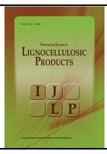

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Degradation analysis of lignocellulosic fillers infused coir epoxy composites in different environmental conditions

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Abstract

India has large resources of natural fibres such as Banana, Khus, Sisal, Korai grass, Talipot, Palm leaf, Coconut, Pineapple, Screwpine, Golden grass, Jute and Sabai among others. In the past, trade of the village folk depended on harvesting fibres grown in their villages. Natural fibers have recently gaining attraction of researchers due to their low cost eco-friendly and biodegradability characteristics they are exploited as a replacement for the conventional fibre, such as glass, aramid and carbon. Use of lignocellulosic fillers offers both cost saving and reduction in density as compared to inorganic particulates. This paper deals with fabrication of composite samples using coconut coir, epoxy resin (with hardener) and lignocellulosic particulate fillers such as cow dung, wheat husk and rice husk. after fabrication composite samples were exposed to different environmental conditions such as ultraviolet radiations, soil burial, high temperature, water (at room and high temperature) to study degradation behaviour of coir fiber reinforced epoxy composites filled with organic lignocellulosic fillers. Composite samples were fabricated using compression moulding technique. Degradation was confirmed by change in mechanical properties (Tensile, flexural and impact strength) and microstructure.

Keywords: Degradation studies, Environmental conditions, Mechanical properties, Microstructure.

1. Introduction

Processing of plastic composites using natural fibers as reinforcement has increased dramatically in recent years (Singleton et al., 2003; Keller, 2003; Rana et al., 2003; Valadez-Gonzalez, 1999; Oksman et al., 2003, Ashori and Nourbakhsh, 2014). For example automotive applications based on natural fibres with polypropylene as matrix material are very common today. Less work has been done to study composites with matrices, which originate from renewable raw materials. There are many different polymers of renewable materials: for example polylactic acid, cellulose esters, poly hydroxyl butyrates, starch and lignin based plastics. The problems with these polymers have been poor commercial availability, poor processability, low toughness, high price and low moisture stability. The long-term properties of renewable materials are also very important especially if the products are not single use applications. Natural fibres have many advantages compared to synthetic fibres, for example low weight, and they are recyclable and biodegradable. They are also renewable and have relatively high strength and stiffness and cause no skin irritations (Hornsby et al., 1997; Oksman et al., 2002; Oksman, 2001; Heijenrath and Peijs, 1996; Oksman, 2000; Mieck et al., 1994; Sanadi et al., 1994; Bledzki et al., 1996).

On the other hand, there are also some disadvantages: moisture uptake, quality variations and low thermal stability. Many investigations have been made on the potential of the natural fibres as reinforcements for composites and in several cases the results have shown that the natural fibre composites own good stiffness but the composites do not reach the same level of strength as glass fibre composites (Hornsby et al., 1997; Oksman et al., 2002; Oksman, 2001; Heijenrath and Peijs, 1996; Oksman, 2000; Mieck et al., 1994; Sanadi et al., 1994; Bledzki et al., 1996). Nowadays wheat, corn, rye, oats and other cereal crops are used to produce fibers and investigating to reinforce in composites area. Zhao et al. (2009) has focused on flame retardant properties of rice husk/PE composites. Kumar et al. (2010) used rice husk as filler for PP composites. The effect of the rice husk size and composition on the injection molding possibility of rice husk/PE and PP composites were studied by Rahman et al. (2010) and Nascimento et al. (2010). Kim et al. (2010) investigated photocatalytic performance of a carbon/TiO_2 composite with rice husk. Kumagai and Sasaki (2009) were fabricated carbon/ silica composites from rice husk by means of a binder-less hot-pressing. Mehdinia et al. (2014) investigates the physical and mechanical properties of soya stalk flour reinforced HDPE composite.

The aim of this study was to make an investigation of degradation of biodegradable fillers such as cow dung, wheat husk and rice husk infused coir-epoxy composite in different environmental conditions. Degradation of composites was confirmed by weight loss strength loss and micro-structural analysis.

2. Materials and Methodology

2.1. *Materials*

Coconut coir fibers were obtained from local sources and were used as reinforcement in the composite. Araldite LY-556 epoxy resin and HY-951 hardener were supplied by Ciba-Geigy of India Ltd. Lignocellulosic particulates such as cow dung, wheat husk and rice husk were used as fillers.

2.2. *Methodology*

2.2.1. *Composite fabrication*

Coir-epoxy infused particulate composites were prepared at room temperature using compression molding technique. Coconut coir, particulates and epoxy resin (with hardener) were mixed manually. The mixture was transferred into iron molds (Cavity size: 22 x 30 x 1.0 cm) and then pressed using a hydraulic press. A sample was taken out after 24 h. Fifteen samples with different fractions of cow dung, wheat husk and rice husk were fabricated are presented in Table 1.

Table 1. Blend formulation of materials.

Sample Designation	Epoxy (%)	Coir (%)	Particulate (%)		
			Cow dung	Wheat husk	Rice husk
A1	70	25	5	0	0
A2	70	20	10	0	0
A3	70	15	15	0	0
A4	70	10	20	0	0
A5	70	5	25	0	0
B1	70	25	0	5	0
B2	70	20	0	10	0
B3	70	15	0	15	0
B4	70	10	0	20	0
B5	70	5	0	25	0
C1	70	25	0	0	5
C2	70	20	0	0	10
C3	70	15	0	0	15
C4	70	10	0	0	20
C5	70	5	0	0	25

2.3. Degradation of composites in different environmental conditions

2.3.1. UV exposure

For UV exposure composite sheets were cut into dumbbell shaped and rectangular (according to ASTM D-638 and ASTM D-790) specimen. Pre-weighed composite samples were exposed to ultraviolet radiations in a QUV spray with solar eye irradiance control weather meter to simulate outdoor exposure. Test conditions were continuous UV exposure for 24 hours and weighed on completion of the test. After exposure to UV radiations weight loss and mechanical properties of composite samples were determined.

2.3.2. Soil burial analysis

For biodegradability test the composite sheets were cut into dumbbell shaped and rectangular (according to ASTM D-638 and ASTM D-790) specimen. Biodegradability test was carried out in a rectangular tray (38×55 cm) containing farmland soil by maintaining high relative humidity by daily sprinkling of water. Each specimen was buried in soil at room temperature. Specimens were dug out of the soil after 10, 20, 30, 40, 50, 60, 70, 80 and 90 days, respectively, and then washed with water and dried at 60°C in an air oven for 48 h. Biodegradability was determined by measuring loss in weight of the specimens (Muller, 2004). The percentage weight loss was determined by equation 1:

$$\text{Weight loss\%} = \frac{\mathrm{M}_o - \mathrm{M}_d}{M_o} \times 100 \qquad (1)$$

M_o= mass of composite before degradation

M_d = mass of composite after degradation

After soil burial analysis, mechanical properties of composites were determined.

2.3.3. Thermal ageing

The effect of thermal ageing on the properties of composite was determined by using ASTM D-5721. For determining thermal ageing the composites were cut into dumbbell and rectangular shaped (according to ASTM D-638 and ASTM D-790) specimen. The samples were heated at 80°C in air circulating oven for 5 days. After cooling down and conditioning at room temperature, the weight loss (equation 1) and mechanical properties of these samples were measured.

2.3.4. Exposure to water at room temperature and elevated temperature

Effect of water absorption on the properties of composite was determined by using ASTM D-570. For water absorption test the composites were cut into dumbbell and rectangular shaped (according to ASTM D-638 and ASTM D-790) specimen. Samples were immersed in water at 100°C for 24 h under atmospheric pressure and dried the samples in an air oven at 50 °C for a day. Mass change of the samples was recorded using an electronic balance. Mechanical properties of these samples were measured according to ASTM D-638 and ASTM D-790. For room temperature studies, the samples were submerged in water at room temperature for a week, dried in an air oven at 50°C for 24 h and then the weight loss (equation 1) and mechanical properties were measured.

2.4. *Effect on mechanical strength*

2.4.1. Tensile properties

Tensile strength of composite samples was determined by following ASTM D-638 using an Instron model 8801 testing machine. Dumbbell shaped samples of composites with dimension 150×10×3.5 mm were tested. Samples were tested to failure under tension at a crosshead speed of 1.5 mm. An extensometer was attached to the gauge section of the sample for strain measurement. Five samples were tested to check the reproducibility of results. Tensile strength, elongation at break and tensile modulus were recorded.

2.4.2. Flexural properties

Three point bending test of composite samples was performed according to ASTM D-790. Flexural test of composite samples was conducted using Instron Machine Model 8801. Rectangular samples with dimensions 80 ×10 ×3. 5 mm^3 were tested at a cross-head speed of 3 mm/min at room temperature. The support span for the flexural test was 51 mm. An average of five specimens of each type of samples was reported as a result.

2.4.3. Impact properties

Impact test was conducted according to ASTM D-256. Rectangular samples with dimensions 80×10×3.5 mm were tested using an Izod impact tester. For each composition five specimens were tested and average data was reported.

2.5. *Microstructural analysis*

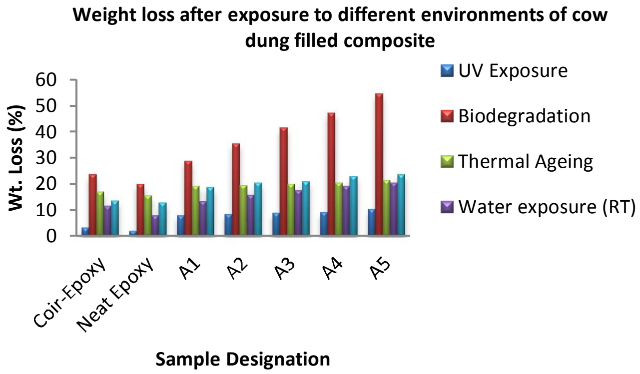
Scanning electron microscopy (SEM) images of the surface of the degraded and normal composites were taken to study the morphology of the surface degradation of composite. Composite samples were cut into 1x1 cm. Electron micrographs were obtained by using JEOL JSM 6390A before and after exposure to different environmental conditions at an accelerating voltage of 10 kV in low vacuum. The specimens were coated with gold to prevent electrical discharge.

3. Results and discussion

3.1. Weight loss analysis

Figure 1 presents weight loss of coir fiber reinforced epoxy composites filled with different particulate fillers after exposure to different environments such as ultra violet radiations, soil, high temperature, water at room temperature and elevated temperature. Composite samples were exposed to accelerated UV radiations for 24 h. Results show maximum weight loss of 10.33% in cow dung composite and minimum weight loss of 9.43% in wheat husk composite. Effect of ultraviolet radiations was increased with addition of lignocelluosic fibers and particulates whereas neat epoxy show maximum weight loss of 0.38%, but after the addition of coconut coir weight loss increases up to 1.98%. There was only 19% loss in weight in neat epoxy after 90 days of burial in soil was observed. This weight loss was increased up to 19.86% after addition of 23.56% coir in neat epoxy. There was a drastic change was observed in loss in weight after incorporation of organic particulate fillers in coir-epoxy composite. It can be observed that percentage weight loss was increased with increase in particulate content up to 25%. Similar results were observed for agro flour filled PBS composite by other researchers (Kim et al., 2005; Tserki et al., 2003; Ratto et al., 1999; Ishiaku et al., 2002).

Cow dung shows maximum weight loss than wheat husk and rice husk. Maximum weight loss of 54.58% was observed after 90 days of soil burial in a composite sample with 25% cow dung. After thermal ageing 15.55%, 16.96%, 21.34%, 21.21% and 20.03% weight loss was observed in neat epoxy, coir-epoxy, cow dung, wheat husk and rice husk composite respectively. With the increase in particulate content weight loss due to thermal ageing was also increasing. After water exposure 7.95%, 11.57%, 20.57%, 16.82% and 15.08% weight loss was observed in neat epoxy, coir-epoxy, cow dung, wheat husk and rice husk composite respectively. With the increase in particulate content weight loss due to water exposure was also increasing. After high temperature water exposure 12.87%, 13.56%, 23.76%, 21.65% and 20.87% weight loss was observed in neat epoxy, coir-epoxy, cow dung, wheat husk and rice husk composite respectively. With the increase in particulate content weight loss due to water absorption at high temperature was also increasing.



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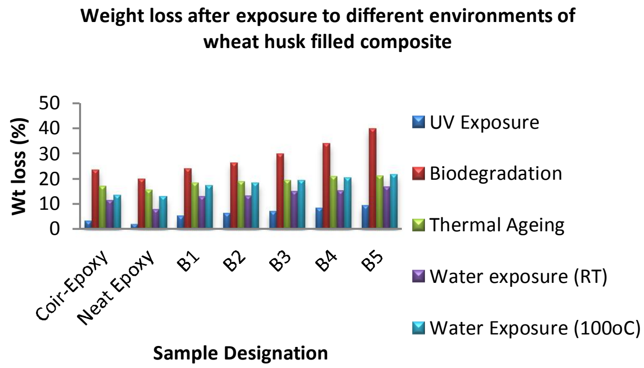

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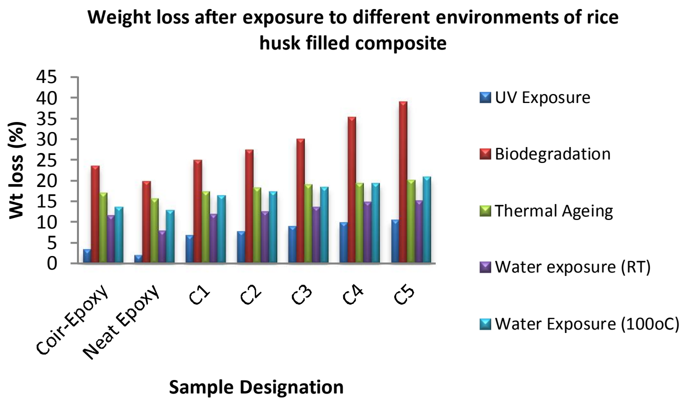

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Figure 1. Weight loss of coir-epoxy composite samples filled with different particulates (a) cow dung (b) wheat husk (c) rice husk after exposure to different environments.

3.2. *Effect on mechanical properties*

3.2.1. Tensile strength

Figure 2 presents the effect on tensile strength of coir fiber reinforced epoxy composites filled with different particulate fillers such as cow dung, wheat husk and rice husk after exposure to different environments such as ultra violet radiations, soil, high temperature, water at room temperature and elevated temperature. Declination in mechanical properties supports material degradation.

Neat epoxy shows a 5.24% decrease in tensile strength after UV exposure. The addition of coir in epoxy shows the slight declination of around 15.34% on tensile strength, but incorporation of cow dung shows 68.53% decline in tensile strength. In case of wheat husk and rice husk 55.58% and 52.47% decline in tensile strength was seen after UV exposure. There was a 9.97 % decrease in tensile strength of neat epoxy after 90 days of soil burial was observed. After addition of coconut coir 36.94% decrease in tensile strength was observed.

The incorporation of organic particulate fillers in coir-epoxy composite resulted in a more rapid decline in tensile strength during soil burial test (Shogren et al., 2003; Tansengco and Tokiwa, 1998). A declination of 84.07%, 81.81% and 81.47% in tensile strength of cow dung, wheat husk and rice husk based composites after soil burial analysis was observed. Result showed noticeable decrease in tensile strength after thermal ageing. 17.26% declination in tensile strength of neat epoxy after thermal ageing was observed. The polymer becomes more susceptible to thermal ageing after incorporation of coir and particulates. Declination of 24.52%, 75.53%, 72.33% and 57.37% were observed after addition coir, cow dung, wheat husk and rice husk respectively. With the increase in particulate content decrease in tensile strength was observed.

Adverse effect on mechanical properties of natural fiber reinforced polymer composites were also reported by some scientists (Chen et al., 2009; Wang et al., 2006). Mechanical properties of natural fiber reinforced polymer composite are considerably degraded after water exposure (Thwe and Lao, 2002). Water diffuses through the matrix and reaches the interface region and reinforcement. Dissolution of matrix, debonding of fiber/matrix interface and degradation of fiber due to water exposure may lead to reduction in mechanical properties of composite (Ghavami et al., 1999). Reduction in tensile properties has ranged from 60-90% in case of cow dung based composite and 30-70% in the case of wheat husk and rice husk based composite. Neat epoxy and coir-epoxy composite showed 10.53% and 20.07% reduction in tensile strength after high temperature water exposure respectively. Reduction in tensile properties has ranged from 70-90% in case of cow dung based composite and 42-68% in the case of wheat husk and rice husk based composite.

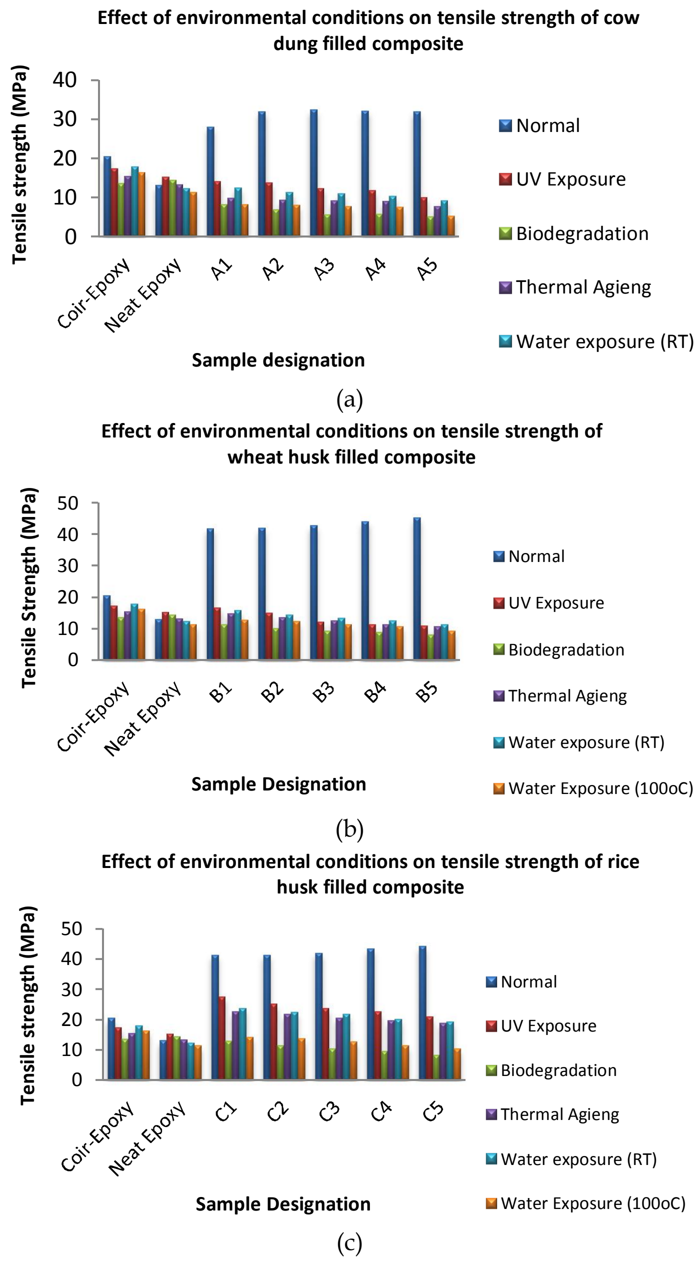


Figure 2. Effect on tensile strength of coir-epoxy composite samples filled with different particulates (a) cow dung (b) wheat husk (c) rice husk after exposure to different environments.

3.2.2. Flexural strength

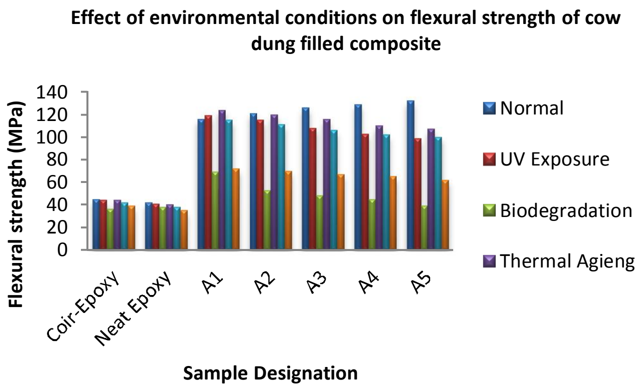
Figure 3 presents effects on the flexural strength of coir fiber reinforced epoxy composites filled with different particulate fillers after exposure to different environments such as ultra violet radiations, soil, high temperature, water at room temperature and elevated temperature. There was 3.28 % decrease in flexural strength of neat epoxy after UV exposure was observed. After addition of coconut coir 4.72% decrease in flexural strength was observed.

The incorporate ion of organic particulate fillers in coir-epoxy composite resulted in a more rapid declination of 25%, 23.81% and 20.88% in flexural strength of cow dung, wheat husk and rice husk based composites after UV exposure was observed. Neat epoxy shows 10.36% degradation after soil burial on flexural strength. The addition of coir in epoxy shows the slight declination of around 20.41% on flexural strength, but incorporation of cow dung shows 70.46% decline in flexural strength. In case of wheat husk and rice husk 65.99% and 61.01% declination in flexural strength was seen after 90 days of soil burial analysis. After the thermal ageing noticeable decrease in flexural strength was observed. 5.64% declination in flexural strength of neat epoxy after thermal ageing was seen.

The polymer becomes more susceptible to thermal ageing after incorporation of coir and particulates. Declination of 7.72%, 18.94%, 17.77% and 16.56% were observed after addition coir, cow dung, wheat husk and rice husk respectively. With the increase in particulate content decrease in flexural strength was noticed. The most prominent decrease of 6.36%, 7.17%, 24.25%, 22.58% and 35.23% in flexural strength of composite occurs after water exposure were observed in a neat epoxy, coir-epoxy, cow dung, wheat husk and rice husk composite respectively. The most major reduction of 13.78%, 17.44%, 53.04%, 50.55% and 51.98% in flexural strength of composite occurs after water exposure at 100 ^{o}C were observed in a neat epoxy, coir-epoxy, cow dung, wheat husk and rice husk composite respectively.

3.2.3. Impact strength

Figure 4 presents effects on impact strength of coir fiber reinforced epoxy composites filled with different particulate fillers after exposure to different environments such as ultra violet radiations, soil, high temperature, water at room temperature and elevated temperature. There was a minor decrease of 7.64%, 8.75%, 60.99%, 59.07% and 56.8% in impact strength of neat epoxy, coir-epoxy, cow dung, wheat husk and rice husk composites was observed after UV exposure. With increase in particulate fraction impact strength of composite was also found to be decreasing. There was a significant decrease in impact strength was observed after addition of particulate fillers in coir-epoxy composite after 90 days soil burial analysis.



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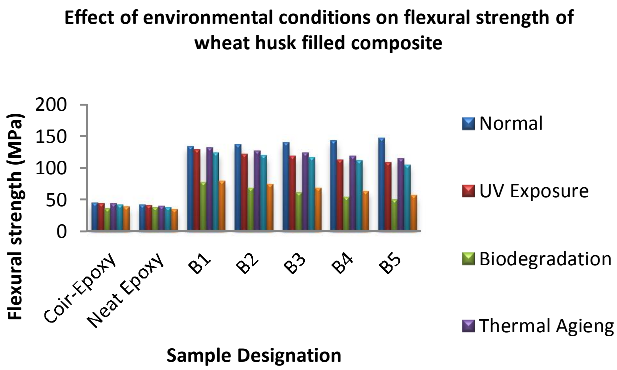

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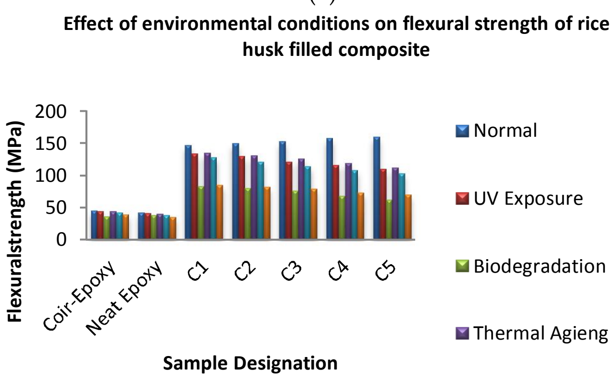

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Figure 3. Effect on the flexural strength of coir-epoxy composite samples filled with different particulates (a) cow dung (b) wheat husk (c) rice husk after exposure to different environments.

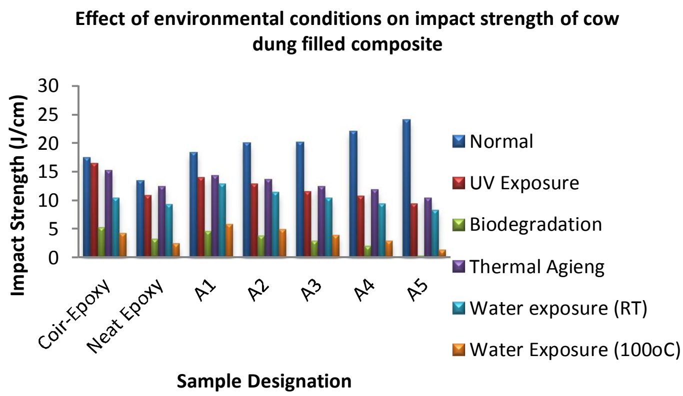
With increase in filler loading impact strength of composite was also found to be decreasing. There was a significant decrease of 15.76%, 43.44%, 98.44%, 94.57% and 88.39% in impact strength of neat epoxy, coir-epoxy, cow dung, wheat husk and rice husk composites was observed after soil burial analysis. Result showed noticeable decrease in impact strength after thermal ageing. 12.87% declination in tensile strength of neat epoxy after thermal ageing was observed.

The polymer becomes more susceptible to thermal ageing after incorporation of coir and particulates. Declination of 11.01%, 56.41%, 55.07% and 52.89% were observed after addition coir, cow dung, wheat husk and rice husk respectively. With increase in particulate content decrease in impact strength was seen. Cow dung based composite demonstrated a greater decrease of about 50% in impact strength than wheat husk and rice husk composite. This is because cow dung contains highly hydrophilic substance nitrocellulose for which its water absorption ability is superior to wheat husk and rice husk (Fakhrul and Islam, 2013). After water absorption at room temperature significant decrease of 18.52%, 20.7%, 65.31%, 61.99% and 59.56% in impact strength of neat epoxy, coir epoxy, cow dung, wheat husk and rice husk based composite was observed respectively. Cow dung based composite established a greater decrease of about 60% in impact strength than wheat husk and rice husk composite after exposure to water at 100^{o}C. This is because the superior water absorption capability of cow dung due to the presence of highly hydrophilic substance nitrocellulose. After water absorption at 100^{o}C major decrease of 33.31%, 54.25%, 94.32%, 90.19% and 87.15% in impact strength of neat epoxy, coir epoxy, cow dung, wheat husk and rice husk based composite was observed respectively.

3.3. Microstructural analysis

Degradation in composites is confirmed by microstructural analysis before and after exposure to different environmental conditions. Figure 5 represents the microstructures of coir-epoxy infused cow dung composite before and after exposure to different environmental conditions.

Microstructural analysis before degradation showing densely embedded fiber and particulate in epoxy matrix with epoxy surrounding completely the reinforcement and particulate which maintain their circularity and suggest good strength (Figure 5a). After microbial attack lumping of epoxy is clearly visible (Figure 5b), the lumping of epoxy causing hardening of epoxy. After UV exposure micrograph indicate degradation in epoxy (Figure 5c) and formation of voids in composite which also causing reduction in strength. After thermal ageing micrograph indicates delamination of reinforcement and voids in composite (Figure 5d). After soil burial analysis micrograph showed markedly damage and voids in composite (Figure 5e).



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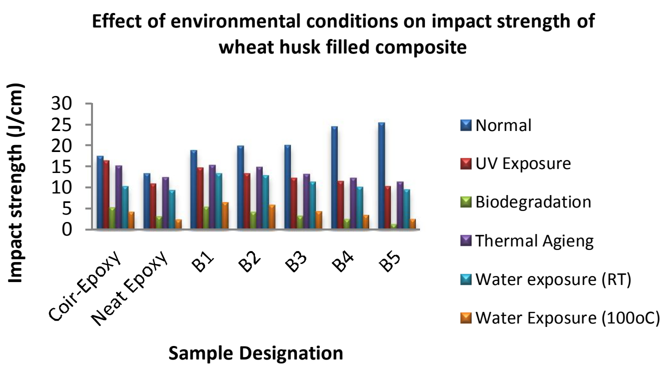

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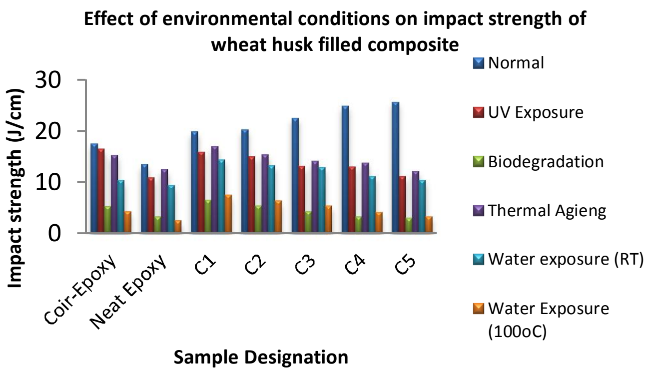

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Figure 4. Effect on impact strength of coir-epoxy composite samples filled with different particulates (a) cow dung (b) wheat husk (c) rice husk after exposure to different environments.

Figure 6 represents the microstructures of coir-epoxy infused wheat husk composite before and after exposure to different environmental conditions. Microstructures of coir-epoxy infused wheat husk composite before degradation showing densely embedded fiber and particulate in epoxy matrix with epoxy surrounding completely the reinforcement and particulate which maintain their circularity and suggest good strength (Figure 6a). After microbial attack lumping of epoxy is clearly observed, the lumping of epoxy causing hardening of epoxy (Figure 6b). After UV exposure micrographs clearly indicate degradation in epoxy and the formation of cracks in composites which also causing declines in strength (6c). After thermal ageing micrographs indicates extensive degradation of epoxy, formation of cracks and voids in composite (Figure 6d). After soil burial analysis micrograph markedly indicates smearing of the surface and formation of voids in composite (Figure 6e).

Figure 7 represents the microstructures of coir-epoxy infused rice husk composite before and after exposure to different environmental conditions. Microstructures of coir-epoxy infused rice husk composite before degradation showing tightly entrenched fiber and particulate in epoxy matrix with epoxy surrounding entirely the reinforcement and particulate which retain their circularity and suggest good strength (Figure 7a). After microbial attack lump formation in epoxy is clearly seen, the lumping of epoxy causing hardening of epoxy and reduction in mechanical properties (Figure 7b). After UV exposure micrographs clearly indicate clumping in epoxy and the formation of voids in composite which also causing a decrease in strength (Figure 7c). After thermal ageing micrograph indicates matrix degradation and formation of voids in composite (Figure 7d). After soil burial analysis micrograph noticeably indicate damage and formation of voids in composite (Figure 7e).

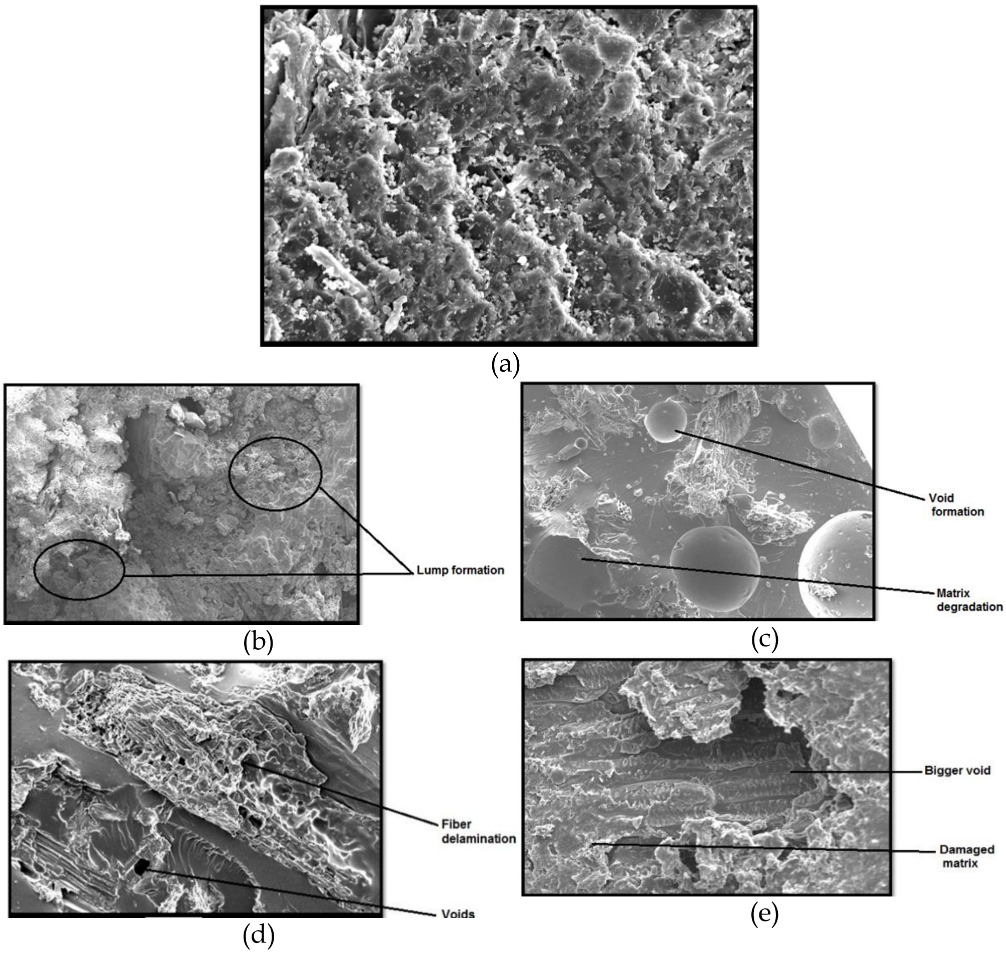

Figure 5. Microstructures of coir-epoxy filled cow dung composite (a) Before degradation (b) microbial degraded (c) UV exposed (d) thermal degraded (e) biodegraded.

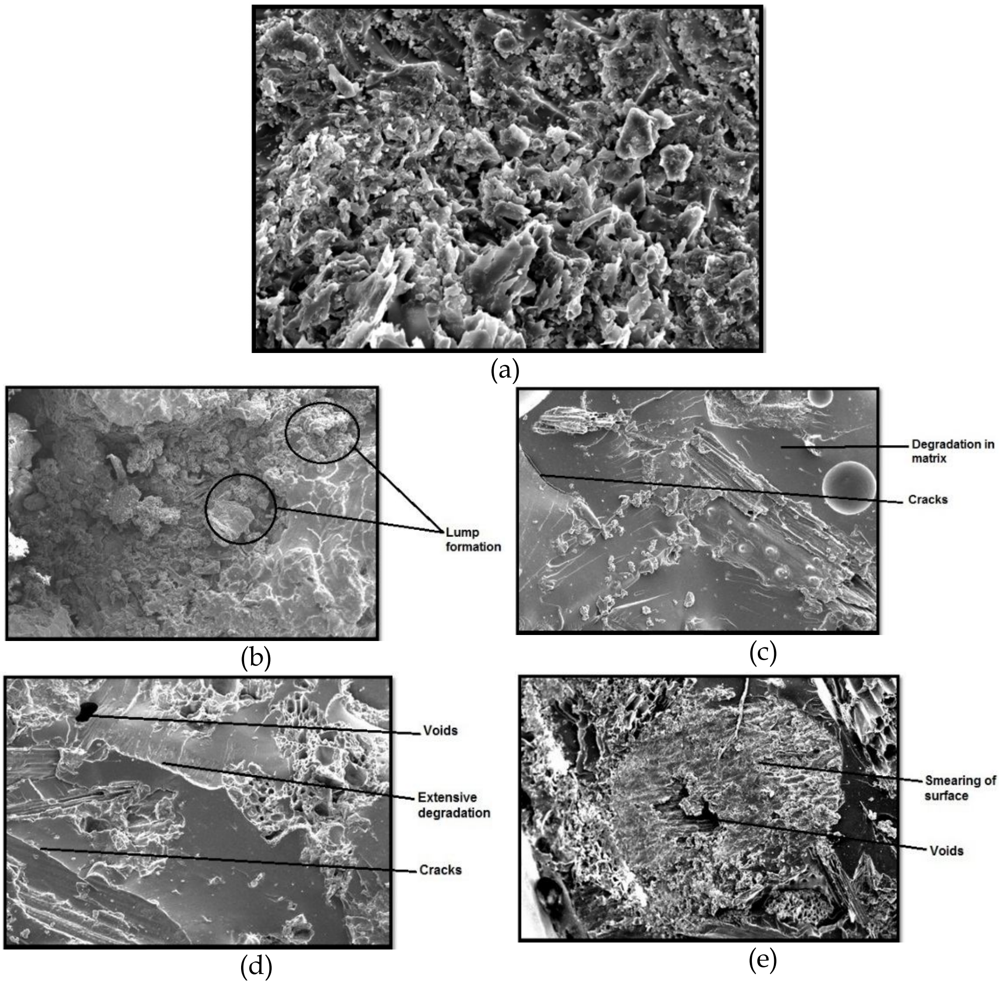

Figure 6. Microstructures of coir-epoxy filled wheat husk composite (a) before degradation (b) microbial degraded (c) UV exposed (d) thermal degraded (e) biodegraded.

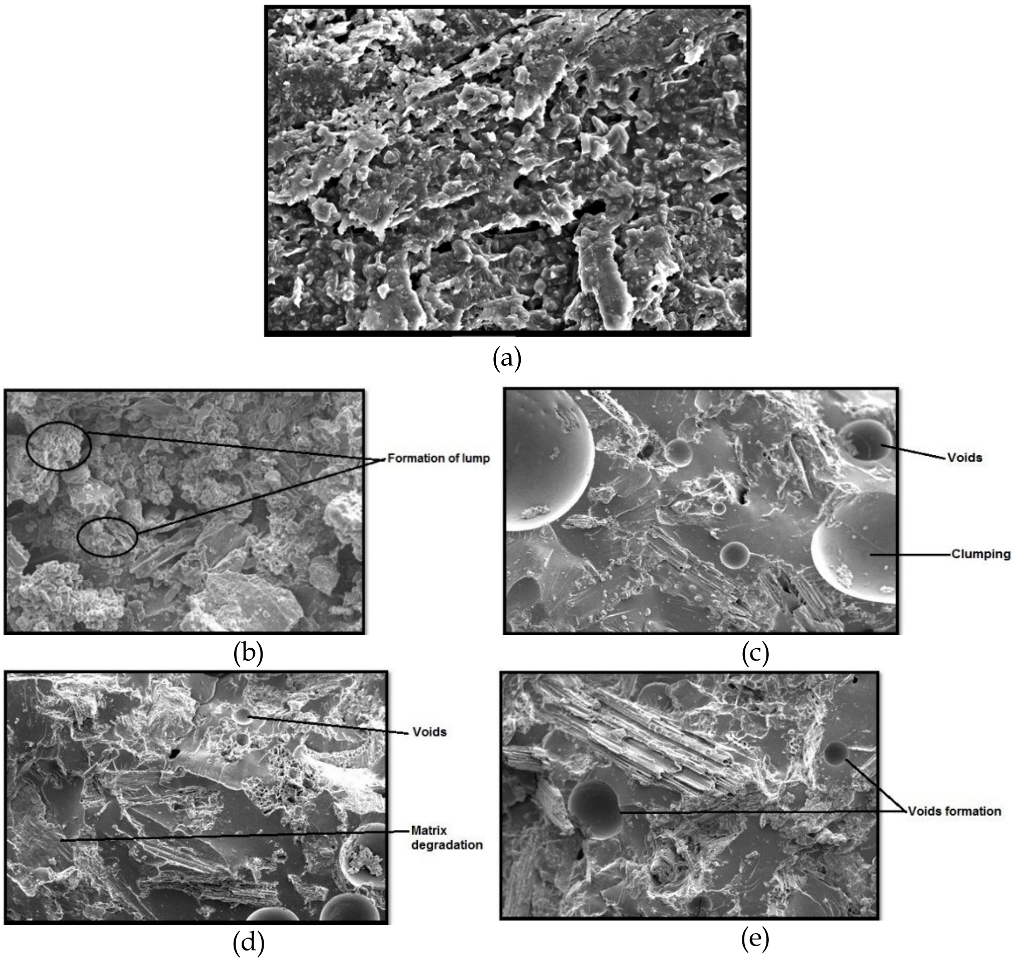

Figure 7. Microstructures of coir-epoxy filled rice husk composite (a) Before Degradation (b) Microbial degraded (c) UV exposed (d) Thermal degraded (e) Biodegraded

4. Conclusions

Natural lignocellulosic fillers are susceptible to degradation, thus composites based on them face higher risk of degradation when subjected to outdoor applications as compared to composites with synthetic fillers. Different components of lignocellulosics fillers have different influence on their properties and degradability. For instance, cellulose is responsible for strength, hemicelluloses for thermal, biological and moisture degradation, while lignin for UV degradation and char formation.

Effect of various environmental conditions such as UV radiations, biological degradation, microbial attack, chemical exposure, water exposure at room temperature and high temperature on microstructure, mechanical properties and chemical structure of coir-epoxy infused particulate composite has been reported. While the primary focus of this study has remained on degradation of material in different environmental conditions and also attempted at the same time to open up a new pathway for value added utilization of cow dung and agricultural wastes (wheat husk and rice husk).

Lignocellulosic particulate filled coir fiber reinforced epoxy composites are most susceptible to soil burial analysis and showing the major degradation of about 55, 40 and 35% in cow dung, wheat husk and rice husk filled composites respectively. Presence of natural lignocellulosic fillers is the major factor affecting water absorption of composites as it enhances matrix porosity by creating more moisture path into the matrices. Poor adhesion between filler particles and the polymer matrix generates void spaces around the filler particles causing degradation of about 15-20% at room temperature and about 20-25% at 100^{o}C.

Higher fiber volume composites immersed in water generally have greater decrement in tensile and flexural properties compared to dry samples. Exposure of particulate filled coir-epoxy composites to high temperature showed degradation of about 19-24%. Outdoor weathering causes degradation of polymer composite through photo-radiation, thermal degradation, photo-oxidation and hydrolysis. These processes result in changes in their chemical, physical and mechanical properties. Water enhances the rate of degradation through swelling of fiber and this leads to further light penetration which resulted decrease in mechanical properties. After exposure to UV radiations about 9-11% degradation was observed in particulate filled coir-epoxy composites. Order of susceptibility of particulate filled coir-epoxy composites after environmental exposure: - Soil burial analysis > Thermal ageing > Water at 100^{o}C > Water at room temperature > UV radiations.

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