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Characterization and Wear Behavior of Carbon Black Filled Polymer Composites

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Abstract

Carbon black derived from wood apple shell, was obtained by pyrolysis of wood apple shell particles at 400°C, characterized by EDS analyzer and uses as filler in polymer composites. An experimental study was conducted to compare the erosive wear behavior of both raw and carbon black wood apple shell particles filled epoxy resin matrix composites. The effect of wood apple shell particles concentration with different impingement angles (30°,45°,60°and 90°) at constant impact velocity 48 m/sec on the erosion rate of composite has been analyzed. However, it is found that the carbon black particulates composite shows minimum wear as compared to raw particulate composite. It also shows semi ductile type failure and maximum erosion rate is observed at 60° impingement angle. Further, the erodent worn surface morphology is examined by using scanning electron microscope (SEM).

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Key words:wood apple shell; carbon black; wear; EDS; SEM

1. Introduction

Historically composite are old but in1960 it start capturing the attention of industries with the introduction of polymeric-based composites. Polymer based composite materials have been used widely in home appliances, construction, automotive industry, packaging application, aircraft engine blades due to their excellent specific mechanical and tribological properties for thousands of years by Guadagnoa et al. (2009), McIntyre et al. (2005) and Leman et al. (2008). In 1999, the construction sector was the world's second largest consumer of polymer composites representing 35% of the global market by Yan et al. (2000).

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In that period synthetic fibers like glass and aramid used as reinforcement in polymer composite. Synthetic fibers provide advantages of high stiffness and strength compared to conventional construction materials, i.e. wood, concrete and steel. In spite of these advantages, synthetic fibers have a tendency to decline because of their high-initial costs and pollution generated during the production and recycling.

In recent period bio fibers have attracted their attention of researchers because of their advantages over other established materials. These bio fibers are fully biodegradable, environmentally friendly, renewable, abundantly available, high stiffness, cheap, low density and high degree of flexibility during processing. Literatures survey reveals that various attempts made to develop polymer composites modified with various fillers (such as jute, banana, bamboo, coconut, silica, carbon black, Al_2O_3, $CaSiO_3$, etc.) in other to improve the performance of this matrix by Murali Mohan et al. (2007), Wetzel et al. (2003), Dandekar et al. (2005) and He et al. (2009). Xing and Li (2004) investigated the effect of silica particle size on the wear behavior of epoxy composites at low levels of filler content. In that study, it was proved that the smaller sized particles seemed to be more effective in improving the wear resistance. Recently, conductive polymer composites obtained by filling polymer matrices with various carbon blacks were reported by Krzesinska et al. (2008).

Particulate fillers of which carbon black is a notable example are widely used as reinforcing in polymer industries for advanced application by Zhang et al. (2008). Carbon blacks are produced from the agricultural and forestry waste materials but over the last century those carbon black were obtained from thermal cracking of natural gas, furnace black which is produced by incomplete combustion of oil feed stocks. The production of carbon black is relatively very expensive; therefore researchers found the alternative source i.e. renewable resources such as agricultural waste from which carbon black can be prepared. According to their investigation carbon black can be prepared by the pyrolysis of coal, wood, coconut shell, oil palm shell and other lignocellulosic materials because they are carbonaceous in nature and reach in organic materials. This biomass can be converted into carbon black thereby reducing unwanted, low-value agricultural residues and underutilized crop into useful, high-value materials by Abdul Khalil (2007). Carbon black is widely used for adsorption of pollutants from gaseous and liquid streams, for recovery of solvents, due to their high adsorptive capacities, porous size and relatively high mechanical strength. It is also used for coating, ink and inkjet application. Carbon materials provide excellent properties for a large spectrum of industrial applications by Adinata (2007). From carbon we obtain the strongest fibers (carbon fibers), one of the best solid lubricants (graphite), one of the best electrically conducting materials (graphite electrodes), the best structural material for high temperature tribological application (carbon–carbon composites). Among the various bio fibers wood apple shell is also a carbonaceous fiber. Still there is no work has been so far done on the chemical modification and analysis of wear behavior of polymer composite incorporating wood apple shell particulates. Some researchers like G.anusha (2011) and Ahmad (2011) used wood apple shell particulates as an absorbent for the removal of iron or Congo red dye from waste water using wood apple shell carbon.

The objective of this paper is to fabricate a new set of carbon black particulate filled polymer composite using bio waste wood apple shell. The effect of solid particle erosion wear and morphological behavior of the composite was investigated. In addition, the wood apple shell particles were also characterized by EDS analyzer to find out the elemental composition present in the wood apple shell particles. Some results, previously published by Adinata et al. (2007), Dandekar et al. (2005) and Abdul Khalil et al. (2007), were very encouraging and point towards the possibilities of structural and thermal applications of these composites.

2. Materials and methods

2.1. Epoxy resin

Epoxy LY556 (liquid diglycidyl ether of biphenol -A (DGEB-A) resin is used for this experimental study. It has been used extensively due to superior strength, excellent adhesion, good chemical resistance and excellent performance at elevated temperatures. Epoxy resin having density 1.2 g/cm^3, equivalent weight and viscosity is 182-192(gr/eq) and 11000-14000 MPa.s at 25°C.

2.2. Hardener

The curing agent was clear epoxy hardener HY-951 [NN0 (2-amineethylethane-1, 2- diamin)] is also used with epoxy resin with an amine value of 260–284 (mg KOH gm^{-1}). Both the epoxy resign and curing agent were obtained from supplier.

2.3. Wood apple shell

Wood apple (Aegle marmelos) belongs to family rutaceae is highly reputed medicinal tree commonly known as the 'Stone apple' or 'Bael'. It is an indigenous fruit of India and found abundantly from sub-Himalayan forest, Bengal, central and south India. Among the different natural fibers, wood apple shell (Aegle marmelos) appears to be promising material because of the high hardness and toughness.

The wood apple shells used in the present investigation are initially washed with water to remove the impurities and dried in an oven at 110°C for 24 hours to remove excess water content and moisture. The dried shells are converted to fine powder using a ball milling process for 24 hours and followed a sieve analysis to measure a particle size. The shell particles chosen for the experiments are in the range of 212 to 1µm is shown in Figure 1.

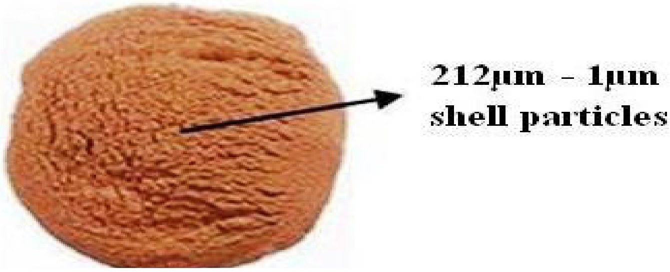


Fig. 1 Photomarcograph of raw wood apple shell particles

2.4. The processing of the bael shell (Carbonization)

The main purpose of carbonization process is to increase the carbon content and to create an initial porosity in the carbon black. During carbonization process most of the non-carbon elements, hydrogen, oxygen, nitrogen and sulphur etc. are first removed in gaseous form by pyrolytic decomposition of the raw materials.

Carbon black was prepared by carbonization process from raw wood apple shell particles. Wood apple shell particles were loaded on a ceramic boat which was placed inside a muffle furnace. The shell particles were heated up to a carbonization temperature at 400°C a heating rate of 5°C/min and were held for at least 2h at the carbonization temperature under inert atmosphere. After carbonization a soaking time of 1h was given after which the furnace was switched off. The carbon black produced which is shown in Figure 2, was cool to room temperature and subsequently ground and sieved. The process is usually carried out at a particular temperature in a continuous stream of an inert atmosphere.

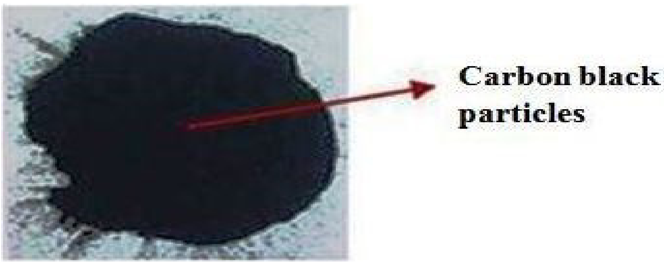


Fig. 2 Photomarcograph of the sieve carbon black wood apple shell particles

2.5. Characterization of the carbon black wood apple shell particles

Samples were analyzed by the combination of a scanning electron microscope (SEM) and an Energy dispersive spectrometer (EDS) analyzer. To characterize the morphology, eroded surfaces and to identify the mode of material removal, the eroded samples were studied using a NOVA NANO SEM 450 Scanning Electron Microscope at an accelerating voltage of 15 kV.

2.6. Composite preparations

The carbon black shell particulate composites were prepared by hand layup technique. Five different types of composites are prepared with varying weight fractions (5, 10, 15 and 20 wt.%) of particulates and epoxy. A wooden mold of (150x60x5) mm^3 is used for manufacturing the composite. Ten percentage of hardener HY951 is mixed in the resin earlier to reinforcement and proper stirring is done with mechanical stirrer for uniform mixing of particulates. Carbon black particulate was added carefully and gradually to avoid the loss of carbon black during the process and was observed to ensure that the carbon black blended well with matrix. For quick and easy removal of the composite a mold release sheet is placed on the top and bottom of the wooden mold. Mold release spray is also applied on the inner surface of the mold wall to facilitate easy removal of the composite specimen. Due to apply of load some polymer squeezes out from the mould. Care is taken to ensure that the expel polymer not to squeezes out from the mould. The each composite are cured under a load of about 30 kg for 24 h before it removed from the mould. Test specimens of suitable dimensions are cut with a diamond cutter from the composite sheets for wear and thermal test as per ASTM standard.

3. Erosion Wear Test Set-Up Description

One of the most important characteristic is the erosion behavior of polymer composite because in application parts of polymer composite very often operate in industry or dusty environments. This is the reason why in the last three decades the erosion behavior of polymers and related composites has been intensively studied.

Various studies have been made on tribological analysis of NFRPs like betel nut fiber-reinforced polyester by Nirma et al. (2010), sugarcane fiber-reinforced polyester (SCRP) by El-Tayeb et al. (2008), kenaf/epoxy by Chin et al. (2009) attempted to use kenaf fibers reinforced epoxy composite for bearing application. The wear test is conducted on erosion wear test apparatus according to the ASTM G76-95 standard test method. The test evaluates the wear characteristics of the developed carbon black filled wood apple shell particulates composite. The schematic of erosion test apparatus is shown in Figure 3. The details of the erosion test parameters for the experiment conducted are given in table 1. The weight loss is recorded for subsequent calculation of erosion rate.

Table 1 Experimental conditions for the erosion test

Test parameters	
Erodent	*Silica sand*
Erodent size (μm)	200±50
Erodent shape	Angular
Hardness of silica particles (HV)	1420±50
Impingement angle (α^0)	30, 45, 60 and 90
Impact velocity (m/s)	48
Erodent feed rate (gm/min)	1.467±0.02
Test temperature	RT
Nozzle to sample distance (mm)	10

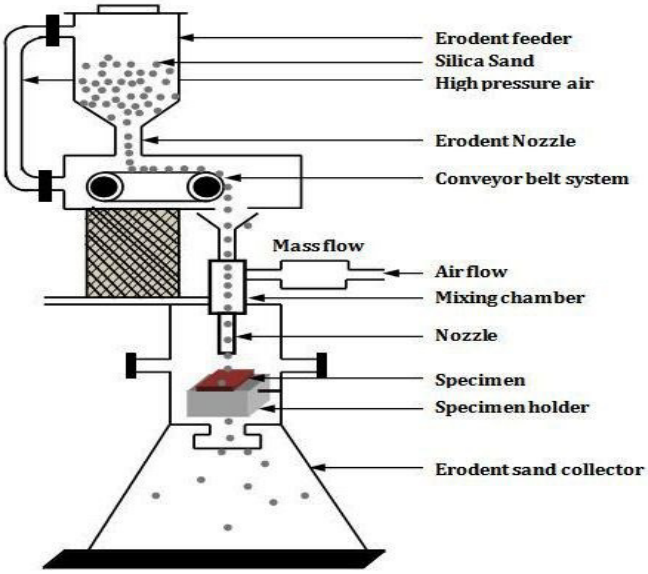


Fig. 3 Schematic diagram of Erosion test Rig

4. Results and discussion

Figures 4 show the inspection spectra of wood apple shell particulate surface elements acquired for wood apple shell. The particulates surface of wood apple exhibit spectra containing mainly carbon, oxygen, silicon, aluminum and small amount of, zirconium and calcium. However these elements are slowly removed from raw wood apple shell particulates by pyrolytic decomposition of shell particulates at 400°C. Also from the Figure 5 it can be observed that the carbon percentage increases from raw to carbon black particles at carbonization temperature. The presents of hard element like SiO_2 and Al_2O_3, the raw wood apple shell powder can be used as particulate reinforcement in various polymer matrixes.

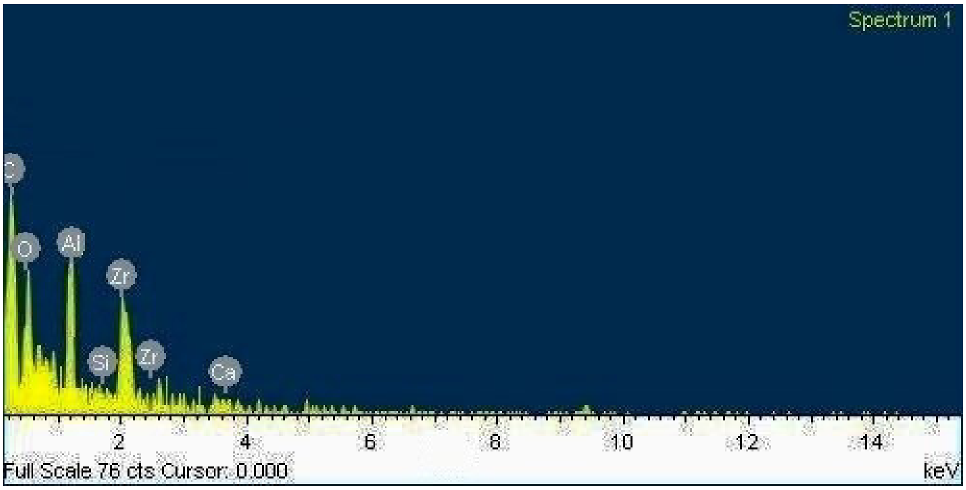


Fig. 4 EDS spectrum of raw wood apple shell particles

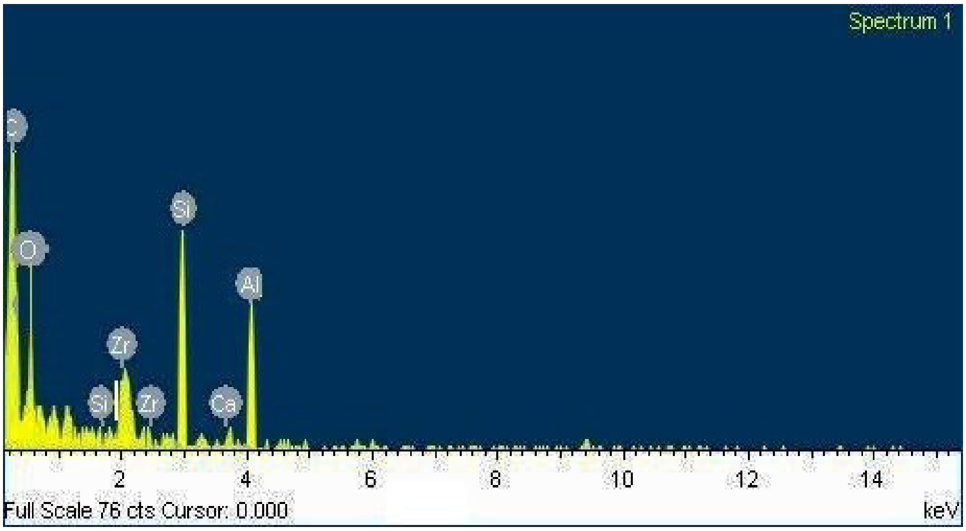


Fig. 5 EDS spectrum of carbon black wood apple shell particles

Erosion wear behavior of raw wood apple shell particulates composites with varying filler percentage at impact velocity 48m/s are presented in Figure 6. It is clearly evident from the figure that impingement angle significantly influenced on erosion rate. In case of neat epoxy composite maximum erosion occur at 90°C impingement angle. Therefore neat epoxy shows brittle nature. After adding raw wood apple shell particulates with epoxy, the maximum erosion wear occur at 30° to 45°C impingement angle of for all composite samples irrespective of filler loading as well as the nature of the composite changes from brittle to semi-ductile and it shows slight increase in erosion rate. Among all the composites the minimum erosion wear rate occurs for 10% wood apple shell particulates filler composite. But on further increase in impingement angle from 45° to 60°, almost all the composites showed minimum erosion rate. So the wood apple shell composite is behaving like semi ductile mode of erosion wear.

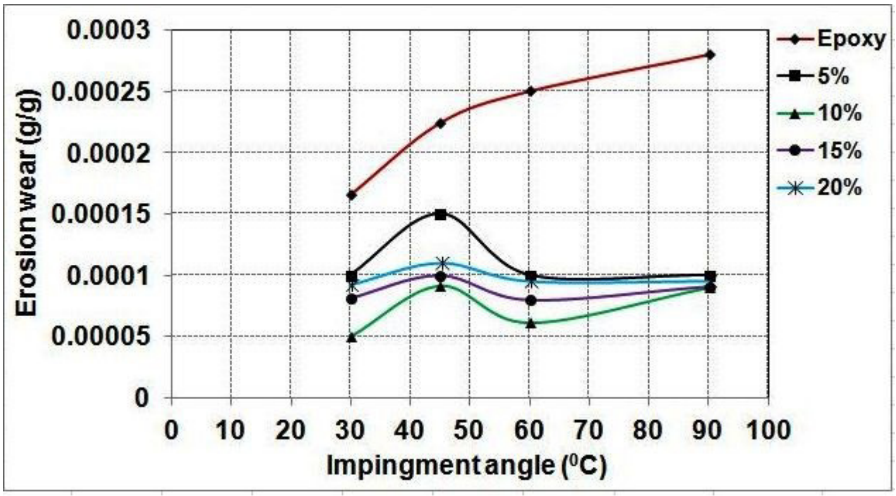


Fig. 6 Erosion wear behaviour of raw wood apple shell particulates composite with varying filler percentage at impact velocity 48m/s

Erosion wear behavior of carbon black particulates composites with varying filler percentage at impact velocity 48m/s is shown in Figure 7. It is observed from the figure that after adding of carbon black particulates with neat epoxy, the maximum erosion occurred at 60° impingement angle of for all composite samples irrespective of filler loading as well as the nature of the composite changes from semi ductile to semi brittle and it shows slight decrease in erosion rate as compared to raw particulates filler composite.

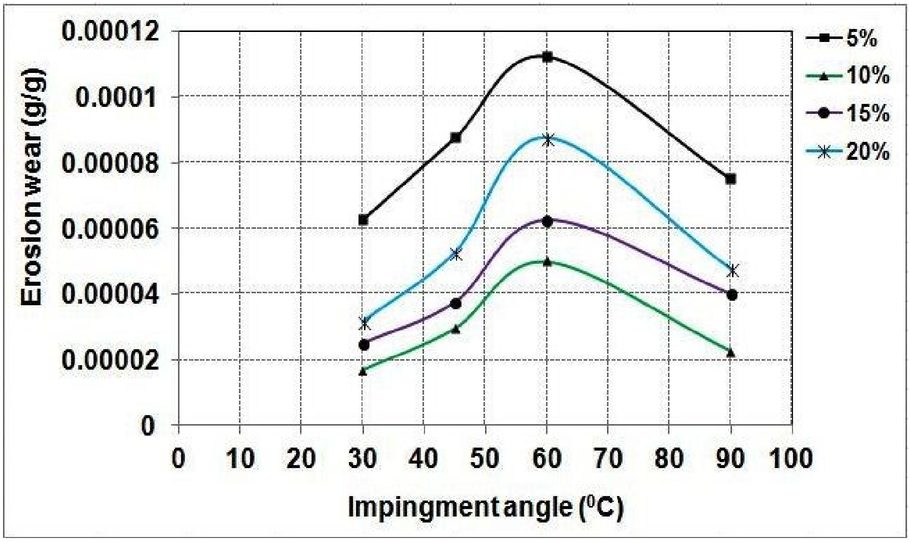
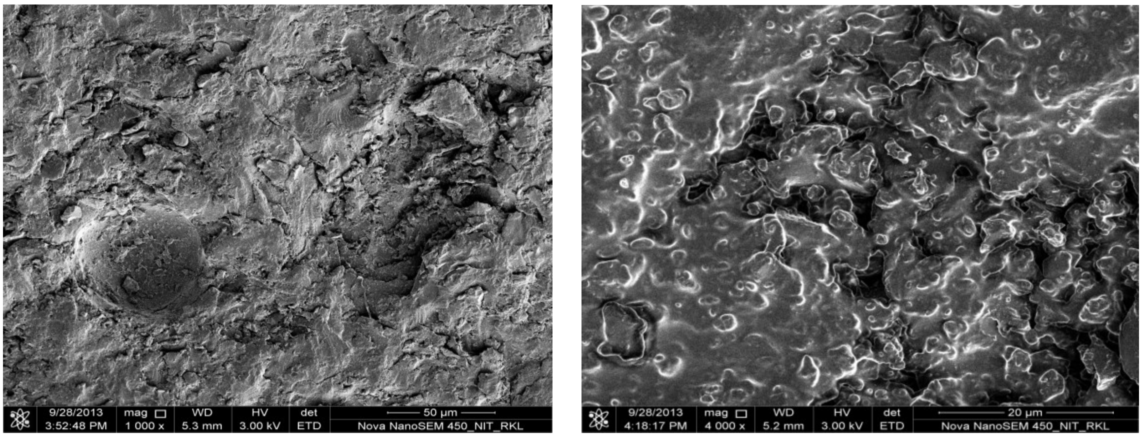


Fig. 7 Erosion wear behaviour of carbon black wood apple shell particulates composite at 400°C carbonization temperature with varying filler load at constant impact velocity 48m/s

SEM analysis of raw 10 wt% and carbon black 20 wt% filler composites with constant impact velocity 48m/s and impingement angle 45º are shown in figure 8 (a) (b). It is observed from the figure 8(a) that some materials have removed from the eroded surface of the 10 wt% filler composite. It shows the slightly good interfacial boding between the filler and matrix. After addition carbon black particulates with epoxy the surface of the composite becomes hard as compared to raw composite due to increases of carbon percentage in filler particles. Hence no voids and cracks found on the surface of the eroded composite shown in figure 8(b) and there is a good interfacial bonding between them.



(a) (b)

Fig. 8. SEM of eroded composite surface with impact velocity 48m/s , and impact 45º angle of (a) 10 wt.% filler (b) 20 wt% filler

5. Conclusions

The carbon percentage in wood apple shell particles greatly increase due to the pyrolytic decomposition of shell particulates at 400°C due to non-volatile substances are removed from the particles.

Study of influence of impingement angle on erosion rate of the composites filled with different weight percentage of filler loading reveals their semi ductile nature with respect to erosion wear. The peak erosion rate of raw composite is found to be occurring at 45° to 60° impingement angles for both raw and carbon black filler composite under various experimental conditions irrespective of filler loading. But the minimum erosion wear occurred for carbon black composite at 20 wt% filler as compared to raw 10 wt% filler composite.

From the SEM it is clear that there is good interfacial bonding between the filler and matrix material in carbon black composite as compared to raw composite.

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