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RICE HUSK ASH AS A NANO-FILLER TO SYNTHESIZE THERMOSETTING POLYMER NANOCOMPOSITES AND EVALUATION OF ITS TRIBOLOGICAL BEHAVIOR

Salsabeel K. Burhan[1], Meethaq M. Abed[2], Moslih A. Salih[3]
[1] MSc Polymers and Composite Materials. Lecturer, Al-Furat Al-Awsat Technical University, Technical Institute of Babylon, Babil 51015, Iraq. salsabeel.k.b@gmail.com
[2] MSc Polymers and Composite Materials, Karbala Technical Institute, Al- Furat Al - Awsat Technical University, 56001, Karbala Iraq, mee1sa2012@gmail.com
[3] PhD Civil Engineering, Head of Department, Al-Furat Al-Awsat Technical University, Technical Institute of Babylon, Babil 51015, Iraq. moslih.a.salih@gmail.com
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ABSTRACT

This paper deals with synthesizing of nanocomposites of unsaturated polyester (UP) filled with nano silica and investigation the effect of silica content on the tribological behavior of UP and nanocomposites . Silica nanoparticles were prepared by burning rice husk ash (RHA) as an agricultural waste material. Three different percentages as 2%, 6% and 10% of nano silica were added to the unsaturated polyester and dispersed by using ultrasonic waves method. As a result of agglomeration and sedimentation of nano RHA particles, dispersing by ultrasonic waves was very effective to terminate these challenges. X-Rays diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and scanning electron microscopy (SEM) technics were used for characterization of materials. RHA-UP nanocomposites were tested for their tribo-performance by pin-on-disc machine. XRD demonstrated that white rice husk ash consists of amorphous silica. SEM demonstrated that silica particles were distributed uniformly and well embedded within the polyester matrix. Results showed an improvement in wear resistance with relatively stable friction coefficient with the addition of 10% nano silica. Adding 2 wt% nano RHA negatively affected the wear resistance of RHA-UP nanocomposite .The best tribological performance of 10% RHA-UP nanocomposites achieved at load 10N. Incorporation of RHA with unsaturated polyester has advantages of reducing pollution, making the pollutant RHA as a valuable material to produce low cost and lightweight products with modified properties. Silica enriched RHA could prove to be cheaper anti-wear additives for producing high performance unsaturated polyester composites such as brake pads.

KEYWORDS: Nano-silica, Rice husk ash, Nanocomposites, Anti-wear, FTIR, SEM.

1. INTRODUCTION

Recently, nanocomposite materials, renewable sources, recycling and ecofriendly materials attract attention of many researchers around the world (García et al., 2004), Emad et al., 2015), Chang and Sun, 2014), Rajendra et al., 2015), Meethaq, 2017). Polymer nanocomposite (PNC) is a composite material of polymeric matrix filled with dispersed nano-fillers (Kazım, 2007), Vikas, 2015). Nano fillers may be nanoparticles, nanofiber or nanotube (Rao and Kurt, 2011), Tai and Kim, 2003). Inorganic nanoparticles such as alumina, silica, clay and ZnO and metallic nanoparticles such as gold and silver had been used to synthesize nanocomposites (An and Lim, 2003), Pedro et al., 2009).

Rice husks considered to be one of the renewable resource which can be used to prepare silica (SiO_2) (Della et al., 2002). Rice husks are agricultural wastes accumulated in millions of tons at rice producing factories (Soltani et al., 2015), Ghassan and Hilmi, 2010), Jassim, 2014). Some researchers reported that burning of rice husks produces ash containing high percent of silica reaches 97 % with some traces of Al, Fe, Mg, Ca, K, C, P and Na (Ayswarya et al., 2012), Khalida et al., 2016), Virginia et al., 2013). RHA characterized by high thermal shock resistance, low thermal conductivity and tough (Ayswarya et al., 2012), Alfaro et al., 2013). Using RHA as a source rich in silica as a major constituent to synthesize composite materials would provide a low cost products with better mechanical and wear properties (Dora and Chintada, 2014), Navin et al., 2010). Polyester based materials are extensive in applications. Reinforcement of unsaturated polyester opens doors for various applications range from house hold, pipes, tanks, ducts, brake pad formulation to helicopter rotor blades and pump impeller blades (Emad et al., 2015), Arun and Alok, 2012), Mutlu., 2009).

Generally, polyesters characterized by low cost, very good electrical insulating properties, chemical and heat resistance, good adhesion and good mechanical properties (Mei and Raman, 2004), James, 1999).Wear represents an important phenomenon that occurs in all applications of polymers and it can be quantitatively measured in term of the mass or volume loss from sliding or eroding contacts surfaces (Awham et al., 2010). Many researchers have published studies about rice husk ash – polymer composite. Navin et al were studied the tribological behaviour of polyvinylchloride filled modified rice husk ash and showed that an positive effect of rice husk ash additive which improved the tribological and mechanical properties (Navin et al., 2010). Sihama et al were studied the mechanical and thermal properties of polymers filled rice husk ash at various condition and showed that the increasing of mechanical and thermal properties as increased calcined temperature (Sihama et al., 2014). Also, Aseel et al were

studied the influence of epoxy polymer coated by natural based materials (rice husk ash) on erosion wear behaviour and showed that the erosion wear resistance increasing when coating by rice husk ash (Aseel et al., 2015).

The present work focuses on the selection of SiO_2 in nanoscale, prepared by burning of rice husk as a filler for the unsaturated polyester and investigation the filler content influence on the tribological behavior of unsaturated polyester nanocomposites.

2. EXPERIMANTAL WORK

2.1. Materials

2.1.1. RHA (Rice Husk Ash)

Ash of rice husk was used as the main source to produce SiO_2 nano particles. It was brought from local source. After burning in closed oven at 650 °C for up to 120 min, it turns to a white ash. The white ash was milled for 15 hrs by (SFM-1 Desk-Top Planetary Ball mill, China), alumina balls were used at various radius 2, 4, 6 mm at 450 r.p.m.

2.1.2. The mixture of the Unsaturated Polyester resin (UP)

The unsaturated polyester resin was used as the main matrix. Cobalt nephthalate was used as an accelerator, the addition percentage was (0.5 g to each 100 g) of the resin. MEKP (Methyl ethyl ketone peroxide) was used as catalyst to solidify the unsaturated resin, the addition was (2% by weight of the matrix). The chemical materials were supplied by IRC Saudi Arabia Company.

2.2. Synthesis of nanocomposites specimens

The nano silica powder was dried to remove any moisture at 100 °C for 1 hr. Three different percentages of nano silica as 2%, 6%, and 10% by weight of the resin were prepared. The unsaturated polyester resin was poured into a container and then the nano silica was added and mixed manually for 10 min to avoid any agglomeration. An advanced mixing for 45 min was applied to the mixture by using ultrasonic waves machine (Ultrasonic cell crusher-SJIA-1200W, MTI corporation) which is followed by adding the catalyst and the accelerator. The final mixture was poured into cylindrical molds (5 mm * 15 mm) which are allowed for 24 hours for curing at room temperature . After the 24 hours of curing, the molds were cured in the oven at 100°C for 1hour to complete curing. Fig. 1 shows preparing process of RHA - UP Nanocomposites.

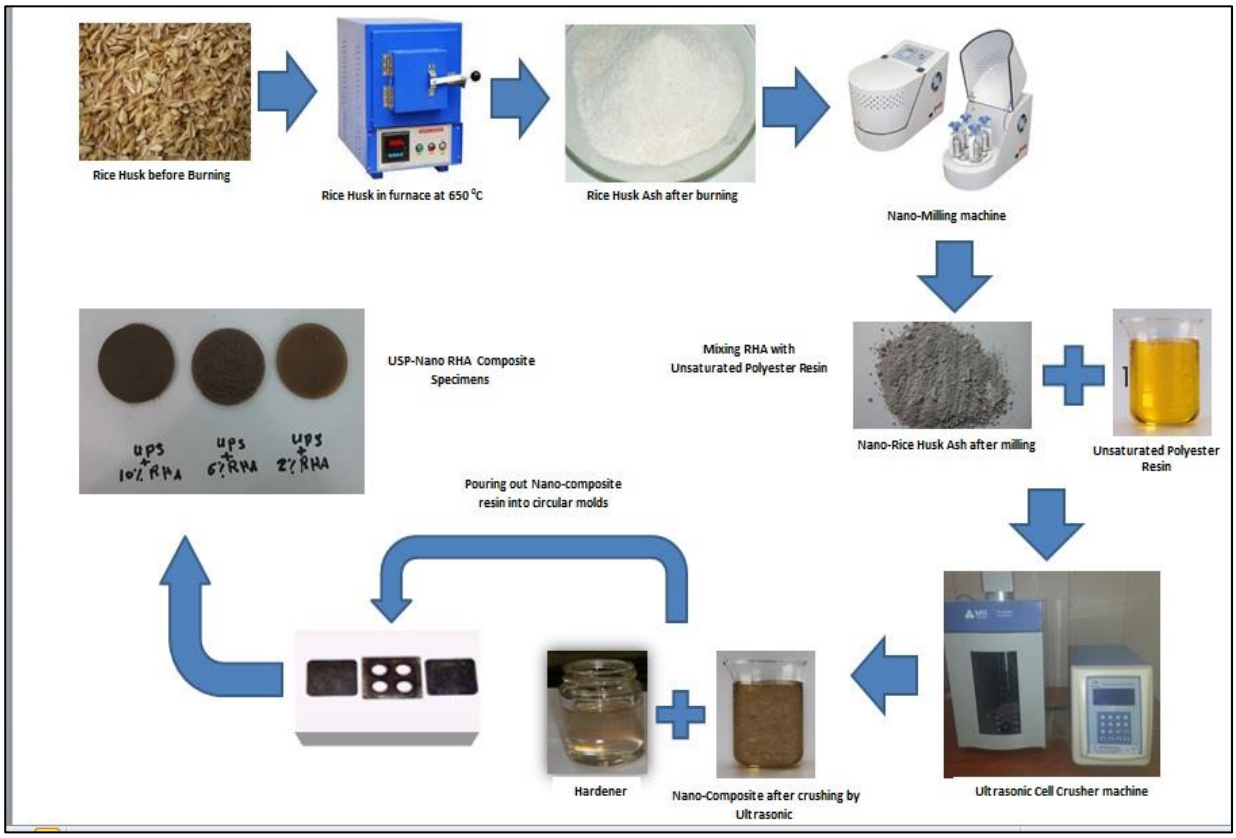

Fig. 1. Preparing of RHA - UPS Nanocomposite.

2.3. Characterization

2.3.3. X-Ray Diffraction (XRD)

Shimadtzu X-ray diffractometer (XRD-6000) was used to characterize rice husk ash by detecting the resulted phases. The test conditions: target (Cu ; kα = 1.54056 Å), voltage (40kV), of 2θ (20 – 70) and scan speed (6.000 deg/m) distinguish.

2.3.4. FTIR Spectroscopy

The nano silica, unsaturated polyester resin and nano composites were characterized by FTIR spectroscopy (FTIR: MODEL ALPHA T, Bruker, Germany). FTIR test was carried out to characterize RHA by distinguishing any formed bonds between RHA and UP matrix.

2.3.5. Scanning Electron microscopy (SEM)

UP-SiO_2 nanocomposite was scanned by SEM (Inspect S50, Bruker, Germany) to show the morphology of the composite and the size of silica particles.

2.4. Wear test

Wear behavior of the unsaturated polyester reinforced RHA was evaluated by pin on disk type friction and wear monitor (Microtest, MT4003, Spain) as shown in Fig. 2. The data acquisition

system was used to provide the magnitudes of (friction coefficient). The final results represent the relation between the coefficient of friction and sliding distance.

This machine was designed for studying wear under sliding condition. Generally, a stationary pin slides on a rotating disc. D.C motor rotates a disc at speed range (0 – 700 r.p.m). In this experimental study, a steel ball with 6 mm radius slides against the nanocomposite specimens rotate 250r.p.m. with wear track diameter 6 mm. At ambient conditions, tests were performed, with different normal loads (5N, 10N, 15N, 20N, 25N) for 30 min, then a 5 N load was applied for (30, 60, 90, 120, and 150 min). Before and after each test, the specimens were well cleaned and weighted to calculate the wear rate according the equation (Awham et al., 2010):

$$W.R = \frac{\Delta m}{2\pi r n t} \quad (1)$$

W.R = rate of wear (g/m)

Δm = missing mass (g)

r= wear track radius (m)

n= revolution per minutes

t= time (min)

Fig. 2. Pin -on- Disk machine.

3. RESULTS AND DISCUSSION

3.1. XRD test

From the XRD pattern of the white extracted powder of rice husks ash (Fig. 3) it can be seen that the extracted material is amorphous silica . The XRD results show that compound name is silicon oxide, chemical formula is SiO_2, mineral name is crystobalite, crystal data [cell vol. : 0.00 Å3] .The Rice husk ash after burning is a silica in amorphous form transformed into crystobalite – like structure at 2θ (21.7, 30.13, and 50.16); however, it contains some peaks at 2θ (35), and at 2θ (60) which reflect amorphous random phase. Music et al., 2011 and Ghassan et al., 2010 recorded a broad XRD peak for amorphous silica at 2θ close to these measurements (Music et al. (2011), Ghassan et al. (2010)).

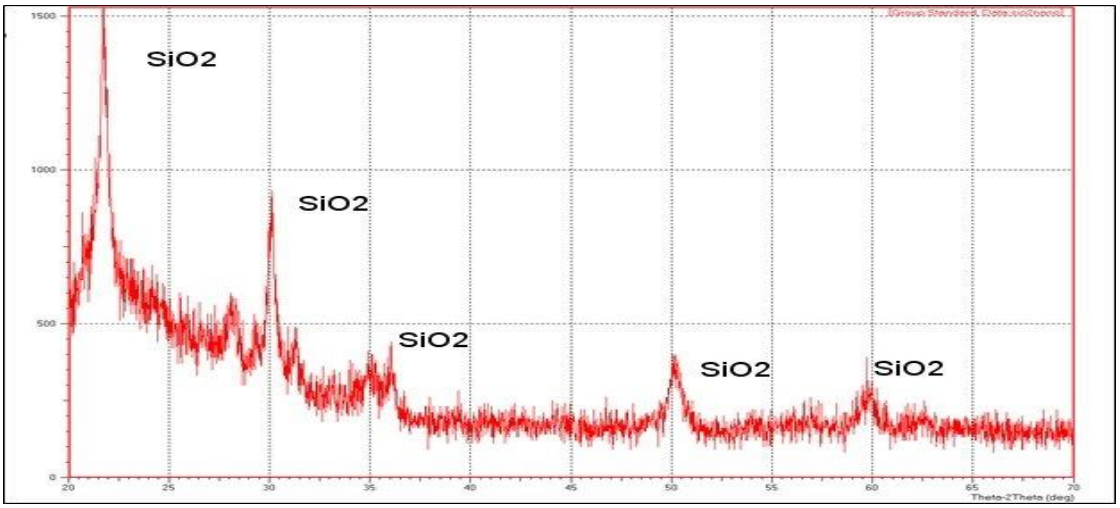

Fig. 3. XRD Pattern.

3.2. Scanning Electron Microscope test

Scanning electron microscopy of UP-RHA composite (Fig. 4) demonstrates that silica particles in nano size range of 50 nm to 300 nm have uniformly dispersed and well embedded in the unsaturated polyester matrix. The uniform dispersion of nano silica particles (hydrophilic) within the polymeric matrix (hydrophopic) may be due to the use of the ultrasonic waves energy which be enough to break the filler aggregates into small agglomerates, so that the contact surface area increases and improving wettability. Therefore, a better matrix-filler interaction satisfied, in addition to avoid using of coupling or compatibility agents which act as link between the filler and matrix.

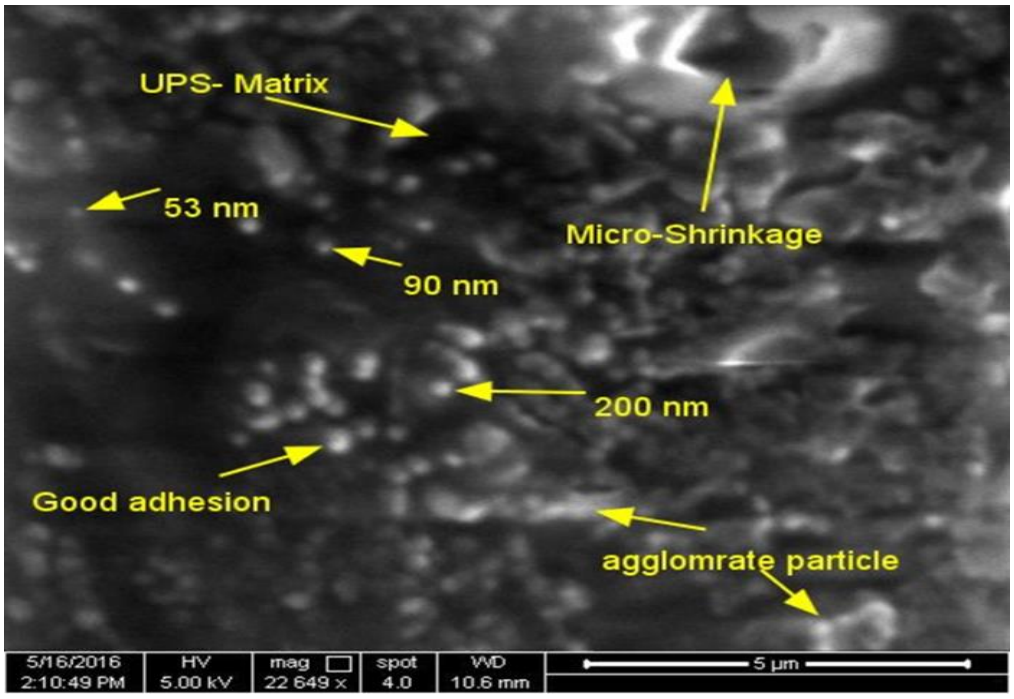

Fig. 4. SEM micrograph of nanocomposite (RHA-UPS).

3.3. FTIR test

Fig. 5 show the spectra of pure RHA, pure UPS and RHA-UPS nanocomposites with the addition of 2%, 6%and 10% by weight respectively. RHA-Pure shows the characteristic peak at 2394.7 cm^{-1} could be assigned to Si-O group (stretching vibration) in the amorphous silica. The very strong band at 1054 cm^{-1} is assigned to Si-O-Si stretching vibration. The IR band at 786 cm^{-1} could be assigned to Si-O-Si. The band at 459 cm^{-1} is due to O-Si-O. UPS-Pure shows the characteristic peaks at 2918 cm^{-1} (C-H stretching vibration), 1708 cm^{-1} (C=O stretching vibration), 1440 cm^{-1} (C-C in ring, stretching), 1610 cm^{-1}, 1240 cm^{-1} and 1098 cm^{-1} (C-O stretching vibration) and 686 cm^{-1} (=CH out of plane) . RHA-UPS nanocomposites (2%, 6%, 10%) show the new characteristic peaks at 2360 cm^{-1} and 488 cm^{-1} (Si-H). The new peaks indicates polymer-RHA interactions.

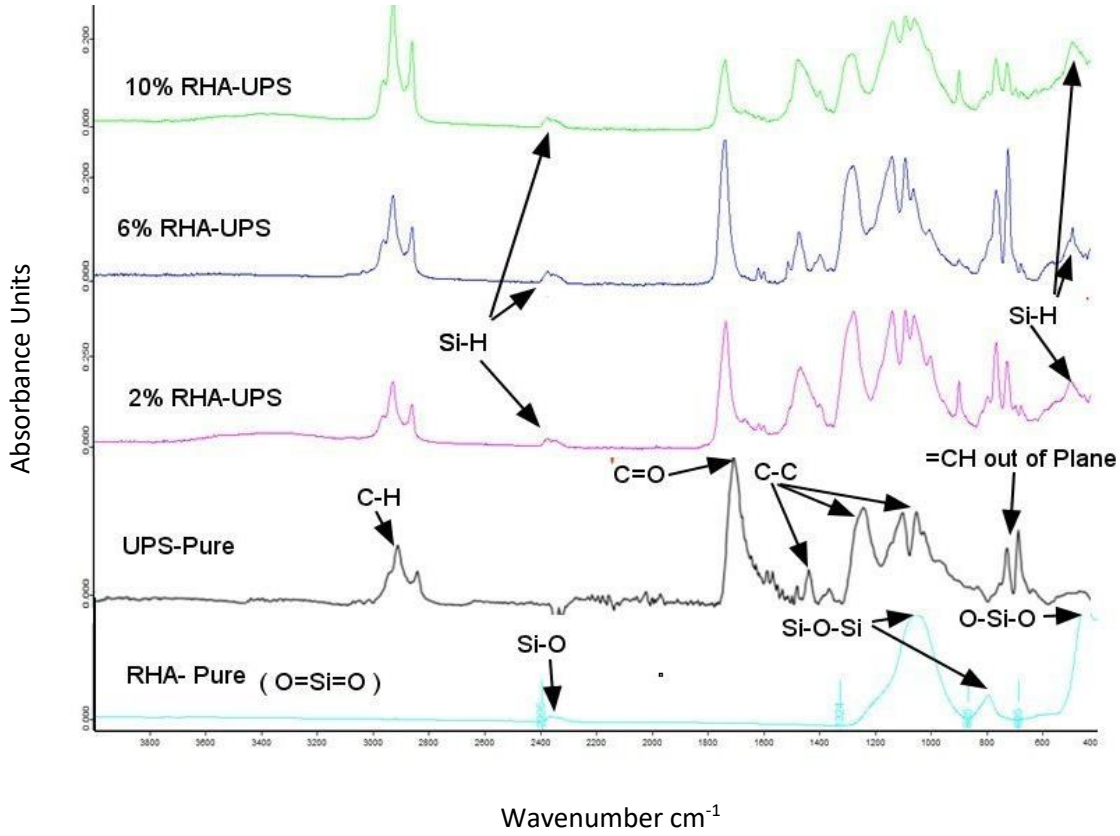

Fig. 5. FTIR of pure RHA, pure UP and RHA-UPS Nanocomposites.

3.4. Wear rate test

As can be seen from Fig. 6, the wear rate results with 10% nanocomposites RHA-UP samples showed the highest wear resistance in comparative with samples contained pure unsaturated polyester, 2%, and 6% RHA. Also, it can be noted that at load 5 N, all samples suffered high wear rate and this can be due to the presence of asperities on the fresh surfaces .The highest wear rate was registered with 2% RHA-UP in comparative with pure UP, 6% and 10% RHA-UP composites. This behavior may be attributed to the effect of ultrafine silica content which act as plasticizer; moreover, it reduces the cross-linked network in the unsaturated polyester structure.

The test results presented in Fig. 7 showed that samples with 10% RHA-UP resisted wear at different rates of loading which are 10N, 15N and 20N and reached a steady state; however, it suffered wear at 25N. When metal counter face slides against polymer composites, abrasion take place and remove asperities generated from the wear. Removed asperities retained within the interface till they escape or attach and fill the valleys on surface during the wear process, a thin polymer film is formed by adhesion called "transfer polymer film" which act as protective

layer until steady state reached. After this stage, a back transferred film case occurred and wear raised (Khalida et al., 2016), Sawyer, 2003).

At higher RHA content, the nanoparticles percent per unit volume increased and carried part of load and as well-known that the nanoparticles are difficult to rub by the counterpart face and the polyester based composite surface compared with larger particle size, therefore 10% RHA significantly improved the wear properties of the polymeric matrix.

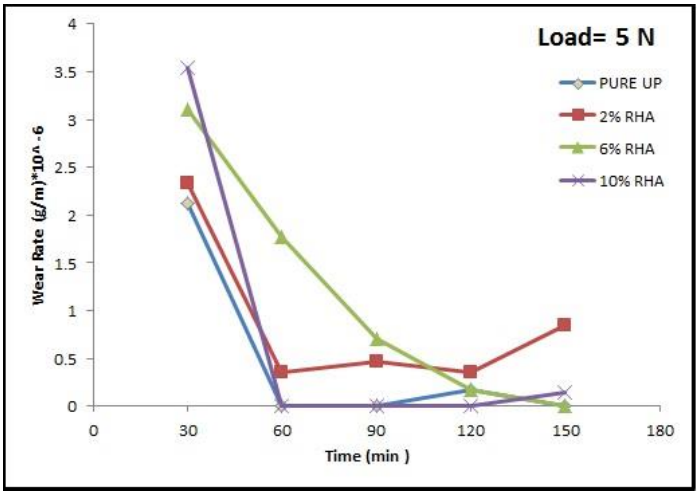

Fig. 6. Variation of wear rate for RHA-UP nanocomposites at constant load (5 N) and different times.

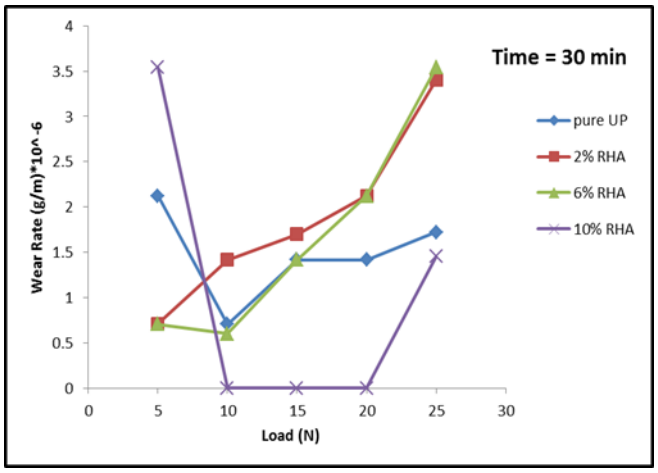

Fig. 7. Variation of wear rate for RHA-UP nanocomposites at constant time (30 min) and different loads.

3.5. Friction coefficient

Similarly, Fig. 8 shows the friction coefficient profiles for the neat UP and nanocomposites samples under the described conditions. It was noted that the friction coefficient of 2% RHA was relatively unstable. Also, it can be observed that friction coefficients of 6% RHA and 10%

RHA composites were in steady state. 10% RHA was the lowest friction coefficient in comparative with 6% RHA and 2% RHA.

At load 5N, coefficient of friction of unfilled UP was less than of nanocomposites but when load increased up to 10 N, friction coefficients of nanocomposites became the lowest as shown in Fig. 8-b. As a result of the smoothing effect of the nanoparticles embedded tribo-film at contacting surfaces. According to the friction mechanism of nanocomposites when load increased, the friction coefficients of nanocomposites increased. Embedded nano silica particles carried part of load and acted as anti-wear factor therefore friction may be happened between the metallic surface and the hard ceramic particles. Pure UP showed the lower values of friction coefficient. This may be a result to the softer wear debris of neat UP produced during the sliding in comparative with those of nanocomposites containing hard nano silica (Kazım, 2007).

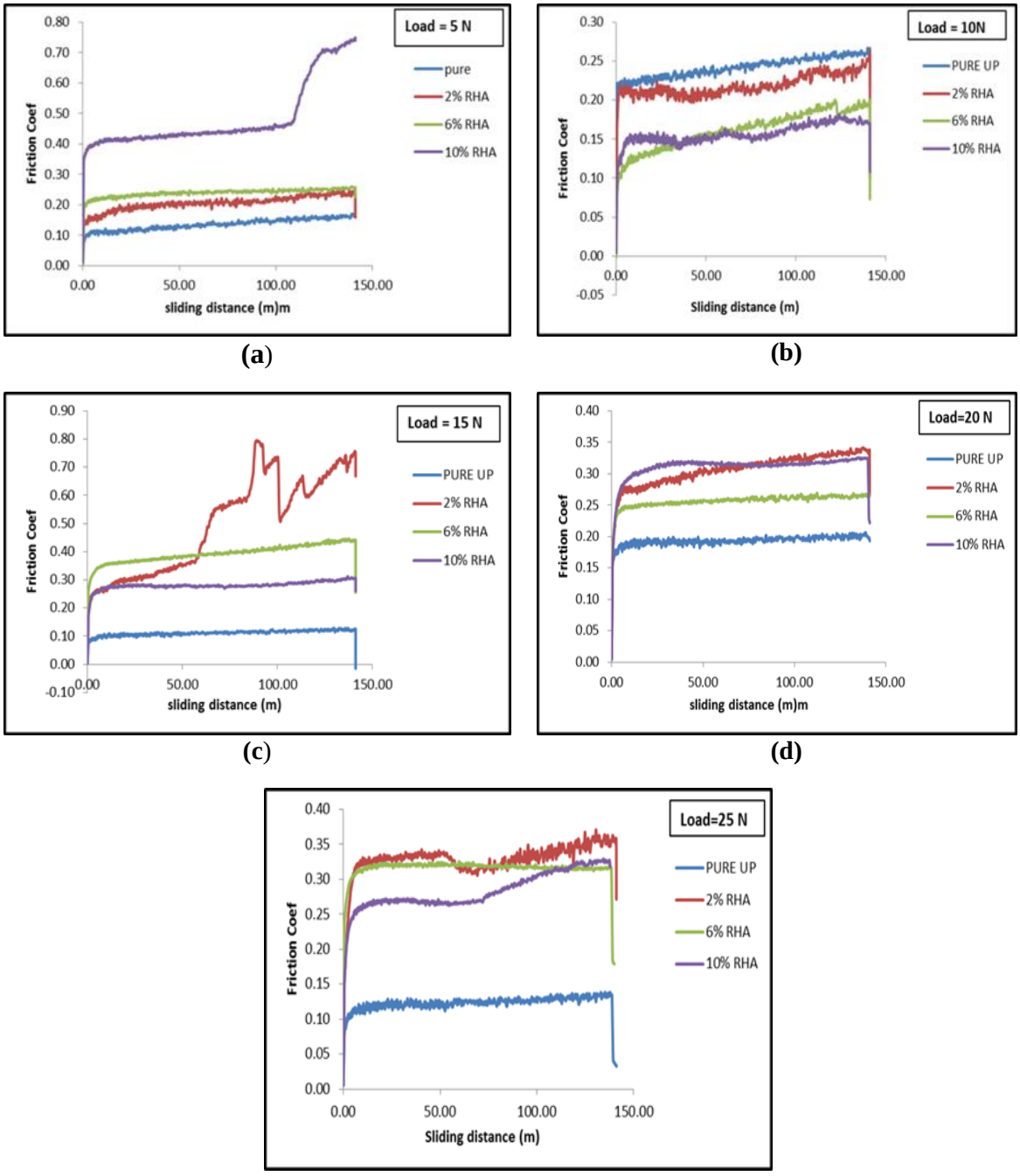

Fig. 8. Friction coefficient profiles of Pure UP, 2%RHA, 6%RHA, 10%RHA-UP composites at loads (a) : 5N, (b): 10N, (c): 15N, (d) : 20N, (e) : 25N.

4. CONCLUSIONS

1. The results showed that using nanosilica from RHA increased the wear resistance for the unsaturated polyester.
2. Wear resistance increased with the increase of the nanosilica percentages and reached the highest resistance with 10% addition.

3. The best improved tribo-performance of 10% RHA-UP nanocomposites attributed to the uniform distribution of the nanoparticles within the polyester matrix, with combination of particular mechanical and antiwear properties of nanosilica .
4. 2% RHA-UP nanocomposite exhibited the lower tribo-performance, this was ascribed to the low RHA content which act as plasticizer reduces the cross-link network in the UP structure.
5. Friction coefficient values stayed relatively stable during the test of 6% RHA-UP and 10% RHA-UP nanocomposites comparative with 2% RHA-UP nanocomposites.
6. Producing nano-silica from RHA can be cheaper anti-wear additives for the preparation of high performance unsaturated polyester composites.

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