

Strength development of soil-fly ash geopolymer: Assessment of soil, fly ash, alkali activators and water

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Abstract

In this study, fly ash was added to residual soil to produce soil-fly ash geopolymer bricks. This study investigated the effects of fly ash/soil, alkali activator/ash, $\text{Na}_2\text{SiO}_3/\text{KOH}$ (or NaOH), additional water content, curing condition and curing temperature on the compressive strength of soil-fly ash geopolymer. The results showed that the optimum compressive strength was obtained when the ratios of alkali activator/ash, $\text{Na}_2\text{SiO}_3/\text{KOH}$ (or NaOH) and additional water were 0.6, 0.5 and 10 % respectively. Higher amount of alkali activators was required for strength development in soil-fly ash geopolymer than conventional fly ash-based geopolymers. The formation of the rigid structure at low ratios of alkali activator/ash and $\text{Na}_2\text{SiO}_3/\text{KOH}$ (or NaOH) was unlikely. Compressive strength decreased when additional water was increased. High curing temperature and long curing duration showed negative effect on strength development. The compressive strength of soil-fly ash geopolymer varied as different mixing sequences of raw materials were used, indicating the importance of the formation of geopolymer gel in the structure.

29 Compressive strength results obtained in this study demonstrate that soil-fly ash geopolymer can
30 be a potential alternative to traditional clay fired brick.

31

32 **Keyword**

33 Soil; fly ash; geopolymer; alkali activators; compressive strength

34

35 **Introduction**

36 Clay fired brick is commonly used as construction material. The making process requires firing
37 process which is kilned under high temperature for strength development. During the firing process,
38 it consumes high energy derived by burning charcoal to increase the temperature. Carbon dioxide
39 emission due to high energy consumption pollutes the atmosphere by contributing to the
40 greenhouse effect. Developing a greener material can effectively reduce the exposure of hazard to
41 the surrounding environment, thus being more environmentally friendly. Different types of
42 industrial by-product such as fly ash, sawdust, waste bricks, biomass waste and olive pomace ash
43 have been reported and added into fired clay brick by other researchers (Bories et al. 2015; Casa
44 and Castro 2014; Velasco et al. 2014a; Zhang 2013).

45

46 Fly ash, an aluminosilicate material, is an industrial by-product from the combustion of coal at
47 power stations. Geopolymer, an inorganic aluminosilicate polymer, is produced by activating the
48 aluminosilicate materials such as fly ash, bottom ash, metakaolin and slag in an alkaline
49 environment at the low curing temperature (Davidovits 2008). Various types of alkali activator
50 can be used to activate fly ash such as sodium hydroxide, sodium silicate, potassium hydroxide
51 etc. (Criado et al. 2007; Heah et al. 2012; Nematollahi and Sanjayan 2014; Ryu et al. 2013) to

52 produce geopolymer gel. The combined use of sodium hydroxide and sodium silicate as alkali
53 activators for making geopolymer exhibited good compressive strength (Hardjito and Rangan
54 2005). The formation of three-dimensional polymer chains can be attributed to three main stages,
55 i.e. dissolution of silica and alumina from the aluminosilicate source, reorganisation and
56 polycondensation (Davidovits 2008). The formation of geopolymer gel in the structure was
57 confirmed through the microstructure images (Komljenovi et al. 2010; Tennakoon et al. 2014),
58 contributing to the strength development. Geopolymer has been used as a sustainable construction
59 material in structural elements of buildings such as beams, columns and piles (Power Pile 2013;
60 Sarker 2008; Shrest 2013; Urettek 2014), besides also being viable as a ground treatment method
61 when mixed with weaker clayey soils (Cristelo et al. 2013; Cristelo et al. 2012; Zhang et al. 2013).
62 Being an environmentally friendly construction material, the production of geopolymer emits 80 %
63 less carbon dioxide than manufacturing traditional Portland cement (Duxson et al. 2007).

64

65 Other than making fly ash based geopolymer, fly ash can be used to produce clay-fly ash
66 geopolymer (Phetchuay et al. 2014; Sukmak et al. 2013a; Xu et al. 2005). Sukmak et al. (2013a)
67 reported that the amount of alkali activators and fly ash/clay content correspond directly to its
68 compressive strength. The maximum compressive strength was obtained at the ratio of alkali
69 activator/ash of 0.5 to 0.6 and the ratio of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ of 0.7. The increase of amorphous phase
70 of SiO_2 and the Al_2O_3 due to higher ratios of fly ash increases the strength capability (Lingling et
71 al. 2005). However, SiO_2 and Al_2O_3 in clay particles are mostly of the crystalline phase (Phetchuay
72 et al. 2014), indicating the formation of geopolymer gel due to the chemical reaction amongst the
73 clay particles and alkali activators is unlikely. The water content within the sample influent the
74 compressive strength of geopolymer (Phetchuay et al. 2016).

75

76 Diop et al. (2014) reported that different curing temperature and curing time led to different
77 strength capability. High curing temperature and long curing time corresponded to higher
78 compressive strength. The formation of geopolymer gel due to better geopolymerisation enhanced
79 the strength capabilities (Phetchuay et al. 2014; Phummiphan et al. 2016a). The geopolymer
80 structure became more rigid after curing for certain period in comparison to its early stage (Diop
81 et al. 2011). This could be due to the geopolymer that formed became more stable as the curing
82 time increased. Zhang et al. (2012) explained the importance of pre-curing process at room
83 temperature. As water content within the sample evaporated during the pre-curing process,
84 cracking at the later curing stage at high temperature was prevented. Hence, strength development
85 was improved. The formation of sponge-like aluminosilicate species was observed amongst the
86 clay particles in the geopolymer structure (Ogundiran and Kumar 2015). The formation of
87 geopolymer gel in clay-fly ash geopolymer was confirmed through scanning electron microscopy
88 by researchers (Diop et al. 2014; Phetchuay et al. 2014; Phummiphan et al. 2016b; Sukmak et al.
89 2013a; Xu et al. 2005). It bound the clay particles together thus forming a rigid structure (Diop et
90 al. 2014). However, the morphology studies on the soil-fly ash geopolymer at different ratios of
91 fly ash/soil and additional water content are found to be rather limited.

92

93 In this study, residual soil is used to produce soil-fly ash geopolymer. The main purpose of this
94 study is to assess the effect of the ratio of fly ash/soil, the ratio of alkali activator/ash, the ratio of
95 $\text{Na}_2\text{SiO}_3/\text{KOH}$ (or NaOH), additional water content, curing condition and curing temperature on
96 the compressive strength of soil-fly ash geopolymer. This study aims to develop further the

97 application of fly ash other than producing the conventional fly ash-based geopolymer, utilizing
98 the method, which is analogous to the brick-making industry to produce soil-fly ash geopolymer.

99

100 **Experimental Procedure**

101 *Materials*

102 *Fly ash*

103 In this study, Sarawak fly ash which is available in the local industry was used. It was obtained
104 from Sejingkat Power Station, which is located in the Sarawak state capital of Kuching in Malaysia.
105 Sarawak fly ash has been successfully used in a handful of studies (Leong et al. 2015; Leong et al.
106 2016a; Leong et al. 2016b). **Table 1** shows the chemical compositions of the fly ash using WD-
107 X-ray Fluorescence Spectrometer (WD-XRF). It is classified as Class F type in accordance to
108 ASTM-C618 (2005), containing low calcium content and hence having poor self-cementing
109 properties. Alkali activators are essentially required to activate the fly ash content for
110 geopolymerisation and strength development.

111

112 *Soil*

113 The residual soil was sourced from the outskirts of Kuching city, Malaysia. It is classified as reddish
114 brown slightly sandy silt with a natural moisture content of 53 %. It consists of 11 % gravel, 13 %
115 sand, 34 % silt and 42 % clay. The liquid limit, plastic limit and plasticity index of the residual
116 soil are 66 %, 36 % and 30 % respectively. The optimum moisture content (OMC) and the
117 maximum dry density (MDD) of the residual soil are 47 % and 1184 kg/m³. The chemical
118 compositions of the residual soil are tabulated in **Table 1**. It contains relatively lower SiO₂ content
119 but relatively higher Al₂O₃ and Fe₂O₃ contents than fly ash. **Fig. 1** shows the morphologies of fly

120 ash and soil under a scanning electron microscopy (SEM). As seen in the SEM images, fly ash
121 mainly consists of spherical shape whereas soil shows irregular shapes and exhibits porous-like
122 structures. The particle size distributions of both fly ash and soil are plotted in **Fig. 2**. It
123 demonstrates that fly ash has much smaller particle sizes than soil. However, the latter has wider
124 distributions of particle size. It is mainly due to the physical and chemical weathering processes of
125 the parent rock to form the residual soil, making it behaves more heterogeneous rather than
126 homogenous (Wesley 2010). The difference of particle size distributions of both fly ash and soil
127 is postulated to be an important factor for strength development, which will be discussed further
128 in the next section.

129

130 *Alkali activators*

131 8M potassium hydroxide (KOH) solution and 8M sodium hydroxide (NaOH) solution were
132 prepared (Leong et al. 2016a) from KOH pellets (brand: Merck) and NaOH pellets (brand: Hudson)
133 respectively. The combined use of potassium hydroxide or sodium hydroxide and sodium silicate
134 (Na_2SiO_3) (17% Na_2O and 35% SiO_2 by weight) was used as the alkali activators in this study.
135 This combination is the most common used in practice in terms of cost effectiveness to produce
136 geopolymer with good compressive strength than the sole use of alkali silicate or hydroxide
137 (Hardjito & Rangan 2005). High concentrations of alkali are essential to activate fly ash particles
138 to form geopolymer gel for strength development. It also facilitates a greater degree of
139 geopolymerisation (Diop, Grutzeck & Molez 2011). 8M NaOH exhibits the optimal concentration
140 for the optimum compressive strength in term of long term performance (Diop et al. 2014).

141

142 ***Sample Preparation and Mixture Proportions***

143 Different ratios of fly ash/soil were studied (0, 0.3, 0.6, 0.8 and 0.9). The ratio of alkali
144 activator/ash was varied in the range of 0.4 to 0.7 and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ (or KOH) = 0.5, 1 were
145 studied. The residual soil was placed in the oven for it to be dried out for 24 hours at 100 °C. The
146 effect of water content on compressive strength of geopolymer was investigated by adding
147 additional water content (i.e. 10 %, 20 %, 30 % and 40 %) into the mixture. The dry soil was
148 initially premixed with fly ash in a mixer. The alkali activators and additional water content were
149 added into the mixture for further mixing until homogenous state was obtained. The mixture was
150 poured into the modified cube mould (50 mm × 50 mm × 120 mm) as presented in **Fig. 3a**. The
151 compression machine was used to press the sample into cubes with dimensions of 50 mm × 50
152 mm × 50 mm (see **Fig. 3b**) at a press load of 10 kN as shown in **Fig. 3c**. This press load was fixed
153 at 10 kN due to the limitation of the modified cube mould. The mould might deform if a press load
154 higher than 10kN was applied. The samples were then demoulded and cured in an oven at different
155 curing temperatures (i.e. 60 °C, 100 °C or 200 °C) to investigate the effect of different curing
156 temperatures on compressive strength of geopolymer. Different curing time of the samples was
157 also studied (i.e. dried at room temperature for 2 days then cured in oven for 1 day, direct cured in
158 oven for 1 day and direct cured in oven for 2 days). **Table 2** shows the designations of soil-fly ash
159 geopolymers studied in this study.

160

161 *Test*

162 *Workability*

163 The mixture was placed into the conical mould on top of the flow table for the workability test
164 immediately after finishing the mixing process. The test was conducted in accordance to ASTM-
165 C1437 (2013).

166

167 *Compression test*

168 Samples were tested for compressive strength after oven curing. The compressive strength of the
169 samples was obtained using compression test machine in accordance to ASTM-C109/C109M
170 (2005).

171

172 *Particle Size Distributions*

173 The particle size distributions of the fly ash were analysed using the laser particle size analyser
174 (brand: CILAS 1190). However, for the soil, it was obtained using dry sieving and hydrometer test
175 due to its larger particle sizes as compared to the fly ash. The test was performed in accordance to
176 BS-1377-2 (1990).

177

178 *Scanning Electron Microscope (SEM)*

179 The morphology of the soil-fly ash geopolymer was studied using ZEISS SUPRA 40 VP SEM. A
180 layer of gold was coated on the sample prior to the micrograph analysis.

181

182 **Results and discussion**

183 *Effect of additional water content on compressive strength of geopolymer*

184 **Fig. 4** shows the effect of additional water content on compressive strength of soil-fly ash
185 geopolymer using different ratios of alkali activator/ash and $\text{Na}_2\text{SiO}_3/\text{NaOH}(\text{KOH})$. The results
186 show that the reduction of additional water content intensively increases the compressive strength
187 of sample. Samples with the ratio of alkali activator/ash of 0.5 to 0.7 exhibit the highest
188 compressive strength at additional water content of 10%, after which it gradually decreases with
189 the increase of additional water content. It may be due to the reduction of alkalinity in the mixture,
190 which will be discussed here-in-after. Mixture with further increase of additional water appears to
191 be very liquid, particularly at additional water content of 40 %. These mixtures were directly cast
192 into the mould with dimensions of 50 mm × 50 mm × 50 mm as denoted by L in **Fig. 4**.

193

194 Geopolymer made of alkali activator/ash = 0.4 shows different trend in strength. The optimum
195 compressive strength was obtained at additional water of 20 to 30 %. It is most probably because
196 the mixture at additional water of 10 % is very dry. The sample appeared to be easily broken when
197 minimal load was applied on it. As the additional water increases, it acts as lubricant to improve
198 the formation of rigid structure and hence, it leads to higher strength capability.

199

200 The workability of these mixtures is depicted in **Fig. 5**. It shows similar trend of workability as fly
201 ash-based geopolymer as reported by Leong et al. (2016a). The workability increases when the
202 additional water content and alkali activators increase. From the workability point of view, it may
203 be beneficial to brick making industry using extrusion method. It is important to mention that
204 geopolymer with additional water content of 0% was unable to produce. It is because that the

205 sample at this mixture ratio was very dry and it couldn't form into shape. Hence, the comparison
206 between the sample made with and without additional water content was not available.

207

208 Samples made using the press method appear to be more rigid in comparison to those made without
209 using the press method. It is because through pressing the air bubbles/voids within the sample
210 escape into the atmosphere when load is applied to the samples. Hence, entrapped air within the
211 sample is squeezed out from the void spaces thus making the particles closely packed together. It
212 is important to note that the press method is somewhat different from the consolidation process
213 where water content is removed from the sample. Furthermore, the alkali activators within the
214 sample may be further diluted by the excessive additional water content thus reducing the strength
215 capability. The results show that the compressive strength is nearly negligible when the ratio of
216 alkali activator/ash and additional water increase (i.e. when additional water is 40% and the ratio
217 of alkali activator/ash ranges from 0.4 to 0.7). This observation provides a significant insight into
218 the effect of water on compressive strength and the optimum water content for making soil-fly ash
219 geopolymer.

220

221 **Fig. 6** depicts the morphology images of the soil-fly ash geopolymer at different ratios of additional
222 water content but having same ratios of alkali activators/ash, $\text{Na}_2\text{SiO}_3/\text{NaOH}$ and fly ash/soil (i.e.
223 SFG11). Geopolymer at these ratios were selected for morphology studies in terms of good
224 compressive strength and the common use of NaOH and Na_2SiO_3 as alkali activators. **Fig. 6a**
225 shows the SEM image of soil-fly ash geopolymer with additional water content of 10 %. The
226 structure was observed to be very dense and rigid. The geopolymer gel was formed surrounding
227 the soil particles, indicating better geopolymerisation. At optimum water content, it reduces the

228 distance between soil and fly ash particles. Thus, it leads to higher compressive strength (Sukmak
229 et al. 2013). However, the geopolymer structure became looser and cracks were visible within the
230 samples when the additional water content increased (see **Fig. 6b**). Layer of alkali activators in
231 excess seems present in the structure as shown in **Fig. 6c**. It is most probably due to the high water
232 content in the sample, which reduces the contact points amongst fly ash particles and alkali
233 activators for further reaction. At high temperature exposure, the water content within the sample
234 evaporates thus making the alkali activators, which have not used in the geopolymerisation, remain
235 in the structure. Some tiny pores were observed within the sample due to the evaporation of water
236 content. It can be seen that the unreacted fly ash particles were embedded within the sample. This
237 phenomenon is evidently shown in **Fig. 6d**. The large voids that remained were caused by the
238 unreacted fly ash particles. As the concentration of alkali activator was further reduced by higher
239 additional water content, the formation of geopolymer gel was lesser and therefore more unreacted
240 fly ash particles were presented within the sample. As the bonding of the structure is weaker, these
241 unreacted fly ash particles could be easily broken.

242

243 As discussed earlier, the alkalinity of the alkali activator could be reduced by the addition of water
244 subsequently, resulting in reduction of geopolymer strength. The pOH value increased as the
245 additional water increased subsequently reducing the pH value. In high pH environment, the soil
246 particles are expected to be dispersed while the soil particles are flocculated when pH value is low
247 (Mitchell and Soga 2005). As NaOH (or KOH) is categorised as strong base whereas Na₂SiO₃ is a
248 weak base, the decrease in strength is governed by the variation of alkalinity in NaOH. Moreover,
249 Na₂SiO₃ requires dissociation of water to form OH⁻. The evidence of reduction in pH values in
250 NaOH as the additional water content increases is shown in **Fig. 7**. It is noted that the pH values

251 are greater than 14 as shown in **Fig. 7**. Commonly, it is in the range of 1 to 14. It can be attributed
252 to the high initial molarity of alkali activator in this study (i.e. 8M NaOH solution). The
253 concentration of NaOH in the sample also decreases as the water content increases as presented in
254 **Fig. 8**. Hence, less fly ash particles are dissolved due to the lower concentration of alkali activators.
255 The decline of geopolymer strength was reported as a result of less geopolymerisation. It is
256 consistent with the observation in **Fig. 6** that unreacted fly ash particles increase when the water
257 content increases. The example of evaluation of molarity and pH value in NaOH is shown in
258 appendices.

259

260 Soil and water particles are not chemically or biologically inert. Other than the absorbed water in
261 soil particles, there are some interactions amongst soil particles, dissolved ions and water
262 molecules. The possible mechanisms that occur between water and soil particles are identified as
263 hydrogen bonding, ion hydration, attraction by osmosis and dipole attraction (Mitchell and Soga
264 2005). As water molecules have dipolar character, its positive poles are attracted to the negatively
265 charged soil surface particularly in clay particles. The bonds between water molecules and soil
266 particles thus creating an interface layer. It reflects that soil particles are further apart from
267 interacting the fly ash particles or the geopolymer gel formed (due to the reaction amongst fly ash
268 and alkali activators) by the interface layer in close proximity. For the coarser fraction in soil, it
269 has lower specific surface area as compared to the clay particles, causing greater thickness of water
270 layer between soil particles. It indicates that the surface interaction effect is relatively much lower.
271 More water is available and mobilised as the additional water content increases. Therefore, the
272 alkalinity of the alkali activator as mentioned previously could be possibly reduced. It is worth
273 mentioning that soil studied in this study may have positive surface charge. However, the cation

274 exchange capability in soil could be much greater than the anion exchange capability due to the
275 high pH environment (Hendershot et al. 2007) in soil-fly ash geopolymer system.

276

277 *Effect of Na₂SiO₃/NaOH(KOH) on compressive strength*

278 The effect of Na₂SiO₃/NaOH(KOH) on compressive strength of soil-fly ash geopolymer was also
279 studied as shown in **Fig. 4**. Both NaOH- and KOH-based soil-fly ash geopolymer demonstrate the
280 highest compressive strength at the ratios of alkali activator/ash and Na₂SiO₃/NaOH at 0.6 and 0.5
281 respectively. The NaOH-based geopolymer exhibits lower compressive strength than the KOH-
282 based geopolymer. It could be due to more geopolymer precursors that exist in the geopolymer
283 system when KOH solution is used, hence demonstrating higher strength capability (Xu and
284 Deventer 2000). These results show similar findings as those reported in the previous study when
285 fly ash-based geopolymer was studied (Leong et al. 2016a).

286

287 It is suspected that the alkali hydroxide may be more important for strength development than
288 alkali silicate at the optimum additional water content in order to obtain the maximum compressive
289 strength. It is known that alkali hydroxide plays a role of leaching the Si and Al content from the
290 reacting fly ash whereas alkali silicate aids in increasing the Si content in the system for
291 geopolymerisation (Xu and Deventer 2000). Sukmak et al. (2013a) reported that partial alkali
292 hydroxide might be absorbed for soil cation exchange and therefore more alkali hydroxide was
293 required than alkali silicate. However, the effect of Na₂SiO₃/KOH(or NaOH) on compressive
294 strength was further investigated as shown in **Fig. 9**. The results show that the compressive strength
295 increases when Na₂SiO₃/KOH (or NaOH) is increased. However, it decreases with further increase
296 of Na₂SiO₃/KOH(or NaOH). It could be due to an excessive amount of alkali hydroxide within the

297 sample as the ratio increases. It may be presented as salt precipitates which hinder the
298 geopolymerisation thus resulting in strength reduction. Hence, it is suggested that an optimum
299 amount of alkali hydroxide and alkali silicate could possibly obtain the maximum compressive
300 strength. This condition is attainable when $\text{Na}_2\text{SiO}_3/\text{KOH}$ (or NaOH) is 0.5.

301

302 *Effect of curing condition and curing temperature on compressive strength*

303 Soil-fly ash geopolymer at the ratios of alkali activator/ash of 0.6 and $\text{Na}_2\text{SiO}_3/\text{KOH}$ (or NaOH) of
304 0.5 (i.e. the sample exhibits the highest compressive strength) was further studied at different types
305 of curing condition and curing temperature. As shown in **Fig. 10**, the compressive strength
306 gradually increases as the curing temperature increases with the exception of sample cured directly
307 in oven for 1 day. Diop et al. (2014) explained that the strength gain with the increase of curing
308 temperature was most probably attributed to the dehydroxylation, disintegration and
309 polycondensation of aluminosilicate polymer between the soil particles and alkali activators. In
310 this study, it shows that the loss of moisture increases as the curing temperature increases. It is
311 thought that the sample which was initially dried at room temperature for 2 days could aid in
312 removing the water content within the sample thus enhancing the geopolymerisation between fly
313 ash and alkali activators subsequently leading to higher compressive strength. However, on the
314 contrary, the moisture loss at this type of curing condition shows similarity to the other types of
315 curing condition. It shows that room temperature curing only aids in removing the moisture on
316 sample surface. For moisture within the sample, it requires oven heating and thus it explains
317 moisture loss due to oven curing is similar.

318

319 Comparing the geopolymer samples cured in oven for 1 day and 2 days, strength gain over longer
320 curing duration was observed in sample cured at 60 °C only. It is most likely due to the increase
321 of moisture loss. Samples cured at low temperature might need longer duration of oven curing to
322 build up the strength (Sukmak et al. 2013b). However, having the same curing duration at 100°C
323 and 200°C, strength reduction was observed. It could be due to high temperature exposure and
324 more cracks develop in the sample. The loss of moisture from 1-day oven curing to 2-day oven
325 curing is minimal. This implies that most of the moisture had been evaporated within first 24 hours.
326 It also demonstrated that longer duration of oven curing does not necessarily lead to higher
327 compressive strength but resulting in strength reduction in particular to the sample cured at high
328 temperature.

329

330 The highest compressive strength is obtained when the samples were cured directly in oven for 1
331 day. It intensively increased from 60°C to 100°C and decreased from 100°C to 200°C. This
332 suggested that sample cured in oven plays an important role in strength development for soil-fly
333 ash geopolymer. Additionally, it is postulated that sample cured at this type of curing condition
334 (i.e. directly cured in oven for 1 day at 100°C) achieved the initial maximum strength. As
335 mentioned earlier, moisture within the sample might not able to evaporate at low curing
336 temperature. High temperature exposure may result in crack development subsequently reducing
337 the strength capabilities. An adequate curing temperature (i.e. 100 °C in this case) facilitates the
338 moisture evaporation and geopolymerisation for forming a rigid structure within first 24 hours.
339 Therefore, this condition may be adequate for making soil-fly ash geopolymer as it required short
340 curing duration, moderate curing temperature but it exhibits the highest strength capability.

341

342 **Fig. 11a-c** presents the morphologies of the geopolymer samples cured directly in oven for 1 day
343 at different curing temperatures. At 60°C, agglomeration of particles was observed. The
344 geopolymer structure appeared to be non-homogenous and consisted coagulation of fly ash
345 particles and alkali activators around the soil particles. The geopolymer gel formed was distributed
346 in scatter within the sample. Moreover, some unreacted or partially dissolved fly ash particles were
347 also present as marked in the figure. This observation shows that the geopolymerisation process is
348 incomplete. The geopolymer sample depicted in **Fig. 11b** exhibits more homogeneity and a more
349 compact microstructure. Furthermore, the phenomenon described in **Fig. 11a** did not occur. It
350 implied that geopolymer cured at 100°C enhances the geopolymerisation for soil-fly ash
351 geopolymer. When the curing temperature increases to 200°C, different structure was observed as
352 illustrated in **Fig. 11c**. The soil particles and geopolymer gel seem to have fused together due to
353 high temperature exposure. However, the sample showed less dense structure and cracks occurred
354 as marked. Evaporation of the water content at high temperature increased the porosity within the
355 sample thus causing the strength to decrease.

356

357 *Effect of fly ash/soil on compressive strength*

358 The effect of the ratio of fly ash/soil on compressive strength was studied and illustrated in **Fig.**
359 **12**. The compressive strength gradually increased when the ratio of fly ash/soil increased. It is
360 most likely due to the increase of fly ash content, which enhances the geopolymerisation within
361 the sample. It is evident that the geopolymer strength increased as the amount of alkali activators
362 increased regardless of the types of alkali activator. The amorphous phase of SiO₂ and Al₂O₃
363 contents could be leached out from the fly ash. Hence, more geopolymer could be sufficiently
364 formed to bind the soil particles together thus increasing the compressive strength. Although soil

365 contains high SiO_2 and Al_2O_3 contents, it is mostly presented in the crystalline phase (Phetchuay
366 et al. 2014). It acts as fine aggregate (Sukmak et al. 2013a) and contribution to the formation of
367 geopolymer gel is unlikely. It is also noted that Na_2SiO_3 content is lower than NaOH content in
368 geopolymer. As reported in previous study, Na_2SiO_3 provides more Si content to the geopolymer
369 system whereas NaOH dissolves alumina and silica from the fly ash particles (Leong et al. 2016a).
370 However, increase of Na_2SiO_3 may obstruct NaOH to dissolve the essential elements for
371 geopolymerisation. Hence, an adequate amount of Na_2SiO_3 and NaOH should be adopted for good
372 strength result. The SEM images in **Fig. 13** show that more geopolymer gel was formed as the
373 ratio of fly ash/soil increased. The strength results obtained in this study had shown well agreement
374 to the microscopy observations.

375

376 More unreacted fly ash particles were observed at low ratio of fly ash/soil. The structure of the
377 sample becomes more rigid and less undissolved fly ash particles were observed as the ratio of fly
378 ash/soil increased. Samples made using 100% fly ash exhibited the most homogenous structure,
379 however, cracks were obviously seen in the sample due to high temperature exposure. Fly ash
380 particles were mostly unreacted in samples cured at room temperature regardless of the ratio of fly
381 ash/soil as shown in **Fig. 14**. Moreover, no obvious changes of the microstructure were observed
382 over the increase of the ratio of fly ash/soil. This observation was in agreement to the strength
383 results and it explained the reason behind the insignificant strength gain. Although low
384 compressive strength was obtained in samples cured at room temperature, the SEM images proved
385 that geopolymer gel could be formed at room temperature. However, it might require longer
386 duration to achieve the desired strength.

387

388 The strength results of the samples made using 100% soil and 100% fly ash demonstrated that the
389 trend of the strength development in terms of the ratio of fly ash/soil is principally caused by the
390 geopolymerisation. Sample made using 100% soil exhibited very low compressive strength when
391 curing at room temperature. As no aluminosilicate gel was formed, it showed good evidences that
392 the geopolymerisation gel could effectively increase the geopolymer strength when fly ash and
393 alkali activators were added into the soil. The latter type of curing condition also showed that the
394 strength gained (i.e. 0.18MPa) was mainly due to the press method at 10kN. Hence, the remaining
395 strength is built up through the temperature exposure and geopolymerisation. Therefore, strength
396 gained due to these two factors can be evaluated by data presented in **Fig. 15**. The results showed
397 that the increase of compressive strength due to the geopolymerisation at room temperature was
398 minimal as compared to the geopolymerisation at temperature exposure. This results show well
399 agreement to the findings obtained in fly ash-based geopolymer as reported by Leong et al. (2016a).
400 These observations showed that soil-fly ash geopolymer behaved similar geopolymerisation as in
401 fly ash-based geopolymer. Curing temperature plays an important role in strength development
402 regardless of the types of raw material.

403

404 In **Fig. 12** and **Fig. 15**, the mixing sequence of raw materials were investigated and denoted as M1
405 and M2. M1 represents the sample premixed with soil and fly ash prior to the addition of liquid
406 content whereas M2 represents premixing fly ash with the liquid content followed by the addition
407 of soil. It is thought that the formation of aluminosilicate gel in M2 could effectively bind the soil
408 particles thus leading to higher strength capability. However, the results reflected that samples
409 prepared from M1 obtain higher strength gain than sample prepared from M2 as tabulated in **Table**
410 **3**. The possible reasons are schematized in **Fig. 16**.

411

412 It is postulated that the fly ash particles are packed closely between the soil particles during the
413 premixing process in M1 as the former particles show much smaller particle sizes as compared to
414 the latter as evidenced in **Fig. 2**. When alkali activators were added into the mixture, it dissolved
415 the fly ash particles surrounding the soil particles. Hence, the aluminosilicate gel was formed
416 around the soil particles. Moreover, the negatively charged surface of soil particle held some of
417 the cations from the alkali activators, resulting in high concentration of absorbed cations near the
418 surface of soil particles. It implied that the anions such as OH^- and SiO_3^{2-} from the alkali activators
419 are adjacent to the fly ash particles, which are the essential ions for dissolution of fly ash particles
420 and the geopolymerisation process. Combining these two factors, a strong interaction and reaction
421 amongst soil particles, fly ash particles and alkali activators are developed, forming a rigid
422 structure for better strength capability.

423

424 For samples prepared from M2, the formation of aluminosilicate gel due the reaction of fly ash
425 particles and alkali activators may not sufficient to bind the soil particles completely. In this case,
426 the binding effect only occurred in limited region of the sample. Therefore, the structure formed
427 may not be as rigid and homogenous as achieved in M1.

428

429 **Conclusion**

430 The effect of the ratio of fly ash/soil, the ratio of alkali activator/ash, the ratio of $\text{Na}_2\text{SiO}_3/\text{KOH}$
431 (or NaOH), additional water content, curing condition and curing temperature on compressive
432 strength of soil-fly ash geopolymer had been successfully studied. The following conclusions can
433 be made:

- 434 1. Soil-fly ash geopolymer obtains the highest compressive strength when the ratio of alkali
435 activator/ash of 0.6, the ratio of $\text{Na}_2\text{SiO}_3/\text{KOH}$ (or NaOH) of 0.5 and additional water of 10 %.
436 The effect of additional water on compressive strength shows similar pattern regardless of the
437 types of alkali activators.
- 438 2. Water is essential in shaping and forming soil-fly ash geopolymer. However, excessive water
439 and alkali activators were not suitable for press method although it shows high workability.
440 The increase in additional water content decreases the alkalinity and molarity of alkali
441 activator, causing the geopolymer strength to decline.
- 442 3. More alkali activators are required for soil-fly ash geopolymer to obtain maximum
443 compressive strength as compared to fly ash-based geopolymer. It organic matter in soil may
444 behave as absorbent for alkali activators.
- 445 4. The compressive strength increases when the curing temperature increases. However, high
446 curing temperature and long curing duration do not lead to higher compressive strength. Loss
447 of moisture was reported during the curing process. Sample directly cured in oven for 1 day
448 at 100 °C exhibits the highest compressive strength.
- 449 5. The compressive strength increases as the ratio of fly ash/soil increases. The increase of fly
450 ash content is associated with more alkali activators that increases the formation of
451 geopolymerisation gel thus resulting in binding of the soil particles.
- 452 6. Different mixing sequences of the raw materials used were also studied. Premixed fly ash and
453 soil before adding the liquid contents shows better formation of geopolymer gel within the
454 sample, thus resulting more significant strength capability.

455 7. In this study, the compressive strength of soil-fly ash geopolymer ranges from 0.88 MPa to
456 32.05 MPa. It is thus evidenced that fly ash and residual soil can be potentially used to produce
457 soil-fly ash geopolymer, an alternative to traditional clay fired brick.

458

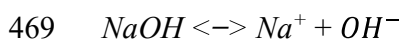
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466

467 **Appendices**

468 **Evaluation of molarity and pH value in NaOH**



470 Assume: 8M NaOH = 106.7 g

471 Water = 50 g

472 Consider 1g/mL,

473 □ Molarity, $M_{NaOH(8M)} = 106.7 \text{ g}$

474 Volume, $V_{NaOH(8M)} = 106.7 \text{ mL}$

475 Number of mol, $n_{NaOH} = (106.7 \text{ mL} \times 10^{-3}) \times 8M$

476 = 0.853 mol

477 Volume of solution = $(106.7 + 50) \times 10^{-3}$

478 $= 0.157 \text{ L}$

479 Molarity of $\text{OH}^- = \frac{0.853 \text{ mol}}{0.157 \text{ L}}$

480 $= 5.45 \text{ M}$

481 $\text{p}(\text{OH}^-) = -\log_{10}(\text{OH}^-)$

482 $= -\log_{10}(5.45)$

483 $= -0.74$

484 $\text{pH} = 14 - (-0.74)$

485 $= 14.74$

486

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604 **Table 1.** Chemical Composition of Fly Ash and Residual Soil

Elements (%)	Sarawak Fly Ash	Residual Soil
SiO ₂	43.8	32.71
Al ₂ O ₃	18.1	25.33
Fe ₂ O ₃	7.7	21.29
CaO	3.9	0.04
MgO	0.5	0.22
MnO	22.8	-
K ₂ O	2.0	0.03
Na ₂ O	0.3	0.07
SO ₃	0.1	0.01
TiO ₂	0.6	-
P ₂ O ₅	0.1	-
LOI	0.5	17.18

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626 **Table 2.** Designations of soil-fly ash geopolymers

Fly ash/ soil	Alkali activator/ ash	NaOH or KOH	Na ₂ SiO ₃ / NaOH (or KOH)	Water (%)	Curing temperature (°C)	Curing time	Mixing sequence	Designation
0.8	0.4	KOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG1
0.8	0.5	KOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG2
0.8	0.6	KOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG3
0.8	0.7	KOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG4
0.8	0.4	KOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG5
0.8	0.5	KOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG6
0.8	0.6	KOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG7
0.8	0.7	KOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG8
0.8	0.4	NaOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG9
0.8	0.5	NaOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG10
0.8	0.6	NaOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG11
0.8	0.7	NaOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG12
0.8	0.4	NaOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG13
0.8	0.5	NaOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG14
0.8	0.6	NaOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG15
0.8	0.7	NaOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG16

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0.8	0.6	KOH	0.1, 0.3, 0.5, 1	10	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG17
0.8	0.6	NaOH	0.1, 0.3, 0.5, 1	10	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG18
0.8	0.6	KOH	0.5	10	60, 100, 200	Dried at room temperature for 2 days then cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG19
0.8	0.6	KOH	0.5	10	60, 100, 200	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG20
0.8	0.6	KOH	0.5	10	60, 100, 200	Directly cured in oven for 2 days	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG21
0.8	0.6	NaOH	0.5	10	60, 100, 200	Dried at room temperature for 2 days then cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG22
0.8	0.6	NaOH	0.5	10	60, 100, 200	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG23
0.8	0.6	NaOH	0.5	10	60, 100, 200	Directly cured in oven for 2 days	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG24
100% soil, 0.3, 0.6, 0.8, 0.9, 100% fly ash	0.6	NaOH	0.5	10	Room temperature	Directly cured at room temperature for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG25
100% soil, 0.3, 0.6, 0.8, 0.9, 100% fly ash	0.6	NaOH	0.5	10	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG26
100% soil, 0.3, 0.6, 0.8, 0.9, 100% fly ash	0.6	NaOH	0.5	10	100	Directly cured in oven for 1 day	Fly ash and the liquid content were premixed followed by the addition of soil (M2)	SFG27

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638 **Table 3.** Strength increment due to sequence of mixing

Ratio of fly ash/soil	M1 (%)	M2 (%)
0.3	45.08	66.95
0.6	86.22	78.25
0.8	92.30	80.29
0.9	90.81	77.53

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Table 1. Chemical Composition of Fly Ash and Residual Soil

Elements (%)	Sarawak Fly Ash	Residual Soil
SiO ₂	43.8	32.71
Al ₂ O ₃	18.1	25.33
Fe ₂ O ₃	7.7	21.29
CaO	3.9	0.04
MgO	0.5	0.22
MnO	22.8	-
K ₂ O	2.0	0.03
Na ₂ O	0.3	0.07
SO ₃	0.1	0.01
TiO ₂	0.6	-
P ₂ O ₅	0.1	-
LOI	0.5	17.18

Table 2. Designations of soil-fly ash geopolymers

Fly ash/ soil	Alkali activator/ ash	NaOH or KOH	Na ₂ SiO ₃ / NaOH (or KOH)	Water (%)	Curing temperature (°C)	Curing time	Mixing sequence	Designation
0.8	0.4	KOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG1
0.8	0.5	KOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG2
0.8	0.6	KOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG3
0.8	0.7	KOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG4
0.8	0.4	KOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG5
0.8	0.5	KOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG6
0.8	0.6	KOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG7
0.8	0.7	KOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG8
0.8	0.4	NaOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG9
0.8	0.5	NaOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG10
0.8	0.6	NaOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG11
0.8	0.7	NaOH	0.5	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG12
0.8	0.4	NaOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG13
0.8	0.5	NaOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG14
0.8	0.6	NaOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG15
0.8	0.7	NaOH	1	10, 20, 30, 40	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG16

0.8	0.6	KOH	0.1, 0.3, 0.5, 1	10	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG17
0.8	0.6	NaOH	0.1, 0.3, 0.5, 1	10	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG18
0.8	0.6	KOH	0.5	10	60, 100, 200	Dried at room temperature for 2 days then cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG19
0.8	0.6	KOH	0.5	10	60, 100, 200	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG20
0.8	0.6	KOH	0.5	10	60, 100, 200	Directly cured in oven for 2 days	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG21
0.8	0.6	NaOH	0.5	10	60, 100, 200	Dried at room temperature for 2 days then cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG22
0.8	0.6	NaOH	0.5	10	60, 100, 200	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG23
0.8	0.6	NaOH	0.5	10	60, 100, 200	Directly cured in oven for 2 days	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG24
100% soil, 0.3, 0.6, 0.8, 0.9, 100% fly ash	0.6	NaOH	0.5	10	Room temperature	Directly cured at room temperature for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG25
100% soil, 0.3, 0.6, 0.8, 0.9, 100% fly ash	0.6	NaOH	0.5	10	100	Directly cured in oven for 1 day	Soil and fly ash were premixed prior to the addition of liquid content (M1)	SFG26
100% soil, 0.3, 0.6, 0.8, 0.9, 100% fly ash	0.6	NaOH	0.5	10	100	Directly cured in oven for 1 day	Fly ash and the liquid content were premixed followed by the addition of soil (M2)	SFG27

Table 3. Strength increment due to sequence of mixing

Ratio of fly ash/soil	M1 (%)	M2 (%)
0.3	45.08	66.95
0.6	86.22	78.25
0.8	92.30	80.29
0.9	90.81	77.53

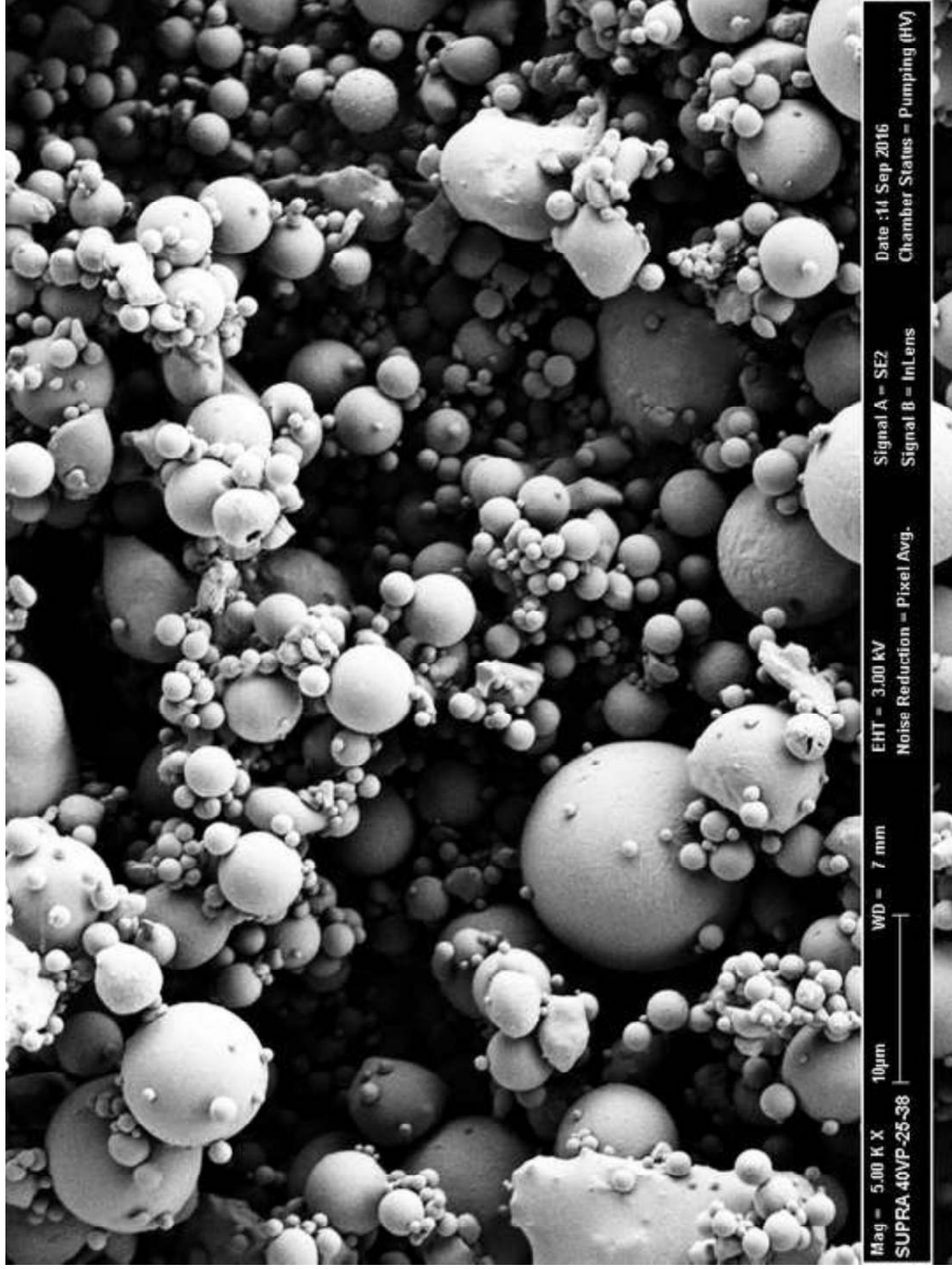


Figure 1a

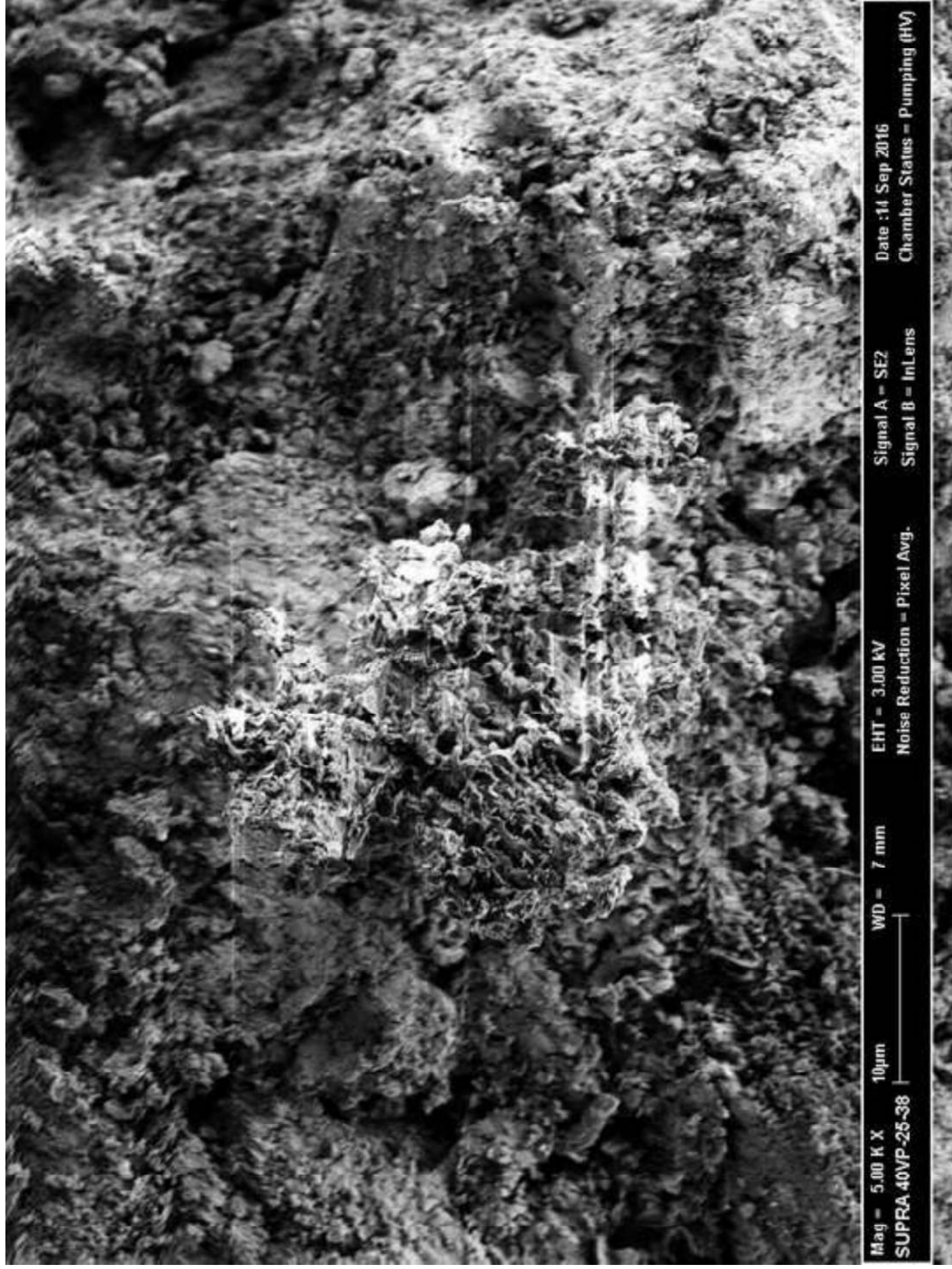


Figure 1b

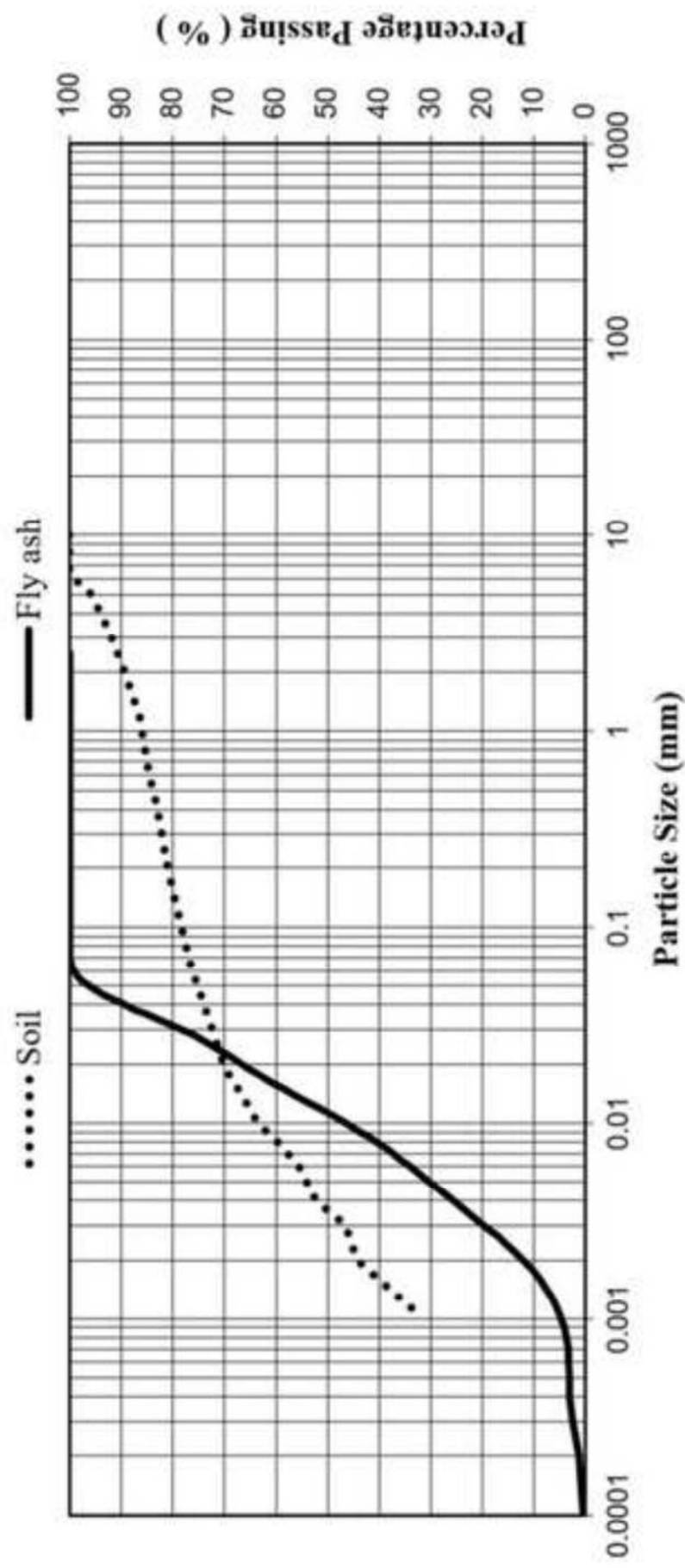


Figure 2



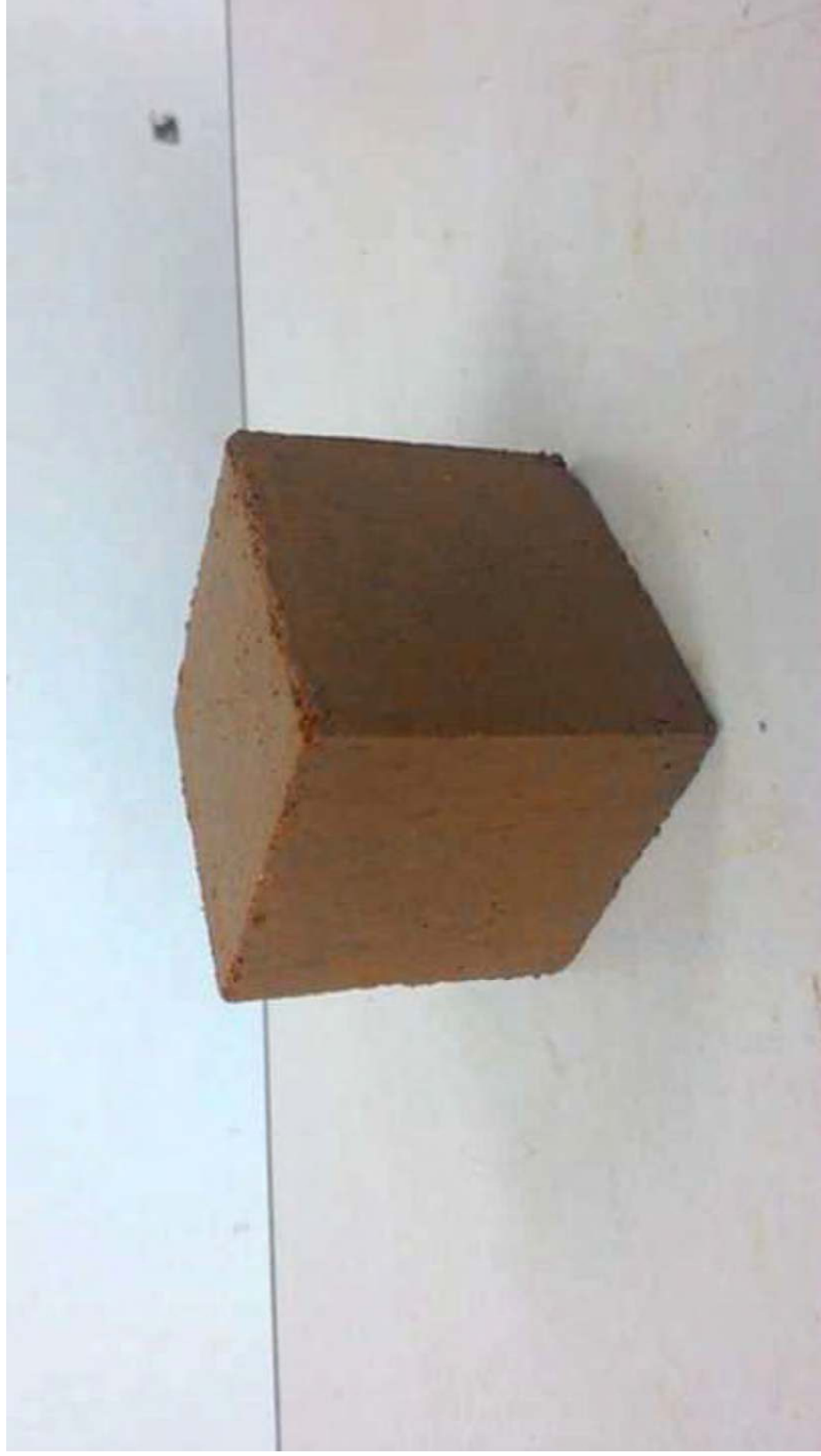
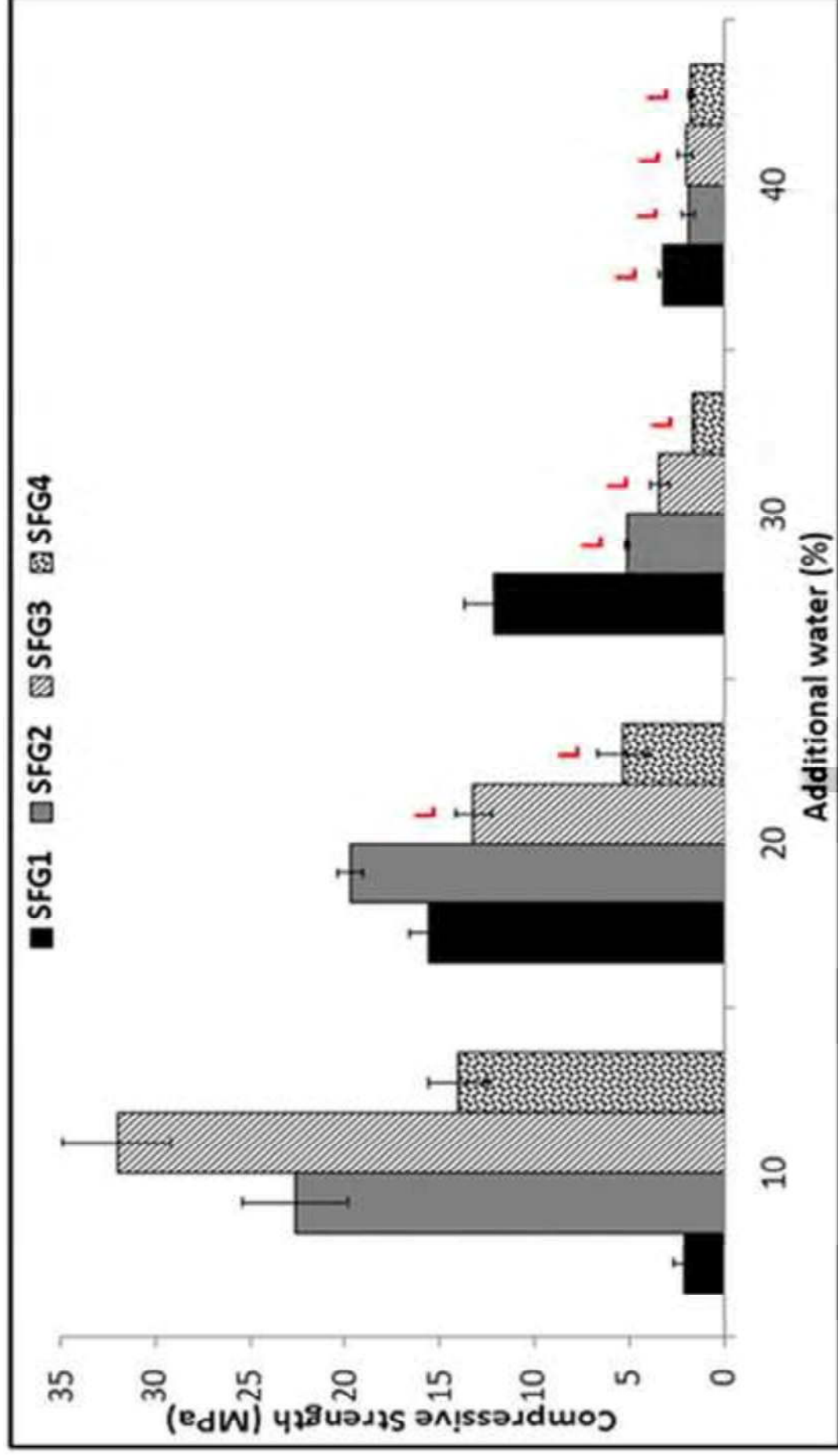


Figure 3c

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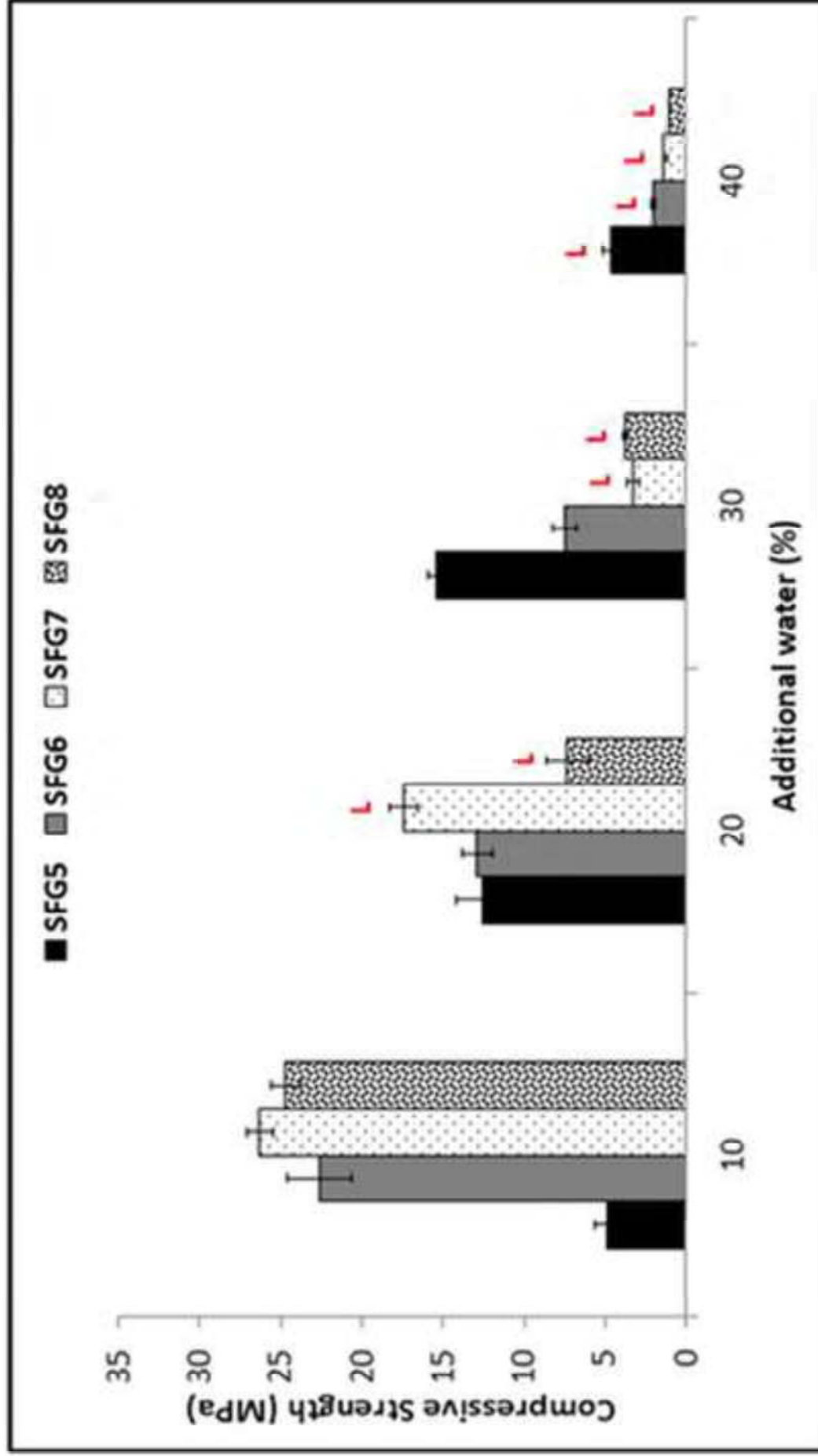


Figure 4b

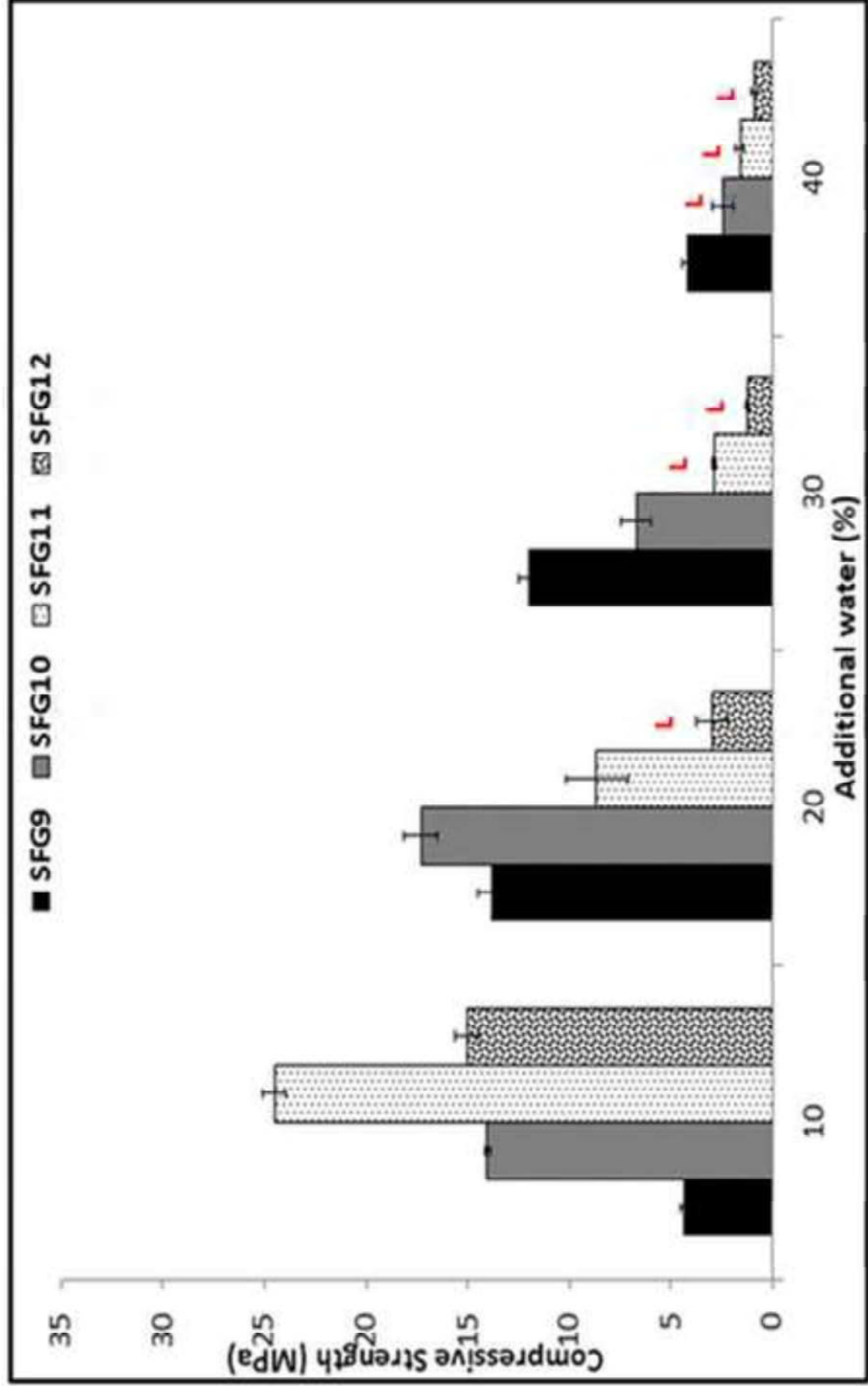
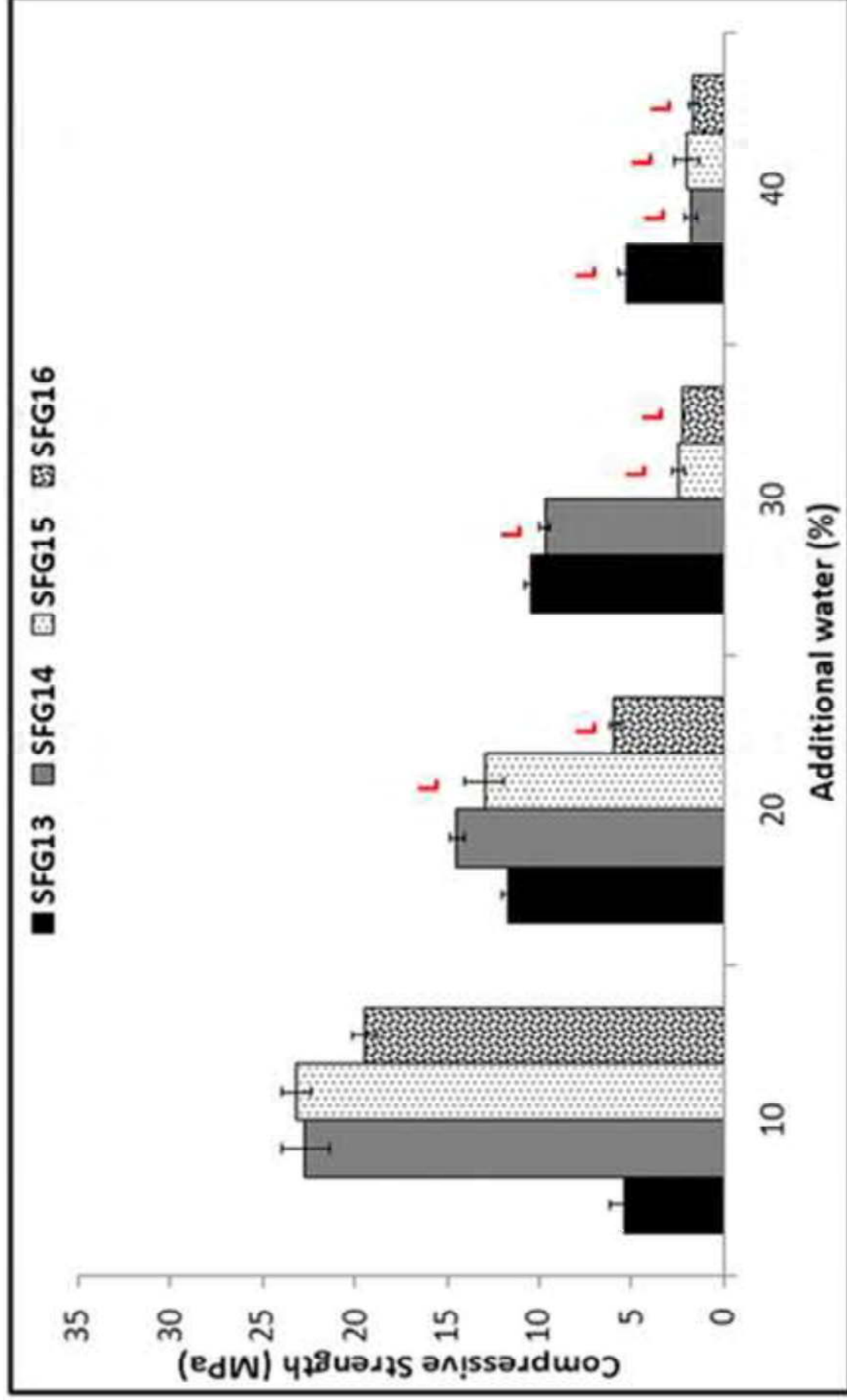
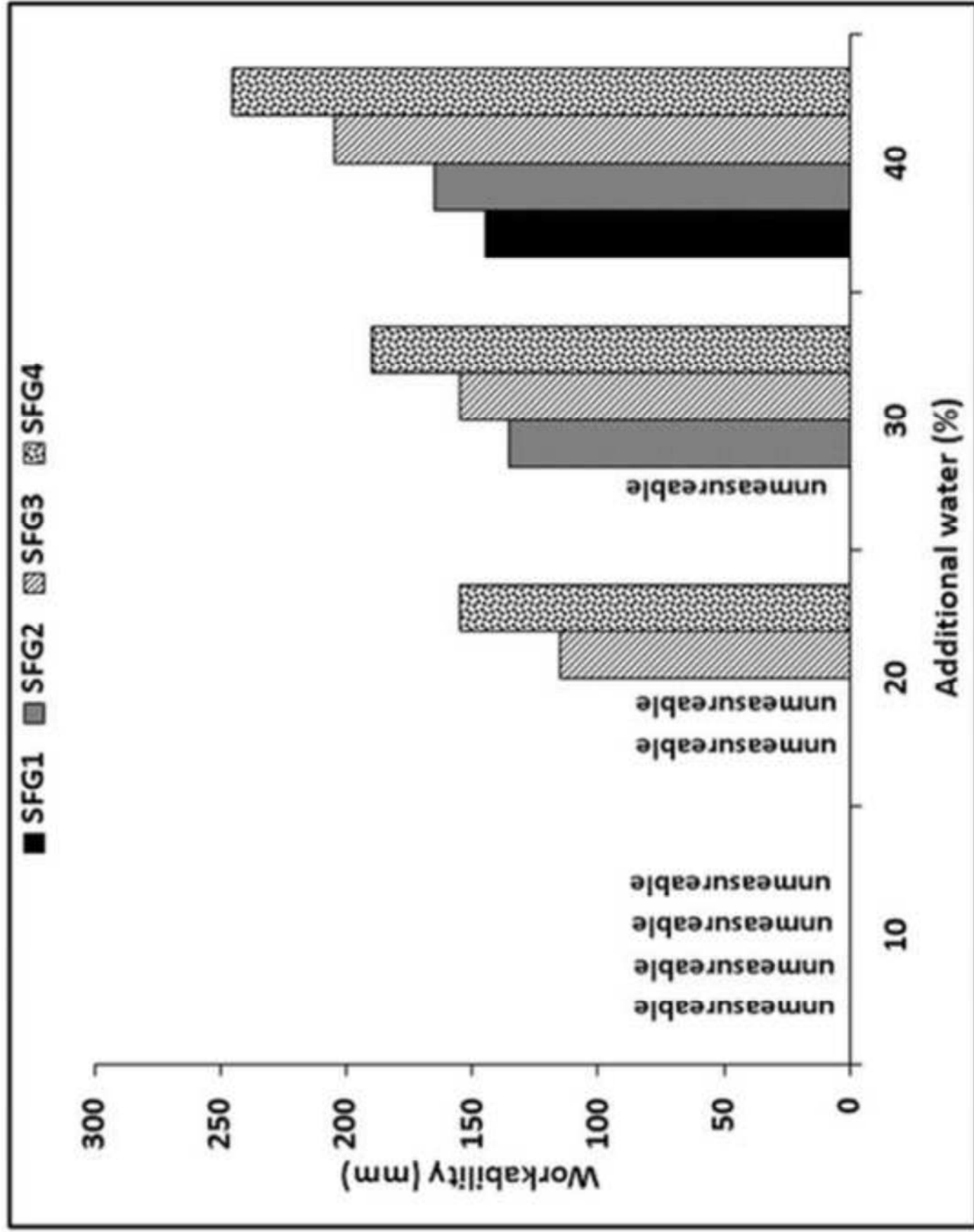
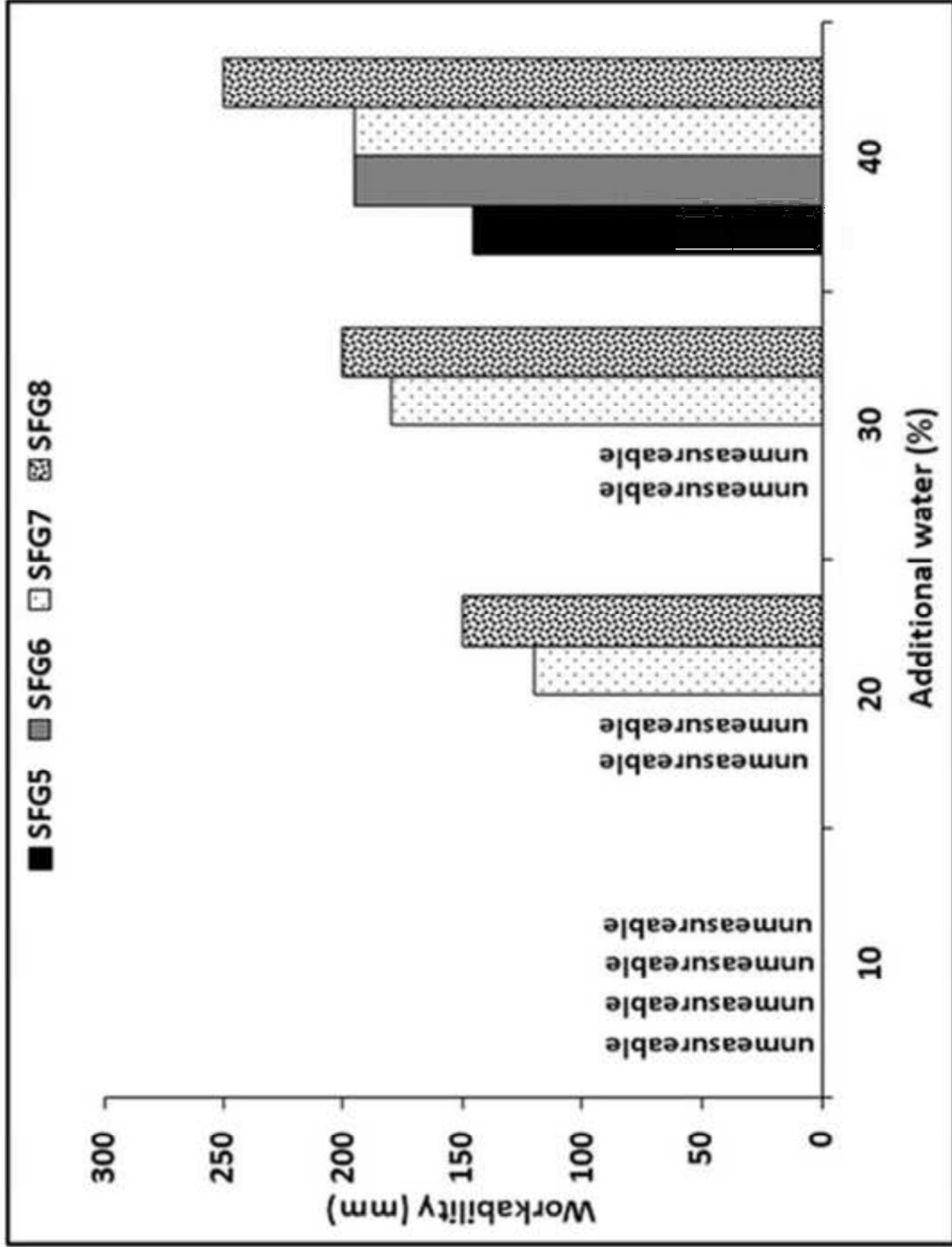
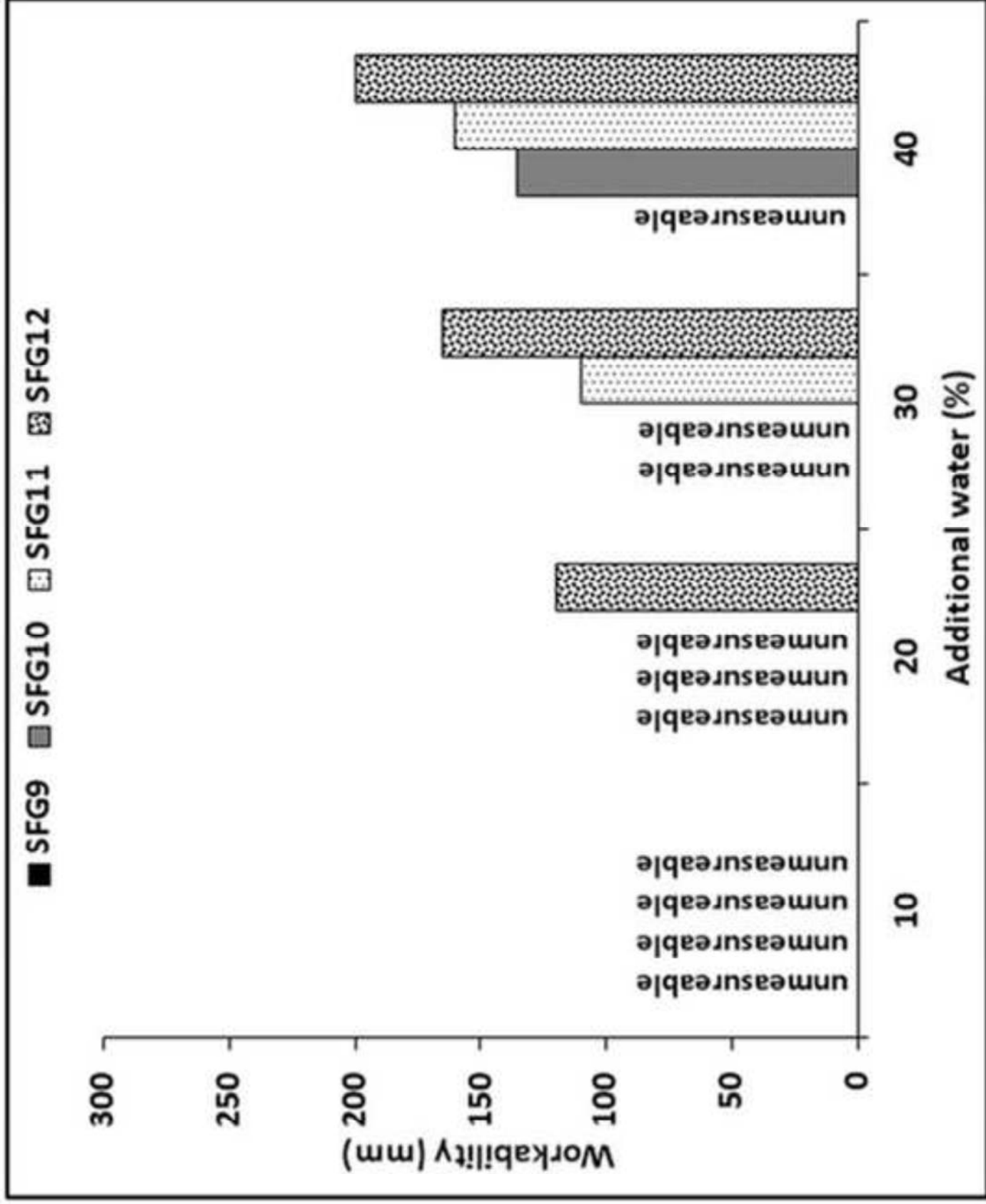


Figure 4c









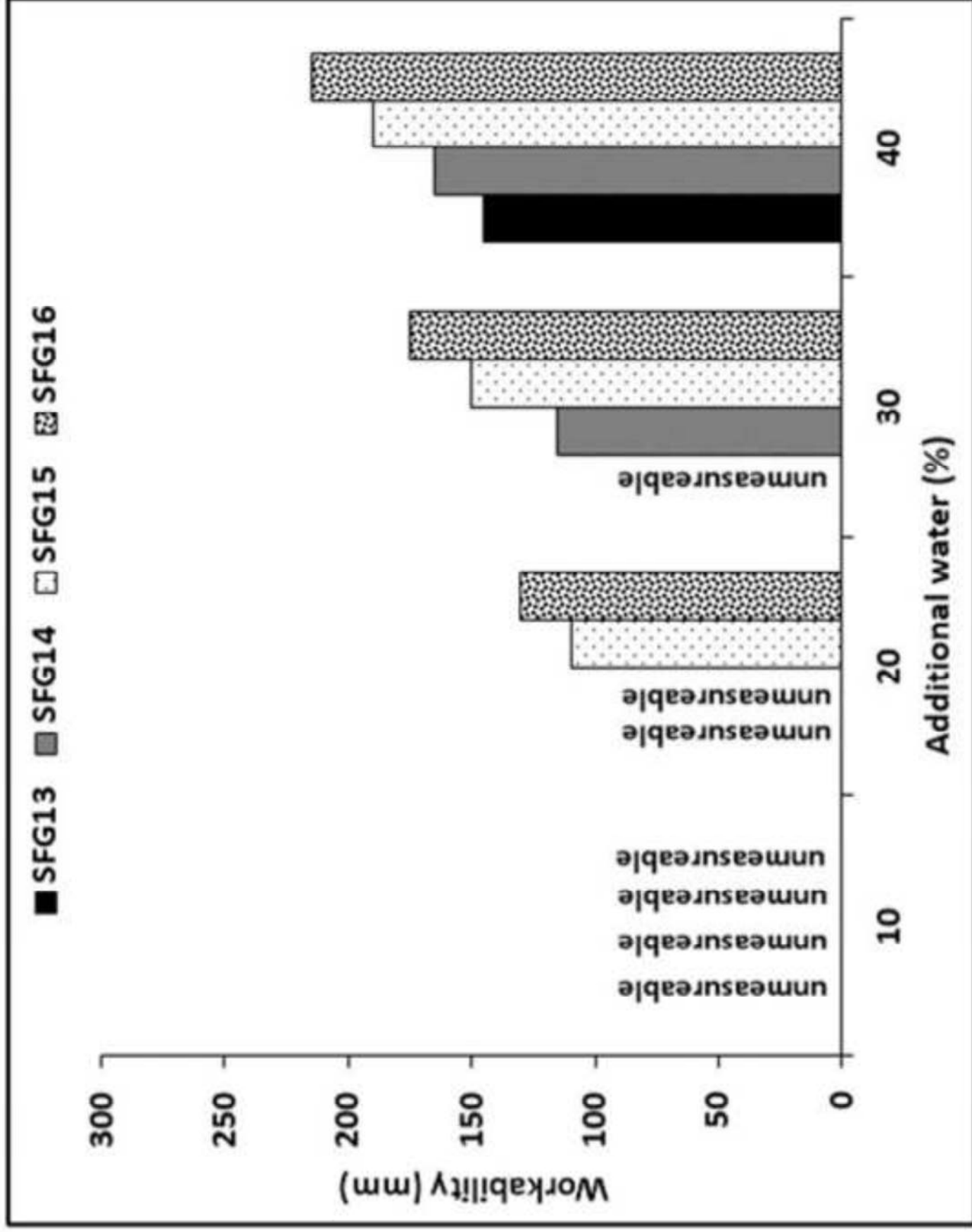




Figure 6a

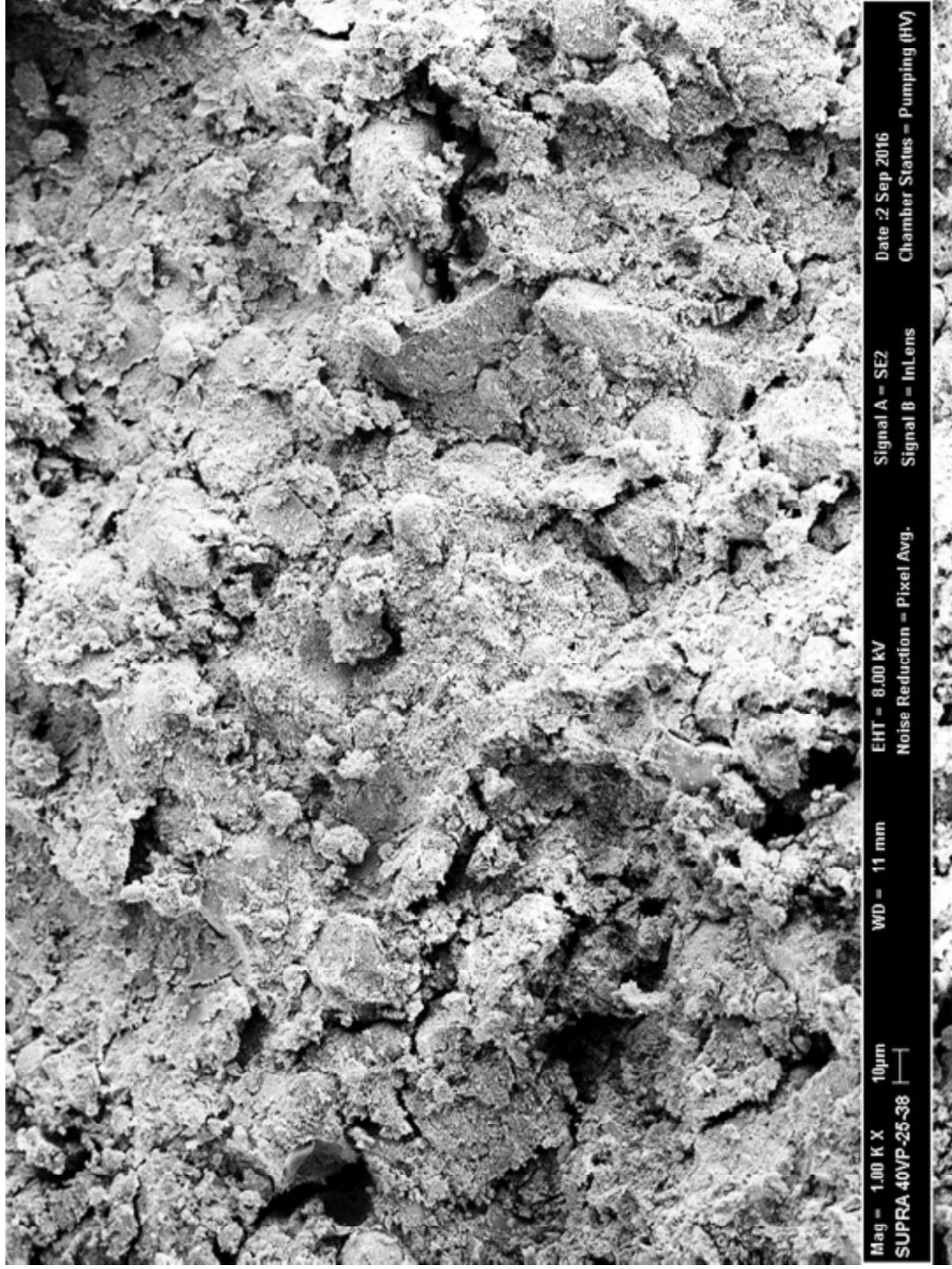


Figure 6b

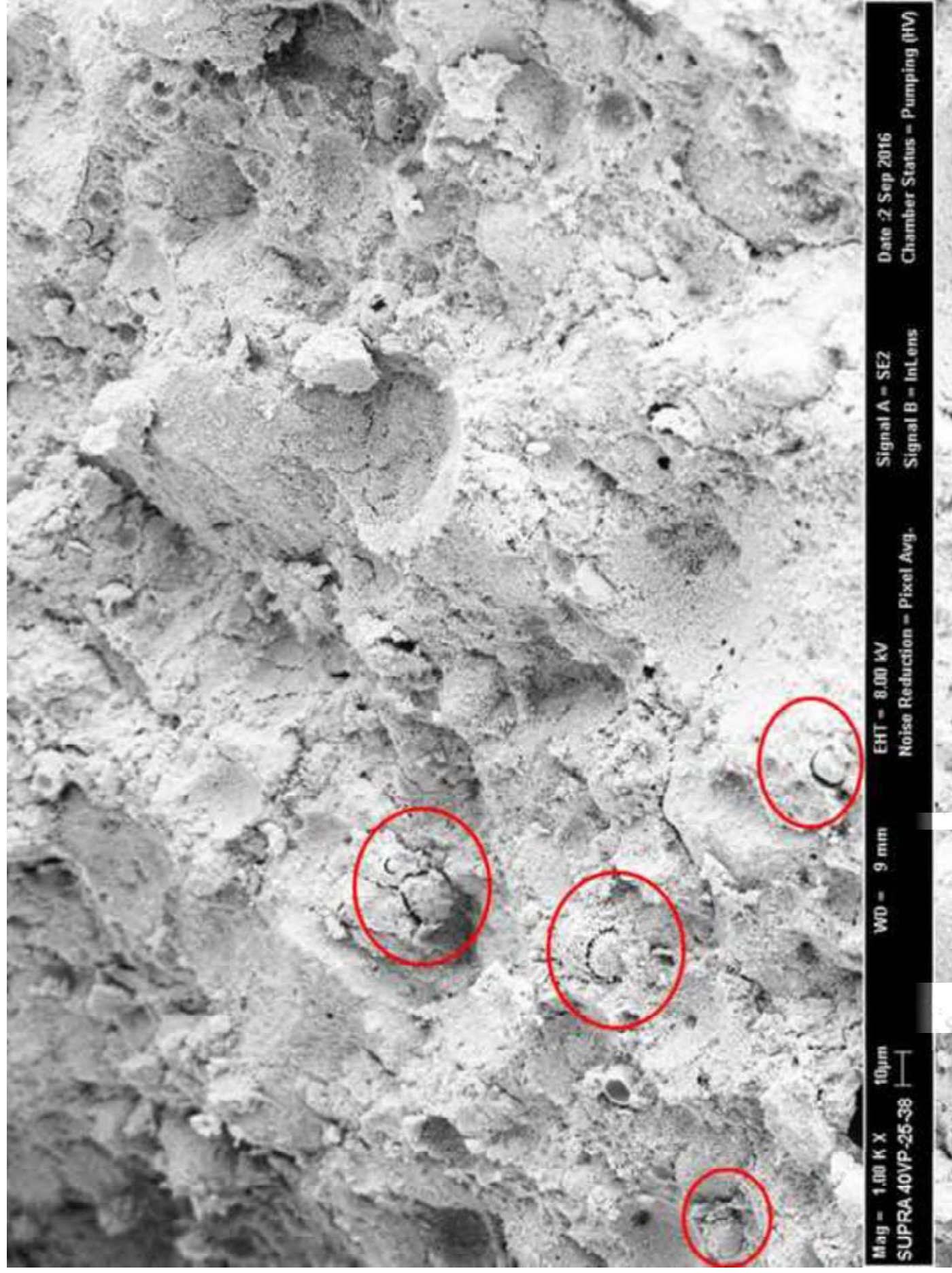
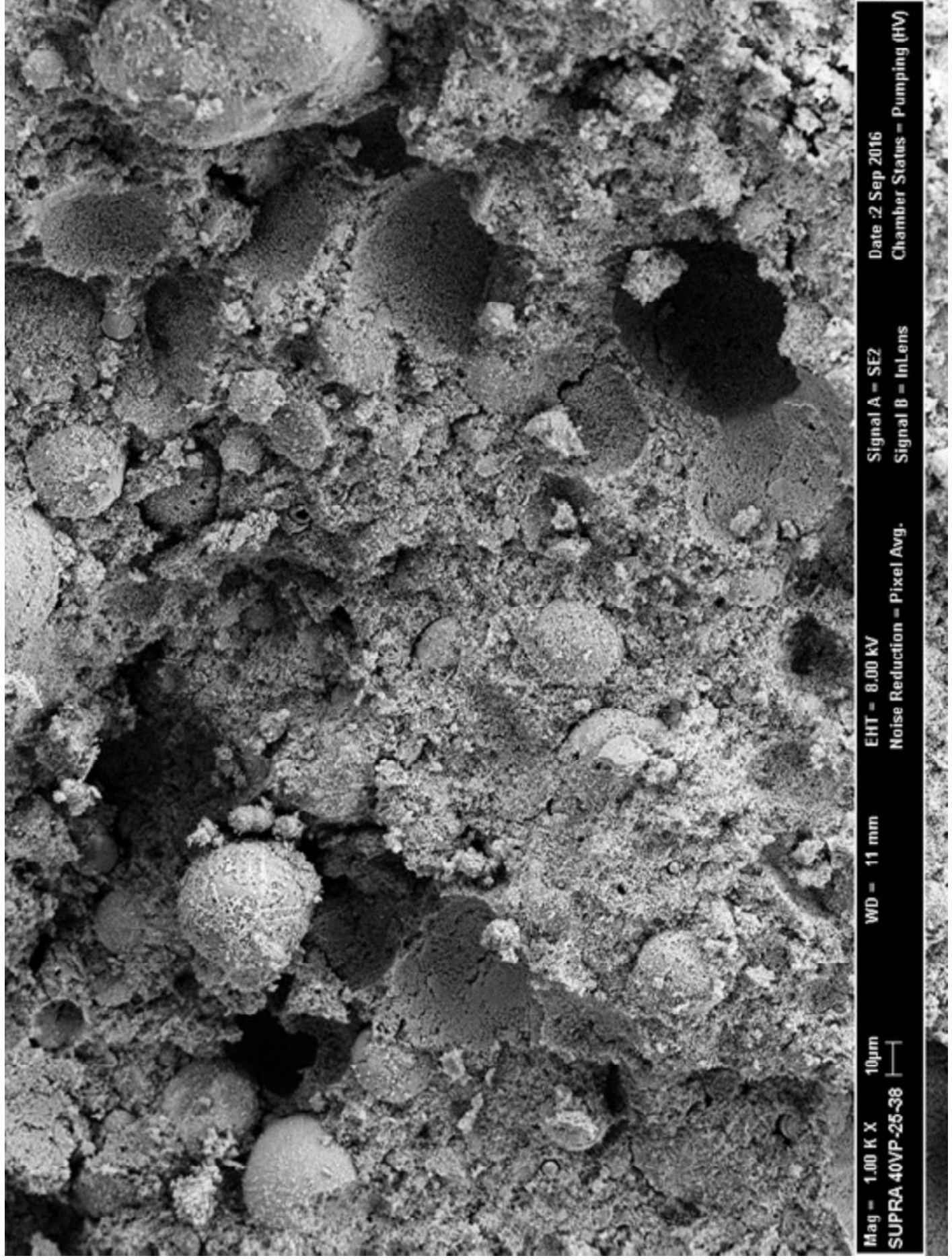


Figure 6c



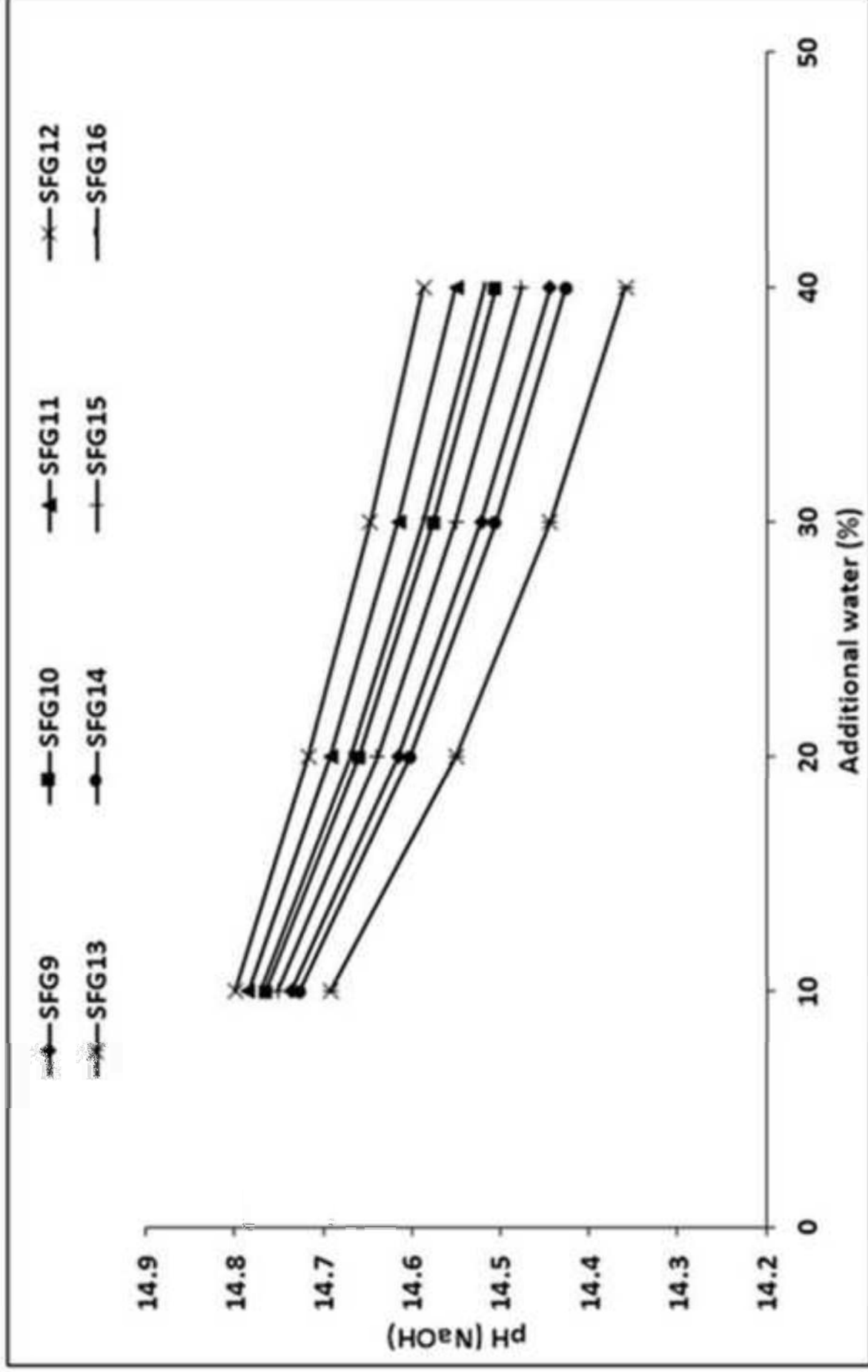


Figure 7

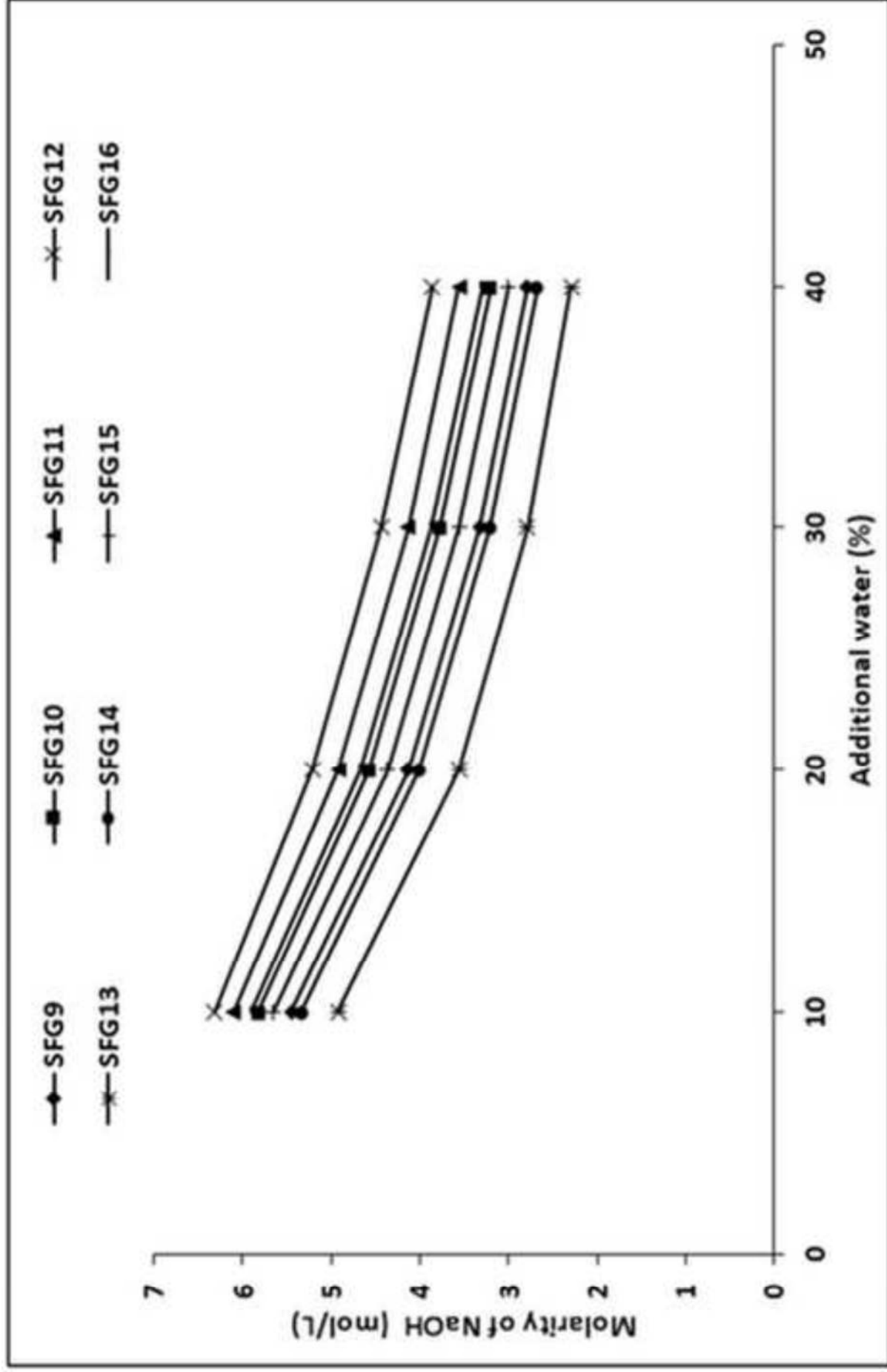


Figure 8

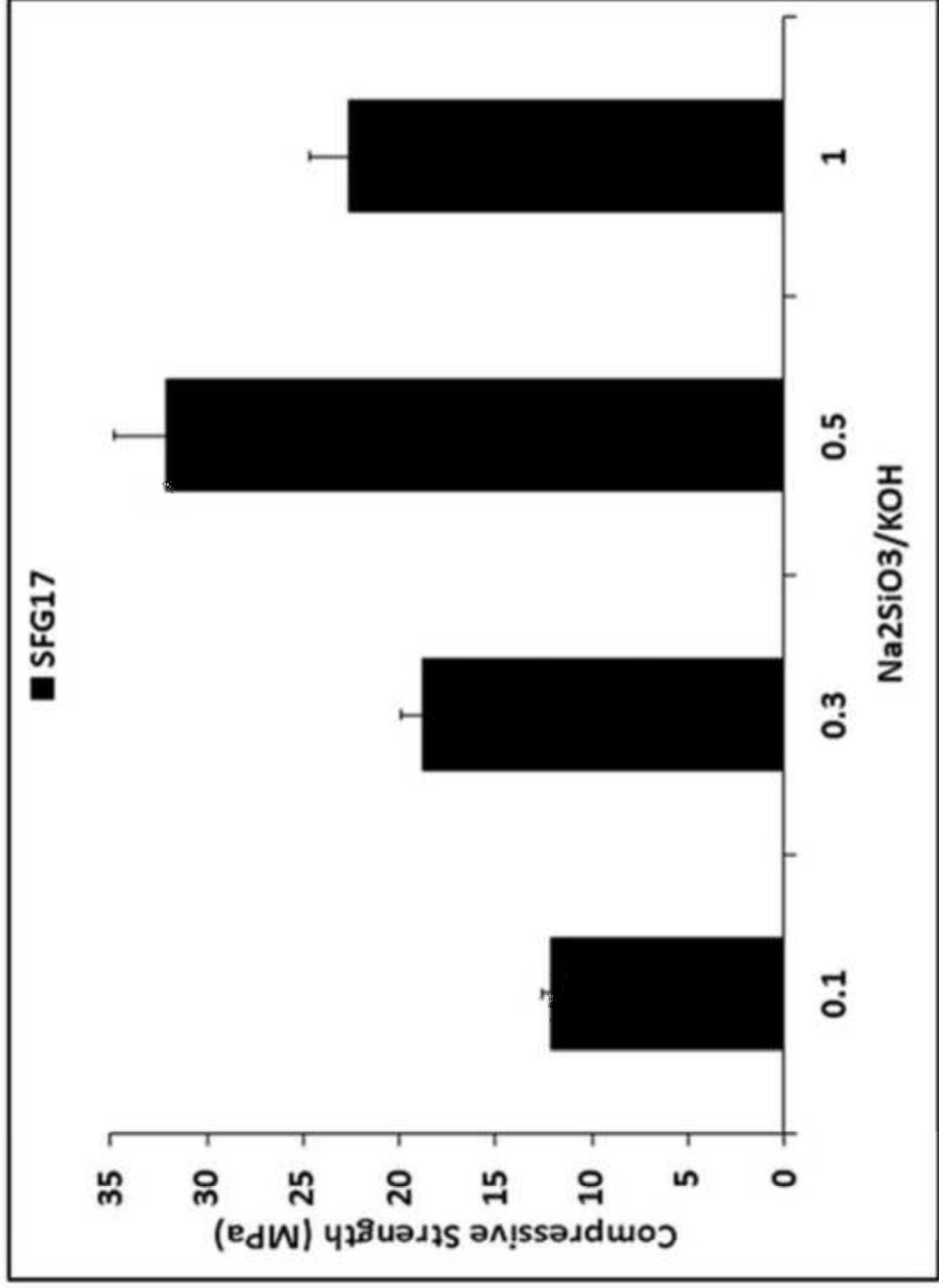


Figure 9a

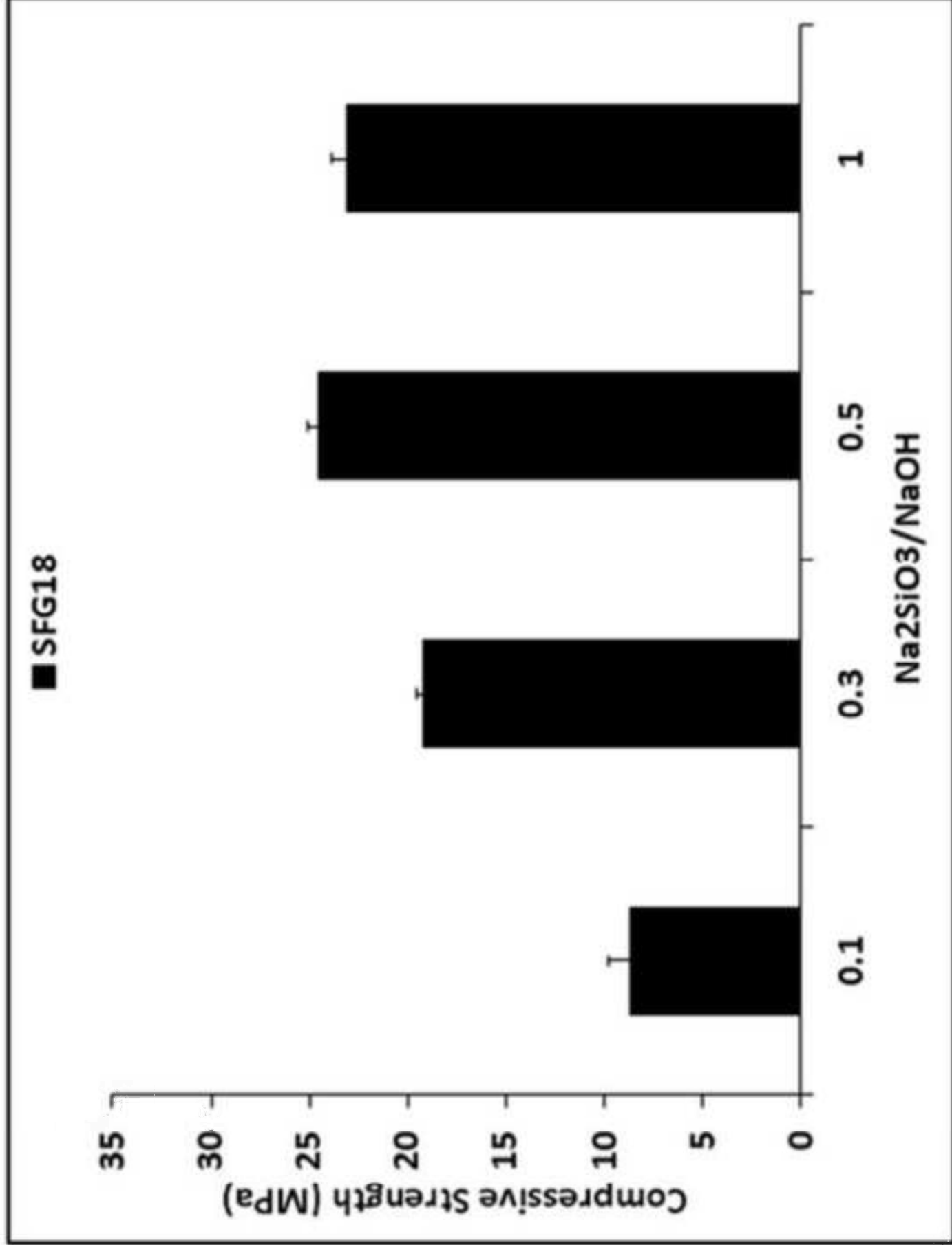


Figure 10a

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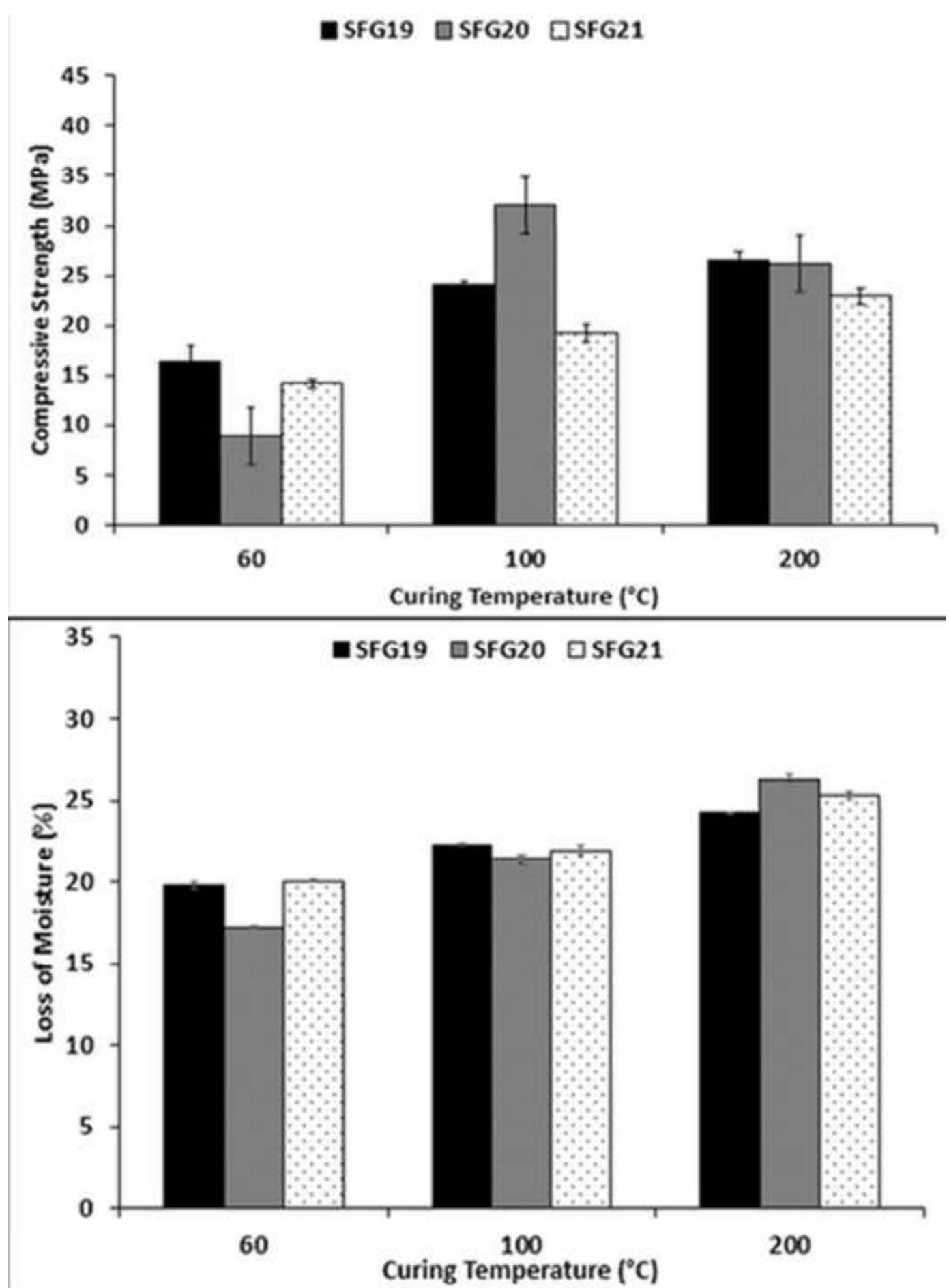
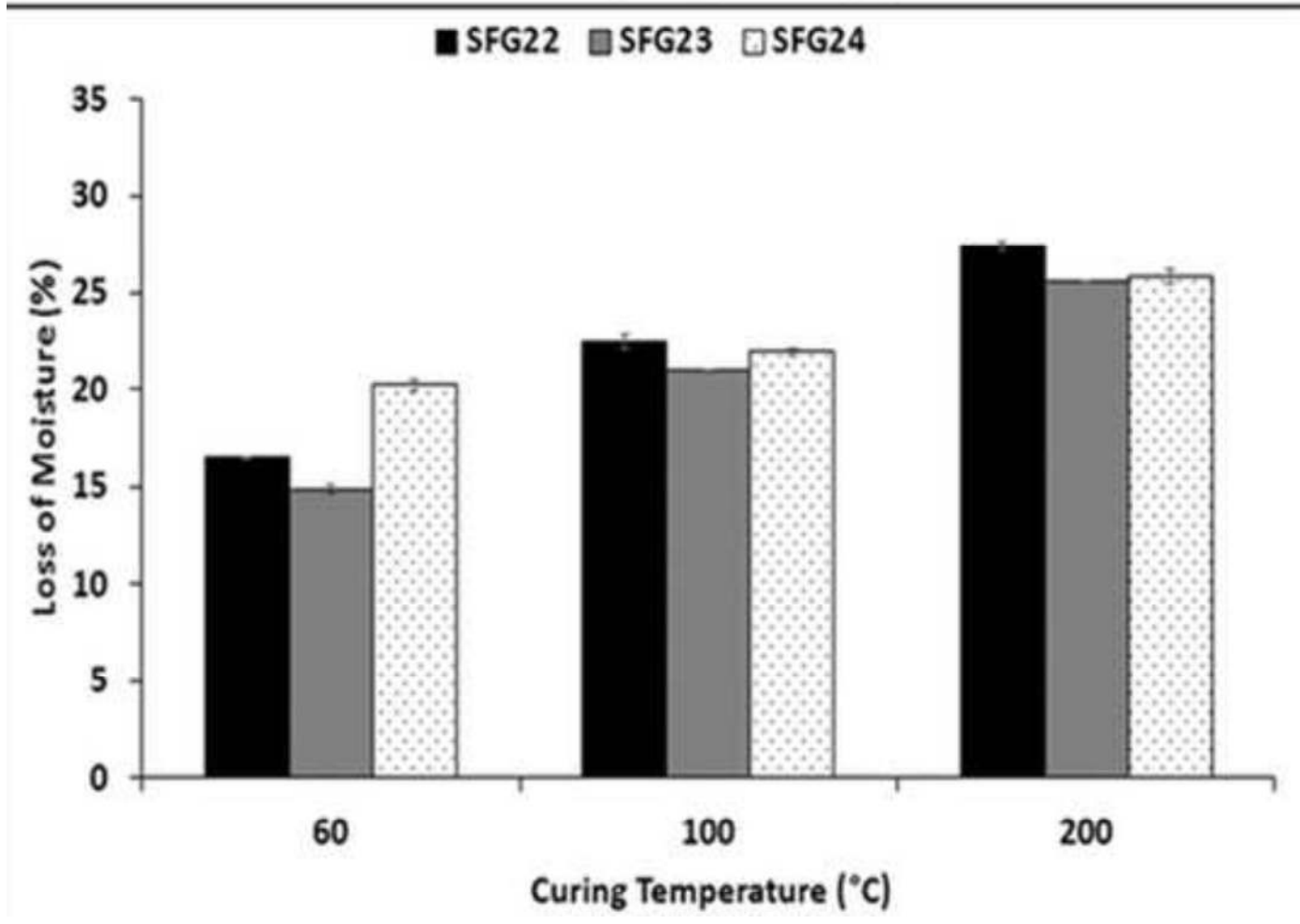
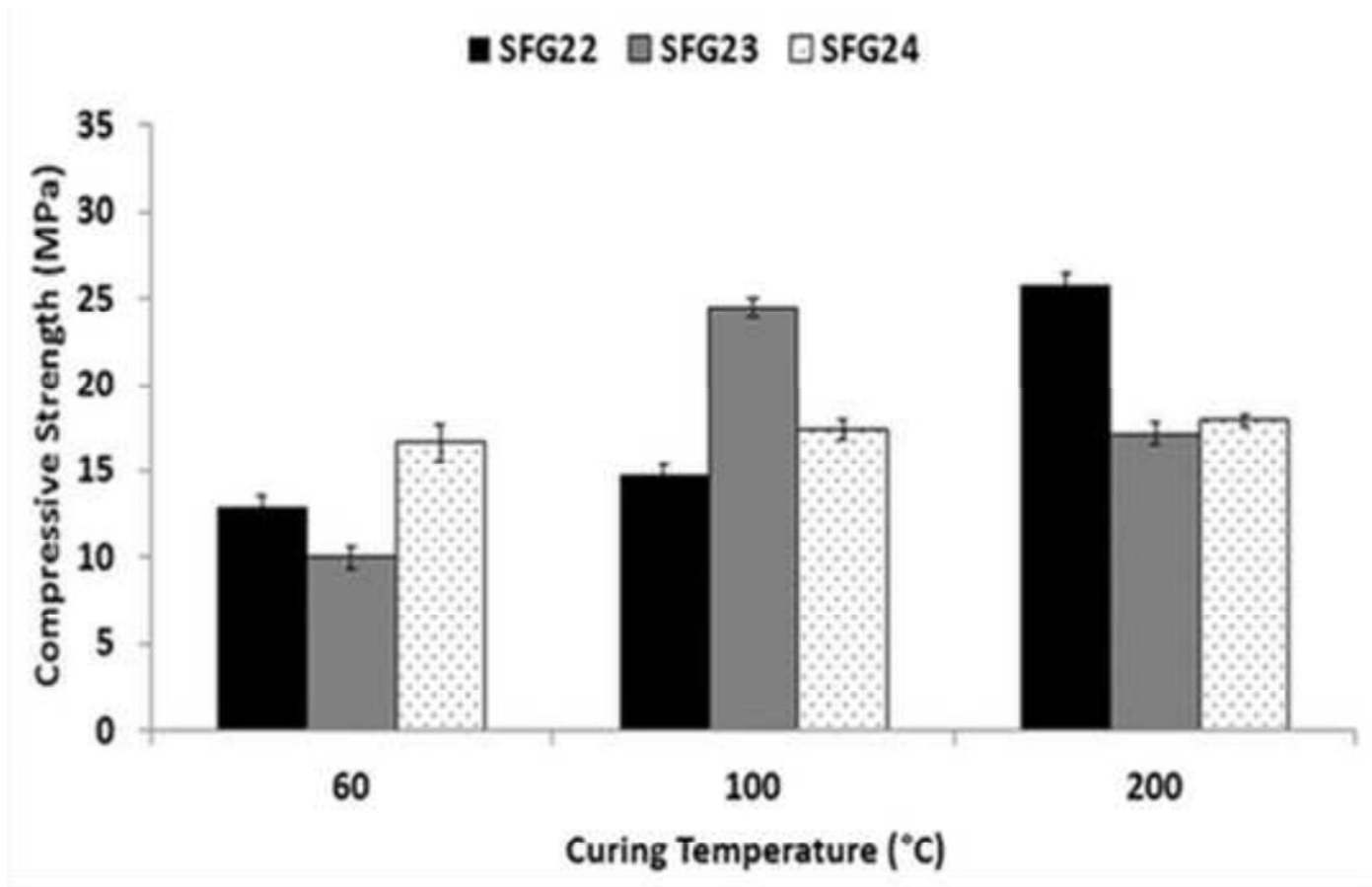


Figure 10b

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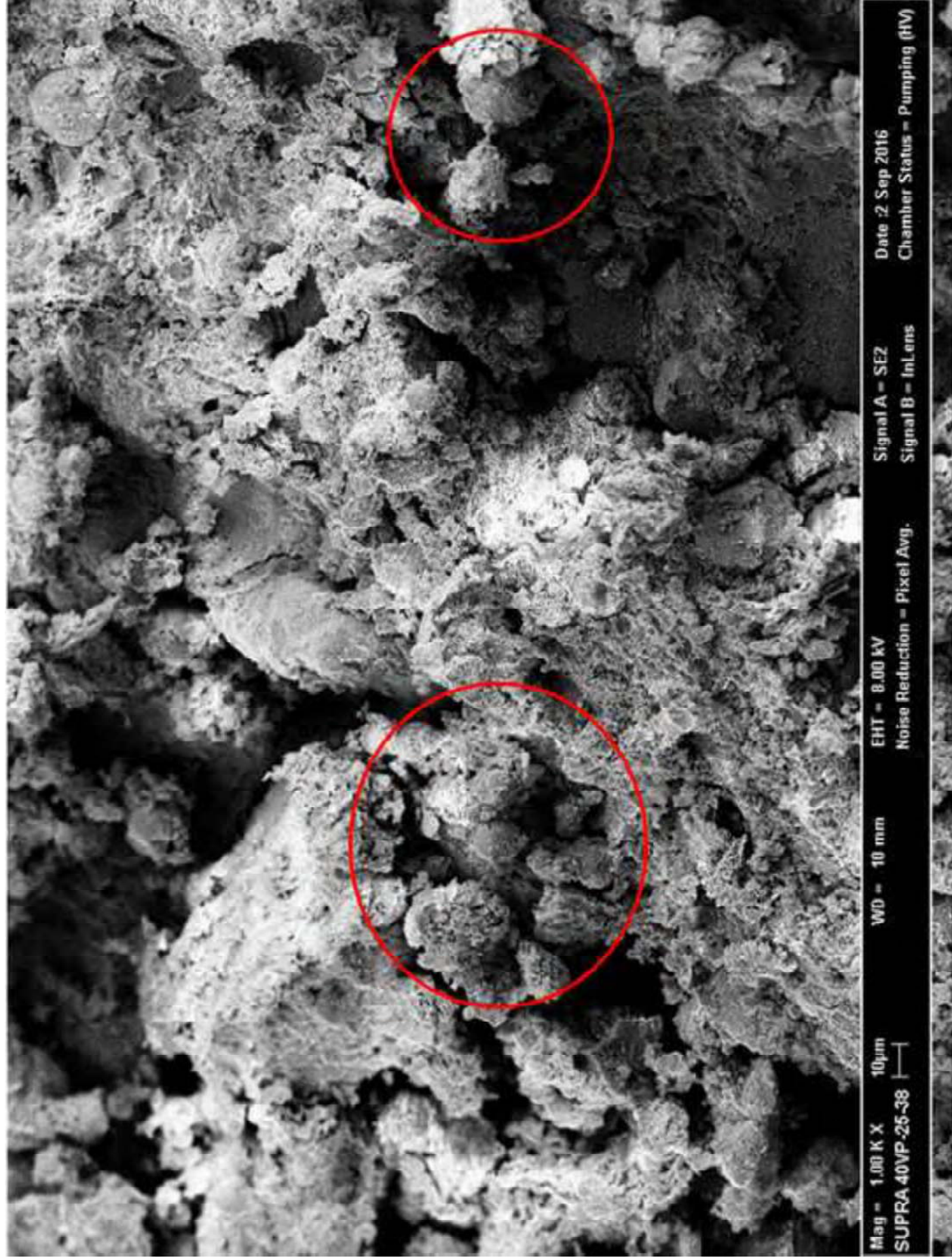


Figure 11a



