

Engineering Properties of Fly Ash-Based Lightweight Aggregate

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Abstract

This study evaluates the feasibility of producing lightweight aggregates (LWA) using off-spec waste fly ash (FA) that would not meet the ASTM C618 standard specifications for direct use in concrete due to high loss on ignition (LOI) and moisture content. The production processes of LWA from off-spec fly ash were first introduced and the physical properties of developed aggregates including oven dry (OD), saturation surface dry (SSD), and apparent specific gravities were measured. Moreover, porosity, vacuum absorption, and water absorption of produced LWA over time were assessed. LWA showed an OD specific gravity ranged from 1.3 to 1.6 with a total porosity ranging from 34% to 46.4%. Except for one type of LWA, produced LWAs had 72h water absorption greater than 5%, which complied with ASTM C1761 as a suitable LWA for concrete internal curing. It was found that the formation of a glassy phase on the surface of the aggregate

23 may result in water absorption reduction. Moreover, different LOI contents led to different
24 physical appearances of the LWAs.

25 **1. Introduction**

26
27 Lightweight concrete is experiencing a “dormant” period according to Mousa et al. [1] due to
28 among many factors, a high unit price and the shortage of lightweight aggregates (LWA), were
29 considered as key obstacles, which create a reluctance for use of lightweight concrete in the
30 market. Improving technologies for LWA production is one possible solution to this problem.
31 Beside natural LWA such as pumice and perlite, most of the available LWAs on the market are
32 synthetic and made by firing shale, clay and slate raw materials in rotatory kilns at elevated
33 temperatures (1000 to 1300 °C) [2]. However, synthetic LWA are only available at certain locations
34 in the U.S, which makes accessing them a challenge.

35 Producing LWA from solid waste materials such as fly ash (FA), bottom ash, sewage ash, waste
36 clay, and slag, that do not meet standard specifications to be directly used in concrete, may be an
37 alternative approach for increasing the local accessibility of LWA, based on power plant locations,
38 for concrete producers. Moreover, converting these waste sources to LWA may lower the final
39 price of LWA and, very importantly, minimize the surface impoundment of solid waste material.
40 Billen et al. [3] proposed a viable process for converting coal bottom ash to LWA. Experiments
41 were guided by thermochemical models that identified the melting temperature for the LWA
42 besides the formation of mineral phases as a function of temperature [4–6]. Korat el al. [7]
43 investigated LWA production from silica sludge by addition of FA and silicon carbide (SiC),
44 where SiC acted as foaming agent and FA was responsible for the formation of a liquid phase on
45 the surface of the aggregate for entrapment of released gas. Kockal and Ozturan [8] investigated

46 the production of LWA from FA by addition of glass powder and bentonite. They observed melting
47 of FA at 1150°C while with the addition of the glass powder and bentonite, the melting point
48 decreased to 1100°C. Moreover, they observed that increasing the amount of glass powder
49 decreased the oven dry specific gravity of FA LWA sintered at 1200°C mainly due to expansion
50 and bloating in comparison with the FA LWA without glass powder.

51 The waste material obtained from different sources offers considerable variety in chemical
52 composition, particle size distribution, formation and amount of liquid (vitreous) phase and its
53 viscosity at elevated temperature. However, for successful production of LWA from waste
54 materials, three main factors are required during sintering as they influence the occurrence of
55 bloating, which has a direct effect on properties such as specific gravity, porosity, and water
56 absorption [2,9]:

57 (1) appropriate amount of liquid phase on the surface of aggregate to turn the LWA's entire body
58 in a viscous state;

59 (2) suitable viscosity of liquid phase for entrapment of evolved gas during sintering; and

60 (3) gaseous phase formation during the sintering for leading to bloating.

61 Figure 1 shows a schematic representation of these factors. The formation of a liquid phase on the
62 surface of the aggregate with optimum viscosity, while the gas is being released, can result in
63 bloating of the aggregate and formation of desirable LWA.

		Viscosity		
		High	Optimum	Low
Gas Release	Yes	No Expansion	Bloating	Collapse
	NO	No Expansion	Softening Without Expansion	Melting Without Expansion

64

65 Figure 1. Schematic representation of required conditions for successful bloating of LWA,
 66 adopted from [2] and [9].

67

68 This study evaluates the feasibility of producing synthetic LWA from waste FAs whose loss-on-
 69 ignitions (LOI) varied from 0.92% to 8.7%. Moreover, the engineering properties of produced
 70 LWA including, specific gravity, porosity, and water absorption was assessed to evaluate whether
 71 producing LWA from FA is an efficient LWA compared to available LWAs in the market.

72 **2. Experimental program**

73 *2.1 Materials*

74 Four types of FAs obtained from U.S. sources were studied whose chemical compositions are
 75 illustrated in Table 1. Based on ASTM C618 [10], FA1 and FA2 can be classified as class C. These
 76 two FAs have similar chemical composition and their main difference is the LOI content. LOI for
 77 FA1 and FA2 is 0.92% and 8.7%, respectively. While FA1 meets ASTM C618 requirements, FA2
 78 LOI content does not meet ASCTM C618 and cannot be used directly in concrete. FA3 and FA4
 79 have exactly the same chemical composition and because of their low calcium oxide content, they
 80 are classified as Class F FA according to ASTM C619. FA3 and FA4 are differentiated by their
 81 LOI content where FA3 has 3.5 % LOI while FA4 contains 5.3%. FA1 and FA3 were studied in

82 this paper to assess the influence of carbon content to create gas phases during sintering and
83 produce porous structure.

84

85 Table 1. Oxide compositions of FAs used in this study obtained from the providers

Chemical Composition (% by mass)	Sample Name			
	FA 1	FA 2	FA 3	FA 4
SiO ₂	38.88	38.19	49.5	49.5
Al ₂ O ₃	19.44	18.76	23.8	23.8
Fe ₂ O ₃	10.86	10.88	15.45	15.45
SO ₃	1.61	3.59	0.75	0.75
CaO	19.39	18.8	3.2	3.2
Na ₂ O	1.27	1.12	0.42	0.42
MgO	4.04	3.6	1.6	1.6
K ₂ O	1.04	0.98	2.3	2.3
P ₂ O ₅	0.73	0.7	-	-
TiO ₂	1.33	1.31	-	-
Total	98.59	97.93	97.02	97.02
LOI	0.92	8.7	3.5	5.3
Initial moisture content	0.1	1.21	0.4	0.4

86

87

88 *2.2 Sample preparation procedure*

89 A four-step process [3] was used to produce the samples. In Step 1, the moisture of ashes was
90 removed in an oven at a temperature of 110 °C ± 5 °C until the mass of the ash reached a constant
91 value (approximately 24 h). In Step 2, the dried ash was mixed with NaOH aqueous solutions with
92 molarities of 2.5 mol/L, 6.25 mol/L and 10 mol/L. NaOH was added as a fluxing agent to lower
93 the melting temperature of the ashes as well as to reduce the viscosity of molten phases during
94 sintering [3]. For FA1 liquid/solid (L/S) ratio of 0.2 was used while for FA2, FA3 and FA4, L/S
95 was selected to be 0.3. The different L/S ratios used resulted in different NaOH additions on a mass
96 basis (mass of solid NaOH per mass of ashes): 2%, 5%, and 8% for FA1 and 3%, 7.5% and 12%

97 for FA2, FA3, and FA4. Moreover, samples with 0% NaOH concentration were produced to
 98 evaluate the effect of NaOH fluxing agent during sintering.

99 The wet mixes were then compacted into a diameter of 16 mm plastic mold under a compressive
 100 load of 18.77 N to form into a spherical fresh pellet. The addition of NaOH enabled chemically
 101 binding the ashes through geopolymerization, which helped to retain the spherical shape of the
 102 fresh pellets before sintering. After removing the green pellets from the mold, moisture was
 103 removed in Step 3 by placing the green pellets in a controlled environmental chamber at 40 °C and
 104 30 % RH for 24 h. Preparation of FA1 samples with 0% NaOH concentration and FA2 samples
 105 with 0% and 3% NaOH concentration were found to be very challenging. Therefore, they were not
 106 included in this study. Reduction in NaOH concentration was accompanied by increasing the water
 107 to ash ratio resulting in a blend with very low viscosity, which made the successful production of
 108 a fresh pellet impossible. In Step 4, the fresh pellets were sintered in a tube furnace at a temperature
 109 of 1160 °C. A residence time of 8 min was used, followed by immediate natural cooling to room
 110 temperature (23 °C). Table 2 shows the samples prepared in this study. Aggregates are abbreviated
 111 by the following format: XX-X%-Xmin where XX represents the ash type (FA1, FA2, FA3, and
 112 FA4), X% is the NaOH concentration (% by mass), and Xmin is sintering time (in min),
 113 respectively. For instance, FA1-5%-8min indicates aggregates that are made from FA1 with 5 %
 114 NaOH concentration (by mass) and sintered for 8 min.

115 Table 2. Sample compositions and preparation conditions

Ash type	L/S	Residence time	NaOH concentration (%)				Code*
							-
FA1	0.2	8 min	-	2	5	8	FA1-2%-8min
							FA1-5%-8min
							FA1-8%-8min

								-
FA2	0.3	8 min	-	-	7.5	12		-
								FA2-7.5%-8min
								FA2-12%-8min
								FA3-0%-8min
FA3	0.3	8 min	0	3	7.5	12		FA3-3%-8min
								FA3-7.5%-8min
								FA3-12%-8min
								FA4-0%-8min
FA4	0.3	8 min	0	3	7.5	12		FA4-3%-8min
								FA4-7.5%-8min
								FA4-12%-8min

116 *For each aggregate type, at least three replicates were produced for testing.

117

118 2.3 Testing procedure

119 2.3.1 Specific gravity

120 Oven dry (OD), saturated surface dry (SSD), and apparent specific gravity (which depends on the
121 solid constituents and does not consider the pore space within the materials) of aggregates was
122 measured using a buoyancy method according to ASTM C127 [11]. Additionally, the vacuum
123 absorption capacity, was measured, which indicates the maximum possible absorption capacity of
124 an aggregate. To obtain the OD mass, samples were dried in an oven at a temperature of $110\text{ }^{\circ}\text{C} \pm$
125 $5\text{ }^{\circ}\text{C}$ for a minimum of 12 h. OD samples were de-aerated with a vacuum pump (pressure of 1.33
126 $\text{kPa} \pm 0.33\text{ kPa}$) for 3 h. followed by introducing deionized water, which was previously de-
127 aerated, to the samples under vacuum conditions. Samples continued to be de-aerated under
128 vacuum for an additional 1 h. Afterward, the samples were taken out of the vacuum chamber and
129 kept in the atmosphere for 6 h. After vacuum saturation of the samples, the surface water was

130 removed and the SSD mass was measured. Samples were then placed in the buoyancy container
131 and the apparent mass was measured.

132

133 2.3.2 Sorption properties

134 Water absorption in the aggregate samples takes place over time; therefore, a volumetric flask
135 method [12] was conducted for the OD samples to evaluate the water absorption as a function of
136 time. Water absorption was measured at the intervals of 6h, 24h, 48h, and 72h and compared with
137 vacuum water absorption values.

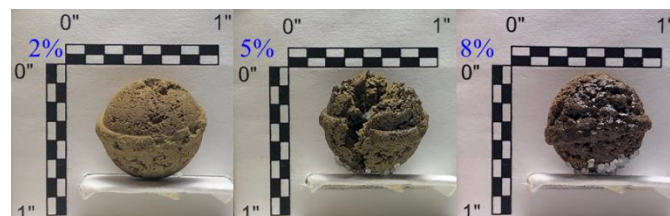
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139 3. Results and discussion

140 3.1 Physical appearance

141 Optical photos were taken from the front view of the samples to present the physical appearance
142 of the aggregate samples. The distance between the camera and the aggregate were kept constant
143 during imaging to maintain the same magnification for all photos. Figure 2 depicts the images of
144 aggregates made from FA1. It was found that by increasing the NaOH concentration from 2% to
145 8% a glassy surface formed. FA1-2%-8min had a yellowish color with no glassy surface, while
146 FA1-8%-8min exhibited a deep brown color with a partial shiny surface suggesting a glassy
147 material. A strong bonding among ash particles on the surface was visually observed which may
148 be due to the formation of liquid phase at the sintering temperature. For all three replicates of FA1-
149 5%-8min, a rupture was observed for the aggregates.

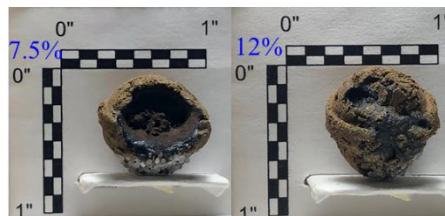
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151 Figure 2. Front views of sintered FA1 at 1160 °C for 8 min (NaOH concentration is indicated on
152 the upper left corner)

153

154 Figure 3 shows samples of aggregates made from FA2. For FA2-7.5%-8min, a partial erupted
155 aggregate was observed through all three replicates. This behavior could be attributed to the
156 formation of large gas bubbles during sintering due to the high LOI content of FA2. Such eruption
157 was also observable for FA2-12%-8min. The dark portion of the aggregate shown in the figure is
158 believed to be the molten ash (liquid phase) in the aggregate.



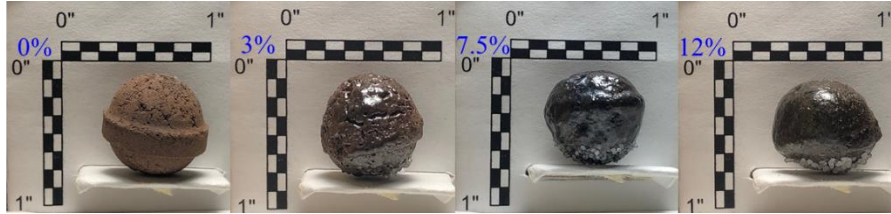
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160 Figure 3. Front views of sintered FA2 at 1160 °C for 8 min (NaOH concentration is indicated on
161 the upper left corner)

162

163 Figure 4 shows the aggregate samples made from FA3. They reveal that sample without addition
164 of NaOH (i.e., FA3-0%-8min) did not form any glassy surface. On the other hand, for FA3-3%-
165 8min, formation of glassy surface was noticeable, which confirms the role of NaOH for reducing
166 softening temperature of the aggregate. The melting incident was more intense for FA3-7.5%-
167 8min and FA3-12%-8min by increasing the NaOH concentration and the aggregates started to lose
168 its original spherical shape. FA3-12%-8min showed a semi-spherical shape with flat bottom which
169 implies the reduction in the viscosity of formed liquid phase. Billen et al. [3] showed that
170 increasing the NaOH concentration could reduce the viscosity leading to deformation of the

171 aggregates. Ruptures or formation of pores were not observed on the surface of the aggregates
172 produced from FA3 which is likely due to low LOI content.



173
174 Figure 4. Front views of sintered FA3 at 1160 °C for 8 min (NaOH concentration is indicated on
175 the upper left corner)

176
177 Figure 5 shows aggregate samples of FA4. FA4-0%-8min and FA4-3%-8min revealed a similar
178 appearance to those aggregates made from FA3 with the same concentration. For FA4-7.5%-8min
179 and FA4-12-8min the spherical aggregate was deformed due to bloating (i.e., entrapment of
180 released gas by the liquid phase formed on the surface of aggregate). A comparison between FA3-
181 12%-8min and FA4-12%-8min suggests that the liquid phase formed in these two aggregates are
182 different, which suggests that the unburnt carbon content in FA3 and FA4 may have some
183 influence on the physical appearance.



184
185
186 Figure 5. Front views of sintered FA4 at 1160 °C for 8 min (NaOH concentration is indicated on
187 the upper left corner)

188

189

190 *3.2 Porosity*

191 The porosity of the FA aggregates was determined using a buoyancy method ASTM C127 [11], and is
192 reported in Table 3. FA1-2%-8min, which did not form a liquid phase on the surface, showed the
193 highest porosity among the aggregates. For FA1, increasing the NaOH concentration led to reducing
194 the porosity of the aggregates. Similarly, FA2-7.5%-8min and FA2-12%-8min have similar porosity
195 and are lower than the aggregates made from FA1.

196 For FA3, increasing the NaOH percentage from 0% to 7.5% resulted in increasing the porosity of
197 the aggregates. This matter suggests that the addition of NaOH and sintering process were both
198 effective in increasing the porosity of aggregate. Among the aggregates made using FA4, FA4-
199 7.5%-8min led to the highest average porosity (i.e., 39.3%).

200

Table 3. Porosity of LWAs made using FA measured by the buoyancy test.

Sample Name	Porosity (%)
FA1-2%-8min	46.4±0.5
FA1-5%-8min	39.2±7.5
FA1-8%-8min	35.5±1
FA2-7.5%-8min	34.0±4.9
FA2-12%-8min	34.0±4
FA3-0%-8min	35.3±4.1
FA3-3%-8min	36.2±2
FA3-7.5%-8min	39.2±2.1
FA3-12%-8min	35.8±4.7
FA4-0%-8min	38.8±1
FA4-3%-8min	35.6±2.2
FA4-7.5%-8min	39.3±0.1
FA4-12%-8min	38.1±0.8

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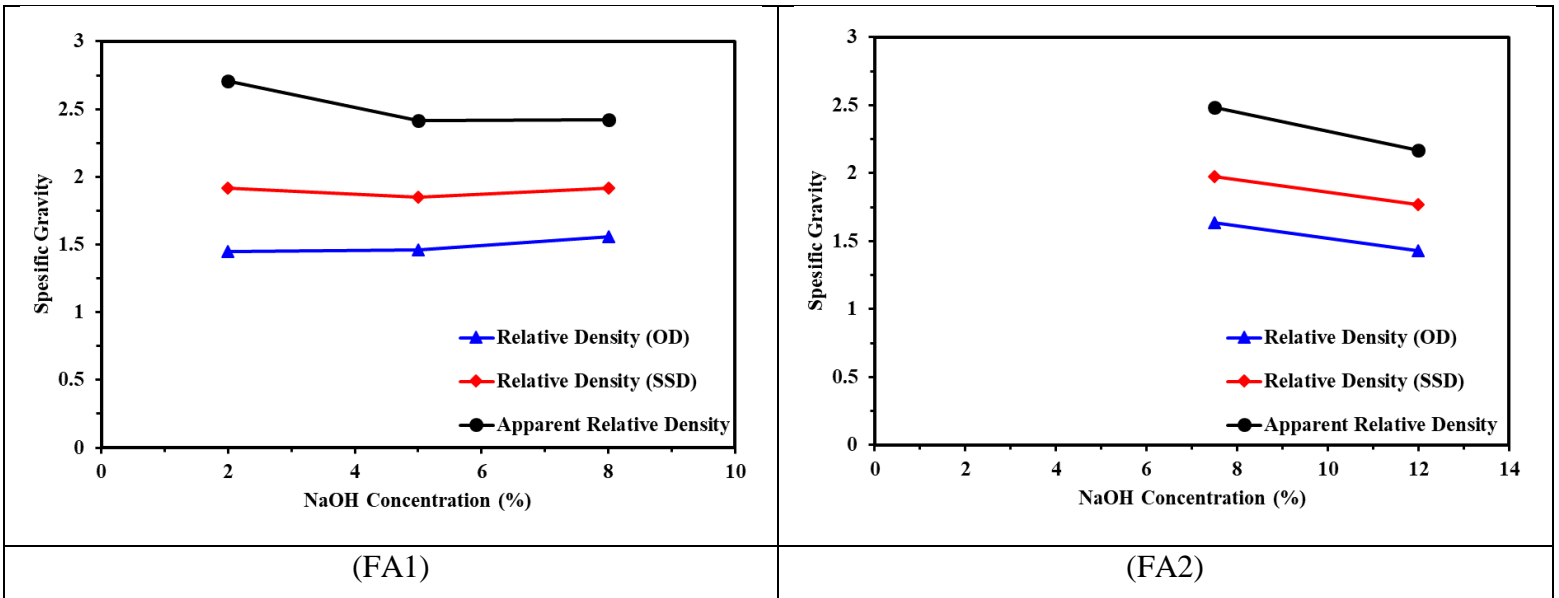
202 *3.3 Specific gravity*

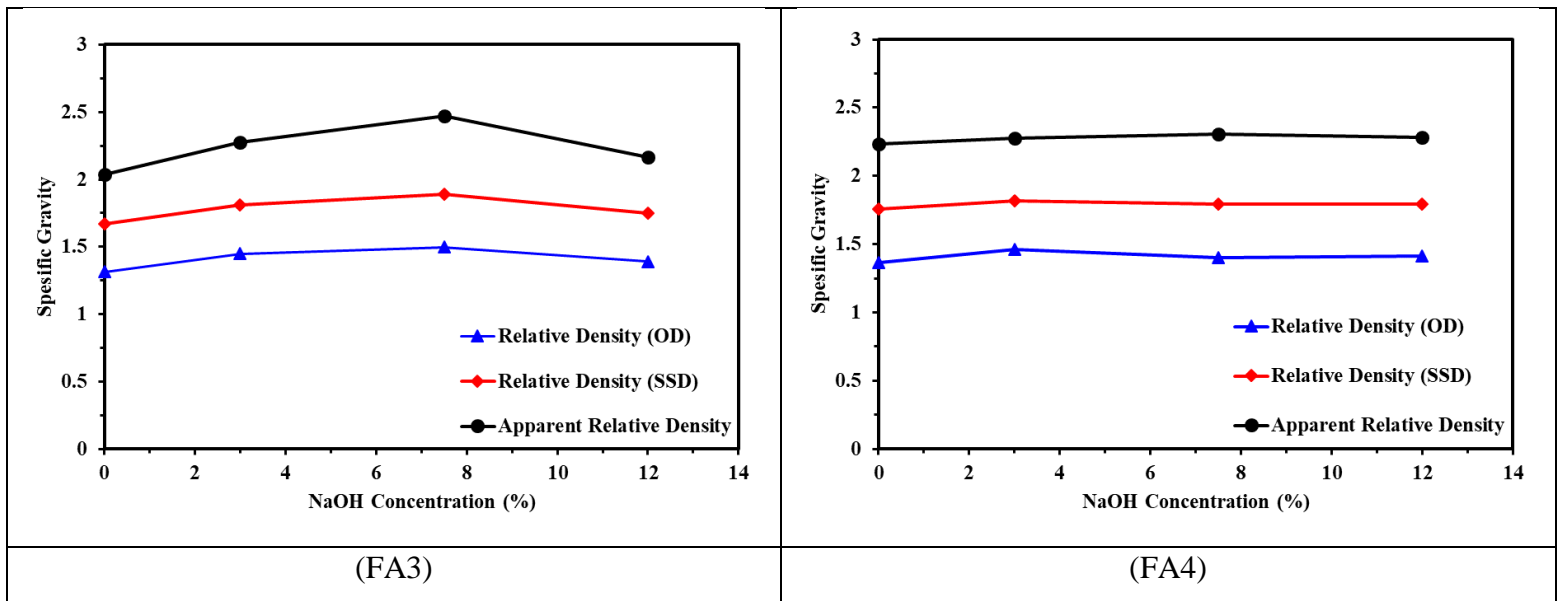
203 For every type of aggregate, three replicates were tested to obtain the specific gravity. Figure 6
204 shows apparent, OD, and SSD specific gravity of the aggregates. Specific gravity for FA1
205 decreased by increasing the NaOH concentration from 2% to 5% and then remained relatively
206 constant after 5%. For FA2, a reduction trend was observed by increasing the NaOH concentration
207 from 7.5% to 12%. This observation may be attributed to the bloating effect at the elevated
208 temperature contributed by the high LOI content.

209

210 Aggregates made from FA3 showed an increase in the specific gravity when increasing NaOH
211 concentration up to 7.5% beyond which the specific gravity decreased. However, the specific
212 gravity for the FA4 aggregates remained constant at all NaOH concentrations. This behavior
213 implies the effect of LOI on the physical properties of aggregates, regarding the fact that FA3 and
214 FA4 had the same chemical compositions.

215





216 Figure 6. Apparent, oven dry, and saturated surface dry specific gravity of aggerates made from
 217 (a) FA1 (b) FA2 (c) FA3, and (d) FA4.

218

219

220 3.4 Water absorption

221

222 Table 4 shows the water absorption of aggerates at 6h, 24h, 72h and their vacuum absorption.

223 Vacuum absorption of the aggerates varied from 20.9% to 32%. For FA1, vacuum absorption

224 decreased by increasing the NaOH concentration. Interestingly, although FA1-2%-8min had 32%

225 vacuum absorption, but 72h water absorption could not exceed 0.8 %. FA1-5%-8min and FA1-8%-

226 8min could almost reach 45% of their vacuum absorption. On the other hand, vacuum absorption and

227 absorption over time for aggerates made from FA2, increased by increasing the NaOH concentration.

228

229 For aggregates made from FA3, vacuum absorption ranged from 25% to 26.9%. FA3-12%-8min and

230 FA3-3%-8min had a 72h water absorption equal to 7.2% and 6.7%, respectively. These values were

231 approximately 25% of their vacuum absorption, which may suggest that formation of glassy phase on

232 the surface of the aggregates limits their water absorption [13]. For FA3-7.5%-8min, the 72h water
 233 absorption was 50% of its vacuum absorption. It can be inferred, although the liquid phase formed on
 234 the surface of aggregates but probably the configuration of connected pores to the surface led to higher
 235 72 h water absorption. FA4-7.5%-8min had the highest water absorption among all aggregates made
 236 from FA4, which was approximately 50% of its vacuum absorption. The high water absorption
 237 property of aggregates can be attributed to many factors such as pore volume, size, tortuosity, and
 238 connectivity.

239

240 Table 4. Water absorption at 6h, 24h, 72h, and vacuum water absorption of aggregate samples
 241 made from different FAs

Sample Name	6h water absorption (%)	24h water absorption (%)	72h water absorption (%)	Vacuum absorption (%)
FA1-2%-8min	0.8	0.8	0.8	32.0
FA1-5%-8min	11.1	12	12	27.0
FA1-8%-8min	9	9.6	9.6	22.8
FA2-7.5%-8min	7.5	10.2	10.2	20.9
FA2-12%-8min	16.7	17.4	17.4	23.9
FA3-0%-8min	8.4	11.1	11.1	26.9
FA3-3%-8min	5.9	6.7	7.2	25.0
FA3-7.5%-8min	13	13.2	13.2	26.2
FA3-12%-8min	6.5	6.5	6.7	26.0
FA4-0%-8min	9.7	9.9	9.9	28.4
FA4-3%-8min	5	6.1	6.1	24.3
FA4-7.5%-8min	9.7	13.3	14.6	28.1
FA4-12%-8min	9.4	11.6	12.6	27.0

242

243

244 **4. Summary**

245 This study investigated the feasibility of LWA production from waste off-spec FA and evaluated
246 their engineering properties including, specific gravity, porosity, and water absorption. The
247 findings from this study are:

248 1- Physical appearance of aggregates made from FA1 and FA2 were completely different due to
249 their difference in LOI content. Aggregates made from FA2, which has 8.7% LOI, showed a partial
250 eruption which may be attributed to excessive gas formation during the sintering. This was also
251 observed with aggregates made with FA3 and FA4, which have the same chemical compositions
252 and different LOI contents, i.e., the different appearance for the sintered aggregates is attributed
253 to LOI content.

254 2- Specific gravity for FA1 and FA2 aggregates showed a decreasing trend by increasing the NaOH
255 concentration. On the other hand, specific gravity for aggregates made from FA3 increased by
256 increasing the NaOH concentration up to 7.5% beyond which specific gravity decreased. Specific
257 gravity of FA4 aggregates remained constant by changing NaOH concentration.

258 3- Aggregate FA1-2%-8min, showed a very low water absorption although it had 32% vacuum
259 absorption. For aggregates made from FA2, with high LOI, increasing the NaOH concentration
260 from 7.5% to 12% increased the 72h water absorption to 70%. However, aggregates made from
261 FA3 and FA4, which formed a glassy phase on the surface, could have a low 72h water absorption,
262 as the liquid phase can act as a barrier for absorption of water.

263 Based on the measured engineering properties, this study shows that produced aggregates from
264 FA had OD specific gravities (1.3 to 1.6) comparable to traditional LWA specific gravities (1.3 to
265 1.7), therefore; can be classified as promising LWAs. Moreover, these LWAs had appropriate

266 water absorption capability over time (0.8% to 17.4%), which may provide opportunities for use
267 in internal curing of concrete. However, further investigation on the effect of LOI and fluxing
268 agent on the formation of liquid phase on the surface of aggregate, pore size distribution and water
269 absorption of these LWAs is required.

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275

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