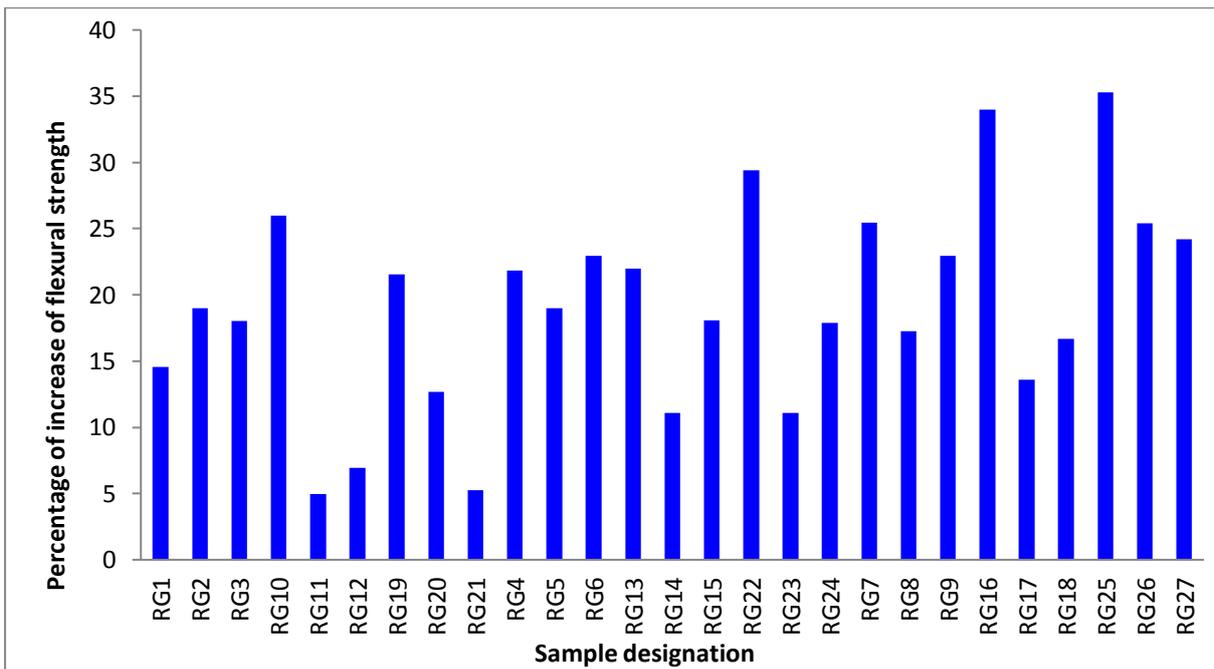
46 Fig. 4. Effect of a) borax to NaOH solution weight ratio, b) alkali activator to fly ash weight
47 ratio and c) strength of the unreinforced paste on flexural strength of RG (reinforced)
48 specimens containing different percentages of steel fibres



49

50

(a)



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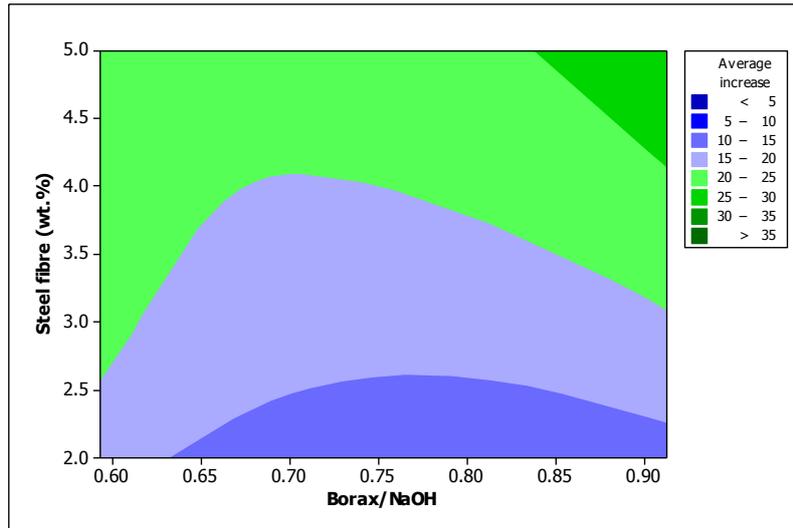
52

(b)

53 Fig. 5. Average percentage of increase of flexural strength in RG series specimens by adding
 54 steel fibres into G series specimens in two different arrangements: a) arranged by increasing
 55 borax to NaOH solution weight ratio from left to right and b) arranged by increasing alkali
 56 activator to fly ash weight ratio from left to right

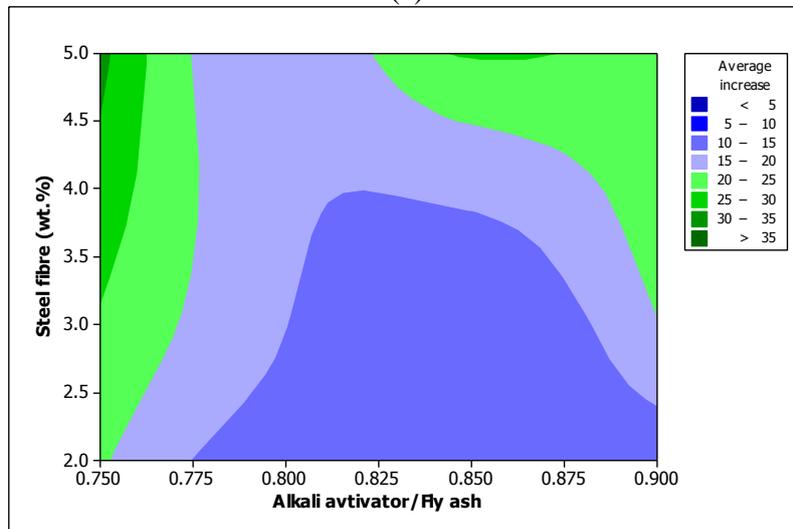
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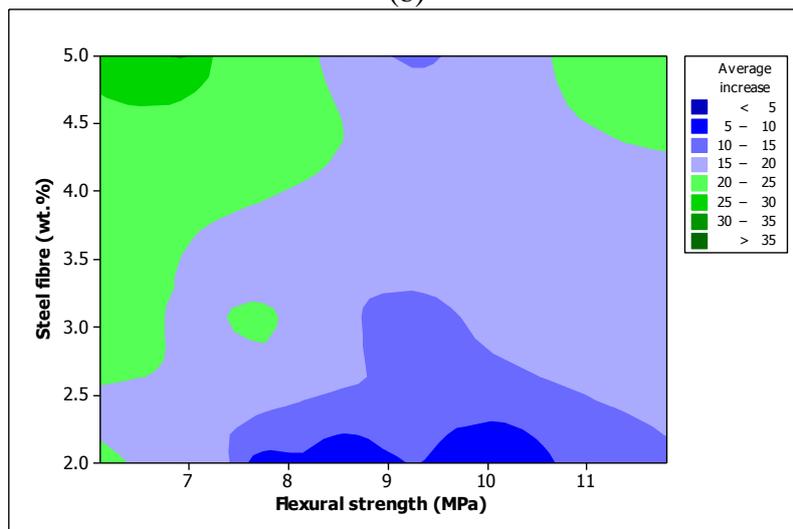
(a)

60
61



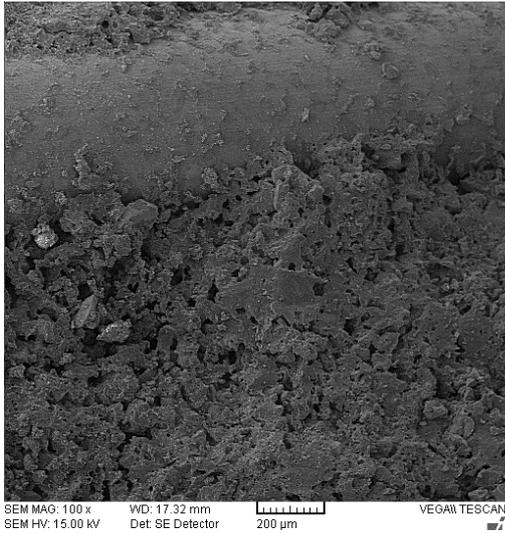
(b)

62
63



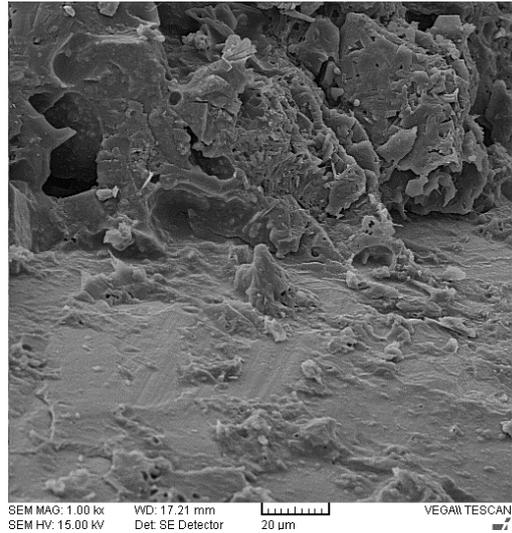
(c)

64 Fig. 6. Effect of a) borax to NaOH solution weight ratio, b) alkali activator to fly ash weight
65 ratio and c) strength of the unreinforced paste on average of increase (%) of flexural strength
66 of RG (reinforced) specimens containing different percentages of steel fibres



67

(a)



(b)

68

69 Fig. 7. SEM images of a) a steel fibre in geopolymeric paste and b) same picture at higher

70 magnification