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Coir-fiber-based fire retardant nano filler for epoxy composites

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Abstract Coir-fiber-based fire retardant nano filler has been developed for epoxy resin (ER). At first, the coir fiber was brominated with saturated bromine water and then treated with stannous chloride solution. After drying, it was grinded to nano dimension and mix well with ER for composites preparations. FTIR, DSC, and TG techniques were used to characterize the brominated coir fiber. Gravimetric analysis shows only 10% by mass of bromination on coir fiber. Bromination decreases the thermal stability of the coir fiber, but it does not affect the final stability of the composites. This study concentrates on the thermal, fire retardant, and morphological properties of nanocomposites prepared by direct mixing. The fire retardancy properties (smoke density and limiting oxygen index) of coir–epoxy nanocomposites have increased significantly.

Keywords Coir fiber · Bromination · Epoxy · Thermal · Fire retardant

Introduction

The behavior of materials in a fire depends on several factors such as ease of ignition, flame spread, fire endurance, rate of heat release, ease of extinction, smoke evolution, and toxic gas evolution. The reduction of any of the above parameters than base material is defined as fire retardant material [1]. Statistical studies of fire death in the world demonstrated that 80% of fire fatalities are due to fire effluents (smoke and toxic gas removal) [2, 3]. Epoxy resins (ER) are one of the popular materials to produce plastics in many application areas, e.g., building construction, transport, recreation, electronic industries, etc. One of the biggest disadvantages of ERs is their flammability and production of smoke in fire. Hence, chemical composition has been modified in technological processes to change simultaneously flammability properties and smoke release of plastic materials to expand their application areas.

Rose et al. [4] have studied the different steps in the thermal oxidative degradation of an ER and predicted the degradation mode. They have proposed that the degradation leads to the formation of a surface carbonaceous material, which may take part in the protection of the resin. They have shown that the oxygen plays a part in the formation of a suitable carbonaceous material in the temperature range of 590–770 K and then in the degradation of the material in the temperature range 770–920 K. The following literature comprises many experimental data referring to reducing the intensity of smoke of ER [1, 5, 6].

Flame retardants can be incorporated into polymeric materials either as additive or as reactive materials. Additive type flame retardants are widely used by blending with polymeric materials, but this creates problem of poor compatibility, leaching and reduce mechanical properties. Reactive flame-retardants are used to overcome this problem. However, these are costly and used for high-end applications like printed circuit board and airline industries.

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A major use of ER is in the manufacture of glass-reinforced printed circuit boards. The principal component for the fire retardant application is diglycidyl ether of tetrabrominated bisphenol A (TBBPA), which is often mixed with antimony oxide for fire retardant properties [7]. Despite the primary use of TBBPA as a reactive flame retardant (covalently bond to the polymer), it has been observed in the environment [8]. The key concern with TBBPA is its similarity in chemical structure to thyroxine [9]. In vitro analysis shows that TBBPA is stronger affinity for binding with the thyroid hormone transport than the natural ligand. Hence, it is restricted in production in many countries. Therefore, a development of an alternative low-cost reactive flame-retardant material is necessary for ER.

Antimony oxide and halogen containing flame-retardants are classical in flame retardancy field. During fire antimony oxide and halogen additive interact to make antimony trihalide. The antimony trihalide partially vaporizes into the flame zone, where it dissociates giving halogen atoms, which scavenge H atoms, O atoms, and OH free radicals. Also, it forms blanket over the burn surface due to high density and removes oxygen from the propagation front [1].

The problem of antimony oxide with halogenated ERs is the dispersion of antimony oxide at the molecular level and the efficiency of the system. The melting point of antimony (III) oxide is 928 K. At the flame temperature, it should react with halogen-free radicals to form antimony halide. For this, halogen vapors should come in contact with antimony oxide for reaction. In this process, there is less possibility of 100% use of antimony oxide as well as for halogen vapors.

We have designed a system which can hold both the halogen and metal compounds covalently. Then, this matrix was grinded to smaller dimensions (nano range) and mixed well with ER. Due to the potential toxicity of antimony, we have used tin compounds for synergistic effects [10]. Bromine and chorine compounds are most generally used for halogen-containing fire retardants. Out of that aromatic bromine compounds are most effective than aromatic chlorine compounds. This has been observed that the synergistic results have been found with combination of chlorine and bromine. The maximum effect was found at a Cl:Br atomic ratio of 1:1 [11].

In this study, we have chosen coir fiber for matrix substances. Coir fiber is a lignocellulosic material having ~46% lignin and 43% cellulose [12]. Lignin is a phenolic compound, which can react easily with bromine [13], and then it is reacted with stannous chloride solution. The possible reaction scheme is as follows.

Coir fiber (Lignin) + Bromine water → Brominated coir fiber (Brominated phenol)

Coir fiber (Cellulose) + Bromine water → Oxidation on fiber surface

Modified coir fiber + Stannous Chloride → $[Sn(Cl)_2$ (Cellulose –OH)]

Tin(II) can accept electron pair through 5p and 5d orbitals, also it can donate electron from $5s^2$ orbital. Due to this reason, Tin(II) has catenation property into larger structural units such as rings or chains [14]. Finally, the fiber was grinded to nanodimensions and mixed thoroughly with ER for composite preparations. Here, we have studied thermal, fire retardant, and morphological properties of coir–epoxy composites.

Experimental

Epoxy resin (araldite) and hardener were obtained from Huntsman advanced materials, Delaware, USA. Coir fiber was purchased from local market for fire retardant filler preparations. Stannous chloride of AR grade was purchased from CDH chemicals, Bombay, India. Liquid bromine was purchased from SD Fine chemicals, Bombay, India, for bromination of coir fiber.

Coir fiber was first segregated from spongy material, and dewaxing of coir fiber was done in a soxhlet apparatus with a mixture of 4:1:1 (by volume) toluene, methylated spirit, and acetone for 4–5 h [15]. The extracted fiber was kept overnight in air oven at 380 ± 5 K for drying. Dewaxed coir fiber was grinded to smaller dimension (pass through 70 mesh size of ASTM standard) and was used for untreated coir–epoxy composite (ERC) preparation. The sample nomenclature is shown in Table 1.

The dewaxed coir fiber was brominated with saturated bromine water for 24 h. After that the fiber was removed and washed thoroughly with tap water for several times

Table 1 Nomenclature of coir fiber, brominated coir fiber, nano powder, cured epoxy resin, coir–epoxy composites, and nano powder epoxy composites which are used in this report

Sample descriptions	Abbreviations
Dewaxed coir fiber	Coir
Brominated Coir fiber	BC
Stannous chloride treated brominated coir fiber (nano powder)	SBC
Amine cured epoxy resin	ER
Amine cured dewaxed coir powder epoxy composites	ERC
Amine cured stannous chloride powder epoxy composites	ERSN
Amine cured nano coir powder (SBC) epoxy composites	ERSBC

(until it is free from acid) and dried in air oven at 380 ± 5 K.

Tin powder of 0.72 g was dissolved in dilute hydrochloric acid. The above brominated coir (BC) fiber was mixed with this stannous chloride solution and boiled until it is evaporate to dryness. Then, the mixture was dried in oven at 380 ± 5 K. In this drying process, coir fiber becomes black. This may be due to the formation of some tin oxide during heating in atmosphere [16]. This black fiber was grinded to powder (defined as nano powder) (SBC), and the particle size was measured with Malvern particle size analyzer (Nano ZS model). The particle sizes were approximately 800–900 nm ranges and were used for preparation of coir–epoxy nano composite (ERSBC).

Fourier transform infra red (FTIR) spectra of dewaxed coir fiber, BC fiber, and stannous chloride treated BC fiber were studied in Shimadzu IR prestige 21 model by diffuse reflectance spectroscopy technique.

The differential scanning calorimetry (DSC) of dewaxed coir fiber and BC fiber was studied in Waters DSC 10 model from 225 to 670 K. The DSC of cured epoxy and its composites were studied from room temperature to 770 K. In all the cases, heating rate was 10 K min^{-1}, and the atmosphere was nitrogen.

Thermogravimetry (TG) of coir fiber, modified coir fiber, and their epoxy composites were carried out in Shimadzu DTG 60 model from room temperature to 970 K at the heating rate of 10 K min^{-1} in nitrogen atmosphere.

Liquid epoxy resin (Araldite) and hardener (amine) of mass ratio (1:0.8) were taken in a plastic container and mixed thoroughly for 10 min. Then, it was poured into a plastic mold and kept at room temperature for 24 h. After that the pellet was removed from the mold and heated at 380 ± 5 K in air oven for 3 h. Compositions and sample nomenclature are shown in Table 2.

Smoke density was carried out in a Platon smoke density chamber from S.A. Associates, New Delhi, designed to meet ASTM D-2483 flammability test requirement. The sample dimensions were 2.54 × 2.54 × 0.5 cm^3, and the experimental procedure was as follows. The sample was kept on a wire net and ignited by a flame of 40 psi pressure keeping the burner at an angle of 45°. Smoke generation is measured as obscuration posed by smoke to a light source. For this purpose, a bulb of 5.80 W was used in the chamber. Obscuration was noted after every 15 s with an alarm sound. Sixteen readings were noted, and each test takes 4 min for completion. The total smoke produced is determined by measuring the area under the curve, and this area in percent is used for the smoke density rating makes a visual comparison of smoke density. The maximum smoke density is taken as maxima on the smoke density vs. time curve.

Table 2 Epoxy resin composites with coir powder, nano powder, and stannous chloride powder are prepared with the following compositions

Sample designation	Epoxy/g	Hardener/g	Filler/g
ER	20	14	0
ERC	20	14	1.6 (Dewaxed coir)
ERSBC	20	14	1.6 (Nano powder)
ERSN	20	14	0.24 ($SnCl_2$)

Limiting Oxygen Index (LOI) is the minimum percentage of oxygen in an atmosphere of oxygen–nitrogen mixture (by volume), which is required to sustain candle like burning of the specimen for 3 min and is calculated using the following formula: LOI = oxygen/(oxygen + nitrogen) × 100.

In this test, specimen was clamped at the bottom in a closed chamber. The sample was ignited at the upper part of the sample. The flow rate of the mixture of gases was at 10.6 L min^{-1}. Electronic digital meter connected with the instrument gives the percentage of oxygen in the mixture. Five samples were tested for each sample, and an average value was reported.

The morphological properties of the composites were studied to investigate the state of dispersion of fiber particles and the interaction between the filler and matrix interface. The samples were coated with gold prior to scanning. The scanning electron microscopy (SEM) micrographs were recorded on a Carlzeiss EVO50 scanning electron microscope.

Results and discussion

Characterization of coir fibers

Coir fiber possess substantial amount of cellulose (~43%), which is a natural polymer made of β glucose units with abundant primary and secondary hydroxyl groups. It also possesses lignin (~46%) having a network type of structure with phenolic hydroxyl groups. Bromine water can react both as lignin and carbonyl of sugar unit. To our knowledge, no literature is available on the surface modification of coir fiber by bromine water. The SEM picture of coir fiber shows that lot of porosity is present on the surface of the coir fiber [17]. Bromine water can easily enters into the pores and react with the inner layer. Gravimetric analysis of BC fiber shows 10% by mass of bromination on the coir fiber. FTIR, DSC, and TG method were used to characterize the coir fiber.

Fourier transform infra red spectroscopy (FTIR)

Figure 1 is the FTIR spectra of dewaxed coir, BC, and stannous chloride treated brominated coir fiber (SBC). The spectral behavior of coir fiber changes after reaction with bromine water. The peak near 3600 cm^{-1} is due to the phenolic–OH group, which reduces after bromination. Also, the carbonyl group intensity near 1720 cm^{-1} reduces after reaction with bromine water. This indicates that the reaction has taken place on the fiber surface.

However, the formation of carbon halogen bond is not prominent. This may be due to the small amount of reaction on the fiber surface. Meng et al. [18] also could not observe the carbon bromine bond by FTIR spectra on the cellulose surface although it was present on the cellulose surface. The SBC shows broad peak above 3000 to 3400 cm^{-1}. This is due to the presence of some amount of hydrated stannous chloride. The broad peak for SBC sample near 605 cm^{-1} is due to the metal halogen bond.

Differential scanning calorimetry (DSC)

Figure 2 is the DSC curve of dewaxed coir fiber, BC fiber, and SBC fiber. The dewaxed coir fiber shows three peaks: one endothermic peak near 370 K and other two exothermic peaks near 560 and 625 K. The endothermic peak is mostly due to adsorbed moisture and the exothermic peaks are due to the degradation of the coir fiber. On the other hand, BC fiber shows a broad endothermic peak near 400 K. This is due to the adsorbed moisture on fiber surface. The broad peak is due to the change in surface composition after bromination. In case of BC fiber, the other endothermic peak comes near 520 K and above 570 K the graph shows exothermic behavior but no peak has been observed. In all the cases, the heat change for BC fiber is small compare to coir fiber.

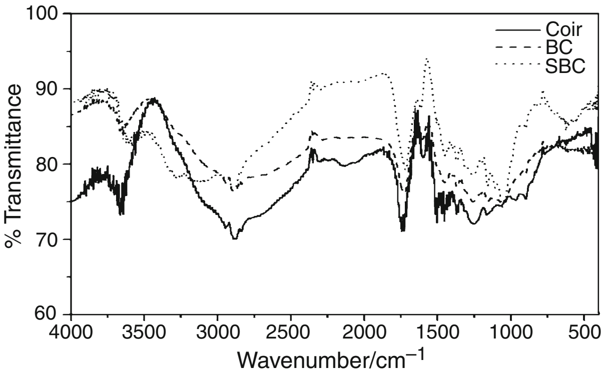

Fig. 1 FTIR spectra showing the difference in peak position and intensity of dewaxed coir, brominated coir, and stannous chloride treated brominated coir fiber

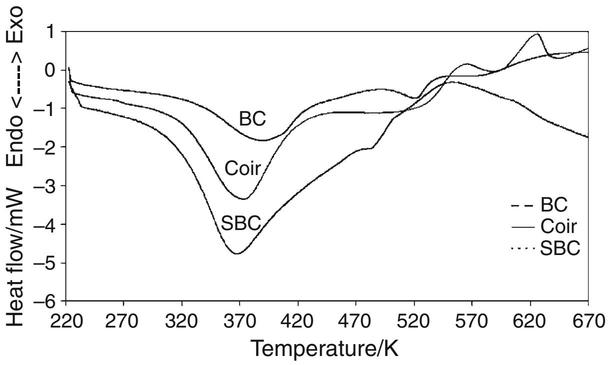

Fig. 2 DSC graph shows the different exothermic and endothermic transition of coir fiber, brominated coir fiber (BC), and stannous chloride treated brominated coir fiber (SBC)

The thermal behavior of SBC fiber is different from others. Only one broad endothermic peak observes near 370 K, which is due to the presence of moisture in the sample. The most interesting point is that at higher temperature (above 560 K), the curve shows only endothermic transition, where as in other cases the degradation is exothermic. This indicates low amount of heat evolve during degradation. This is favorable for fire retardant properties.

Thermogravimetry (TG)

Figure 3 is the TG of coir fiber; BC fiber, and SBC fiber. All the samples show the initial mass change from room temperature to 350 K. This is due to the adsorb moisture present in the sample and is endothermic in nature which is observed in DSC curve. In case of coir fiber, the degradation starts above 500 K, where as the other sample starts degrading near 480 K. This indicates that the bromination decreases the thermal stability of the coir fiber.

The degradation of all material is sharp up to 620 K, but after that the mass change is gradual. In case of coir fiber, a

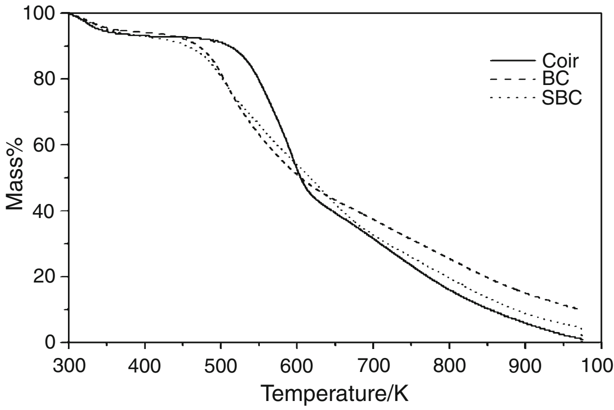

Fig. 3 TG plot showing the mass change at different temperatures of coir fiber (Coir), brominated coir fiber (BC), and stannous chloride treated brominated coir fiber (SBC)

comparatively steep change in mass loss takes place from 500 to 620 K. The similar thermal behavior was also reported for jute fiber [19]. The important point is that in this region the thermal change (DSC) is exothermic for coir fiber, whereas for BC fiber and SBC fiber it is endothermic. Above 620 K, the degradation of coir fiber is faster compare to others, and the residue is almost zero. The residue at 920 K is higher in case of BC fiber than coir fiber. This indicates more char product is formed for brominated sample. This is also favorable for fire retardant properties.

Properties of coir–epoxy composites

DSC of coir–epoxy composites

Figure 4 is the DSC curve of amine cured ER, coir fiber epoxy composite (ERC), and coir-fiber-based nano filler epoxy composites (ERSBC). This shows large thermal change of ERC sample than ER and ERSBC samples. This may be due to large sizes of the coir fiber and the presence of moisture in the sample. In the entire cases, ERSBC sample is showing fewer changes in thermal behavior than pure epoxy composites. This may be due to the smaller dimension and low amount of moisture in the coir nano powder.

All the samples show endothermic behavior up to 700 K, but after that exothermic behavior is observed. First endothermic transition near 370 K is due to the glass transition temperature of epoxy matrix. The second endothermic transition near 500 K is due to the removal of water through dehydration of secondary alcohol group. The third endothermic transition is near 630 K is due to the degradation of network structure [4].

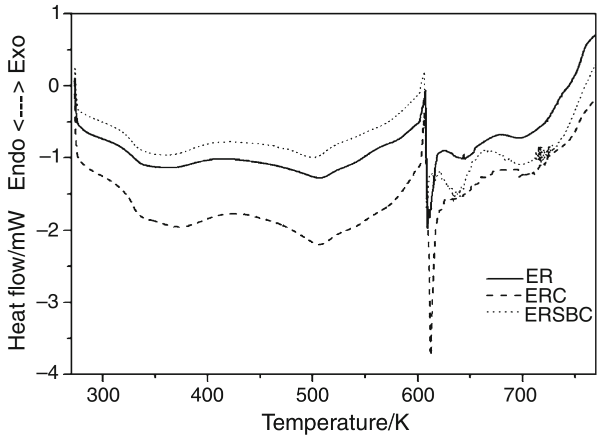

Fig. 4 DSC plot showing the exothermic and endothermic transition at different temperatures of amine cured epoxy (ER), coir fiber epoxy (ERC), and nano powder epoxy (ERSBC) composites

TG of cured coir–epoxy composites

Figure 5 is the TG curve of amine cured pure epoxy (ER), coir fiber epoxy (ERC) and nano powder epoxy (ERSBC) composites. In all the cases, mass change takes place in two major steps. The first step is up to 600 K and another is from 600 to 750 K. The similar behaviors are also reported in the literature [4, 20]. The first step involves elimination of water through dehydration of secondary alcohol groups and formation of unsaturated structures, which are endothermic in nature in DSC curve. The unsaturation generates weak aliphatic C–O and C–N bonds in the β position, which breaks down giving formation of phenolic chain ends and secondary amine terminal functions, respectively. In the second step of thermal decomposition with a maximum near 670 K, scission of the aliphatic C–N and C–O, together with C–phenyl bonds of bisphenol A leads to almost complete volatilization. A small amount of char residue generates, which completely volatilizes at higher temperature. Literature reported that the inorganic filler change the decomposition behavior of ER [21, 22].

All the graphs are almost similar up to 670 K. However, after that ERSBC sample (fire retardant composite) shows slow degradation than others. Also the residue above 750 K is higher in case of fire retardant composite than pure epoxy. This indicates fewer amounts of volatiles generates during fire. The interesting point is that the thermal stability of all the composites is same. Although bromination reduces the thermal stability of the coir fiber, however, it is unaffected in the composites.

Fire retardant properties

Table 3 is the fire retardant properties of epoxy composites. Two different types of fire retardant properties are investigated. One is smoke density and another is LOI. Lower smoke density and higher LOI are preferable for fire retardant applications. The smoke density of nano filler

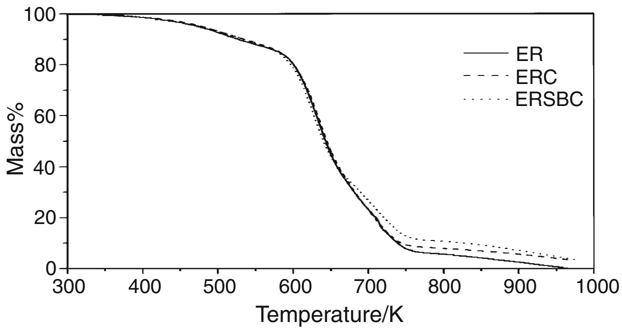

Fig. 5 TG plot showing the mass change at different temperatures of amine cured epoxy (ER), coir fiber epoxy (ERC), and nano powder epoxy (ERSBC) composites

Table 3 Smoke density of nano powder reinforced epoxy (ERSBC) composites decreases, whereas LOI values of nano powder reinforced epoxy composites increases than amine cured epoxy resin (ER) and Stannous chloride containing epoxy resin (ERSN)

Sample designation	Smoke density/%	LOI/%
ER	47	31
ERSN	50	36
ERSBC	34	39

epoxy composite (ERSBC) is 27% lower than epoxy amine network (ER). On the other hand, the smoke density of ERSN (only stannous chloride containing epoxy) sample is higher (6%) than epoxy (ER) sample. Hence, only stannous chloride cannot be used directly as fire retardant applications.

The LOI value of both the ERSN and ERSBC samples increases than the pure ER. The LOI value for ERSN is 16% higher than ER sample, where as the LOI value for fire retardant (ERSBC) nano filler is 26% higher than pure epoxy composites (ER). From smoke density and LOI value, it may be considered that tin containing BC fiber (nano filler) can be used as fire retardant material for ER.

Generally, the bromine content in electronic board is 6–7% by mass of bromine [23]. Here, the nano composite (ERSBC) contains 0.45% by mass of bromine and 0.32% by mass of tin (which is equivalent to 0.41% by mass of tin oxide). Therefore, coir-fiber-based fire retardant nano filler (ERSBC) is efficient fire retardant material.

Morphological properties of the coir–epoxy composites

In order to study the morphology and interfacial adhesion of filler with matrix of composites, the SEM studies were carried out. Figure 6 is the SEM picture of nano powder (ERSBC) epoxy composite. This shows that the fiber distributed equally throughout the matrix. Figures 7, 8, and 9 are the SEM photographs of cross-sectional view of the fractured surface of the tensile specimens.

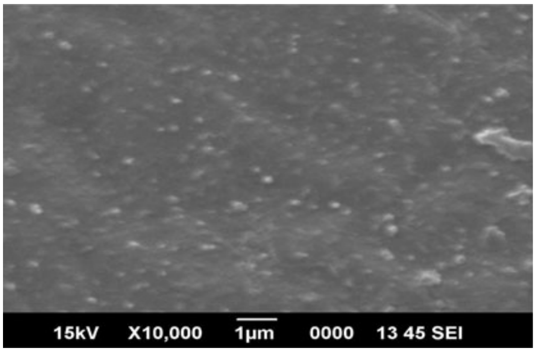

Fig. 6 SEM picture showing the dispersion of filler in coir–epoxy composites

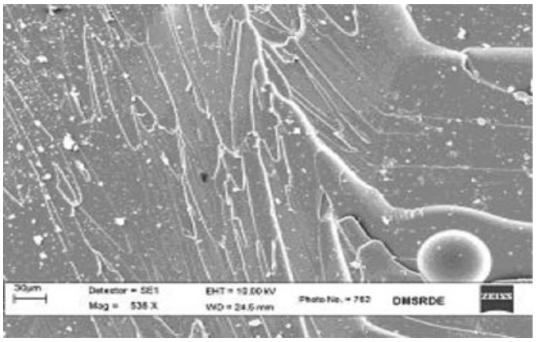

Fig. 7 SEM picture showing many crack lines of the fracture surface of epoxy resin (ER)

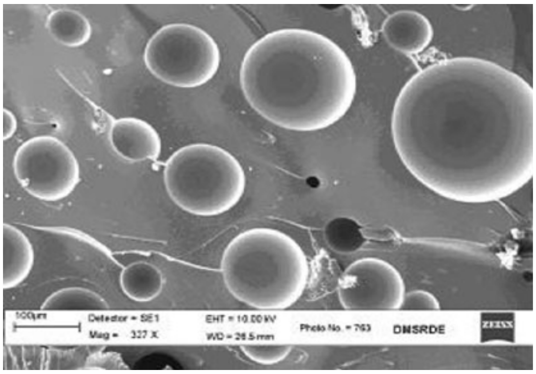

Fig. 8 SEM picture showing the deformation of fracture surface of coir fiber epoxy composites (ERC)

Epoxy resin is a brittle plastic, and hence, many crack lines are appeared in the fractured surface (Fig. 7). Introduction of filler (heterogeneous phase) into the brittle material decreases the crack lines (Figs. 8, 9), but in both the cases matrix deformation takes place. This indicates the strong adhesion between the filler and the matrix. This is expected as the filler contains large number of polar hydroxyl group, which can form bond with the hydroxyl and amine group of ER.

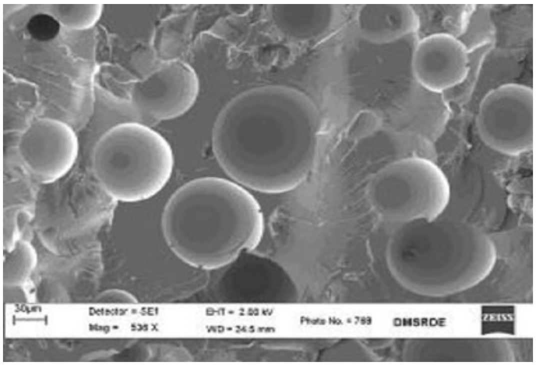

Fig. 9 SEM picture showing the deformation of fracture surface of nano powder epoxy composites (ERSBC)

Conclusions

The present investigation was undertaken with a prime objective of improving the fire retardant performance of ER composites with natural fiber employing simple chemical modification and surface treatment. The important findings of the present investigation are as follows.

FTIR spectra show the bromination has taken place on fiber surface. Thermogravimetry shows low amount of heat change during degradation of BC fiber and even lower for SBC fiber than normal coir fiber. Although the thermal stability of the BC fiber and SBC fiber are slightly lower than coir fiber, however, it is not affecting the overall thermal stability of the epoxy composites.

A strong interfacial adhesion takes place between the filler and the matrix. In spite of the small quantity of the bromine and tin in the final composite, the fire retardant properties of ER like LOI and smoke density properties improve significantly in the final composite. Based on these studies, it can be concluded that the coir-fiber-based fire retardant filler can be used as a fire retardant material for epoxy resin.

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