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Flame retardant diglycidylphenylphosphate and diglycidyl ether of bisphenol-A resins containing *Borassus* fruit fiber composites

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Abstract Natural fiber composites containing diglycidylphenylphosphate (DGPP) resin were prepared from DGPP, diglycidyl ether of bisphenol-A (DGEBA), and *Borassus* fruit fiber. Morphology, thermal, and mechanical properties of blends and fiber-reinforced composites were investigated. The tensile strength, flexural strength, and tensile modulus increased up to 10% addition of DGPP and decreased with high percentage of DGPP. The flexural strength of composites was increased up to 15% addition of DGPP due to good dispersion and toughening of DGPP in DGEBA. As observed by the SEM analysis, the matrix–fiber adhesion was poor in the case of 20% DGPP containing composites and failure occurred through fiber pullout whereas for composites with 5 and 10% of DGPP, interaction of fiber and matrix was strong and failure occurred through fiber breakage rather than fiber pullout. Addition up to 15% DGPP improved desired thermal and mechanical properties of these composites.

Introduction

Natural fiber containing polymer composites are of increasing interest in recent years, because of their low cost, low density, good mechanical properties, and ample supply of fibers from renewable resources [1–8]. As a consequence, many reports have focused on natural fiber-based composite materials [9–14]. Among them sisal, coir, pineapple, jute, and bamboo have gained attention of researchers [15–22]. Although preliminary studies on *Borassus* fruit fibers were reported in the literature [23], a significant study on coarse and fine *Borassus* fruit fibers was carried out by Obireddy et al. They have characterized these fruit fibers on morphology and mechanical behavior, and X-ray diffraction studies suggested that these fruit fibers can be utilized as reinforcement component for green composite [24]. *Borassus* (Palmyra Palm) is a genus of six species of fan palms, native to tropical regions of Africa, Asia, and New Guinea, economically useful and widely cultivated. The fruits are eaten either roasted or raw, and the young, jellylike seeds are also edible [25].

Natural fibers are organic materials and sensitive to flame and hence, improvement of flame retardancy of composite materials has become important to comply with safety requirements. The expeditious method used to acquire flame retardancy is the incorporation of flame retardants into epoxy matrix system that can interfere with combustion for satisfactory flame retardancy [26]. Among the various flame retardant systems, organophosphorus compounds were identified as better fire retardant materials in terms of environmental and health safety, which generate less toxic plume than halogen-containing counterpart. Most of them were incorporated as additives to operate individually or collectively in the matrix for enhancing fire retardancy along with other special characteristics like processability, plasticisability [27], thermal stability [28], and adhesion [29]. Nevertheless, additive type approach is associated with variety of problems, such as poor compatibility, leaching, reduction in mechanical properties, and weakened attraction. Therefore reactive type flame retardants were found to be more effective in fire retardant

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applications [30–43]. Recently, we have synthesized phosphorus-containing epoxy resin namely diglycidylphenylphosphate (DGPP) and used for the preparation of organophosphorus liquid crystalline thermosets and MMT-clay-based nanocomposites [44, 45]. This study deals with the investigation on the effect of DGPP content (blended with DGEBA) on flammability and mechanical properties of *Borassus* fruit fiber composites prepared using DGPP/DGEBA blends as matrix.

Experimental

Material

DGPP was synthesized based on the reported procedure [46]. *Borassus* fine fruit fibers (*Borassus flabellifer*) were extracted from dried ripened fruits (Fig. 1) and used as reinforcement. Epoxy resin was a liquid diglycidyl ether of bisphenol-A (DGEBA) (Ciba Geigy, Araldite LY 556) with an equivalent weight per epoxide group of 195 ± 5. The ambient temperature hardener used herein is an aliphatic polyamine (Ciba Geigy, HY 951).

Fabrication of blends and composites

The composites were prepared as follows: a glass mold covered by Teflon sheet having dimensions of 160 × 160 × 3 mm was used. A stoichiometric amount of LY 556 and various percentages (5, 10, 15, and 20%) of DGPP were thoroughly mixed with room temperature curative (HY 951), then the mold was loaded with matrix mixture and *Borassus* (chopped fiber length in the range of 1–1.5 cm) fruit fibers in random orientation. To minimize voids and avoid accumulation of excess matrix resin, the mold was squeezed using a hand roller [47]. The excess resin was removed and mold allowed to cure for 48 h at room temperature and finally post-cured in an oven at 100 °C for 4 h as post-curing at higher temperature is necessary for completion of reaction due to slow of diffusion process [44]. All samples were allowed to cool down gradually to room temperature. The blends of DGPP and DGEBA were prepared without fiber in the similar manner for comparison.

Thermal properties

Thermogravimetric analysis was performed on a Mettler TA 3000 thermal analyzer in a dry nitrogen atmosphere at a heating rate of 10 °C/min with sample weight of 3–5 mg in the temperature range of 35–700 °C. Curing studies and glass transition temperature (T_g) of the samples were performed on a DSC instrument (DuPont 910) at a heating rate of 10 °C/min, using a sample of about 3–5 mg, under nitrogen atmosphere in the temperature range of 35–350 °C.

Mechanical properties

Tensile and flexural strengths were measured on a standard computerized Instron 3369 Universal testing machine. The tensile strength was measured for 150 × 25 × 3 mm specimens at a cross-head speed of 5 mm/min as per ASTM D 3039-76. The flexural strength of samples with a size of 100 × 15 × 3 mm was measured at a cross-head speed of 2 mm/min as per ASTM D5943-96 in the three point bending mode. Impact test was performed employing

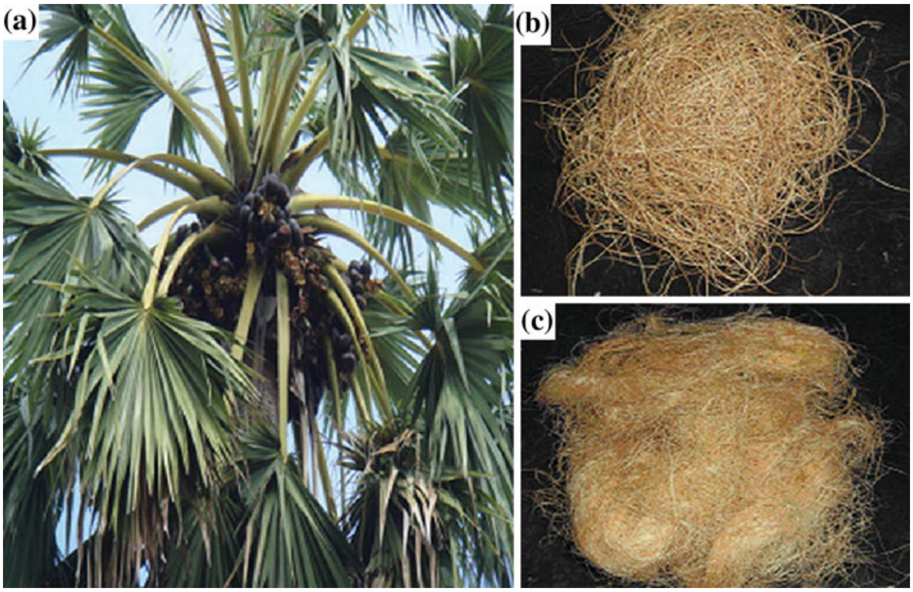

Fig. 1 Photographs of **a** *Borassus* tree with fruits; **b** *Borassus* coarse fibers; and **c** *Borassus* fine fibers

an Izod impact testing machine, supplied by International equipments, Mumbai, India. Rectangular strips of 122 × 13 × 3 mm were used as per ASTM 256-88 specifications. The impact test was carried out at room temperature and impact energy was reported in J m^{-1}. Five test samples were used for each test and the average value was recorded.

Morphology

The morphology of fractured surface of the samples was examined using a scanning electron microscope (SEM; JEOL JSM Model 6360). The fractured surface of the samples was sputter coated with gold (JEOL JFC-1600) an Auto Fine Coater before the fractographic examination.

Flammability test

Flammability of the samples was carried out by horizontal burning test and limiting oxygen index test as per ASTM D 635 and ASTM D 2863, respectively. In the horizontal burning test, the sample was held horizontally and exposed the flame from Bunsen burner one end of the sample for 30 s. The sample was allowed to burn until it either extinguished itself or burned past a gauge mark (100 mm) on the bar. If the sample did not burn past the 100 mm gauge mark, time and extent of burning were recorded. If it burns past the gauge mark, average burn rate, in centimeters per minute, was reported. In the limiting oxygen index test (LOI) a specimen was positioned vertically in a transparent test column and a mixture of oxygen and nitrogen was forced upward through the column. The specimen was ignited at the top end. The oxygen concentration was adjusted until the specimen just supported combustion. The concentration was recorded in volume percent. For each test three samples were used and average value was noted.

Results and discussion

The aim of this study was to develop flame retardant natural fiber composites by utilizing flame retardant epoxy resin (DGPP) of various weight percentages with commercial epoxy resin (DGEBA) and 5% by weight of *Borassus* natural fruit fibers as reinforcing materials using aliphatic polyamine as hardener. Chemical structures of the epoxy resins and hardener are shown in Fig. 2. Ten formulations were prepared, wherein five formulation blends of DGPP and DGEBA are named as A1, A2, A3, A4, and A5 and the remaining five fiber-reinforced composites are named as A6, A7, A8, A9, and A10. The details of all formulations are given in Table 1.

DSC analysis

To understand the processability and completion of curing of the blends and composites, DSC study was conducted for the formulations A1–A5. The thermograms are shown in Fig. 3 and the characteristic temperatures are given in Table 1. Thermograms showed the absence of residual exothermic peak indicating the completion of curing reaction. Table 1 shows that the T_g of the formulations (A2–A5) decreased with increase in DGPP content ascribed to plasticizing effect of DGPP arising by two factors, (i) is the pendant phenyl ring in DGPP which increases the distance between polymer chains and (ii) the weaker P–O–C bonds in DGPP. No abrupt change in T_g was noticed for samples A2–A5 as compared to blank sample A1. Thermal stability decreased with increase in DGPP content, clearly identified from exothermic peak of DSC curve observed above 265 °C attributed to decomposition which is in accordance with TGA data.

Thermal stability and flame retardant properties

Thermal stability of polymeric materials is significant while used as flame retardant that mainly concern the release of decomposition products and formation of char. TGA provides direct information about thermal stability and degradation mechanism by measuring weight loss of sample as a function of temperature. Thus, cured samples were compared with 5, 50% and stages I and II weight loss temperatures and char yield at 700 °C as shown in Table 1. Temperature corresponding to 5% weight loss in TGA was viewed as a rough index of thermal stability. TGA results indicated the formulations with and without fibers showed (Fig. 4a, b) similar trend. Thermograms for neat DGEBA sample (A1) and its fiber composite (A6) commenced to lose their weight at 292 and 305 °C, respectively, and degraded in the range of 292–345 °C with 50% weight loss leaving little residue at 700 °C. TGA curves distinctly demonstrated the effect of phosphorus content on decomposition temperature and char formation as follows. Samples A2–A5 and A7–A10 degraded in two stages and have a lower initial decomposition temperature (IDT) of around 288–265 and 285–260 °C, respectively. Temperature for the maximum rate of pyrolysis was lowered and the amount of residual char at 700 °C increased with increasing DGPP content attributed to P–O–C bond linkage in DGPP [48]. Thus, while the resin is burning, the phosphorus segments degrade in the first step and form phosphorus-rich residue to prevent further decomposition of epoxy resin by enhancing second decomposition at higher temperature contributing to high char yield [49]. Due to its more volatile nature, DGEBA (A1) and its composite (A6) without flame retardant (DGPP) begins to decompose first

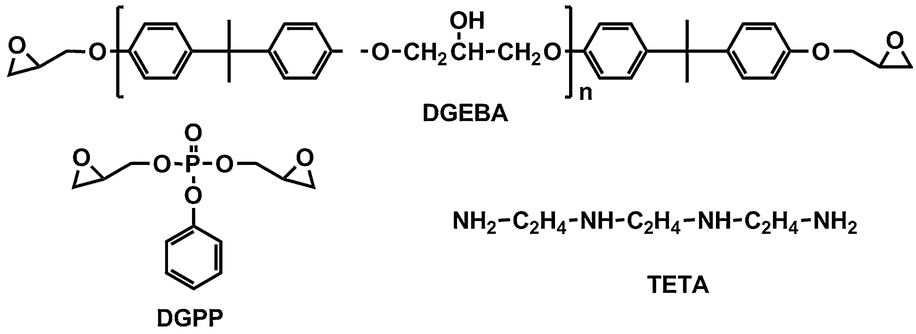

Fig. 2 Chemical structures of epoxy resins and hardener

Table 1 Thermal and flame retardant properties of cured blends (A1–A5) and composites (A6–A10)

Formulation	Formulation code	T_g (°C)	Weight loss		T_{max} (°C)		Char yield (%)	UL-94 (mm/min)	LOI (%)
			5% (IDT)	50%	Stage 1	Stage 2			
Neat DGEBA	A1	131	292	340	326	–	5	35	19
DGEBA + DGPP5%	A2	125	288	342	320	370	12	26	27
DGEBA + DGPP10%	A3	120	274	375	301	372	18	23	28
DGEBA + DGPP15%	A4	117	268	402	295	385	22	20	30
DGEBA + DGPP20%	A5	111	265	428	276	410	25	18	32
DGEBA + Fiber	A6	–	305	338	320	–	7	33	20
DGEBA + DGPP5% + Fiber	A7	–	285	340	318	375	14	25	28
DGEBA + DGPP10% + Fiber	A8	–	280	351	302	409	20	21	29
DGEBA + DGPP15% + Fiber	A9	–	266	380	285	415	22	20	31
DGEBA + DGPP20% + Fiber	A10	–	260	410	275	430	27	16	32

IDT Initial decomposition temperature; T_g glass transition temperature

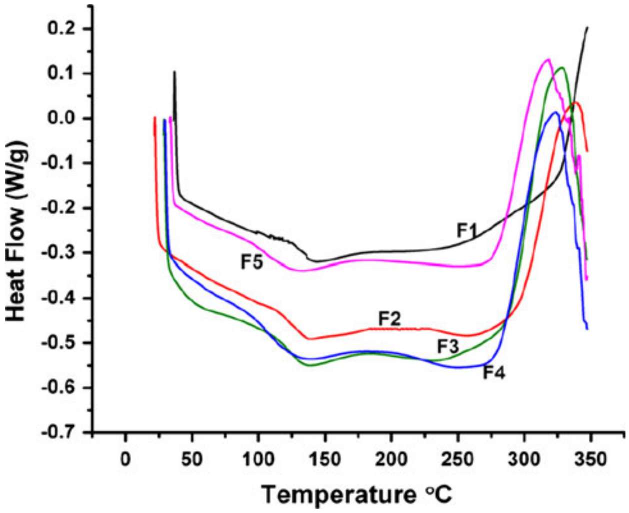

Fig. 3 DSC thermograms of cured blends (A1–A5) show the decreasing T_g with increase in DGPP content

around 292–300 °C, and completely degraded at 700 °C. However, initial decomposition temperature is lower with the addition of DGPP to DGEBA matrix and the overall thermal stability of samples (A2–A5 and A7–A10) increased up to 700 °C as expected.

Flame-retardant properties of cured samples (A1–A10) were evaluated by UL-94 (horizontal burning test) flammability (Fig. 5) and limiting oxygen index (Fig. 6) tests and results are listed in Table 1. Both the formulations (A1–A5 and A6–A10) established similar trends in flame-retardant property. The more volatile nature of samples A1 and A6 caused higher burning rate and lower LOI values than DGPP containing samples. The burning rate decreased and LOI increased for samples A5 and A10 with 20% of DGPP to about half the value of samples A1 and A6, respectively. Generally LOI values for polymers should be >26 to meet the requirements for flame-retardant applications and specimens must not have a horizontal burning rate greater than 37 mm/min for thicknesses between 3 and 12 mm [50]. Table 1 indicates that all the phosphorus-containing samples (A2–A5 and A7–A10) show LOI value between 27 and 32 and horizontal burning rate in between 16 and 26 mm/min. It is clear that the incorporation of phosphorus-containing DGPP in DGEBA thermosets promoted excellent flame retardancy as compared with flammable DGEBA curing system.

The char yield, LOI, and UL-94 values of A2–A5 are lower than those of A7–A10 (Table 1), which may be attributed to the formation of acids during pyrolysis that increase dehydration of fiber components and char

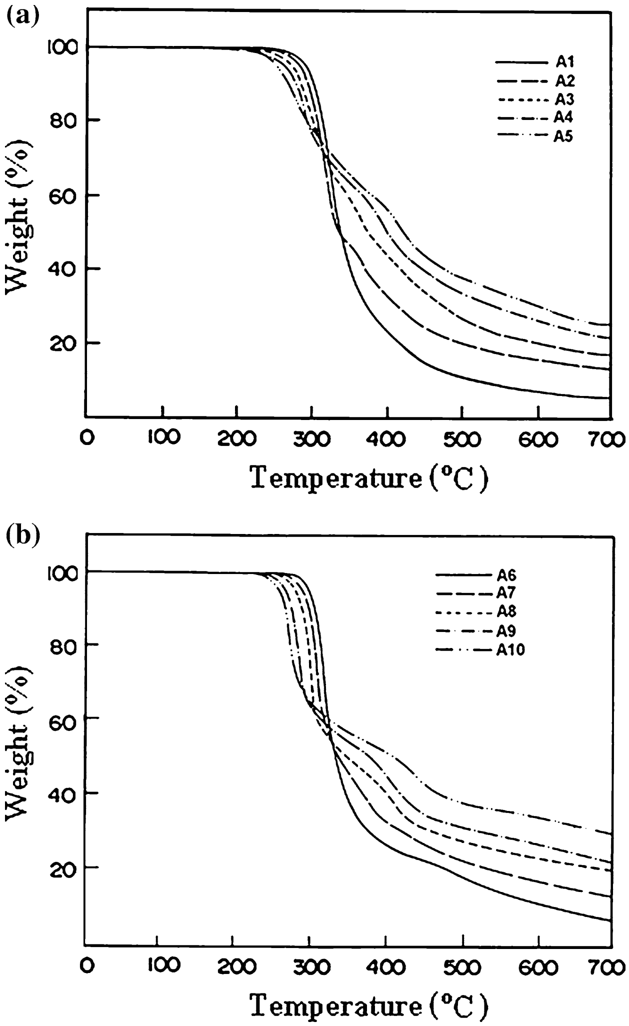

Fig. 4 TGA thermograms of cured **a** blends (A1–A5) and **b** composites (A6–A10)

formation. The char forms a thermal insulation barrier as well as a diffusion barrier for oxygen and volatile combustible components [51, 52].

Mechanical properties

Tensile, flexural, and impact strengths

The measured tensile, flexural, and impact strengths of cured samples A1–A10 are shown in Figs. 7, 8, 9, respectively, and the data is presented in Table 2. The tensile and flexural strengths of A2–A3 and A7–A8 were found to be higher than that of the neat epoxy system (A1) and its corresponding composite (A6). This is attributed to –P=O group in DGPP which is more polar in nature and can form hydrogen bond with residual –OH groups generated in the curing process and the –OH groups of natural fibers, assisting in good compatibility of DGPP resin with DGEBA which in turn acts as crack stoppers and increases tensile and flexural strength as well as modulus. But the tensile and flexural strengths of A4, A5 and A9, A10 were decreasing with increase in DGPP content which is ascribed to lower bond strength of P–O–C bonds in DGPP will provide more flexibility and plasticizing effect to the cured system. Rigidity of the system also reduced.

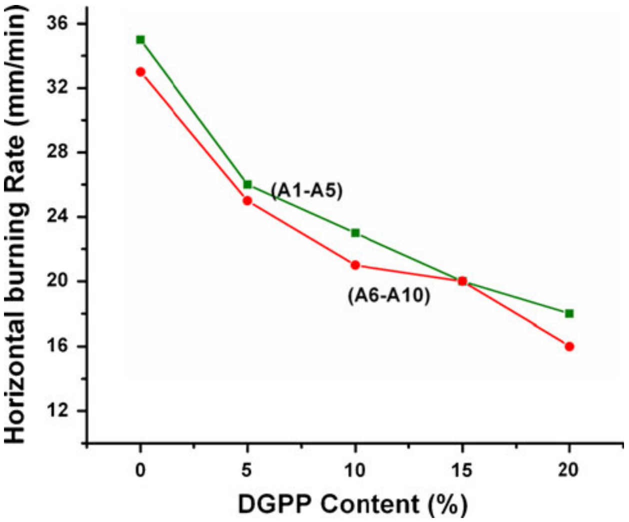

Fig. 5 Horizontal burning rate of cured blends (A1–A5) and composites (A6–A10) shows the burning rate decreases with increasing DGPP content

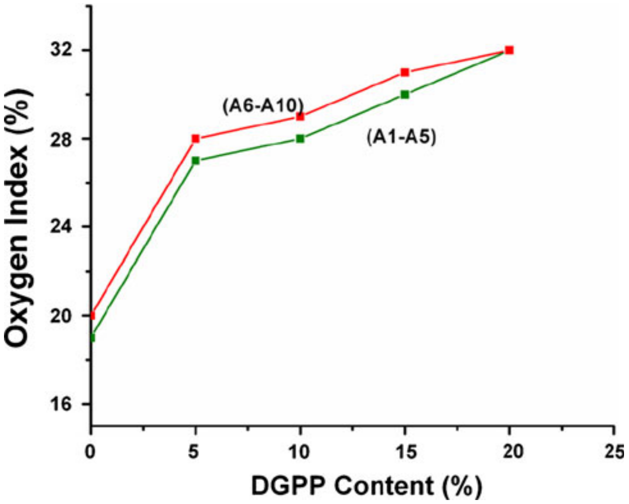

Fig. 6 Limiting oxygen index of cured blends (A1–A5) and composites (A6–A10) shows the oxygen index increases with increasing DGPP content

The impact strength (Fig. 9) of cured samples (A1–A10) showed a dissimilar trend in their tensile and flexural strengths. Impact strength of A4 and A9 systems is somewhat higher than the remaining systems of respective series. The same samples (A4 and A9) evidenced lower tensile and flexural strengths (wide Table 2) indicating the samples A1–A3 and A6–A8 are comparatively rigid than

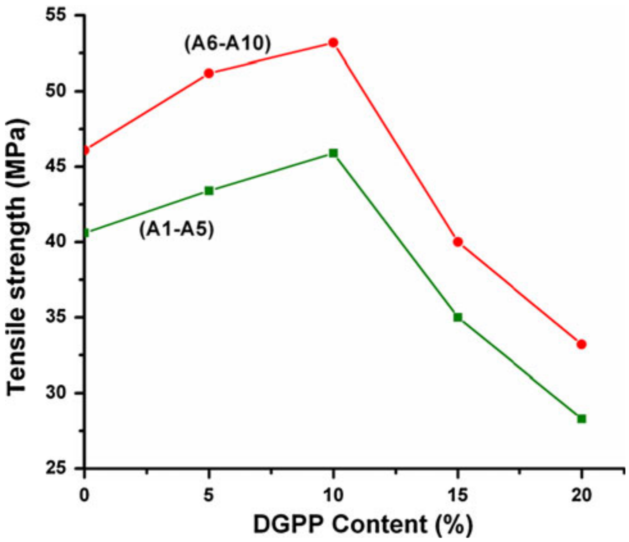

Fig. 7 Tensile strength of cured blends (A1–A5) and composites (A6–A10)

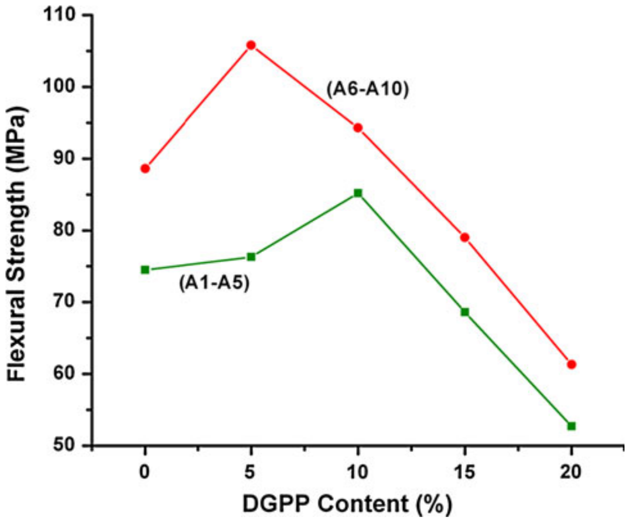

Fig. 8 Flexural strength of cured blends (A1–A5) and composites (A6–A10)

A4 and A9, respectively. The lower percentage of elongation values for samples A1–A3 and A6–A8 than samples A4–A5 and A9–A10 too indicates the rigidness of A1–A3 and A6–A8. Albeit, there is a decrease in the flexural and tensile strengths with increasing DGPP content samples A2–A4 and A7–A9 have the good impact strength. The improvement in impact strength is ascribed to the presence of DGPP content which acts as toughening agent up to 15%. In the case of fiber composites, it was reported that the strong interface between fiber and matrix reduces impact strength of the composites [53]. At high level of adhesion, the failure mode is brittle and relatively little energy is absorbed as indicated by the data on samples

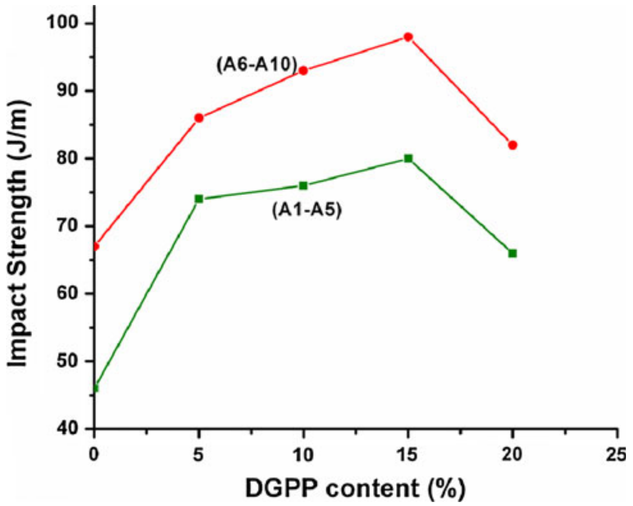

Fig. 9 Impact strength of cured blends (A1–A5) and composites (A6–A10)

A6–A8. In the case of a weak interface, triaxial stresses at the tip of an advancing crack causes debonding to occur and a crack bunting mechanism takes place that improves toughness of the material [51]. However, very low adhesion may result in the lowering of toughness as it can be ascertained in the SEM photograph (Fig. 10e) of A10.

Morphology

Representative SEM microphotographs for fracture surfaces of blends and their fiber composites are shown in Fig. 10a–f. Figure 10a, b indicate a smooth and homogeneous fractured surface with cracks in different planes, for the neat epoxy (A1) and 10% DGPP blend (A3). This indicates a brittle fracture, which accounts for its poor impact strength. Nevertheless, it was observed some partially co-continuous nature of fractured surface for samples A3 and A5 (Fig. 10b, c) with increasing DGPP content from 5 to 20%. Good dispersion of DGPP in DGEBA leads to enhanced toughness of samples A2–A4. In the case of A5, this dispersion leads to plasticizing (or flexibility) effect with increase in DGPP content. The fracture of composite (A10) occurs by fiber pullout and it is indicated by holes and fiber ends (Fig. 10f), as a result of poor adhesion between fiber and matrix. Consequently the mechanical properties are poor (Table 2). A similar effect was observed in the case of A6 (Fig. 10d) to some extent in addition to brittleness, hence mechanical properties were poor. With respect to A9, adhesion between the matrix and fiber may be intermediate which leads to progressive de-lamination that requires additional energy. Hence A9 shows improved impact strength. Figure 10e shows that the failure occurs by fiber breaking rather than fiber pullout, indicating better interaction between fiber and matrix in

Table 2 Mechanical properties of cured blends (A1–A5) and composites (A6–A10)

Property	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
Tensile strength (MPa)	40.6	43.4	45.9	35.0	28.3	46.1	51.2	53.2	40.0	33.2
Tensile modulus (GPa)	2.00	2.18	2.07	1.97	1.91	2.19	2.64	2.33	2.33	2.29
Elongation (%)	2.6	2.9	2.8	3.0	3.0	2.4	2.3	2.4	2.8	2.9
Flexural strength (MPa)	74.5	76.3	85.2	68.6	52.7	88.6	105.8	94.3	79.0	61.3
Flexural modulus (GPa)	2.61	2.88	2.73	2.13	2.35	2.9	3.22	3.41	2.64	2.58
Impact strength (J/m)	46	74	76	80	66	67	86	93	98	82

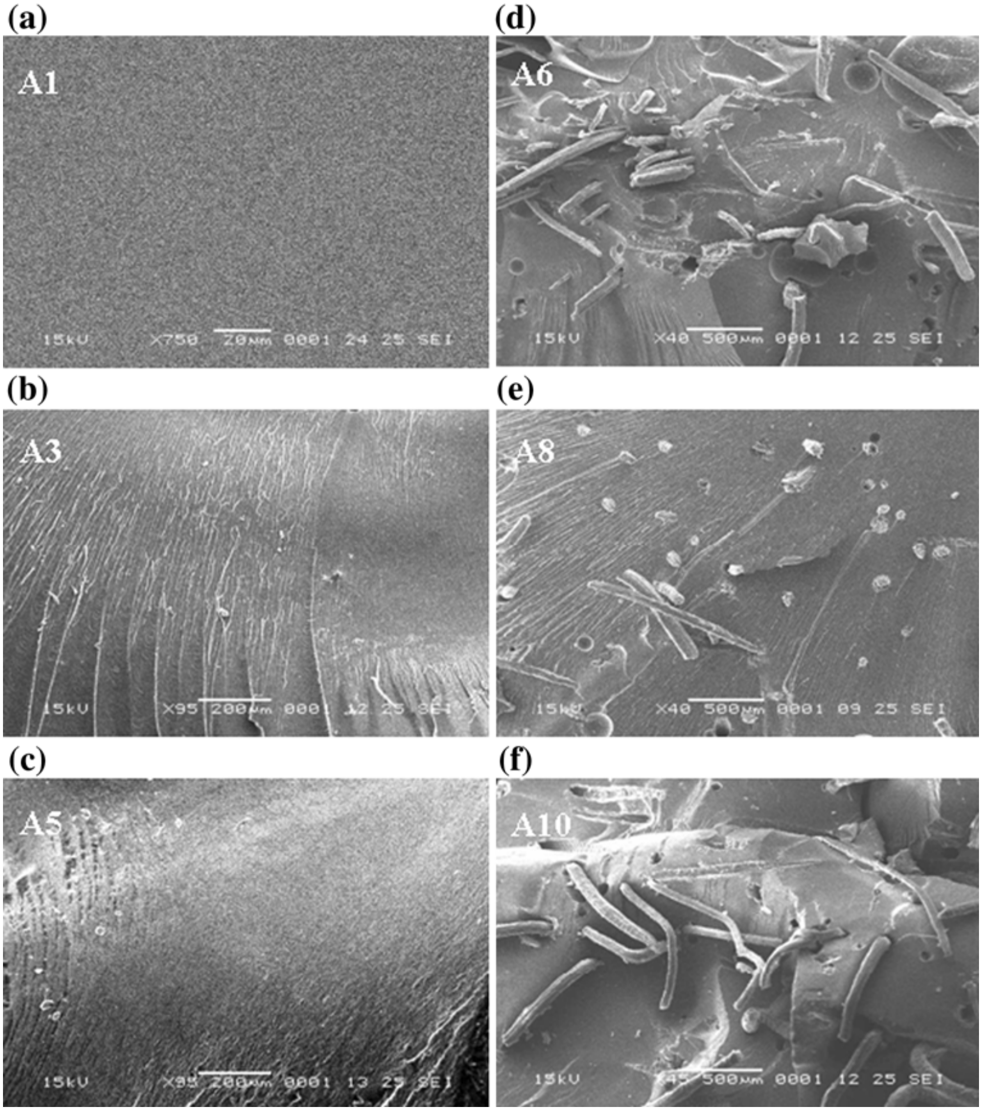

Fig. 10 SEM microphotographs of the fractured surface of blends (**a**–**c**) and fiber composites (**d**–**f**). **a** Morphology of fracture surface exhibits smooth surface of blend. **b** Exhibits brittle fracture. **c** Shows some co-continues faces. **d** The failure of the composite occurs by fiber pullout and fiber breakage. **e** The strong interaction of fiber and matrix in which failure occurs through fiber breakage. **f** Failure occurs through fiber pullout with increasing DGPP content

A8. It is understood from the literature that if fiber–matrix adhesion is strong, the mechanism of failure changes from fiber de-bonding and pullout to brittle failure, and this reduces the impact strength [54].

Conclusions

This investigation had an objective to develop green composites utilizing *Borassus* fruit fiber and fire retardant resin DGPP blending with commercial epoxy resin (DGEBA) incorporating various weight percentages and to study the effect of DGPP content on the properties. The DGPP was blended with DGEBA by 5, 10, 15, and 20% by weight (formulations A1–A5) and corresponding fruit fiber composites were prepared (A6–A10) as well. Although the thermal stability of blends (A2–A5) and composites (A7–A10) were decreased with increasing DGPP content to some extent as compared to neat epoxy (A1) and its composite (A6), the fire-retardant properties significantly improved. The T_g values of blends (A1–A5) were moderate in the range of 111–131 °C. Information on T_g would help to understand the processing conditions of blends and corresponding fiber composites. The tensile and flexural strengths as well as modulus were reasonable and increased up to 10% DGPP content and decreased for 15 and 20% whereas the impact strength of these formulations showed inverse trend. The impact strength was improved till 15% of DGPP addition ascribable to good dispersion and toughening effect of DGPP. SEM analysis of blends and composites provided a clear picture about dispersion of DGPP and bonding nature of fiber and matrix and concluded that the formulations A2–A4 and A7–A9 have superior properties than the rest.

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