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Research paper

Effect of Thermally Activated Alum Sludge Ash and Nanoclay on the Mechanical Properties and Microstructure of Kenaf Fiber Reinforced Mortars

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Abstract

This paper investigates the effect of thermally activated alum sludge ash (AASA) and nanoclay (NC) inclusion at various proportions on the mechanical properties and microstructure of kenaf fiber (KF) blended cementitious composites (KFRBCC). The first objective is to establish a blended cementitious (BC) composite with high mechanical strength. The BC materials used in this investigation comprises of various proportions of ordinary Portland cement (OPC), AASA and NC. Next, the influence of AASA and NC proportions on the properties of KFRBCC such as density and compressive, split tensile and flexural strengths, and microstructure are studied. The results show that the inclusion of AASA and NC improve the mechanical properties of the cementitious composite compared to all OPC mortar with or without KF. The enhancement in compressive strength is 50%, whereas the enhancement in split tensile and flexural strengths are 5.4% and 15%, respectively at optimum content of AASA and NC of 50 wt. % and 1 wt. %, respectively. The results also reveal that the microstructure of the composites is denser due to filler effect where the uniformly dispersed AASA and NC nano-particles had filled the voids/pores in the matrix compared to control specimens. AASA is proved to be benificial as one of cementitious replacement materials and its applicability as cement replacement in concrete should be explored further.

Keywords: *cementitious composite; kenaf fiber; microstructure; mechanical strength; thermally activated alum sludge ash.*

1. Introduction

In recent years, concrete technology development has revealed that adding pozzolanic materials have potential in enhancing the strength and durability of concrete, and they are important for production of high performance concrete (HPC) [1]. There has been a growing interest in the use of natural and artificial pozzolanic materials in concrete due to economic benefits (partial replacement of ordinary Portland cement (OPC) with natural pozzolans or cheaper industrial by-products), environmental protection (for lowering greenhouse gases as that emitted during OPC production), and durability enhancement of the concrete. According to Ghrici et al. [2], the methods for reducing energy consumption and carbon dioxide emissions (CO_2), are by adding natural pozzolans with calcined processing method and grinding into specific fineness and/or adding artificial pozzolans. Some examples of natural and artificial pozzolans are nanoclay (NC), metakaolin (MK), and kaolin, while the latter are silica fume (SF), fly ash (FA), and ground granulated blast slag furnace (GGBS). Studies by several researchers [3]–[6] have shown that natural and artificial pozzolans have been widely used as substitutes for OPC in many applications. Partial replacement of OPC or modification of the matrix with pozzolanic materials had affected the properties of concrete such as increase in strength [7], [8] and durability of the composites [9], [10].

In addition to the above-mentioned pozzolanic materials, various studies have been conducted to investigate the possibility of recycling sludge, an industrial by-product from water treatment plant, known as alum sludge (AS) ash, to be used in cement composites [11]. The AS ash is classified as a Class N (natural) pozzolan while thermally activated alum sludge ash (AASA) is similar to thermally activated kaolin after calcination at 800 °C [12]. These findings are also supported by Tantawy [13], who reported that calcination at 800 °C is adequate to improve the pozzolanic properties of AS. The compressive strength of the cement composite improves with the presence of AASA as partial replacement of OPC, but the optimum replacement level of OPC by AASA to accelerate maximum long term strength enhancement and durability has been reported between 10 to 30% [14]–[16] with the replacement levels of AASA are in percentage by weight of the total binder materials, which is quite small replacement percentage.

Several types of nano materials have been incorporated into concretes and the most popular is nanosilica, carbon nanotubes, nano-metakaolin and nanoclay (NC) in order to improve the mechanical and durability properties of cementitious matrix. However, nanomaterials are added in the concrete or other cementitious composites with very small quantities [17] because of their high reactivity and resulting in

a very low cement replacement with cost effectiveness. Although the use of nanomaterial was first considered as filler to partially replace cement, some studies have shown advantages of using 1% nanoparticles in terms of compressive strength and economic benefits as compared to cement and other supplementary cementitious materials [18]. Nano materials, such as nanosilica also compensate the strength loss of concrete when used with very high volume of pozzolanic materials such as fly ash and the concrete with high volume of fly ash replacement when combined with nano silica even has a very high resistance against high temperature [19].
In this study, NC is chosen as the nano material. Application of AASA and NC are anticipated to produce composites with high compressive strength than all OPC mortar. The inclusion of fibers will help to improve the mechanical strength, especially the split tensile and flexural and for crack bridging in impact resisting application. The advantages of using natural fibers in composites include low density, flexibility and the high modulus [9, 10] as well as biodegradable, renewable and recyclable [11]. Apart from that, kenaf fibre (KF) was chosen as reinforcement with the fact that KF can be used to reinforce composite materials in improving the tensile/flexural strength and fracture resistance properties [20]. As well as biodegradable, KF are cheaper and lighter than synthetic fibers. However, KF needs to be treated first to reduce/eliminate the hydrophilic nature. Treated fibers makes it possible in obtaining high level of ductility. In this paper, the use of AASA and NC in treated KFRBCC are studied to determine the effect of their inclusion in mortar to improve the mechanical strength treatment of KF to overcome the hydrophilic nature of KF in cementitious composites.

2. Experimental procedures

2.1. Materials

Type I OPC complying with ASTM C150 [21], AASA and NC were used to produce cementitious composites specimens reinforced with randomly oriented short treated KF. Physical properties and chemical compositions of OPC, AASA and NC are provided in Table 1. Raw alum sludge (AS) was oven dried at 105±5 °C for 24 h and later the dried AS was calcined in oven at heating rate of 5 °C/min at 800 °C for 2 h to achieve the thermal steady state [12]. Then, the AASA was allowed to cool gradually to room temperature. AASA was ground in steel ball mill until the fineness reach the specification recommended by ASTM C618-17 [22] and sieved using 45 µm sieve which can increase the pozzolanic reaction. The fine aggregate used was obtained from local river sand passing through 4.75 mm sieve with specific gravity and water absorption of 2.82 and 0.99% accordance to ASTM C128 [23] and ASTM C70-13 [24], respectively.

Table 1: Chemical composition of type 1 OPC, AASA and NC

Chemical composition (%)	OPC	AASA	NC
SiO_2	20.18	47.00	45.87
Al_2O_3	5.23	41.94	32.11
Fe_2O_3	3.34	4.86	4.12
CaO	64.4	0.41	0.34
MgO	1.80	0.40	0.28
Na_2O	0.07	0.09	0.77
K_2O	0.44	0.99	0.81
SO_3	2.98	0.10	2.02
Loss of ignition	2.17	2.64	26.68
Specific gravity	3.12	2.53	2.60
Fineness (m^2/kg)	338	1160	0.25
Median Particle Size	16.9 µm	10.1 µm	25 nm
Pozzolanic Activity Index (7 d)	100	84	85
Pozzolanic Activity Index (28 d)	100	93	96

10 mm length KF were treated with 6% w/w $NaHCO_3$ alkali solution and immersed for 72 h. After the treatment, KF were thoroughly washed with running water to remove absorbed alkali from the fiber surface. The washed KF were allowed to dry at room temperature for 24 h and then oven dried for thermal treatment at 70 °C±5°C for 24 h to release lignin and increase the crystallinity index (CrI) of the KF before added into the mixes. Superplasticizer (SP) was used to control the flow of mortar. 4.5% of low viscosity liquid Darex Super 20 is the maximum dose for mixes but the doses used are varied to comply with the designed flow rate.

2.2. Mix proportion

A total of eleven mixes were prepared by blending binary AASA with OPC and NC with OPC, and a tertiary combination of AASA and NC with OPC. The mixes were cast with 2 wt. % volume fraction of KF by volume of total mix design (constant for all mixes) into standard cube 100 mm^3, cylinder 100 diameter × 200 mm height and prism of 100 ×100×500 mm^3 for physical and compressive, split tensile strength and flexural tests with cement/sand (s/c) ratio of 1:2 and superplasticizer (SP) dosage vary from 3.5%. Optimum water/cement (w/c) ratio was obtained at 0.4 and keep constant for all mixes after three different ratio of 0.3 0.4, and 0.45 were used for trial mix. The amount of AASA (20, 30, 40, and 50 wt. %) and NC (2 to 6 wt. %) were added into mixture for partial replacement of OPC. The mix proportions of the composites are given in Table 2. The composite with 100% OPC and cured in normal curing was used as control specimen.

Table 2: Mortar mix proportion

Index	w/b ratio	Cement (g)	Sand (g)	AASA (g)	NC (g)	Wa-ter (g)	%SP	%Fiber
OPC1	0.30	192	384	0	0	58	0	0
OPCR2	0.40	151	378	0	0	76	3.5	2
AASA20	0.40	161	378	28	0	76	4.5	2
AASA30	0.40	151	378	38	0	76	4.5	2
AASA40	0.40	132	378	57	0	76	4.5	2
NC1	0.40	185	378	0	0.04	76	4.0	2

NC2	0.40	181	378	0	0.08	76	4.5	2
NC3	0.40	178	378	0	0.11	76	4.5	2
ANC1	0.40	142	378	0.40	0.08	76	4.5	2
ANC2	0.40	132	378	0.49	0.08	76	4.5	2
ANC3	0.40	123	378	0.59	0.08	76	4.5	2

2.3. Preparation of specimens

The blended cementitious materials of AASA, NC, and OPC were mixed together for 5 minutes to produce homogenous mixture according to ASTM C305 [25]. The dry materials including sand, cement, and pozzolans (AASA and NC) were poured into the mixer, allowed to rotate for 1 minute for evenly mix and to obtain the effect of fineness grinding of blended cement. Then, water and SP were slowly added to achieve smooth mixes and to prevent the loss of water. KF was slowly added into the mixes and mixed until there were no fiber balls. The mixes were left to spin for 3 minutes to ensure that the SP had reacted effectively. A vibration method was used for compaction of the mortar and to remove the air bubbles. The average time taken for mixes is between 10-15 minutes. Homogeneous dispersion of treated KF in mixes must be achieve according to specifications of ASTM C305 to develop high performance KFRBCC. Reduction of free area within the matrix had increased through uniform distribution of the fibers and enhance the efficiency of the treated KF in the cement matrix.

2.4. Curing

After 24 h of casting, the specimens were demolded and then exposed to wet conditions involving specimen immersed in water outside the laboratory. The cementitious mortar specimens were removed from curing conditions 24 h before testing and left in the laboratory to dry. The average temperature recorded in the laboratory was 27± 2 °C with relative humidity (RH) 85±5%. The maximum curing age was 180 days and it is generally agreed that the compressive strength of HPCs must be tested at 90 days, because most of these concretes contain cementitious materials (SF, slag, FA etc.) which hydrate at a slower rate than OPC [26].

3. Test procedures

3.1. Bulk density

Saturated surface dry specimens from three cube samples of 100 mm^3 were used for each mix to determine the density of cementitious composites following the procedures specified by ASTM C948-81 [27].

3.2. Compressive strength

The average of three samples were tested for compressive strength test following ASTM C109 [28]. The specimen were weighted after removal from the curing tank and placed centrally in a compression testing machine with applied load at a rate of 150 kN/min. The reported compressive strengths were determined at various ages of 28, 56, 90 and 180 days.

3.3. Split tensile and flexural strength

The splitting tensile test was conducted following ASTM C496 [29] for cylindrical specimens, and the flexural test used ASTM C348 [30] with a Universal Testing Machine. For both tests, three specimens were surface-dried and tested at curing ages of 28, 56 90 and 180 days.

3.4. Microstructure

The scanning electron microscope (SEM) was used for identification of the changes occurred in the microstructure of the specimens. The effect of KF and BC materials on the structure of the composites was examined through microscopic analysis using Zeiss Evo MA 10 (UK) VP field emission scanning electron microscope (VPSEM). The samples were covered with a thin layer of gold using a sputter coater and operated at 10 kV using the secondary electron mode with images collected digitally.

4. Result and discussion

4.1. Density

Figure 1 shows the bulk density of all KFRBCC after curing in water and OPC1 (control specimens) shows the highest dry density between 2200 and 2250 kg/m³. While density of OPCR2 with the addition of untreated KF dispersed randomly showed a reduction of 7%. However, the bulk densities for samples with AASA and NC with treated KF shows gradual decrease of approximately 2% as the amount of the materials increase from 20 to 40 wt. % and 2 to 6 wt. % of binder, respectively after 28-day curing period. The density of the mixture containing BC of AASA and NC reinforcing with treated KF is lower than that of the control specimen by approximately 8.4 to 10%, respectively, after 28-day curing. According to Owaid et al. [12], the decrease in bulk density is due to the low specific gravity of pozzolanic materials that reduces the mass per unit volume of the material.

In addition, the particle size of AASA and NS reinforced with treated KF are relatively smaller than OPC1 and that they also contribute to further decreasing the bulk density of the BC materials [31]. From this figure, the bulk density of ANC1, ANC2, and ANS3 decreases in a linear manner. It is clearly evident that due to the wide particle distribution of AASA and NC leading to lower densities, which proves that powdering AASA to fine sizes of 45µm was a right decision. The bulk density of the treated KFRBCC is relatively lower than OPC1 and OPCR2, corresponding to the study reported by Chakraborty et al. [32] in which the bulk density of the cement composite decreased substantially with the addition of fiber. The result is correlated with the current study in which the addition of pozzolans and nano materials, together with randomly oriented short KF, had decreased the density of the composites [33]. The addition of raw and treated KF to the mixes gives a tendency to trap more air during mixing and compaction. According to Poon et al. [34], the air voids are created and form a pore matrix that reduces the density of the composite. This is a common phenomenon for nano particles due to small sizes, and high surface area to volume ratio of nanoparticles (van der Waal's force) [29].

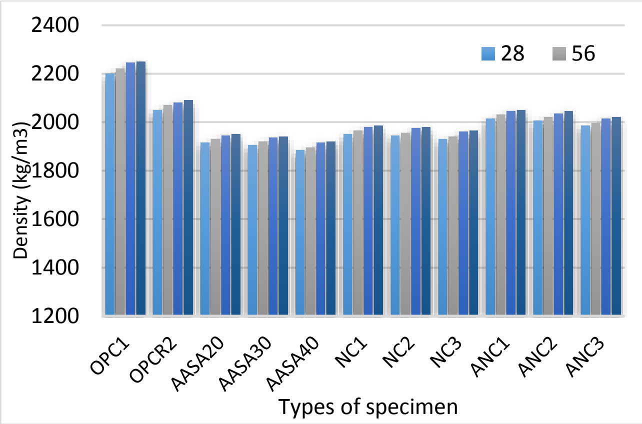

Fig. 1: Bulk density of specimens

4.2. Compressive strength

Figure 3 shows the compressive strength development results of KFRBCC for age of 28, 56, 90 and 180 days. From the figure, when OPC was replaced by 20 wt. % of AASA, compressive strength of specimen is increased up to 30.6% compared to control specimen (OPC1 and OPCR2) for 28-days. The compressive strength of BC composite increased about 34.3 % after 28-days with optimum incorporation of 40 wt. % AASA of OPC. The improvement was related to the effectiveness of AASA particle which provide larger surface contact between fibers and mortar. Thus, generate a friction that accelerate high performance of composites. Compressive strength continued to increase by an average rate of 10% for each specimen up to the age of 180-days and the rate after 28-days are more significant for the strength development. This maybe owing to the higher pozzolanic reaction of AASA at later age compared to early age.
The compressive strength of KFRBCC showed an improvement about 37.6% with addition of NC compared to control specimens. The use of more than 2 wt. % NC reduces the compressive strength that correlate with denser particle packing and crushing of air voids. There is also an evidence suggesting that during hydration process, the overload quantity of NC present does not enhance the compressive strength of the cementitious composite [35]. Furthermore, the effects of pores filling of AASA and NC, the gradation of fine aggregate, and the presence of treated fibers are the secondary possible effects that can contribute to the results which affect the mechanical properties. This result extend the finding from previous study in literatures.

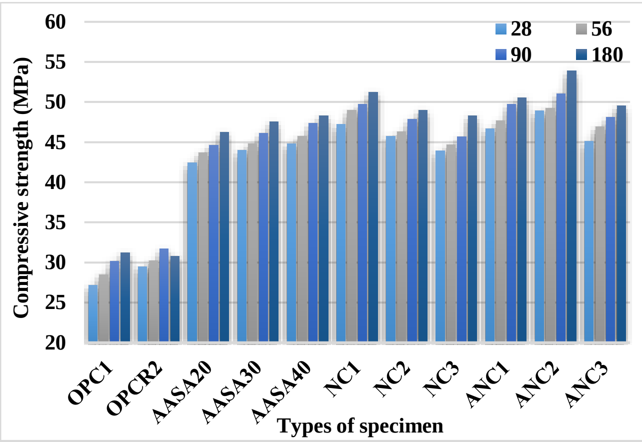

Fig. 2: Compressive strength

The results presented above, suggested that the blended AASA and NC can be effectively used to improve the compressive strengths of cementitious composites. In addition, the 28-days compressive strengths of BC material were found to be higher than mixture containing AASA alone. As for mixture containing NC, similar results was reported and suggesting that NC is more effective in increasing the strengths of KFRBCC. This shows that the AASA and nano material has the effect of filling and pozzolanic properties in the mixes with fiber dispersed randomly.

4.3. Splitting tensile strength

Figure 3 shows the results of the BC composites specimens for splitting tensile strength and the strength for all specimens can be observed between 1.80-3.90 MPa, with BC mixtures of AASA and NC (ANC2) recorded higher tensile strength compared to all specimens. When the AASA replacement for BC composition increased from 50% to 60%, the tensile strength for normal curing period of 28, 56, 90 and 180-days was found to decrease about 3 to 5 %. These findings revealed that the use of more than 50 wt. % replacement of AASA could generate a decrease in tensile strength as also reprted by [36] although 2 wt. % NC was added into the mixture. This could be related to weak zones that had increased the porosity due to poor dispersion and clumping of the AASA in the cement matrix at higher AASA contents. Moreover, mixes using AASA has shown a significant increase in tensile strength with average of 35, 39, 38, and 38.2 % respectively for different curing period but still lower than NC mixes and the BC mixture of AASA with NC. From the results, AASA40 has shown the highest tensile strength. In addition, treated KF added randomly into the mixes is capable in enhancing maximum potential in splitting tensile strength, the increase strength are associated with increasing in pozzolans volume content. This reveals the beneficial effects of alkali and heat treatment of the fibers, which mark better interface bonding with increased fiber strength.

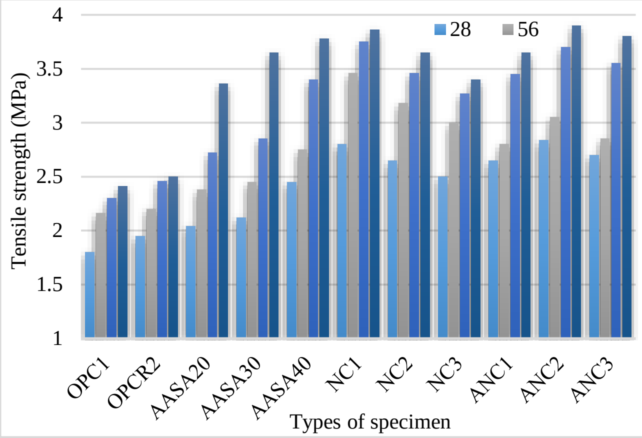

Fig. 3: Tensile strength

Regardless, the tensile strength of the composite had improved with the presence of NC, in which preventing crack extension and strength reduction. These findings was in line with Hakamy et al. [37] which successfully enhance the tensile strength of cementitious composite by adding NC reinforced with sisal fibers into the mixture.

4.4. Flexural strength

Figure 4 shows the flexural strengths for all specimens with strength values between 5.64-7.65 MPa. The flexural strength behavior of the specimen reinforced with treated KF increase with the increase in the total replacement of AASA with OPC. The optimum content of AASA suggested are 40% with flexural strength increased from 6.35 to 7.3 MPa for different curing period, about 15% increase compare to OPC1 and OPCR2. Despite of having pozzolanic effects, AASA and treated KF enhance the mechanical properties and adhesive of the fibers of BC matrix by introducing closely-spaced fibers. It has been suggested by Mader et al. [38] that a combination of matrix and fibers modification has the absolute effect on strength enhancement of the cementitious composite by offering rigidity of fibers through improved crystallinity that will delay tensile cracks and transfer maximum strengths of composite [39]. The increased in the strength for all specimens is associated both in modification of cement matrix and fiber, in which stimulate the interface bonding between fiber-matrix, lowering porosity and to prevent debonding, or pull out fiber and supporting previous study by Papatzani [18].

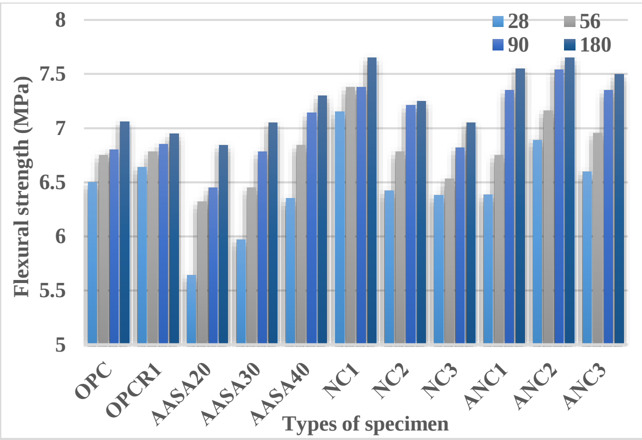

Fig. 4: Flexural strength

The use of 2 wt. % of NC in the mixes resulted in relatively high mechanical properties, but when the content of NC is increased to 4 wt. %, the addition resulted in decreased mechanical properties significantly. The addition of NC with the percentage of over 6 wt. % in mixes is prohibited due to low workability which requires more liquid added into the mixture. However, BC with AASA and NC had shown increasing flexural strength values of 15% compare to AASA40 and control specimens after 180-days curing. The increase in flexural strength is expected due to increase in flexible effective compaction, homogeneity distribution of fibers in the mixes and capacity of fibers to resist and prevent the developing of cracks in composite [40].

4.5. Microstructure

The microstructure of KFRBCC matrix are denser due to micro-pore filling and compaction by pozzolanic activities was enhanced.
Figure 5 shows that the results are supported by SEM analysis of microstructure of BC mortar containing maximum 50 wt. % and 2 wt. % replacement of AASA and NC. Figure 5a and 5b show SEM images of AASA and NC. While in Figure 5c, 40 wt. % AASA demonstrates slightly denser microstructure and point out the effectiveness of pozzolanic reaction and filling effect in the matrix. It also shows good fiber-matrix interfacial bonding as in Figure 5d. Fig 5e show the SEM image of 2 wt. % NC, the microstructure is more compact and denser which provide better fiber-matrix interfacial bonding as in Figure 5f. A small signs of fiber–matrix degradation is spotted but still shown the increased in interfacial zone (Figure 5f) compare to AASA (Figure 5d). Figure 5g shows the SEM image of BC containing 50 wt. % AASA and 2 wt. % NC. The structure is more compact and denser when compared to the matrix of AASA and NC. While in Figure 5h show a SEM images of the fracture surface of BC AASA and NC after compression test.

In addition, the microstructure of KFRBCC of 50 wt. % and 2 wt. % replacement of AASA and NC are consistent with increments of 42%, 38%, and 8% in compressive, tensile and flexural strength respectively after 180-days curing. The replacement of OPC with BC materials enhanced the strength and microstructure of BC mortar through the effect of uniformly dispersed of a small quantity of the nano-particles in the BC mortar which accelerated the hydrate products of cement [41]. Later, the nano particles had further promoted and accelerated cement hydration due to their high activity and provide good microstructure with the uniformly distributed conglomeration.

However, KFRBCC suffered higher porosity due to decrease in density if more than 2 wt. % NC were added into the mixtures. This is related to poor dispersion and agglomerations of the NC which create more voids in the matrix. Other than that, the effectiveness of AASA and NC provide filling effect in the matrix has led to denser microstructure along with the modification of KF by $NaHCO_3$ solution. Furthermore, 50 wt. % and 2 wt. % replacement of AASA and NC had significantly prevented the treated KF from degradation in cementitious matrix. Optimum replacement of 50 wt. % AASA reduce the density, enhanced the mechanical strength and significantly prevented the treated KF from the degradation in cement matrix.

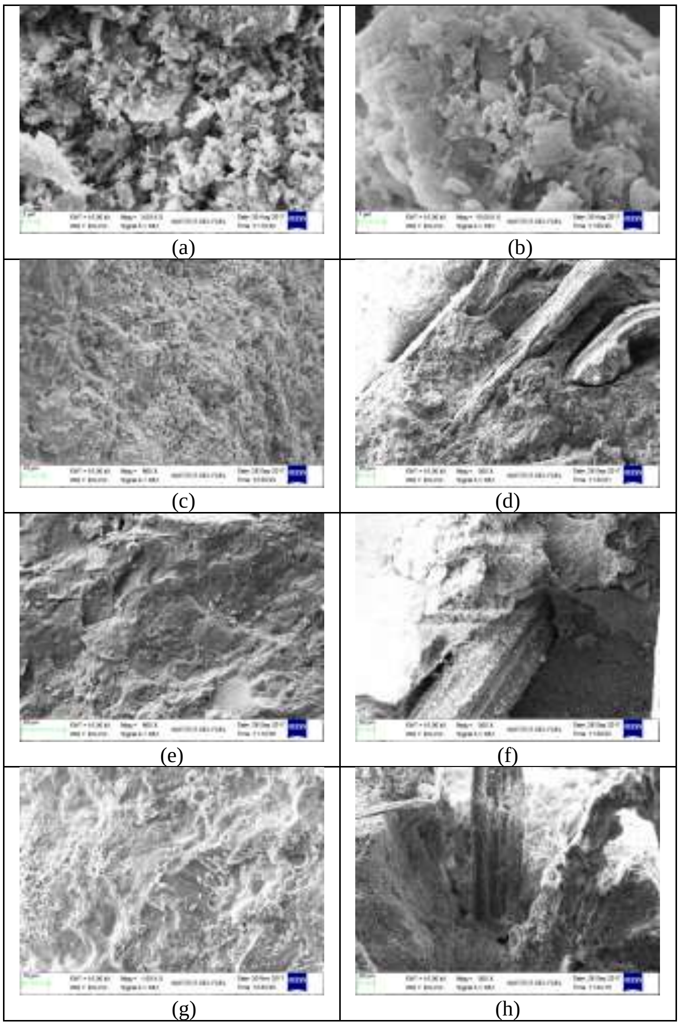

Fig. 5: SEM images of AASA and NC

5. Conclusions

The evidence from this study showed that the optimum contents of AASA and NC are found to be 40 wt. % and 2 wt. % respectively. But when blended together, the AASA and NC, the optimum content are 50 wt. % and 2 wt. % replacement of OPC, respectively. In the case of BC AASA and NC, reduction in density is about 13% and enhancement in compressive strength about 50%, tensile strength 5.4%, and flexural strength 15%. However, the decrease in mechanical strength take place when more AASA and NC are added due to high clay contents and poor dispersion of BC which generate voids and require more liquid. Therefore, adding lower percentage of NC in composites had led the changes in density and mechanical strength. The results reveal that by adding AASA with NC give significant effect on microstructure of BC and enhance the mechanical and physical properties. Grinding AASA into fine particles has helped increase the physical properties of the material. Besides the addition of nano sized NC also provide the filling effect in the matrix and strengthen interface bonds of fiber-matrix. This study confirms that the AASA and NC materials possess large surface area and big aspect ratio though their particles sizes are not arranged uniformly. However, it is suggested that more research are required to overcome the AASA and NC agglomerations when added at exceeding optimum content.

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