Polypropylene fiber reinforced cement mortars containing rice husk ash and nano-alumina				
Ehsan Mohseni <sup>1*</sup> , Mojdeh Mehrinejad Khotbehsara <sup>1</sup> , Farzad Naseri <sup>2</sup> , Maryam Monazami <sup>3</sup> and Prabir Sarker <sup>4</sup>	3 4			
<sup>1</sup> Department of Civil Engineering, University of Guilan, Rasht, Iran	5			
<sup>2</sup> Department of Civil Engineering, Islamic Azad University-East Tehran Branch, Tehran, Iran	6 7			
<sup>3</sup> Department of Civil Engineering, University of Shahrood, Shahrood, Iran	8			
<sup>4</sup> Department of Civil Engineering, Curtin University, Perth, Australia	9			
*Corresponding Author: Mohseni@msc.guilan.ac.ir; Ehsan.mohseni172@gmail.com Tel: +98 9125590423	10 11 12			

## Abstract

This paper presents the effects of incorporating two supplementary cementitious 14 materials: rice husk ash (RHA) and nano-Alumina (NA) in polypropylene fiber 15 (PPF) reinforced cement mortars. RHA is an agricultural waste material and thus 16 recycling of this material has substantial economic and environmental benefits. 17 Compressive strength, flexural strength, water absorption and drying shrinkage of 18 the hardened composites were investigated. The interfacial transition zone and the 19 microstructures were studied by using Scanning Electron Micrograph (SEM) and X-20 ray Diffraction (XRD) analysis. A slight increase in compressive strength of mortar 21 was observed by using up to 10wt% of RHA as a replacement of cement. However, 22 addition of nano-Alumina helped the compressive strength of mortar remain 23 approximately equal to that of the control specimen even when 20 or 30wt% RHA 24 was used. Addition of polypropylene fibers resulted in significant increase in the 25 flexural strength of the mortar specimens. It was also observed that NA and PPF 26 could reduce water absorption by pore blocking effect. The positive interactions 27 between polypropylene fibers and RHA resulted in the lowest drying shrinkage of 28 the fibrous mortar containing RHA. XRD analysis showed that the intensity of Alite 29 and Belite phases decreased and new peak of portlandite produced with the addition 30 of NA. The addition of RHA enhanced the late strength of the cement composites. 31 Consequently, the combined addition of RHA, NA and PPF has resulted in 32 increasing of flexural strength and reduction in both water absorption and drying 33 shrinkage of SCMs. 34

**Key words:** Polypropylene fiber; Rice husk ash; Nano-Al<sub>2</sub>O<sub>3</sub>; Compressive and 35 flexural strength; Water absorption; Drying shrinkage. 36

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## **1. Introduction**

Concrete can be made greener by using waste materials that reduce its 39 environmental impact such as the reduction of CO<sub>2</sub> emission in cement production. 40 According to the European Cement Association, the concrete technology today 41 allows for new buildings to be built with 60% less energy use and CO<sub>2</sub> emissions 42 over the lifecycle of the building than conventional buildings constructed 20 years 43 ago [1]. In the mix design, there are also extra steps taken to improve the 44 sustainability of a structure with a long life cycle. Fly ash, blast furnace slag, 45 recycled concrete, power plant waste, rice husk ash, waste glass, red mud, burnt 46 clay, etc. are some of the waste materials used in concrete. Rice husk ash (RHA) is a 47 carbon neutral green product and several ways are currently being used for disposing 48 them [2-4]. Annually a large amount of rice is produced in the rural areas of 49 different countries such as Iran, Malaysia, Thailand, Bangladesh, India, Pakistan, 50 Myanmar etc. Huge quantities of rice husk are produced during the processing of 51 this rice. The rice husk is used as fuel which generates the rice husk ash. 52 Incorporating rice husk ash in concrete as a cement replacement has many 53 environmental and economic advantages. RHA is a good pozzolan that can be used 54 to make concrete. Using rice husk ash in concrete has been examined by many 55 researchers around the world. The initial and important reviews on the use of RHA 56 in concrete focused on the mechanical performance of concrete [2-9]. However, 57 recent studies have mostly worked on the concrete properties when RHA with 58 additional admixtures were incorporated [10-14]. Using wood fiber waste (WFW), 59 rice husk ash (RHA), and limestone powder waste (LPW) as cement replacement 60 materials in lightweight concrete blocks was studied by Torkaman et al. in 2013 61 [10]. The results indicated that the bulk density and water absorption of concrete 62 samples were significantly reduced by the addition of RHA. Sharma [11] reported 63 that up to 10% mass of cement can be replaced by RHA that is mixed with plastic 64 fibers with nearly equivalent compressive strength. 65

RHA was incorporated in different types of concrete such as normal concrete, self-66 consolidating concrete and high performance concrete [12-18]. Madandoust [12] 67 reported that the use of 20wt% RHA in normal concrete reduced the early-age 68 strength development. Safiuddin et al. [13] investigated the flowing ability of self-69 consolidating concrete and its binder paste and mortar components with rice husk 70 ash. The results showed good improvement in the flow ability of the mixtures when 71 the rice husk ash was added to the binder. The test results revealed that the w/b ratio 72 and RHA content significantly influenced the flowing abilities of the binder pastes, 73 mortars and concretes. However, using RHA may also decrease the flowing ability 74

of the self-compacting concretes. In another study, Safiuddin et al. [14] showed that 75 the flowing ability of mortar mixtures decreased as the amount of RHA was 76 increased. Robler et al. [15] reported that due to the mesoporous structure of RHA, it 77 absorbs free water, so the w/b ratio in the cementitious matrix is reduced and the 78 compressive strength is increased consequently. 79

Nanoparticles have been recently used as a cement replacement to tailor the 80 mechanical properties of concrete [19-24]. These particles increase the cement 81 hydration and act as a filler to reduce the porosity of mortar. Noorvand et al. [20] 82 examined the effect of nano-TiO<sub>2</sub> in black rice husk ash concrete. The results 83 showed improvement in both mechanical and micro-structural properties of mortars 84 with black rice husk ash. In their study, the compressive strength was found to 85 increase by up to 18% when black rice husk ash and nanoparticles were used in the 86 mortar. However, the compressive strength decreased by about 27% when black rice 87 husk ash was used singly. 88

Polypropylene fiber (PPF) was incorporated in concrete for many years to enhance 89 the flexural properties and also restrain the progression of cracks [25-31]. It also 90 improves the strength of concrete against impacts and fatigue. Medina [30] reported 91 that the cracking control ability of PPF on the exposed concrete surface reduced 92 water permeability and  $CO_2$  diffusion. It also increased porosity and reduced bulk 93 density and the ultrasonic modulus of natural pozzolan cement concretes. 94

The ingress of various ions from the environment and its movement through 95 building materials are the main reasons for deterioration of structures. So, the control 96 of the permeability of concrete plays an important role in providing resistance to 97 aggressive environment [32]. In this study, a novel combination of nano-alumina 98 (NA), RHA and PPF was used in concrete. The aim was to partially replace Portland 99 cement by RHA and make mixtures of improved properties in hardened stages by 100 the addition of NA and PPF. 101

2. Experimental program

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#### 2.1 Materials

Natural river sand and ordinary Portland cement type I conforming to ASTM C778105[33] and ASTM C150 [34], respectively were used. According to the ASTM106standard, rice husk ash (RHA) can be used as a pozzolanic material with cement107[35]. The particle size distribution curve of RHA is given in Fig. 1. The physical108properties of RHA substantially depend on the burning conditions. Particularly, the109temperature and period of burning influence on the microstructure and crystallinity110111110

complete burning results in either white or gray RHA. Hwang & Chandra [37] 112 suggested that burning rice husk at temperatures below 700°C provides amorphous 113 silica. In this study the husk was sourced from Rasht, Iran and an electric kiln was 114 used to burn the rice husk pellets. Heating cycles were conducted by an electric 115 system with a heating rate of 5°C/min. Each sample was held at temperatures 116 between 500 and 700°C for 6 hours. The XRD results illustrate that when the RHA 117 was burned within 300°C to 700°C, it turned into amorphous silica. For instance, the 118 XRD pattern of the ash indicates the presence of noncrystalline silica as seen in Fig. 119 2. RHA is mainly composed of silica, which constitutes 91% of the total mass. This 120 amount of silica is seen as Quartz in XRD diagram of RHA. The chemical 121 composition and physical properties of cement and RHA are given in Table 1. Nano-122  $Al_2O_3$  in dispersed suspension form with an average particle size of 20 nm, specific 123 surface area of 200m<sup>2</sup>/g and purity of higher than 98% was used in this study. Fig. 2-124 a and 2-b show the XRD diagrams of the nano-Al<sub>2</sub>O<sub>3</sub> and RHA. The scanning 125 electron micrographs (SEM) of nano-Al<sub>2</sub>O<sub>3</sub> and RHA are given in Fig. 3. It can be 126 seen from these figures that nano-Al<sub>2</sub>O<sub>3</sub> particles are spherical while the RHA 127 particles are of irregular shape. In order to achieve the desired fluidity and better 128 dispersion of the nanoparticles, a polycarboxylate type superplasticizer (SP) 129 conforming to ASTM C494 [38] with a density of 1.03 g/cm<sup>3</sup> was utilized. The 130 content of superplasticizer was adjusted for each mixture to keep the slump flow of 131 mortars  $25\pm1$  cm. 132

Polypropylene fibers (PPF) produced from recycled raw materials was chosen133because of its high resistance to corrosion and chemical leaching, its resilience134against impact and freezing, and its environmental benefits. A photograph of the135fibres is shown in Fig. 4 and the properties of the fibres are given in Table 2. The136fibres with a length of 6 mm and a diameter of 20 micron making an aspect ratio of137300 was utilized at a dosage of 0.3% by volume.138

## 2.2 Mix proportions

Twenty six mixtures were prepared with different amounts of RHA, NA, PPF and 140 SP. The percentage of RHA was varied between 0 and 30% by weight of the total 141 binder. The percentage of nanoparticles was 0, 1, 2 and 3%, and PPF was used in 0 142 and 0.3% of the binder. The amount of SP varied between 0.2 and 1% by weight of 143 the binder. The water to binder ratio (w/b) was kept constant at 0.49 for all mixtures. 144 Detailed mix proportions of the mortars are given in Table 3. In labeling of the 145 mixtures, the number after RHA, NA and PPF represents the percentage of rice husk 146 ash, Nano-Alumina and Polypropylene fibres respectively. 147

# 2.3 Production of specimens

Nanoparticles may not always show a uniform distribution in the mixture due to 149 their large surface area [39]. As this could directly affect the physical and 150

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mechanical properties of the mortars, the specimen production procedure used in this 151 study was carried out in accordance with the ASTMC305 standard [40]. However, 152 some change in the mixing procedure was necessary due to presence of 153 nanoparticles. Masks and gloves were used to avoid direct contact with the fine 154 particles during the mixing. First, the cement and RHA were dry mixed in the mixer 155 at a moderate speed (80rpm) for 1 min. Then, these components were mixed at a 156 high speed (120rpm) for 90 seconds with the nanoparticles, 90% of the water and the 157 specified amount of fibers. The sand was then gradually added over a period of 30 158 seconds while the mixer was running at a moderate speed (80rpm). Eventually the 159 superplastisizer and remaining water were added and stirred at high speed (120rpm) 160 for 30 seconds. After this, the mixture was allowed to rest for 90 seconds and then 161 mixing was continued for 1 minute at a high speed (120rpm). This mixing procedure 162 was followed to facilitate the distribution of the nanoparticles and the fibers in the 163 mortar. 164

Fresh mortar was cast into  $50 \times 50 \times 50$  mm cubes for compressive strength and water165absorption tests and in  $50 \times 50 \times 200$  mm steel moulds for flexural and shrinkage tests.166The specimens were compacted using a tamping rod to exclude the air bubbles from167the mortar. The specimens were demolded 24 hours after casting and cured in water168at  $23 \pm 3$  °C until they were tested.169

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## 2.3 Test procedure

Compressive strength test was conducted in accordance with the ASTM-C109171standard [41] using a hydraulic testing machine at a loading rate of 1350N/s. The172three-point (i.e. center-point) loading flexural test was carried out with the span of173180mm and at a loading rate of 44N/s. The compressive and flexural strength results174were determined at 28 and 90 days of curing. The average value of the test results of175three specimens was reported.176

The water absorption test was carried out at 28 days of age. Saturated surface dry 177 specimens were kept in an oven at 110°C for 72 hours. After determination of the 178 initial weight, the specimens were immersed in water for 72 hours. The final weight 179 was then determined and the absorption was calculated to assess the permeability of 180 the mortar specimens. The absorption value was obtained by taking an average of 181 the test results of two specimens.

To determine the drying shrinkage,  $(50 \times 50 \times 200)$  mm prisms were tested in 183 accordance with ASTM C157-89 [42]. Two gauge studs were inserted at the two 184 ends (along the center axis) of each specimen immediately after casting of the 185 specimens. The specimens were demoulded 24 hours later and placed in water for 7 186 days. After this the specimens were removed from water and the initial dial gauge 187 readings were taken immediately. Then the specimens were stored in dry room of 188 the laboratory with temperature of  $23\pm 2^{\circ}$ C and a relative humidity of  $50\pm 4\%$ . The 189

length change tests were taken at ages (7, 28, 60 and 90) days. The length change of190specimens was measured by means of a length comparator conforming to the191requirement of ASTM C490-00a [43]. The specimen was rotated slowly in the192measuring device, while the measurement of length was being made. The accuracy193of the dial gauge of the measuring device was 0.002 mm. The average value of four194readings from two prisms was adopted for each mix.195

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# 3. Results and discussion

## 3.1. Compressive strength

The compressive strength values of mortar specimens are given in Table 4 and the 199 variations of strength with age are plotted in Fig. 5. The results show a slight 200 increase in compressive strength by the addition of RHA up to 10wt% and a 201 reduction in strength with further increase in the RHA content. The improvement in 202 the strength of mortars incorporating RHA, especially at later ages is due to its 203 pozzolanic action. The calcium silicate hydrate (CSH) gel produced by the reaction 204 of the high silica content of RHA with the Ca(OH)<sub>2</sub> generated from the hydration of 205 cement contributes to the continued increase of strength at the later ages. Similar 206 effect of RHA was also observed in previous works [12]. Giaccio et al. [44] showed 207 in the experiments that incorporating RHA as 10wt% of the binder improved the 208 compressive strength of concrete samples. Gastaldini et al. [45] also reported 209 enhancement in compressive strength, when RHA was used as partial replacement of 210 cement. However, excessive amounts of RHA could decrease the compressive 211 strength. Figs. 6-a, 6-b and 6-c show the SEM images of control specimen, a 212 specimen with 10% RHA, and a specimen with 10% RHA and 3% NA, respectively. 213 The solid phase of mortar is composed of 5 parts: C-S-H, C-H, ettringite, 214 monosulfate and residual unhydrated cement. These 5 parts are different in their 215 volume fraction, density, dimensions, morphology and Christianity. 216 So characterization is usually done by SEM micrograph, especially for understanding 217 the morphology and dimensions of solid parts. Unreacted materials are materials that 218 do not resemble to any products of the hydration. The black spaces in SEM photos 219 are the capillary pores which are decreased, when nano- alumina is added. As a 220 definition, capillary pore is the space which is not taken up by the cement and 221 hydration products (dependent on w/c); 2.5-50nm in size in well-hydrated concrete. 222 These pores are irregular in shape, also size and amount are related to w/c and 223 degree of hydration. Micropores with diameter less than 50 nm are more crucial for 224 drying shrinkage and creep and macropores with diameter more than 50 nm are 225 more significant for strength. As it is seen in Fig. 6-b, the amount of pores is reduced 226 by the addition of RHA in comparison with that of the specimen without RHA as 227 shown in Fig. 6-a. However, couple of unreacted particles can be clearly seen in Fig. 228 6-b. 229

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Using nanoparticles fostered the compressive strength of specimens substantially. 230 The effect of nanoparticles in mortars can be summarized in the following 3 steps: 231

1. Nano-Alumina works as fillers to fill the pores, so the compactness of samples is232enhanced [46]. As it can be seen in SEM images of the specimen with RHA and NA233in Fig. 6-c, there are fewer and smaller pores and the microstructure is more234compacted when NA is added. This is due to the fact that nanoparticles are expected235to affect the kinetics and hydration of cement substantially and yield greater filling236of voids of cement-based composites in comparison with the mineral additives in237virtue of their larger surface area and greater electrostatic force.238

2. Hydration of cement is a sum of chemical reactions between cement and water. A
third type of material may affect the hydration process. High specific surface of
nano-alumina particles accelerates the hydration process by rapid dissolution of
cementitious compounds. This led to the formation of clusters of calcium
aluminosilicate (C-A-S-H) gel.

3. Nanoparticles make the cement matrix homogenous and makes the structure
244 compacted. As it is shown in Fig. 6-c, there are less unhydrated cement and more
245 homogenous cement matrix in comparison with the control sample.
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Table 4 also shows that when the amount of RHA is 30%, using NA more than 2 % 247 has an inverse effect and decreases the compressive strength. When a mixture 248 contains 30% RHA, for the rest of 70% cement, homogeneous hydrated 249 microstructure cannot be formed, because the nanoparticles cannot be well 250 dispersed. By addition of excessive amount of nanoparticles, they replace part of the 251 cementitious materials, nonetheless this does not affect the strength and the excess 252 silica will leach out and cause a deficiency in strength. Dispersion of nanoparticles is 253 the main reason of the formation of homogeneous structure that is an important 254 factor in compressive strength of a specimen. So despite the best result was achieved 255 when 3% NA was used, using 2% NA gives the best average mechanical and 256 economical results. 257

As expected, the results also show that addition of PPF did not have a significant 258 effect on the compressive strength of samples. Also in some specimens like 259 RHA20NA3 addition of PP decreases the compressive strength slightly. According 260 to the Table 5, RHA10NA3PP0.3 showed the best result, as the increase percentage 261 was 18.2 and 20.1 at 28 and 90 days respectively. 262

Also the outstanding impact of age on improvement of compressive strength can be263seen in Table 4. In all samples, percentage of decrease was lowered or the264percentage of increase was heightened, when the curing time increased from 28 days265to 90 days. In the mixtures with RHA, the amount of cement is lower than in the266control sample. So at the early ages, the structure was less compact and the volume267

of pores increased as the amount of RHA increased. As the age of curing increased,268the pozzolanic reactions increased that caused a higher density of the product.269Presence of nanoparticles was the other way of filling the pores when RHA is270incorporated. The similar comparison was also reported by Chao-Lung et al. [47].271The results are similar between early age and 28 days of curing. As a result, the272process of improving compressive strength and the pozzolanic reactions are started273at early ages and continued at least up to the age of 90 days.274

#### 3.2. Flexural strength

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The flexural strength results are presented in Table 5, while the variation of flexural 276 strength with time is shown in Fig. 7. The results indicate the outstanding effect of 277 adding PPF in the improvement of flexural strength. It can be seen that the mixes 278 with PPF and RHA showed higher values in comparison with that of the control 279 sample. Also, flexural strength of specimens incorporating nanoparticles in most 280 cases was higher than that of the control sample. However, these particles strengthen 281 the weak region that is between cement paste and RHA. The SEM images of 282 specimens incorporating RHA, NA and PPF, which are given in Figs. 9-a and 9-b, 283 confirm this statement. The nanoparticles fill the pores especially porous portlandite 284 crystals which array in the interfacial transition zone (ITZ) between cement matrix 285 and RHA. 286

It can be seen in Table 4 that the addition of 30wt% RHA lowered the flexural287strength up to about 14%. Instead the addition of 0.3% PPF boosted flexural strength288up to 14%, which was equal to the value for the control sample. By comparison of289Fig. 6-b and Fig. 8-a, it is evident that using PPF can balance out the negative effects290of RHA incorporation. As it is shown, PPF in mortars look like very small bars that291can control the crack propagation.292

The results also show that when the amount of RHA is 30%, adding nanoparticles 293 more than 2% lowers the flexural strength by about 1.5%. This result was also 294 obtained in compressive strength test. The effect of aging on improvement of 295 flexural strength is also evident. In all samples the percentage decrease was 296 improved when specimens were tested after 90 days of curing. 297

It is verified that specimens that were not reinforced with PPF exhibited brittle 298 failure. These failures started with micro cracks in the cement-aggregate interface 299 that then propagated as the load increased. The PPF reinforced specimens, however, 300 exhibited ductile failure. The initial cracks started in the cement matrix, but they did 301 not propagate as fast as in the case of plain mortar specimens. This is due to the 302 reinforcing ability of PPF to limit cracking from its unique high strength bond with 303 the cementitious materials. Microscopic analysis of mortar fracture surface (Fig. 9a 304 and 9b) shows that micro cracks exist at the aggregate-matrix interface of the plain 305 mortar specimen even before any load has been applied to the mortar. The formation 306 of such cracks is due primarily to the strain and stress concentrations resulting from 307 the incompatibility of the elastic moduli of the aggregate and paste components. On 308 the other hand, the strong bond between the fibers and cement paste in the PPF 309 reinforced mortar specimen reduces such cracks (Fig. 9c). The composite system of 310 mortar reinforced with PPF is assumed to work as if it were unreinforced until it 311 reaches its "first crack strength." It is from this point that the reinforcing fibers take 312 over and hold the mortar together. 313

Typical flexural load displacement response of different mixtures containing 0.3% 314 PPF and 3% nano-Al<sub>2</sub>O<sub>3</sub> at 90 days are represented in Fig. 10. The test was 315 controlled automatically by computer with a constant cross head movement of 316 1mm/min. As it is evident, unreinforced mortar demonstrated brittle behavior. The 317 samples fully fractured with the increase of mid span displacement after peak load, 318 while fiber reinforced mortar exhibited ductile behavior. Study of the load-319 displacement curve showed that mortar containing nano-Al<sub>2</sub>O<sub>3</sub> was obviously more 320 brittle than that of control mortar, however, integrating PP fibers somewhat 321 compensated this shortage. A relatively big increase was observed when increasing 322 the PPF content to 0.3%. When cracks occurred and propagated, the fibers were able 323 to bridge across the cracks, preventing the crack-face separation. The fibers 324 sustained the load until they pulled out from the matrix. This mechanism provides an 325 additional energy absorption which led to a stable fracture process and higher 326 fracture energy. The presence of nano-Al<sub>2</sub>O<sub>3</sub> enhanced the efficiency of the transfer 327 of load from matrix to fiber by increasing the friction coefficient between the fiber 328 and the matrix. Hence the sample containing PP fibers and nano-Al<sub>2</sub>O<sub>3</sub> showed a 329 higher peak-load with long post-peak curve as compared to the other samples, as 330 shown in Fig. 10. When enough PPF is distributed in the matrix to bridge any 331 growing micro crack, the additional energy is consumed in breaking or pulling out 332 the fibers, hence, leading to higher failure load and toughness to the material. 333

#### 3.3 Water absorption test

Water absorption of concrete is a measure of the capillary forces exerted by the pore335structure that causes fluids to be drawn into the body of the materials [48].336

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The experimentally obtained results are presented in Fig. 11. Each of them is the337mean value of three samples tested at 28 days of curing. In general, the results338showed small reductions in the absorption values when RHA was added.339

Using fine particles such as RHA leads to the segmentation of large pores and 340 fosters nucleation sites for precipitation of hydration products in cement paste. 341 However, the replacement of cement by RHA up to 10% also reduced the water 342 absorption of specimens. Addition of RHA more than 10% leads to an increment of 343 water absorption. The increase in water absorption in samples with a relatively high 344 content of pozzolans is associated with the decrease of ordinary Portland cement, 345

which reduces the hydration products in specimens. As shown in Fig. 6, it was 346 observed that the incorporation of RHA in mortars could lead to extensive pore 347 refinement in both the matrix and the interfacial zones. The resulting pore 348 refinement can effectively decrease the water permeability. 349

However increase in the amount of nano-Alumina and also PPF further reduced the350water absorption. Due to very high specific surface area, the nanoparticles contained351some free water on the surface, which resulted in continuous hydration that made the352matrix more compacted. This seems like that external curing time for mortar353samples. The percentage of water absorption is related to the porosity of the354hardened mortar which is engaged by water in a saturated state [5].355

#### 3.4 Drying shrinkage

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Fig. 12 shows the variations of drying shrinkage over time for mortars made with 357 different percentages of RHA and PPF. The drying shrinkage of mortars is highly 358 related to the stiffness of aggregates and their porosity. As seen from the figure, both 359 RHA and PPF reduced the drying shrinkage of the mortar mixtures. All the 360 specimens started to shrink gradually after removal from water. This is in virtue of 361 the modification of pore size distribution and the interfaces beside the rapid loss of 362 moisture from the surface of the specimens having high cement content. The results 363 show that drying shrinkage values increased with time and the rate decreased after 364 about 10 weeks of age. The drying shrinkage of the mortars with RHA after 90 days 365 ranged from 801 to 843 microstrains. Mortars made with RHA exhibited lower 366 drying shrinkage after 90 days compared with that of the control specimen. Drying 367 shrinkage (after 90 days) decreased by 5% to 12% when RHA increased from 10% 368 to 30%. The variation in shrinkage with water content may be introduced by the 369 difference in types of water lost at a wide range of stages of drying. It is also 370 associated with the modulus of elasticity of the sample. Mortar with high water 371 content has a lower strength and lower modulus of elasticity and hence has a greater 372 tendency to shrinkage. Due to the higher surface area of RHA in comparison with 373 cement, such fine particles (RHA) can absorb more water, which leads to reduction 374 of water content and consequently the drying shrinkage is reduced. On the other 375 hand, incorporating PPF also reduced the drying shrinkage of mortars. At 90 days, 376 the drying shrinkage values were 729, 680, 651 and 622 microstrains for mixtures 377 PP0.3, RHA10PP0.3, RHA20PP0.3 and RHA30PP0.3, respectively. Malhotra et al. 378 [49] concluded that polypropylene fiber reinforced high volume fly ash concrete has 379 very low drying shrinkage property. Liu et al. [50] and Salih and Al-Azaawee [51] 380 stated that polypropylene fiber mixed into cement mortar decreased its dry-381 shrinkage. They concluded that the reduction in drying shrinkage increased with the 382 increasing volume fraction of fibers. Kirca and Sahin [52] supported this finding and 383 reported that the use of polypropylene fibres restrained the movements in micro 384 level by bridging and stitching the fine cracks. This is attributed to the ability of 385

polypropylene fibers to minimize the density of cracks, crack length and width, 386 which can limit the shrinkage. 387

## 3.5 X-ray Diffraction

Fig. 13 illustrates the XRD analysis of mortar with and without Al<sub>2</sub>O<sub>3</sub> nanoparticles 389 at 7 days of curing. Ettringite, portlandite, Alite and Belite were found to be major 390 phases for the specimens. Changes in peak height and formation of new peaks were 391 found at 7 days. Intensity of Alite and Belite phase decreased and a new peak of 392 portlandite (2 theta of 16, 28 deg.) were found. However, no other new crystalline 393 phase was found with nano-Al<sub>2</sub>O<sub>3</sub> addition. The results indicate that, Ca(OH)<sub>2</sub> 394 crystals (portlandite) which needs for formation of C-S-H gel appears in mortars 395 containing nanoparticles, while for sample without nanoparticles, it is not appeared 396 demonstrating synergic influence of nanoparticles on formation of subsequent C-S-397 H gel. 398

# Conclusion

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The following conclusions are drawn from the current study on mortars with 400 incorporation of RHA, nano-Al<sub>2</sub>O<sub>3</sub> and polypropylene fibers: 401

- The addition of RHA increased the 28-day compressive strength of samples up to 1% from their original level when cement was replaced by 10% RHA. Nevertheless addition of 20% and 30% RHA decreased compressive strength by up to 11% and 14%, respectively. However, this rate decreased at the age of 90 days.
- The addition of RHA reduced the 28-day flexural strength of samples up to 2%, 406 11%, and 14% from their original level when cement was replaced by 10%, 20% and 407 30% RHA. However, higher strength development was observed at 90 days when 408 RHA content was increased from 10% to 30%. Incorporation of nano-Al<sub>2</sub>O<sub>3</sub> 409 enhanced the compressive and flexural strengths of samples containing 10% and 410 20% RHA. However, strength of the specimens containing 30% RHA enhanced 411 when cement was replaced by 2% nano-Al<sub>2</sub>O<sub>3</sub>. The most advantageously effective 412 amount of nano-Al<sub>2</sub>O<sub>3</sub> was 3% by weight of the binder. For instance, the 413 compressive strength increased up to 18% and 20% when 3% nano-Al<sub>2</sub>O<sub>3</sub> was added 414 to samples with 20% RHA at 28 and 90 days, respectively. Also, the 28-day and 90-415 day flexural strengths were enriched by up to 34% and 41% respectively, when 3% 416 nano-Al<sub>2</sub>O<sub>3</sub> was added to the samples with 10% RHA. Therefore, incorporation of 417 nano-Al2O3 compensated the strength loss caused by 10% and 20% cement 418 replacement by RHA. Furthermore, the strength development from 28 to 90 days 419 was more in samples containing RHA at all levels. 420
- Water absorption of the specimens slightly decreased by the addition of 10% RHA and the values were same as that of the control sample with further increase of RHA up to 30%. Water absorption further decreased with the increase of nano-Al<sub>2</sub>O<sub>3</sub> 423 dosage up to 3%.

Incorporation of polypropylene fiber enhanced the flexural strength of samples remarkably. The 28-day and 90-day flexural strengths increased by 18.6% and 23.1% respectively when 0.3% PPF was added to mixtures containing 10% RHA and 1% NA.

- SEM images confirmed the formation of denser microstructure with nano-Al<sub>2</sub>O<sub>3</sub> 429 addition. The densification of the microstructure is attributed to the formation of 430 additional CSH by the RHA and CASH by the Al<sub>2</sub>O<sub>3</sub>. The compact microstructure 431 improved bonding of the fibres with the matrix, which improved the flexural 432 strength of the mortar specimens. 433
- Presence of RHA and PPF in mortars, both separately and together reduced drying 434 shrinkage. The positive interaction between polypropylene fibers and rice husk ash resulted in the lowest drying shrinkage of the fibrous mortars. 436

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	Constituents (wt.%)	Cement	RHA
Chemical composition	SiO <sub>2</sub>	21.75	91.15
	$Al_2O_3$	5.15	0.41
	$Fe_2O_3$	3.23	0.21
	CaO	63.75	0.41
	MgO	1.15	0.45
	SO <sub>3</sub>	1.95	0.62
	K <sub>2</sub> O	0.56	6.25
	Na <sub>2</sub> O	0.33	0.05
	L.O.I	2.08	0.45
Physical properties	Surface area $(cm^2/g)$	3105	4091
	Specific gravity (g/cm <sup>3</sup> )	3.15	2.07

Table 1: Chemical composition and physical properties of cement and RHA

Table 2: Properties of polypropylene fiber

0.0.0.01
0.9-0.91
Hydrophobic
300-400
100-600
175
0.12
6
20

Table 3: Mix details of mortars

Sample ID	Cement (kg/m <sup>3</sup> )	RHA (kg/m <sup>3</sup> )	NA (kg/m <sup>3</sup> )	PPF (kg/m <sup>3</sup> )	Water (kg/m <sup>3</sup> )	Sand (kg/m <sup>3</sup> )	SP (kg/m <sup>3</sup> )
СО	450	0	0	0	220	1430	0.9
RHA10	405	45	0	0	220	1415	1.8
RHA10NA1	400.5	45	4.5	0	220	1410	1.8
RHA10NA2	396	45	9	0	220	1400	1.8
RHA10NA3	391.5	45	13.5	0	220	1395	1.8
RHA20	360	90	0	0	220	1400	2.25
RHA20NA1	355.5	90	4.5	0	220	1390	2.25
RHA20NA2	351	90	9	0	220	1385	2.25
RHA20NA3	346.5	90	13.5	0	220	1380	2.25
RHA30	315	135	0	0	220	1380	3.25
RHA30NA1	310.5	135	4.5	0	220	1375	3.25
RHA30NA2	306	135	9	0	220	1370	3.25
RHA30NA3	301.5	135	13.5	0	220	1360	3.25
PP0.3	450	0	0	2.7	220	1430	1.5
			_				
RHA10PP0.3	405	45	0	2.7	220	1415	2.25
RHA10NA1PP0.3	400.5	45	4.5	2.7	220	1410	2.25
RHA10NA2PP0.3	396	45	9	2.7	220	1400	2.25
RHA10NA3PP0.3	391.5	45	13.5	2.7	220	1395	2.25
RHA20PP0.3	360	90	0	2.7	220	1400	3.25
RHA20NA1PP0.3	355.5	90	4.5	2.7	220	1390	3.25
RHA20NA2PP0.3	351	90	9	2.7	220	1385	3.25
RHA20NA3PP0.3	346.5	90	13.5	2.7	220	1380	3.25
		105	0			1000	
RHA30PP0.3	315	135	0	2.7	220	1380	4.5
RHA30NA1PP0.3	310.5	135	4.5	2.7	220	1375	4.5
RHA30NA2PP0.3	306	135	9	2.7	220	1370	4.5
RHA30NA3PP0.3	301.5	135	13.5	2.7	220	1360	4.5

	Compressive strength (MPa)					
Sample ID	28 days	% of increase or decrease	90 days	% of increase or decrease		
СО	45.1		50.2			
RHA10	45.3	0.44	50.3	0.2		
RHA10NA1	45.6	1.11	51.1	1.8		
RHA10NA2	50.3	11.5	57	13.5		
RHA10NA3	52.6	16.6	59.6	18.7		
RHA20	40.1	-11.1	45.7	-9.0		
RHA20NA1	41.6	-7.7	47.3	-5.8		
RHA20NA2	43.2	-4.2	49.1	-2.2		
RHA20NA3	46.7	3.5	53.1	5.8		
RHA30	38.7	-14.2	44.1	-12.1		
RHA30NA1	40.1	-11.1	45.7	-9.0		
RHA30NA2	40.3	-10.6	45.9	-8.6		
RHA30NA3	39.6	-12.2	45.3	-9.8		
PP0.3	46.9	4.0	53.4	6.4		
RHA10PP0.3	45.4	0.6	51.9	3.4		
RHA10NA1PP0.3	47.2	4.7	53.4	6.4		
RHA10NA2PP0.3	51.6	14.4	58.4	16.3		
RHA10NA3PP0.3	53.3	18.2	60.3	20.1		
RHA20PP0.3	41.6	-7.8	47.3	-5.8		
RHA20NA1PP0.3	41.9	-7.1	47.5	-5.4		
RHA20NA2PP0.3	42.9	-4.9	48.9	-2.6		
RHA20NA3PP0.3	45.9	1.8	52.1	3.8		
RHA30PP0.3	39.5	-12.4	45	-10.3		
RHA30NA1PP0.3	40.2	-10.9	45.7	-9.0		
RHA30NA2PP0.3	41.6	-7.8	47.3	-5.8		
RHA30NA3PP0.3	40.9	-9.3	46.3	-7.8		

 Table 4: Compressive strength of mortars

Flexural Strength (MPa)					
Sample ID	28 days	% of increase or decrease	90 days	% of increase or decrease	
СО	6.76		7.78		
RHA10	6.64	-1.8	7.8	0.3	
RHA10NA1	6.75	-0.1	7.92	1.8	
RHA10NA2	7.55	11.6	8.84	13.6	
RHA10NA3	7.89	16.7	9.24	18.8	
RHA20	6.02	-10.9	7.08	-9.0	
RHA20NA1	6.24	-7.7	7.33	-5.8	
RHA20NA2	6.48	-4.1	7.61	-2.2	
RHA20NA3	7.01	3.6	8.23	5.9	
RHA30	5.81	-14.1	6.84	-12.1	
RHA30NA1	6.02	-10.9	7.08	-9.0	
RHA30NA2	6.05	-10.5	7.11	-8.6	
RHA30NA3	5.94	-12.1	7.02	-9.8	
PP0.3	7.97	17.9	9.72	24.9	
RHA10PP0.3	7.72	14.2	9.44	21.3	
RHA10NA1PP0.3	8.02	18.7	9.72	24.9	
RHA10NA2PP0.3	8.77	29.7	10.63	36.6	
RHA10NA3PP0.3	9.06	34.0	10.97	41	
RHA20PP0.3	7.07	4.6	8.61	10.7	
RHA20NA1PP0.3	7.12	5.3	8.65	11.2	
RHA20NA2PP0.3	7.29	7.8	8.90	14.4	
RHA20NA3PP0.3	7.80	15.4	9.48	21.8	
RHA30PP0.3	6.72	-0.5	8.19	5.3	
RHA30NA1PP0.3	6.83	1.0	8.32	6.9	
RHA30NA2PP0.3	7.07	4.6	8.61	10.7	
RHA30NA3PP0.3	6.95	2.8	8.43	8.4	

Table 5: Flexural strength of mortars



Fig. 1. Particle size distribution of RHA





Fig. 2. XRD diagram of nano-Al<sub>2</sub>O<sub>3</sub> (top) and RHA (bottom)





Fig. 3. SEM micrograph of the a) nano-Al<sub>2</sub>O<sub>3</sub> and b) RHA



Fig. 4. Polypropylene fibers



Fig. 5. Compressive strength values at 28 and 90 days of curing





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Fig. 6. SEM images of a) Control b) RHA10 and c) RHA10NA3 mixtures at 28 days of curing	652 653
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	657



Fig. 7. Flexural strength values at 28 and 90 days of curing

a) UD27 28.0kV K580 60.04m 660

661



Fig. 8. SEM images of a) RHA10PPF0.3 and b) RHA10NA3PPF0.3 mixtures at 90 days of curing

a) 


- Fig. 9. SEM Micrograph of mortar fracture surface. (a) Control sample @ 15000 Res. b) Control sample @ 3000 Res. (c) Containing PPF @ 1000 Res at 90 days of curing



Fig. 10. Load-displacement curve for mortars reinforced with PPF at 90 days of curing.



Fig. 11. Water absorption values at 28 days of curing





Fig. 12. Effect of RHA and polypropylene fibers on the drying shrinkage of mortars at various ages 



Fig. 13. XRD analysis of mortars a) without NA and b) with NA at 7 days of curing