

Properties of rice straw reinforced alkali activated cementitious composites

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1	Properties of rice straw reinforced alkali activated cementitious composites							
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14								
15	HIGHLIGHTS							
16	• Characteristics of rice straw were studied by SEMs and XRDs							
17	• Characteristics of rice straw alkali activated cementitious composites were investigated via							
18	strengths, density, water absorption, drying shrinkage and wet/dry cycling.							
19	• Alkali treatment enhances bond at rice straw and alkali activated cementitious paste interface							
20	• Mechanisms of deterioration of the composites under wet/dry cycling was studied by SEMs							
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Properties of rice straw reinforced alkali activated cementitious composites ABSTRACT

36 The paper investigates the characteristics of rice straw reinforced alkali activated cementitious 37 composites (AACC). The untreated and NaOH treated rice straw at the proportion of 1%, 2%, 38 3% by weight of binder was added to the mixes. Characteristics of rice straw have been 39 studied by using SEM and XRD. Mechanical properties, water absorption, drying shrinkage 40 and durability under wet/dry cycling have been investigated to evaluate the performance of 41 the AACCs. SEM was also used to investigate the mechanism of deterioration of the AACC. 42 The results show that rice straw has very significant positive effects on the performance of 43 AACCs including increase in flexural and compressive strength, durability under wet/dry 44 cycling, large reduction in drying shrinkage, and water absorption. In addition, alkali treatment is an effective method for enhancing bond between the rice straw and the matrix 45 46 leading to improved performance of AACCs.

47 Keywords: Alkali activated cementitious composite (AACC); Rice straw; Alkali treatment;
48 Strength; Durability; Drying shrinkage; Water absorption.

49 **1. Introduction**

50 CO₂ emission is a key contribute to global warming and the production of Portland cement 51 provides about 5% of global CO₂ emission [1]. Efforts to reduce the use of Portland cement in 52 concrete have led to research on alkali activated cementitious materials (AACMs) in order to 53 provide sustainable material for the construction industry. Alkali activation is the chemical 54 reaction between a solid aluminosilicate precursor and an alkaline activator to make hardened 55 materials. The curing temperature mainly depends on the content of calcium within the 56 aluminosilicate. The alkali activated aluminosilicate containing high calcium content can be 57 hardened at ambient temperature; whereas thermal treatment should be applied to induce the 58 setting of alkali activated aluminosilicate without calcium source [2-4]. The main precursors 59 used for producing AACMs are industrial by products such as granulated blast furnace slag derived from the steel manufacture, fly ash derived from coal combustion and natural clays 60 61 (metakaolin) [5].

62 Characteristics of alkali activated cementitious materials have been studied by many 63 researchers. Compressive strengths of alkali-activated, blast furnace slag high-calcium binders 64 and fly ash-based low-calcium binders can be achieved up to 110MPa [6]. Drying shrinkage 65 of AACMs is typically higher than that of Portland cement- based binders, when 66 manufactured and cured under ambient temperatures, but can be controlled by good mix 67 design of the AACM concrete [3,7] or by using shrinkage reducing admixtures [8]. The sorptivity of AACMs is within a comparable range with similar grade concretes [4]. The 68 69 capillary sorptivity is reduced by employing a lower water content and a higher silica 70 modulus activator [3]. AACM shows greater durability potential than OPC as it has lower 71 porosity [9]. Some tests have shown that superior chloride resistance of the AACM concrete 72 compared with a similar grade of normal Portland cement-based concrete [7]. Carbonation 73 results of AACMs under accelerated carbonation testing often show inferior performance to 74 Portland cement binders, which contradict data obtained under long-term natural exposure [4]. 75 Many studies have shown better frost resistance of AACM concrete compared with similar 76 grade conventional concretes exposed to the same conditions [10,11].

77 Natural fibres have been used to replace synthetic fibres to reinforce concrete or mortar for78 sustainable development. The natural fibres including sisal, flax, hemp, bamboo and coir are

79 cheaper and lighter than synthetic fibres. They are an abundant, renewable and low cost 80 resource [12-15]. There has been far less research on cellulose fibre reinforced cement composites compared with synthetic fibres. The main drawbacks for the use of cellulose 81 82 fibres as reinforcement for cement composites are the shortage of information on production 83 techniques, failure mechanism, fibre strength, effectiveness of fibre shape and size on the 84 performance of composites [16]. Therefore, it is necessary to conduct more research on the 85 performance of cellulose fibre reinforced cement composites, including the effect of fibre 86 dispersion on the matrix properties.

87 Rice straw is a worldwide by -product of rice production. There are some current solutions for 88 disposal of rice straw such as livestock feeds, burning in-situ in the farm or incorporating into 89 the soil. Each solution shows its own advantages and disadvantages. Burning at the field is 90 considered as the cheapest method but it causes air pollution as it releases CO_2 [17]. The use 91 of rice straw to provide natural fibres has been studied and rice straw shows a high potential 92 for fibre reinforcement in cementitious mortar and concrete [17-19].

93 The paper investigates the characteristics of a novel alkali activated cementitious composite 94 (AACC) for low impact buildings by incorporating rice straw fibres from agricultural waste products. The characteristics of rice straw have been determined by Scanning Electron 95 96 Microscope (SEM) and X-ray powder diffraction (XRD). The performance of AACC has 97 been investigated with a wide range of tests including compressive strength, flexural strength, 98 water absorption, drying shrinkage and durability under wet/dry cycling. A SEM was used to 99 study the bond at the rice straw interface with the alkali activated cementitious matrix in order 100 to explain its influence on strength and durability of the composites material. In addition, 101 XRD analysis was also used to determine the effect of chemical reaction between rice straw 102 and AACM matrix on the crystalline structure of the matrix.

103 2. Experimental programme

104 2.1 Materials

105 The alkali activated cementitious composites consists of ground granulated blast furnace slag 106 (GGBS) based binder, fine aggregate (sand), sodium silicate and hydroxide based activator, 107 retarder R42 and rice straw (RS) fibres. The activator was based on a sodium silicate solution 108 of molarity 6.5 mol/L and silica modulus 2% together with NaOH of molarity 4.8 mol/L. 109 GGBS was supplied by Hanson Heidelberg cement Group, UK and its chemical composition 110 is shown in **Table 1**. The XRD pattern of GGBS is presented in **Fig. 1** which was obtained by 111 using a Philips X-1 Pert X-ray diffractometer, operated with a Cu Ka radiation source (40 KV 112 and 40 mA, wavelength λ =1.5406nm [6.07 x10-9 2 in.]) by scanning from 5° to 75° at an 113 angle of 2 Θ , the scan step size is 0.0131303. X-ray data were fitted using the pseudo-Voigt 114 profile function and refined by means of Rietveld. The GGBS shows a peak hump between $2\Theta = 20^{\circ}$ and 40° because of the amorphous components. Fig.1 also shows that there is no 115 116 significant crystalline material observed. Fine aggregate (sand) obtained from a supplier in 117 Sheffield, UK was oven dried at 60°C for 24hours. It was sieved to remove particles retained 118 on a 2mm size sieve then left in the laboratory air for three days before mixing and casting.

119



Fig. 1. X-Ray diffractograms of GGBS

Rice straw was received from a supplier in China. It was cut into 15-20mm lengths by using a scissors and classified as two types of fibre namely untreated rice straw (URS) and treated rice straw (TRS). For untreated fibre, the chopped rice straw was stored in an air tight bag until its use. For treated fibre, the chopped rice straw was soaked in 5% NaOH solution for 48hours in a fume cupboard, then washed with tap water until clear and oven dried at 45°C for 24 hours. The fibre was left in the laboratory air for 24hours and then sealed in an air tight bag until its use in the AACC mixes (see Fig. 2). This alkali treatment has proved to be effective for eliminating organic impurities and low molar mass hemicellulose thereby enhancing the fibre strength and fibre-matrix adhesion [13, 20, 21] including rice straw [18]. This treatment method also breaks down fibre bundles into thinner fibres, thereby, increasing their effective surface area [22] as can be seen in Fig. 2. No additional physical treatment was applied to thin up the straw fibres.



Fig. 2. (a) Untreated and (b) treated rice straw fibres

144 Table 1

145 Chemical composition of GGBS

Constituents	GGBS
Na ₂ O %	0.786
MgO %	11.122
Al ₂ O ₃ %	16.352
SiO ₂ %	32.972
SO ₃ %	2.109
K ₂ O %	0.667
CaO %	34.919
TiO ₂ %	0.24
MnO %	0.265
Fe ₂ O ₃ %	0.17
SrO %	0.154
BaO %	0.244

146 **2.2 Details of mixes**

147 Details of all mixes are given in Table 2. The mix composition of the control sample (M0) are 148 1: 1.94 (by weight) of binder to sand with liquid activator/binder of 0.56 and extra 149 water/binder of 0.1. Extra water was used to improve the workability of the rice straw 150 composites and also to compensate for the dry state of fine aggregate and fibre. The retarder 151 R42 at ratio of 0.76% by weight of binder was added to the mix for increasing the setting 152 time. The URS and TRS fibres were added at 1%, 2% and 3% of total binder weight, to 153 produce composite mixes M1U, M2U, M3U and M1T, M2T, M3T respectively. The rice 154 straw was used to replace its weight of sand in all mixes.

155 **Table 2**

156 The mix proportions of AACC

	AACM		R42	T • • • • • • • • • • •	Water/	Rice straw		
ID	Binder	Sand (%)	(%	Liquid activator/ Binder	Binder Ratio	Amount	Treatment	
	(%)	(,-)	binder)	Ratio		(%Binder)	methods	
M0	34	66.00	0.76	0.56	0.1	0	-	
M1U	34	65.94	0.76	0.56	0.1	1	untreated	
M2U	34	65.89	0.76	0.56	0.1	2	untreated	
M3U	34	65.83	0.76	0.56	0.1	3	untreated	
M1T	34	65.94	0.76	0.56	0.1	1	treated NaOH	
M2T	34	65.89	0.76	0.56	0.1	2	treated NaOH	
M3T	34	65.83	0.76	0.56	0.1	3	treated NaOH	

157 2.3. Casting and curing

158 The AACM binder and sand were mixed for one minute and then the liquid activator was 159 slowly added to the mix. Mixing continued for 5 min until a uniform texture was produced. 160 The retarder admixture R42 was then slowly added while mixing continued. The rice straw 161 fibres were gradually added to the mix together with extra water and mixing continued for 10 162 minutes. The same procedure was applied for the control AACM mix without adding rice

- straw fibres. Specimens for all mixes were cast in 40x40x160mm and 50x50x50mm steelmoulds and compacted by the vibration table in two minutes.
- 165 All specimens were left in the moulds covered with polythene sheets for 24 hours in the 166 laboratory air ($20 \pm 2^{\circ}$ C, RH 65±5%). After 24 hours from casting, the specimens were 167 demoulded and cured in accordance with different test procedures as detailed in the next 168 section.

169 **2.4. Test procedures**

170 2.4.1. Rice straw characteristics

171 The properties of URS and TRS fibres were determined in accordance with procedures 172 described in [18]. SEM images of URS and TRS fibres were obtained with an ETD detector, a 173 working distance of about 10 mm, an accelerating voltage of 5 kV and a spot size of 4 nm. 174 XRD plots of URS and TRS fibres were obtained by using a Philips X-1 Pert X-ray 175 diffractometer, operated with a Cu Ka radiation source (40 KV and 40 mA, wavelength λ =1.5406nm [6.07 x10-9 2 in.]) by scanning from 5° to 75° at an angle of 2 Θ , the scan step 176 177 size is 0.0131303. X-ray data were fitted using the pseudo-Voigt profile function and refined 178 by means of Rietveld.

179 *2.4.2. Consistency of fresh composites*

180 The workability of fresh mortar was measured with the flow table test in accordance with BS 181 EN 1015-3:1999 [23]. The flow value represented the mean diameter of a test sample of the 182 fresh mortar placed on the flow table disc by means of a defined mould and given a number of 183 vertical impacts by raising the flow table and allowing it to fall freely through a given height.

184 2.4.3. Dry bulk density

Dry bulk densities of hardened mortar were determined in accordance with BS EN 1015-10:1999 [24]. Three cubes of 50x50x50mm were cast and cured in water. At 28 days age, the cubes were oven dried at 60°C for 48 hours to reach the constant mass for calculation of the dry bulk density.

189 *2.4.4. Mechanical properties*

190 The flexural and compressive strengths were determined in accordance with BS EN 196-1: 191 2005 [25]. After demoulding, three prisms of 40x40x160mm dimensions were used to 192 determine the flexural and compressive strength at 1 day age while another 6 prisms were cured in water in the humidity room (20°C, RH45%) to determine strengths at 14 and 28 days 193 194 age. The three point bending test method was used to determine the flexural strengths. The 195 two halves of the broken prisms from the flexural strength tests were used to determine the 196 compressive strengths. Hence, the strength measurements of AACC were made at 1, 14 and 197 28 days age. Each result of the flexural and compressive strength was the average value of 198 three and six specimens respectively.

SEM (QUANTA 650) was also used to observe the bond at the straw matrix interface.
Samples for SEM were taken from the 28 day age specimens, oven dried for 4 hours before
coating with a 20 nm thick layer of gold using a Quorum Q150T. SEM images were obtained
with an ETD detector, a working distance of about 10 mm, an accelerating voltage of 5 kV
and a spot size of 4 nm.

204 XRD was used to study the effect of the treatment method of the rice straw on the 205 mineralogical compositions of alkali activated cementitious matrix at 28 days by testing 206 samples of M0, M2U, M2T. AACM matrix from broken flexural strength test samples, after removing of the rice straw, was crushed by the hammer and powder was sieved under 7.5 µm 207 208 for XRD test samples. XRDs were conducted on a Philips X-1 Pert X-ray diffractometer. 209 They were operated with a Cu Ka radiation source (40 KV and 40 mA, wavelength 210 λ =1.5406nm [6.07 x10-9 2 in.]) by scanning from 5° to 75° at an angle of 2 Θ , the scan step size is 0.0131303. X-ray data were fitted using the pseudo-Voigt profile function and refined 211 212 by means of Rietveld Refinement

213 2.4.5. Water absorption test

Test was conducted in accordance with ASTM C1403-15: Standard test method for rate of water absorption of masonry mortars [26]. Three cubes of dimensions of 50x50x50mm were cast and demoulded after 24 hours. After demoulding, they were put in an air tight plastic bag and cured in a desiccator for 28 days before performing the water absorption test.

218 2.4.6. Drying shrinkage

219 The drying shrinkages of the different compositions of AACC were determined according to ASTM C596 -18: Standard test method for drying shrinkage of mortar containing hydraulic 220 221 cement [27]. Three prism specimens of 160mm length and 40x40 mm cross section were cast 222 and demoulded after 24 hours. Two demec points were attached on each face of the prism at a 223 gauge length of 100 mm to measure the distance (strain) between them with an extensometer. 224 The samples were then cured in water at 20°C. They were removed from water at 3 days after 225 casting, dried with a cloth and the first (datum) strain reading was taken with a demec 226 extensometer. The shrinkage specimens were then cured in the humidity room at 20°C, 45% 227 RH. Subsequent shrinkage readings were taken at regular intervals up to 90 days with the 228 extensometer.

229 2.4.7. Durability under wet/dry cycling

230 Wet/dry cycling tests were performed on six prisms of dimensions 40x40x160mm for each 231 mix. They were demould 24 hours after casting and cured in water until 28 days age. Three 232 prisms (set A, reference) were continuously cured in water until the test date. An other three 233 samples (set B) were subjected to wet/dry cycles. Each cycle consisted of 24 hours in water at 20°C and 24 hours in the oven (60°C). The specimens were dried to attain a surface dry 234 235 condition before being placed in the oven, and they were also allowed to cool to room 236 temperature before being immersed in the water. Therefore, one cycle consisted of 23.5 hours 237 wet at 20°C, 0.5 hour lab air (20°C, 65%RH), 23.5 hours dry (oven, 60°C, 20%RH) and 0.5 238 hour lab air (20°C, 65%RH). At 71 days age (20 wet/dry cycles), all wet/dry curing samples 239 (set B) and water curing samples (set A) were tested to determine the flexural and 240 compressive strengths. The retained strength ratio is defined as the ratio of strength of the 241 samples after 20 wet/dry cycles curing (set B) to strength of the samples cured in water at 242 20oC, 45%RH for the duration of the durability test (set A, reference). SEM tests were also 243 conducted to determine the reason for strength loss such as bond loss at the rice straw fibre/ 244 cement matrix interface or damage/strength loss of rice straw fibre or cracking of the alkali 245 activated cementitious matrix.

246 **3 Results and Discussion**

247 3.1. Characteristics of rice straw

248 3.1.1. SEM study

SEM images of URS and TRS fibres are shown in Fig. 3. The surface of URS fibre is quite regular and covered with a layer of substance which includes oils, waxes and extractives such as lipids, phenolic compounds, terpenoids, fatty acids, resin acids, etc..[18]. The surface of TRS fibre is rough as the NaOH treatment removes extractives, waxes, oil and amorphous constituents such as hemicellulose and lignin from fibre surfaces [28]. It can be seen that the surface of TRS fibres has greater roughness than the URS fibres thus promising greater bond strength with the matrix.

256



257 258

Fig. 3. SEM micrographics of (a) URS and (b) TRS

259 3.1.2 XRD study

260 XRD plots of URS and TRS fibres are presented in **Fig. 4**. There are only two clear peaks 261 appearing at around 2Θ =18° and 22° for both URS and TRS fibres and the spectrums are very 262 similar for both type of fibres. However, it is clear that the peaks of TRS are slightly higher 263 than that of URS fibres due to the removal of the amorphous materials which agree well with 264 previous research [18]. The degree of crystalline Cellulose (I) in the total cellulose can be 265 expressed by the X-ray "crystallinity index CI" as follows [18, 29, 30] using the data in **Fig.** 266 **4**. The results are presented in **Table 3**.

$$CI = 100 \times \frac{I_{002} - I_{am}}{I_{002}}$$

267 In which: *Ioo2* is the intensity of the principal cellulose (I) peak at $2\theta = 22^{\circ}$

268 *I_{am}* is the intensity due to the amorphous part of the sample at $2\theta = 18^{\circ}$.

269





Fig. 4. X-Ray diffractograms of treated and untreated Rice straw

272 Table 3

273	Degree of	crystalline	cellulose of	untreated	and	treated	rice	straw
-----	-----------	-------------	--------------	-----------	-----	---------	------	-------

Fibres	I ₀₀₂	I _{am}	CI (%)
Untreated rice straw (URS)	33463	22509	32.7
NaOH treated rice straw (TRS)	49372	27448	44.4

Table 3 shows that crystallinity index of TRS fibres is about 35% (from 32.7% for URS to
44.4% for TRS) higher than the URS fibres which is due to the part of the amorphous
materials. This result also agrees well with the previous research conducted on rice straw
sources from Egypt [18].

278 **3.2.** Workability

279 The flows of all mixes are given in Fig. 5. The rice straw fibres reduced the flow of the 280 AACC and the workability (flows) decreased with increasing volume of rice straw fibres. The 281 reduction in workability of rice straw composites can be due to water absorption by the 282 hydrophilic natural fibres. The loss of workability of natural fibre composites depends mainly 283 on the fibre aspect ratio and volume fraction in the mixtures [16]. Previous research shows 284 that the increase in length and content of jute fibre resulted in the decrease in workability [31]. 285 It is also reported that the workability of cement composites reduced when eucalyptus pulp, 286 coir or eucalyptus pulp combined with sisal fibres [32] were added to the matrix.

Fig. 5 also shows that the TRS fibres resulted in a higher reduction in workability than the URS fibres. This can be explained by the increase in dry surface areas of TRS fibres compared with the URS fibres (see Fig. 2). For improvement of workability of rice straw composites, the straw fibre can be pre-wetted before adding to the mixtures, or by consideration of the water absorption property of fibres in the mixture design [16] or using superplasticizer.





307 308

Fig. 5. Flow of fresh rice straw composites

295 **3.3. Dry bulk density**

296 The dry bulk density of all composites is given in Fig. 6. It can be seen that 2% and 3% 297 addition of URS reduced the density of the ACCC while there is a slight increase of density 298 when 1% of URS is added. The alkali treated rice straw increases the density of alkali 299 activated cementitious composites when added at the proportion of 1% and 2% by weight of 300 binder but reduced the density of the composite when added at 3% compared with the control sample M0. This agrees well with the water absorption test results in section 3.5. Within the 301 302 range of 1-3% fibre content, the density decreases with increasing rice straw fibre content for both untreated and treated fibres. The largest difference of the density between URS and TRS 303 304 fibres are at 3% fibre content which is only more than 5% (from 1.727 g/cm³ for M3U to 1.818 g/cm³ for M3T). It shows that the difference between density of URS and TRS 305 306 composites is not significant.



Fig. 6. Dry bulk density of hardened rice straw composites

309 3.4 Mechanical properties

310 *3.4.1 Flexural strength*



312

311

Fig. 7. Flexural strengths of AACCs

The flexural strengths of all AACCs are plotted in Fig. 7. It is clear that the URS reduced the 313 314 flexural strength while the NaOH TRS increases the flexural strength compared with the 315 control sample without rice straw at 1 day, 14 days and 28 days, except for the increase in 316 flexural strength indicated at 1 day with the 1% of URS fibre reinforced composite. The 317 largest reductions up to 6.5% in flexural strength are at 2% of URS reinforcement (from 318 5.02MPa for M0 to 4.69MPa for M2U) at 14 days and 13.8% (from 5.54MPa for M0 to 319 4.77MPa for M2U) at 28 days. In contrast, the TRS increases the flexural strength of alkali 320 activated cementitious composites. It also been seen that at early age (1 day) the largest 321 increase in flexural strength is at 2% TRS fibres, however, at later age (14 and 28 days), it is 322 at 3% TRS. This can be explained by the aging effect on the bond of TRS fibre with the 323 matrix. Aging effect on bond has been investigated by many researchers where the bond 324 strength at steel fibre/ cement matrix interface rapidly increased with age [33, 34]. The 325 frictional bond strength of polyethylene (PE) fibre in plain and silica fume matrices under 326 moisture curing increased from 0.5 to 28 days age [35]. The increase in bond strength with 327 age is due to the increase in strength of matrix surrounding the fibres [33, 36-39]. This can 328 also be applied to the cellulose fibres as the increase in bond at rice straw fibre interface is 329 due to the increase in strength of the matrix surrounding the rice straw. Therefore, at 14 and 330 28 days age, increasing percentage of TRS results in the higher increase in flexural strength. 331 The increases in flexural strength of 3% TRS fibre composites compared with the control 332 sample are 23.5% (from 5.02MPa for M0 to 6.20MPa for the M3T) at 14 days and 19.7% 333 (from 5.54MPa for M0 to 6.63MPa for M3T) at 28 days. It is also noted that the rice straw 334 fibres fractured under flexural testing in both URS and TRS samples (see Fig. 8) proving that 335 the bond strength of the rice straw fibres with the matrix was higher than the tensile strength 336 of rice straw fibres.





338

Fig. 8. Failures of a) URS and b) TRS fibres under flexural testing

339 *3.4.2. Compressive strength*





341

Fig. 9. Compressive strength of alkali activated cementitious composites

342 The compressive strengths of AACCs are shown in Fig. 9. The compressive strength of 343 composites reduced at early age (1 day) when URS fibres were added to the AACM mortar, 344 the strength decreased with increasing of fibre content. However at 14 days, 1% and 2% URS 345 increase the compressive strength while 3% URS composite has similar strength to the control 346 samples (M0). At 28 days, all 1%, 2% and 3% URS composites have higher compressive 347 strengths than the control sample M0. The increase in compressive strength of URS 348 composites compared with the control sample at 14, 28 days can again be explained by the 349 aging effect detailed in section 3.4.1 as the increase in the strength of matrix surrounding the 350 fibres. The optimum proportion of URS is 1% within the range of investigation with the 351 increase in compressive strength of nearly 40% (from 26.0 MPa for the control sample M0 to 352 36.29MPa for the M1U) at 28 days.

In contrast to the URS, the TRS fibres generally improve the compressive strength of the composite. At early age (1 day), 1% and 2% TRS slightly reduced the compressive strength while 3% TRS improved the compressive strength. At 14 days and 28 days, the TRS improved significantly the compressive strengths which increase with increasing straw content. The better performance of TRS fibre composite compared with the URS can be explained by the improvement of bond at the interface with the matrix due to the increase in effective surface area [22] and surface roughness as explained in detail in the next section. The maximum increase in compressive strength is at 3% TRS composite which are around (from 23.0MPa for M0 to 36.19MPa for M3T) at 7 days and 73% (from 26.0MPa for M0 to 44.93MPa for M3T) at 28 days. This is not in line with the density results in section 3.3 as 3% of both URS and TRS reduces significantly the density of the composites. The reason is that the compressive strength is less affected by fibre density and also improved by the hydrophilic nature of the fibres helps.

366 The compressive strength of the AAC composites is primarily controlled by the AACM 367 matrix strength. Therefore, the increase in compressive strength of AACCs compared to the 368 control can be explained by the hydrophilic nature of rice straw fibres which leads to a 369 reduction in liquid activator/ binder ratio of the matrix compared to the control. In addition, 370 the liquid activator absorbed in the rice straw fibres also works as the internal curing agent of 371 the cementitious matrix enhancing its strength. While the URS absorbed less liquid activator than the TRS then this hydrophilic effect shows more effectiveness in TRS composites than 372 373 URS composites. This is similar to the effect of porous lightweight aggregates in concrete 374 [39].

375 3.4.3 Scanning Electron Microscope Study

376 Bond at the fibre- matrix interface is an important factor effecting the strength of the 377 composite as the stress can be transferred from the cement matrix to the fibres throughout the 378 interface under loading [40, 41]. One of the drawbacks of using natural fibres in cementitious 379 composites is the poor bond due to the hydrophilic nature of natural fibre. The bond failure at 380 fibre and cement matrix interface under loading is due to poor chemical and physical 381 interfacial interaction between natural fibre and cement matrix [42]. The chemical treatment 382 with NaOH solution was used to eliminate lignin, natural fats, waxes and impurities from the 383 fibre surface to improve the surface roughness of natural fibres [43, 44] and surface 384 modification for enhancing the interfacial interaction [45]. The SEM images in Fig. 10 show 385 that rice straw, after alkali treatment, improved the interfacial interaction since the gap (crack) 386 between the matrix and TRS interface (Fig. 10b) is quite small compared with the URS (Fig. 387 10a). In the case of TRS (Fig.10b) the matrix appears to have merged with the fibre at the 388 interface. It can also be seen that surface roughness of rice straw is improved significantly 389 after alkali treatment enhancing the bond strength (Fig. 3).



390

391 Fig.10. SEM images at (a) URS fibre- matrix interface and (b) TRS fibre- matrix interface
392 *3.4.4. XRD Analysis*

393 Mineralogical compositions of alkali activated cementitious paste at 28 days of M0, M2U, 394 M2T are shown in Fig. 11. It can be seen that the XRD patterns of the three composites are 395 very similar showing presence of quartz, calcite and gypsum. The peaks of quartz at $2\Theta=27^{\circ}$ increased slightly as rice straw was used in the composite and the peak of quartz of TRS 396 397 composite is significantly higher than that of URS composite. The total CaCO₃ reduced 398 slightly when rice straw was added to the mixes with TRS reducing CaCO₃ more than the 399 URS. The rice straw and its treatment methods have slight influence on the mineralogical compositions of alkali activated cementitious paste. This may due to the impurity of rice 400 401 straw surface and the NaOH solution used for rice straw treatment.



402





405 **3.5. Water absorption**

406

407

Fig. 12. Water absorption of alkali activated cementitious composites

408 Fig. 12 shows that both URS and TRS reinforcement improves the water resistance of the
409 composites. The water absorption of 1% and 3 % of both URS and TRS composites are very
410 similar while the water absorption of 2% URS composite is slightly higher than that of TRS

411 composite. The graph also shows that increasing straw content reduces the water absorption. 412 This agrees well with the previous research where a collated cellulose fibre at volume 413 fractions of up to 0.5% was used in concrete and contributed to the reduction in the 414 permeability of concrete [46]. The reduction in water absorption of fibre composites may due 415 to the reduction in bleeding as fibres increase mix stiffness and reduce the settlement of 416 aggregates (sand). This is also not in line with the results of the density in section 3.3 as the 417 water absorption of the composites is less affected by fibre density, but improved by the 418 hydrophilic nature of the fibres. Rice straw fibres absorb more liquid in the mix thereby 419 reducing the liquid activator/binder ratio and providing the liquid as an internal curing agent. 420 This contributes to enhanced quality of the AACM matrix (greater strength and lower 421 porosity), thus leading to a reduction in the water absorption. The largest reduction in water 422 absorption is up to 60% when 3% URS was added to the alkali activated cementitious mortar.



423 **3.6 Drying shrinkage**

424

425

Fig. 13. Shrinkage of the alkali activated cementitious composites

426 The shrinkages of rice straw fibre AACCs (2% and 3% URS and TRS) and the control mortar 427 up to 90 days are shown in Fig. 13. It is clear that both URS and TRS contributed to the 428 reduction in drying shrinkage of the AACM mortar. It is known that the drying shrinkage is 429 due to the evaporation of free water. Apart from 1% rice straw fibres, the drying shrinkage of 430 URS composites is less than the TRS composites and the optimum occurs at 2% URS. At 90 431 days the 2% URS composite (M2U) had a drying shrinkage of about 40% of the control 432 sample (M0) This may be due to the reduction in pore volume of alkali activated cementitious 433 composites as the rice straw was added. This is confirmed by the results of water absorption 434 tests in section 3.4 where rice straw reduces water absorption of alkali activated cementitious 435 composites. The reduction in drying shrinkage of rice straw composites compared to the 436 control can also be explained by the hydrophilic effect of the straw. Rice straw absorbed the 437 liquid and reduced the liquid activator/ binder ratio while providing internal curing enhancing 438 the impermeability of the matrix. This leads to the reduction in drying shrinkage by reducing 439 moisture loss from the matrix.

440 Synthetic fibres such as carbon, steel, glass have a positive effect on the drying shrinkage of 441 cementitious mortar [47-49] while some natural plant based fibres have a negative effect on 442 the drying shrinkage of cementitious composites [50, 51]. Previous research shows that the drying shrinkage decreases by the addition of carbon fibres depending also on the treatment of 443 444 carbon fibre; it decreased by up to 32% when 0.5% of silane-treated carbon fibres, by weight 445 of cement, were added [47]. Steel fibre also reduced shrinkage by 40% to 83% when 1-3% of 446 fibres by volume were added [48, 49]. Previous research shows that the effect of other plant 447 based natural fibres on the drying shrinkage of cementitious mortar depends on the fibre 448 characteristics and fibre content leading to the effect on matrix pore structures [50]. Short 449 sisal and coconut fibres at 2-3% volume increased drying shrinkage. The higher drying 450 shrinkage of composites containing sisal fibre is attributed to the higher water absorption and 451 less smooth surface of sisal fibre compared with coconut fibre [50]. Other research also 452 reported that sisal fibre increases drying shrinkage due to the increased porosity of samples 453 containing sisal fibre [51]. Therefore, rice straw fibre has positive effect on the reduction in 454 drying shrinkage than other natural plant based fibres. Rice straw composite reduced drying 455 shrinkage by up to 40% at 90 days compared with the control sample without rice straw 456 fibres. The drying shrinkage of the rice straw fibre composites also depends on the content 457 and treatment methods of fibres.

458 **3.7. Durability under wet and dry cycling**

459 3.7.1. Effect of wet dry cycling on the compressive strength



460

461

Fig. 14. Compressive strength of AACC under wet/dry cycling

462 The compressive strengths of AACCs (M1U, M2U, M3U, M1T, M2T, M3T) and control 463 sample (M0) of set A (reference) which were cured in water and set B (20 wet/dry cycles) are 464 presented in Fig. 14. The retained compressive strength ratios defined as the ratio of 465 compressive strength of the set B (20 wet/dry cycles) samples to compressive strength of the 466 set A (reference) samples for the duration of the durability test are also presented in Fig. 14. 467 The composites with 1% URS and 1%, 2% and 3% TRS showed an increase in the 468 compressive strength under 20 wet/dry cycles curing compared with the control sample (M0). The compressive strength of the control sample without rice straw (M0) after 20 wet/dry cycles curing is 34.31MPa while the compressive strength of 1%, 2% and 3% URS reinforced composites after 20 wet/dry cycles curing are 41.71MPa, 31.32MPa, and 27.45 MPa respectively; the compressive strength of composites reinforced with 1%, 2% and 3% TRS after 20 wet/dry cycles curing are 44.44MPa, 44.09MPa and 46.73 MPa respectively. The 3% TRS is the optimum composite for compressive strength under wet/dry cycling.

475 *3.7.2 Effect of wet dry cycling on flexural strength*

476



477 478

Fig. 15. Flexural strength of AACCs under wet/dry cycling

479 The flexural strengths of AACC and the control samples without rice straw fibres (M0)) of 480 set A (reference) which were cured in water and set B (20 wet/dry cycles) are presented in 481 Fig. 15. The retained flexural strength ratios defined as the ratio of flexural strength of the set 482 B (20 wet/dry cycles) samples to flexural strength of the set A samples (which were cured in 483 water) for the duration of the durability test are also presented in Fig. 15. Both URS and TRS 484 reinforcement increased the flexural strengths of composites after 20 wet/dry cycles curing 485 compared with the control samples without fibres (M0). The flexural strength after 20 wet/dry cycles curing of the control sample without fibres (M0) is 3.11MPa compared with 486 487 3.39MPa, 3.67MPa and 3.18 MPa for 1%, 2%, and 3% URS fibre reinforcement respectively. 488 The flexural strengths of 1%, 2% and 3% TRS composites after 20 wet/dry cycles curing are 489 3.96MPa, 4.2MPa and 3.53MPa respectively.

490 The retained flexural strength ratio of control sample (M0) is 0.55 while the retained flexural 491 strengths ratios of 1%, 2% and 3% URS composites are 0.66, 0.69 and 0.59 respectively; the 492 retained flexural strength ratios of 1%, 2% and 3% TRS composites are 0.62, 0.64 and 0.51 493 respectively. There were reductions in flexural strength after 20 wet/dry cycles for both the 494 AACCs and control samples (M0). The failure of all specimens under flexure occurred with the fracture of straw fibre under 20
wet/dry cycles (Fig. 16). Although under 20 wet/dry cycles there was visual evidence of bond
defect of the straw- matrix interface the residual strength was still larger than the strength of
the fibres.



499

500

Fig. 16. Failures of treated rice straw after 20 wet/dry cycles under flexural testing

501 Fig. 14 shows that the compressive strength of set A (references) which were cured in water 502 and set B (wet/dry cycles) of all AACCs and the control mix are similar, with the retained 503 strength ratios between 0.9 and 1.09, with most values falling between 0.97 and 1.06. This 504 indicates that the 20 wet/dry cycles exposure does not cause any significant reduction in 505 compressive strength. Since the compressive strength of the AAC composites is primarily 506 controlled by the AACM matrix strength with the rice straw fibres contributing primarily to 507 flexural strength and crack control, this indicates that the wet/dry cycles have not affected the 508 durability of the composites with respect to compressive strength. The flexural strengths of 509 the composites and the control mix M0, however, show a substantial reduction relative to the 510 set A (references) which were cured in water (see Fig. 15). The causes of this reduction are shown in the SEM photos given in Fig. 17. Fig. 17b shows greater micro cracking in the 511 512 matrix of AACCs subjected to 20 wet/dry cycles compared to the continuously wet cured 513 specimens (set A) due to the high temperature curing cycle at 60deg C in the former case. 514 This micro cracking has reduced the flexural strength but the compressive strength has not 515 been similarly affected as it is less sensitive to this micro cracking. The contribution to 516 flexural strength reduction by interfacial bond loss of URS fibres after 20 wet/dry cycles' 517 exposure relative to set A (reference) which were cured in water is shown in Figs. 17c and 518 17d where a significant gap between the fibre interface and the matrix can be observed. 519 However, Figs. 17e and 17f show that the bond between the TRS fibres and the AACM 520 matrix remains relatively intact under both wet/dry exposure and water curing only (set A) 521 but the fibre shows a degree of longitudinal cracking (Fig. 17f) due to the 60 deg C drying 522 cycle.

523 3.7.3. Mechanisms of deterioration under wet/dry cycling

524 Previous research on kraft pulp fibre-cement composites showed that under wet /dry cycling, 525 composite failure is due to the initial fibre-cement debonding due to fibre shrinkage during 526 drying, reprecipitation of relatively low-strength hydration products within the new void 527 space produced at fibre-cement interface and fibre mineralization by the re-precipitation of 528 hydration products, likely calcium hydroxide [52]. The failure mechanisms of rice straw fibre and cementitious matrix under wet/dry cycles can be also proposed as follows: i) initial bond
failure under dry cycles as fibre shrinks during drying at high temperature; ii) new AACM
products filling the void space caused by rice straw shrinkage and protecting the rice straw
fibre during wet cycles iii) rice straw mineralization by the AACM products, leading to
embrittlement of rice straw fibres, iv) Micro-cracks appearing in the AACM matrix under the
60°C drying cycle and reducing flexural strength.



AACM (Set A)

baste

(b) AACM paste cracks

AACM (Set B, 20 wet/dry cycles)



537

535

536

538

URS composite (Set A)

Untreated rice straw



539



URS composite (Set B, 20 wet/dry cycles)



TRS composite (Set B, 20 wet/dry cycles)

541 Fig. 17. SEMs of specimens of set A (reference) and set B (20 wet/dry cycles)

542 **4. Conclusions**

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- 543 The main conclusions from the results reported in this paper are as follows:
- Alkali treatment with NaOH is an efficient method for enhancing the roughness of rice
 straw surface leading to an improvement of bond between the rice straw fibres and the
 alkali activated cementitious matrix.
- Both the untreated (URS) and treated (TRS) rice straw fibres with alkali reduce the workability of alkali activated cementitious composites (AACC). The reduction in workability of rice straw composites can be due to water absorption by the hydrophilic rice straw fibres. The TRS fibres resulted in a higher reduction in workability than the URS fibres since the TRS has higher dry surface areas than the URS fibres.
- The URS fibres reduce the flexural strength while TRS fibres increase the flexural strength in comparison with the control sample. The better performance of TRS fibre composite compared with the URS can be explained by the improvement of bond at the interface with the matrix due to the increase in effective surface area and surface solutions.
- Both the URS and TRS reinforcement improve the compressive strengths at 28 days compared with the control. This is due to the hydrophilic nature of rice straw leading to reduction in the liquid activator/binder ratio. In addition, the liquid absorbed in the rice straw fibres provides an internal curing agent enhancing the strength of AACM matrix. The maximum increase of 73% in compressive strength compared with the control occurs at 3% TRS at 28 days.
- Both URS and TRS reduce the water absorption of alkali activated cementitious composites. This may due to the reduction in bleeding as fibres increase mix stiffness and reduce the settlement of aggregates (sand). Greater volume of rice straw fibres leads to less water absorption. The largest reduction in water absorption is up to 60% compared with the control at 3% volume of the URS fibres.
- Both URS and TRS fibres reduce the drying shrinkage of AACC. The rice straw reduces the porosity of alkali activated cementitious composites because of the reduction in liquid activator/binder ratio caused by the hydrophilic nature of rice straw and the internal curing provided by the moisture held in the straw. The reduction in porosity and the moisture trapped in the fibres reduces shrinkage. At 90 days the 2% URS composite reduced the drying shrinkage by more than 40% compared with the control.
- In general, 1% and 2% of both URS and TRS fibres improve the durability of AACCs under wet/dry cycling as it increases the retained flexural strength ratios of the AACCs (between 0.62 and 0.69) compared to the control (0.55). In addition, 1% and 2% of both URS and TRS fibre reduces insignificantly the retained compressive strength ratios of the AACCs (between 0.9 and 1.06) compared to the control (1.09). The highest retained flexural strength ratio is found at 2% URS composites.
 - Further research on the effect of different sources, lengths, treatment methods of rice straw on the mechanical properties including interfacial bond to the matrix is needed as these parameters are expected to contribute significantly to the properties of AACC.
- Further research is needed to analyse the functional groups of straw cellulose and the binding phases within the hardened matrix of the straw composites.

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