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Fabrication and characterizations of biodegradable jute reinforced soy based green composites

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ABSTRACT

Jute reinforced soy milk based composites using non-woven and woven jute fabrics were developed and characterized. Mechanical properties, viz., tensile strength, elongation at break, flexural strength, tensile and flexural modulus of these composites were measured and reported. Composites having 60 wt% jute felt or jute fabric possessed the best mechanical properties. Hydrophilicity of the composites was assessed by the measurement of contact angle and water absorption after immersing in water at ambient temperature as well as in boiling condition. Biodegradability of the composites was evaluated in compost soil burial condition. Fourier transform infra-red (FTIR) and optical microscope (OM) analyses of the buried samples confirmed the degradation of the composites. The composition of the developed composites is novel. The fabricated composites can be used in various fields for replacing plastics.

1. Introduction

The basic disadvantages of plastics are their non degradability and environmentally hazardous nature. Plastic waste disposal management is now a great challenge due to the unavailability of free land for solid waste disposal in regions of high population density (Mohanty, Khan, & Hinrichsen, 2000). Bio-composites can replace synthetic plastic products and make the environment free of plastic wastes. Over the last decade, research efforts have been directed towards making bio-resin based natural fiber reinforced composites. Natural fiber based bio-composites are already introduced in the market of USA, Canada, and UK as a part of green revolution. A wide variety of different lignocellulosic natural fibers like jute, sisal, coir, banana, etc. were used as good reinforcements or fillers for making strong bio-composites (Liu, Misra, Askeland, Drzal, & Mohanty, 2005; John & Thomas, 2008).

Jute, abundantly available in eastern part of India and Bangladesh, has high tensile strength, high moisture sensitivity, low thermal and electrical insulation properties (Wang, Cai, & Yu, 2008). It is primarily composed of cellulose, hemicellulose and lignin. Lignocellulosic components present in jute help to prepare strong and rigid composites when they reinforce with either bio-resin or thermoplastic/thermoset resin. The hydrophilic nature of jute is a major drawback for its engineering application to prepare bio-composite with hydrophobic thermoplastic resin, but may be an advantage for preparing green composite with hydrophilic resins (Wang, Cai, Yu, & Zia, 2009; Doan, Gao, & Mader, 2006). Researchers world wide have already reported about poor bonding between jute and thermoplastic resins due to hydrophilic nature of jute fiber and hydrophobic nature of polymer (John & Anandjiwala, 2008; Liu & Dai, 2007). Weak bonding of jute with hydrophobic polymer contributes to poor physical and mechanical properties of the concerned polymer reinforced jute composites. For this reason several researchers have done different pretreatment on jute fiber with alkali, acid, polyol, low molecular weight polymer, etc. to improve the interaction and adhesion between fiber and polymer matrix (Saha, Manna, Chowdhury, Sen, Roy, & Adhikari, 2010; Ghosh & Das, 2000; Kumar & Verma, 2006).

Most thermoplastic and thermoset resin reinforced jute based composites are not fully biodegradable, as their disposal in compost condition is not found to be safe enough for their slow degradation rate (Rahman & Bala, 2009). Incineration of these products contributes to the total CO_2 pool in atmosphere due to incomplete combustion (Reddy & Yang, 2011). Bio-resin like soy protein concentrate (SPC), soy protein isolate (SPI) based jute composites are completely degradable and environment friendly (Huang & Netravali, 2007). Soy in the form of SPC, SPI, and soy oil has been used as matrix for development of natural fiber reinforced composites (Reddy & Yang, 2011; Huang & Netravali, 2007; Kumar & Zhang, 2009). But these bio-resins are of high cost and require processing before their use as matrix (Liu, Erhan, Akin, & Barton, 2006). High cost of these soy resins is a challenge to prepare low cost jute composite. Hence soymilk, a low cost and natural bio-resin has been

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used for jute composite fabrication in the present work. Soy milk, extracted from water soaked soy seeds contains both protein and carbohydrate (Zhang, Li, & Yu, 2011). These proteins may crosslink with cellulosic groups on jute surface to prepare rigid natural fiber reinforced composite. Production of soymilk is easy, cost effective and less time consuming than processing of SPC, and SPI from soy seeds, hence developed jute-soymilk composites might be economical and user-friendly. These composites can be treated as a part of green revolution to reduce use of eco-hazardous plastic and their concerned composites.

Though significant amount of work has been reported on natural fiber and bio-resin based composite, no literature report is available on the use of soy milk as resin with jute woven/non woven fabric to prepare eco-friendly jute composite. In this work, fully biodegradable jute reinforced composites were prepared using soy milk as resin along with crosslinking agent and other additives. Both non-woven and woven jute fabrics were used for composite preparation as they offer several advantages such as good impact resistance, damage tolerance, toughness and ease of manufacturing. Mechanical and biodegradation properties of jute, soy matrix and their composites were also evaluated in this study. The novelties of this work are use of water as solvent for soy resin preparation instead of any organic solvent and jute without any chemical treatment. Since both jute and soy resins are agro based natural resources, hence their corresponding composites are expected to be eco-friendly and non-hazardous unlike plastic.

2. Experimental

2.1. Materials

Jute felt (400 gsm) and jute fabrics were obtained from Gloster Jute Mills Limited, Bauria, West Bengal, India. Glyoxal (Merck, India), Glycerol (Merck, India), and Ammonium hydroxide (NH_4OH) (Merck, India) and soy bean were procured from market.

2.2. Extraction of soymilk from soy seeds

Soy seeds (500 g) were washed properly and soaked in distilled water (1:4, w/v) for 4 h. Water soaked soy seeds were crushed in a mixer grinder to get soy paste. Soymilk was extracted from soy paste by squeezing through nylon cloth. About 1500 ml of soy milk was obtained from 500 g of soy seeds. Soymilk was then made alkaline (pH 9) by adding ammonium hydroxide (NH_4OH) in order to stabilize it. Solid content of soymilk was determined by taking 10 ml of soy milk in a dry 10 cm diameter petridish and weighed (W_1). It was kept in an oven at 70 °C for 6 h. After that petridish was kept in a vacuum desiccator at room temperature for 24 h and final weight (W_2) was taken. The solid content of soy milk was calculated as 20% by the following formula:

$$\text{Solid content } (\%) = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

2.3. Preparation of soy resin film (SRF)

Different weight percentages of glyoxal (1, 5, 10, 15, and 20) (w/w, solid content of soy milk) along with 10 wt% of glycerol (w/w, solid content of soy milk) were added to required amount of soymilk and stirred for 30 min to prepare soy resin. 100 ml of soy resin was poured on a flat glass petridish of diameter 15 cm, which was kept in a thermal chamber at 125 °C for 25 min. Then dark brown soy resin film (SRF) was formed. Soy resin films are coded as SRF1, SRF5, SRF10, SRF15, and SRF20 with respect to wt% of glyoxal. The resin film was kept in open air for 30 min to absorb some moisture, which helps for easy peeling. Tensile specimens were prepared from different sets of SRF and tested.

2.4. Preparation of non-woven jute soy (NJS) and woven jute soy (WJS) composites

Non-woven jute felt (400 gsm) was used as reinforcing material for NJS composite preparation. Varying the wt% (40, 50, 60, 70 and 80) of jute, different composites were fabricated and designated as NJS1-5 respectively. Glyoxal (10 wt%, as optimized from tensile testing of SRF) and glycerol (10 wt%, w/w solid content of soymilk) were mixed with soymilk with continuous stirring for 20 min to prepare soy resin. Jute felts were soaked in this resin by dipping process and then partially dried in an oven at 60 °C for 45 min. Partially dried soy resin impregnated jute felts were compressed using a hydraulic press at 125 °C and a pressure of 8 ton for 25 min (curing time) to obtain NJS composites (Chabba & Netravali, 2005). Similarly different wt% (40, 50, 60, 70 and 80) of jute woven fabrics were used as reinforcing material with above soy resin to prepare WJS composites (WJS1-5 respectively) by following the above procedure.

2.5. Testing and characterization

NJS and WJS composites were characterized for their mechanical strength and flexibility, hydrophilicity, surface characteristics, and susceptibility to biological degradation as described below.

2.5.1. Analysis of mechanical properties

Tensile strength of SRF and composites (NJS and WJS) were characterized in accordance with ASTM D638 (ASTM, 2003a) with a cross head speed of 5 mm/min using universal testing machine (HOUNSFIELD H10KS UTM). Six specimens of each sample were tested and average values are reported.

Flexural properties of NJS and WJS composites were measured in accordance with ASTM D790 (ASTM, 2003b) with a cross head speed of 2 mm/min using universal testing machine (HOUNSFIELD H10KS UTM). For each composite, six specimens were tested and average values are reported.

2.5.2. Scanning electron microscopy (SEM) analysis

Morphological characteristics of jute, SRF10, composite and tensile fracture surface of composites were analyzed by SEM (VEGA II, LSV, TESCAN, Czech Republic) instrument operated in the high vacuum mode with secondary electron detector and with accelerating voltage between 5 and 10 kV.

2.5.3. Contact angle measurement

In order to study the hydrophilic properties of jute, SRF10 and composites, contact angles were measured using a SEO Contact angle meter. A drop of water (1–2 μl) was dropped on sample surface through a micro-syringe and a photograph was taken for each test with CCD camera attached to the instrument. At least 6 measurements were taken from different areas on sample surface and average contact angle value is reported in accordance to Alix et al. (2009).

2.5.4. Water absorption analysis

Water absorption of jute, SRF10 and composite specimens were determined at room temperature (35 °C) according to ASTM D570 (ASTM, 2005) using the following formula (2) where, W_i is the initial weight of the specimen before immersing in water, and W_f is the

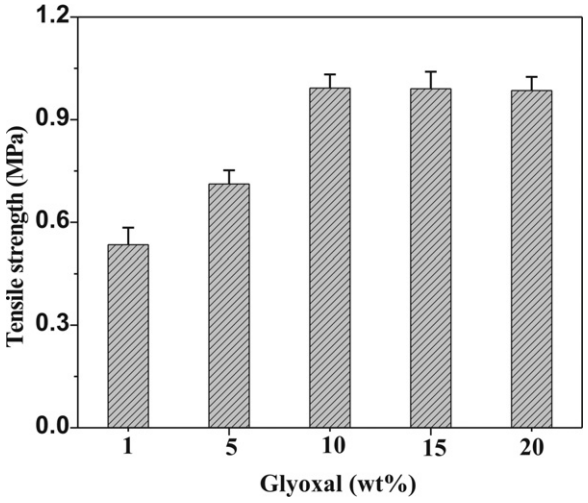

Fig. 1. Tensile strength of soy resin film (SRF).

final weight of the specimen after specific time of immersion in distilled water.

$$\text{Water absorption } (\%) = 100 \times \frac{W_f - W_i}{W_i} \quad (2)$$

2.5.5. Biodegradation study

Soil burial test for biodegradation of jute, SRF10 and composites (NJS and WJS) were conducted as per BIS 1623-1992 (BIS, 1992) standard. The compost for soil burial tests was prepared by mixing garden soil, sand and manure in 2:1:1 ratio (by weight). Specimen to soil weight ratio was maintained at 1:100 (w/w) and the samples were buried under compost within a glass jar. The glass jar was covered and incubated at 32 °C for 60 days. The samples were removed at regular time periods (7, 15, 30, and 60 days), washed with distilled water and dried at 105 °C in an oven for 6 h, weighed and characterized by FTIR and optical microscope to evaluate the degree of degradation.

FTIR spectra of jute, SRF10 and composites (NJS and WJS) before and after 60 days of soil burial degradation, were taken using a Thermo Nicolet, Nexus 870 IR spectrometer. The spectra were recorded from 400 cm^{-1} to 4000 cm^{-1}. Before making KBr pellet the sample powder was dried under vacuum at 105 ± 2 °C for 1 h.

Optical microscopic photograph of jute, SRF10 and composites (NJS and WJS) samples were taken before and after 60 days of biodegradation by an optical microscope (Leica DMLM 2500) to study the degradation.

3. Results and discussion

3.1. Mechanical properties of SRF

The results of the tensile properties of different percentage of glyoxal crosslinked soy resin films are shown in Fig. 1. Soy resin film (SRF10) prepared with 10 wt% glyoxal (w/w, solid soy wt) and 10 wt% (w/w, solid soy wt) of glycerol showed the highest tensile strength of 0.97 MPa, which may be due to better cross linking between soy protein and glyoxal (Chabba & Netravali, 2005). The protein part of soymilk constitutes of 18 different amino acids such as lysine, glutamine, asparagines, arginine, etc., which have more than one NH_2 group (Kumar, Choudhary, Mishra, & Varma, 2008). These NH_2 groups react with glyoxal and produce strong primary electrovalent bonds to improve mechanical strength of the crosslinked resin. The probable crosslinking between different amino acids of soy protein and glyoxal is schematically illustrated in Fig. 2 (Kumar et al., 2008). Addition of excess of crosslinking agent does not increase tensile strength as there are less number of available sites in protein of soy milk for bonding. Hence, 10 wt% of glyoxal was considered optimum as crosslinking agent for preparing non-woven and woven jute reinforced biocomposites.

3.2. Mechanical properties of the NJS and WJS composites

Mechanical properties of NJS and WJS composites are shown in Table 1. As jute loading increased from 40 to 60 wt%, tensile strength of NJS increased from 24.8 to 37.1 MPa. Consequently the tensile modulus increased from 780 to 1040 MPa. Similar trend was also obtained in case of flexural properties as the flexural properties increased from 25.8 to 38.4 MPa and the flexural modulus enhanced from 832 to 1120 MPa with increase in jute loading from 40 to 60 wt%. This indicated a good adhesion between the jute fiber and the soy resin. The presence of side-chain hydroxyl and other polar groups in the jute fiber caused a strong bonding between the fibers and hydrophilic matrix. This may be a consequence of a good dipole-dipole/hydrogen bonding interaction between the cellulose of jute fiber and the protein of soy resin in the presence of glyoxal which helps in cross-linking between them (Fig. 2). However, further increasing in jute loading did not improve the mechanical strength. That may be due to the inadequate wetting of jute fiber surface by soy resin or may be the presence of higher percentage of jute causing easy crack initiation and propagation to the fiber end (Farsi, 2010). NJS3 having 60 wt% jute shows the highest tensile strength of 37.1 MPa and tensile modulus of 1040 MPa which is more than the tensile strength (28.4 MPa) of jute-polypropylene composite (jute without any use of compataibilizer) (Liu and Dai, 2007). Similar trend in mechanical properties were obtained in case of WJS composites. Tensile and flexural strength increased from 22.4 to 35.6 MPa and 21.9 to 33.5 MPa respectively with increase in jute loading (40 to 60 wt%). Tensile and flexural strengths of WJS3 are less than that of NJS3. This may be due to the use of woven fabric which has less wetting capacity with soy resin as compared to jute felt (Rout, Misra, Tripathy, Nayak, & Mohanty, 2001). NJS3 and WJS3 showed highest mechanical strength among their respective NJS and WJS composites and were considered as optimum composite.

3.3. Surface morphology of composites

Scanning electron micrograph of jute, SRF10, NJS3 and fracture portion of NJS3 after tensile testing are shown in Fig. 3. Raw jute fiber has cylindrical rod like structure having a clean and smooth surface (Fig. 3(a)), while SRF10 has smooth surface (Fig. 3(b)). When jute fiber forms composite with soy matrix, a homogeneous soy matrix coated jute surface was obtained (Fig. 3(c)). That leads to increase in adhesion between different soy coated jute fibers to form strong and rigid jute composite. The fracture portion after tensile testing shows voids (illustrated in Fig. 3(d)) and breaking portion of jute fiber inside matrix (showed by arrow) indicates brittleness of jute fiber. The voids in matrix formed under tensile conditions leads to development of fracture in composite.

3.4. Contact angle and water absorption test of composites

Hydrophilicity of NJS3 and WJS3 with respect to jute and soy resin was studied by contact angle and water absorption test and the results are reported in Table 2. Jute and soy resin are hydrophilic in nature showing contact angle values of 42.1° and 50.4° (Fig. 4) respectively, which correspond to water absorption values of 190% and 110.6% respectively after 24 h immersion in water. Higher contact angle value indicates more hydrophobic nature of sample

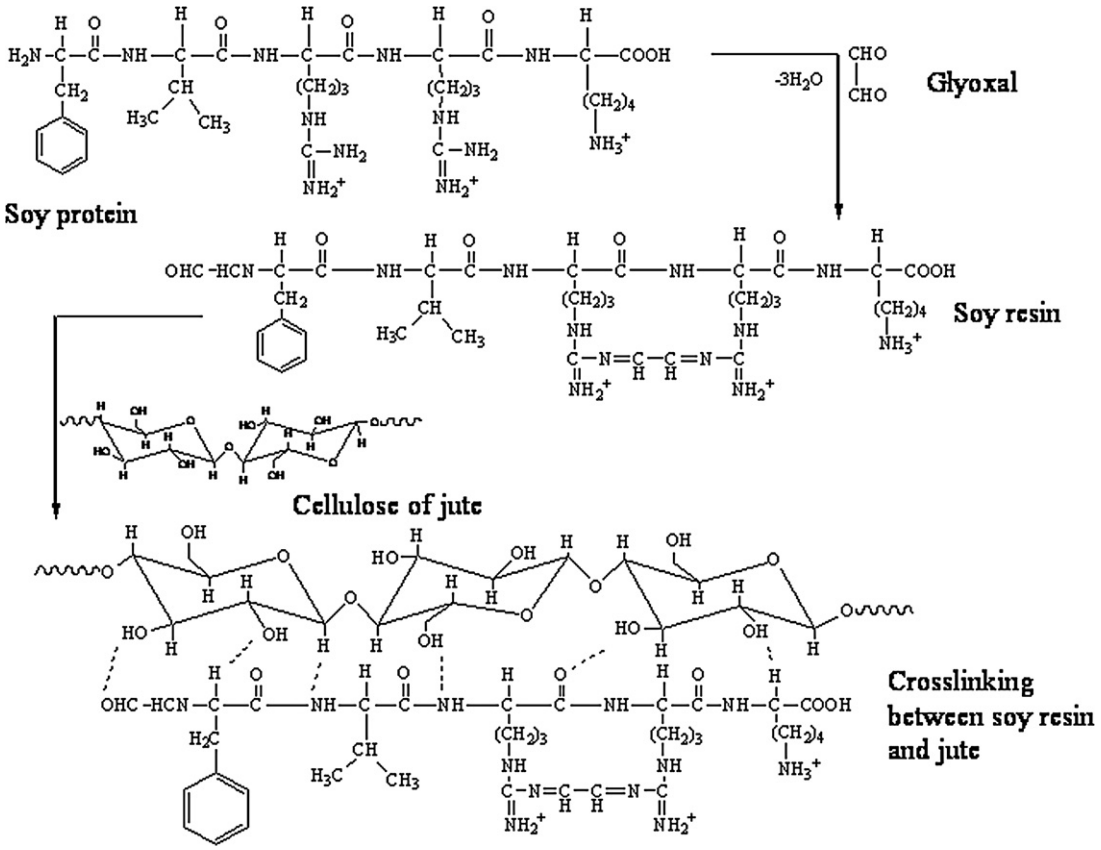

Fig. 2. Schematic representation of probable crosslinking between soy protein (a short amino acid linkage of phenylalanine–valine–arginine–arginine–lysine) and glyoxal (Adopted from Kumar et al., 2008), with cellulose of jute.

Table 1
Mechanical properties of NJS and WJS composites.

Composite	Jute content (wt%)	TS[a] ± SD[g] (MPa)	TM[b] ± SD[g] (MPa)	EB[c] ± SD[g] (%)	FS[d] ± SD[g] (MPa)	FM[e] ± SD[g] (MPa)
NJS1	40	24.8 ± 2.1	780 ± 20	9.3 ± 0.3	25.8 ± 2.0	832 ± 22
NJS2	50	29.6 ± 2.1	824 ± 20	8.7 ± 0.3	30.7 ± 1.9	896 ± 20
NJS3	60	37.1 ± 1.9	1040 ± 20	6.8 ± 0.3	38.4 ± 2.0	1120 ± 21
NJS4	70	35.9 ± 2.0	916 ± 21	6.2 ± 0.3	36.3 ± 2.0	974 ± 20
NJS5	80	31.4 ± 2.0	840 ± 20	5.8 ± 0.3	32.1 ± 1.8	902 ± 21
WJS1	40	22.4 ± 1.9	714 ± 20	10.2 ± 0.3	21.9 ± 2.0	684 ± 20
WJS2	50	27.7 ± 1.9	778 ± 21	8.9 ± 0.3	29.2 ± 2.1	792 ± 20
WJS3	60	35.6 ± 2.1	972 ± 20	7.1 ± 0.3	33.5 ± 2.0	1026 ± 18
WJS4	70	32.5 ± 2.1	804 ± 20	6.8 ± 0.3	31.4 ± 2.1	984 ± 20
WJS5	80	29.4 ± 2.0	764 ± 22	6.1 ± 0.3	28.7 ± 1.9	907 ± 19

[a] TS: tensile strength.
[b] TM: tensile modulus.
[c] EB: elongation at break.
[d] FS: flexural strength.
[e] FM: flexural modulus.
[g] SD: standard deviation.

which is found in case of NJS3 (67.8°). That may be due to better crosslinking of soy resin on fiber surface that does not allow water to easily penetrate. Thickness swelling in case of resin film after water absorption is more as compared to WJS3 and NJS3. Water absorption of NJS3 and WJS3 after 2 h in boiling water is 50.2% and 54.9% respectively, while their thickness swelling is 50.7% and 63.4% respectively. Contact angle and water absorption of jute, resin, WJS3, and NJS3 after 2 h in boiling water and 24 h in distilled water reveal that NJS3 is more hydrophobic than others.

Table 2
Hydrophobicity/hydrophilicity of jute, soy resin, NJS3 and WJS3.

Sample	Jute fiber	SRF10	WJS3	NJS3
Contact angle (°)	42.1	50.4	63.5	67.8
Water absorption (24 h in water) (%)	190	110.6	52.1	46.6
Thickness swelling (24 h in water) (%)	–	84.1	65.7	47.8
Water absorption (2 h in boiling water) (%)	183	118.8	54.9	50.2
Thickness swelling (2 h in boiling water) (%)	–	88.6	63.4	50.7

3.5. Soil burial of jute, SRF, and composites

3.5.1. Weight and tensile loss after different periods of biodegradation

Weight loss after different time periods of biodegradation is shown in Fig. 5(a). After 7 days of biodegradation, soy resin degraded most by 52% weight loss while jute fabric, jute felt, WJS3

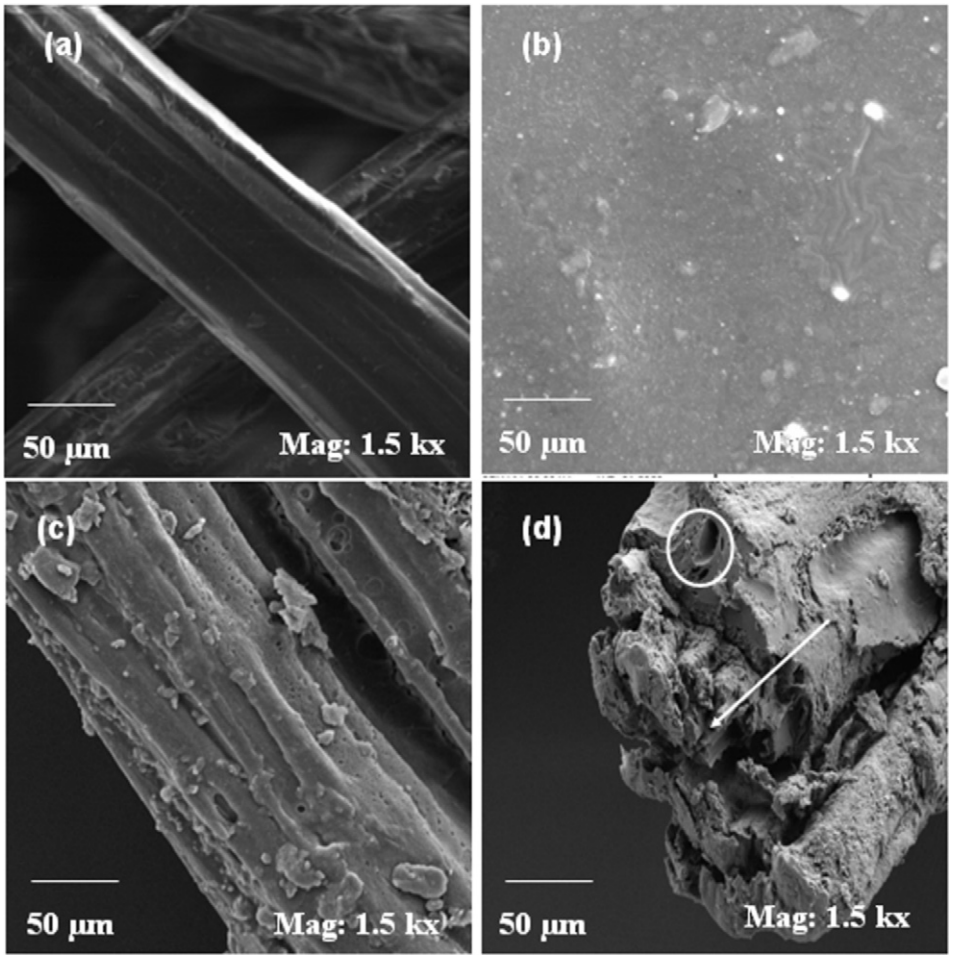

Fig. 3. SEM photograph of (a) Jute fiber; (b) SRF10; (c) Resin coated jute; (d) Fracture surface of resin coated fiber.

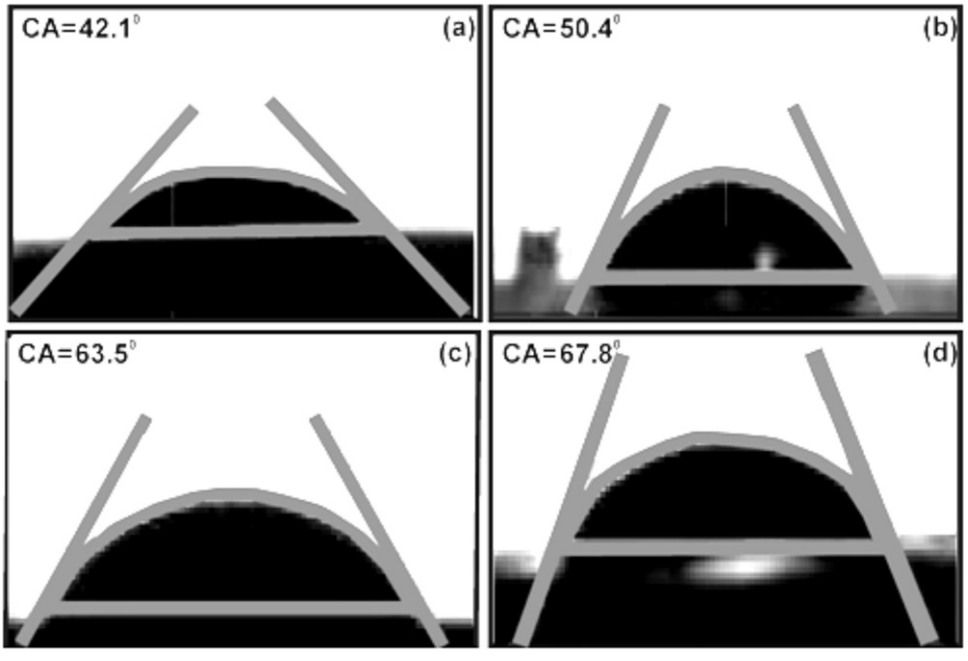

Fig. 4. Contact angle measurement of (a) Jute; (b) SRF10; (c) NJS10; (d) WJS10.

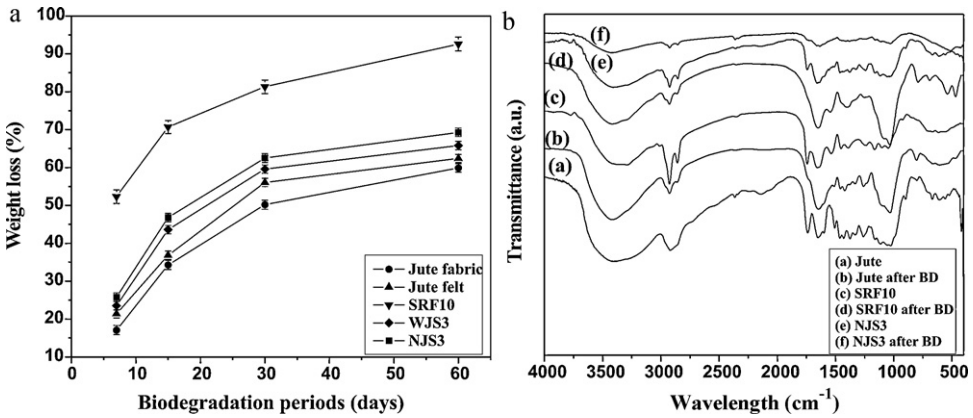

Fig. 5. (a) Weight loss of jute, SRF10, NJS3 and WJS3; (b) FTIR spectra of jute, SRF10, and NJS3 before and after biodegradation.

and NJS3 degraded by 17%, 20% 23% and 26% of their original weight respectively. Due to high hydrophilic nature of soy resin it degraded earlier than other specimens tested. Also soil microbes preferably attack soy protein over cellulose and hemicellulose of jute (Zhang, Rong, & Lu, 2005). Between WJS3 and NJS3, the former degraded less due to stiff weaving nature of jute fabric resisting microbial attack and slowing the degradation process. After 60 days of degradation, soy resin has a weight loss of about 92% while jute fabric, jute felt, WJS3 and NJS3 showed about 52%, 55%, 60% and 64% of weight loss. Such extent of weight losses indicates that prepared composites are bio-degradable and suggest their environment friendly nature after disposal unlike synthetic plastics (Zhang et al., 2005). Reduction in tensile strengths for WJS3 and NJS3 are found 96.3% and 97.6% respectively under compost condition for 30days. The loss in mechanical properties might be due to microbial attack. Rapid degradation of soy matrix from the fiber surface of composites also results in decrement of mechanical properties. Degraded composites after 30 days under compost condition were found to be fragile and hence tensile testing could not be carried out further.

3.5.2. *FTIR spectra of composite before and after biodegradation*

The FTIR spectra of jute, soy resin and NJS3 before and after 60 days of biodegradation are shown in Fig. 5(b). A peak appears at around 3420 cm^{-1} due to —OH stretching, which broadens after biodegradation for all samples. The broad peak between 2700 cm^{-1} and 2900 cm^{-1} in case of jute is attributed to C—H stretching whose intensity reduces for its concerned biodegraded samples

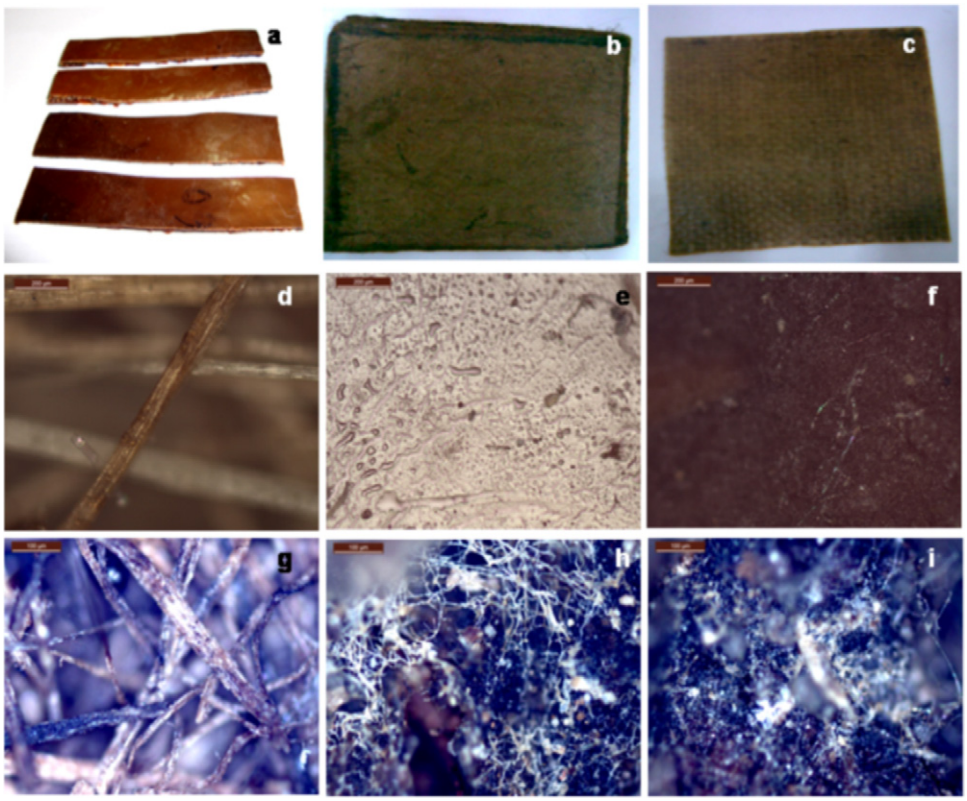

Fig. 6. Photograph of (a) SRF, (b) NJS3 (c) WJS3 and Optical photograph of (d) jute, (e) jute after degradation, (f) SRF10, (g) SRF 10 after degradation (h) NJS3, and (i) NJS3 after degradation.

due to loss of cellulose and hemicellulose. The characteristic peaks at around 1745 cm^{-1} and 890 cm^{-1} are for C=O stretching and bending of ester group of hemicellulose present in jute which after 60 days under compost conditions, vanished completely indicating the degradation of hemicellulose by microbes present in soil (Dash et al., 2002). Peak at around 1240 cm^{-1} corresponds to C—N stretching of proteins and amino sugars present in soy resin and NJS3, and the intensity of these peaks diminished after 60 days of biodegradation, indicating loss of soy protein from composite sample. Similarly for soy resin and NJS3, the C=O stretching band, which occurs at around 1746 cm^{-1} completely disappears after 60 days of biodegradation indicating degradation of sample in soil burial condition (Kumar, Yakubu, & Anandjiwala, 2010).

3.5.3. Optical microscopic observation of degraded surface

Optical photograph of jute, soy resin and NJS3 before and after 60 days of biodegradation are shown in Fig. 6. Raw jute fiber (Fig. 6(d)) broke down and its length decreased after 60 days under soil burial degradation (Fig. 6(g)). Photograph of resin film (Fig. 6(e)) before degradation shows moisture attached on the smooth surface, but after 60 days under soil burial condition the surface topography changed and found degraded surface with full of grooves and pits leaving fibrous structured of degraded portion (Fig. 6(h)). These fibers structure of resin film obtained may be due to degradation of protein part of soy resin by soil microbes. The smooth surface of NJS3 (Fig. 6(f)) changed into rough and dirty after biodegradation (Fig. 6(i)) as a result of removal of resin from composite surface (Cao, Shibata, & Goda, 2007). Some fibrous structure obtained on the composite surface but not so much prominent as compared to that of the soy resin. So among jute, resin and composite, resin degradedation is more rapid as compared to other two, due to its hydrophilic nature.

4. Conclusion

Jute reinforced soy resin composites were prepared by compression molding process without any chemical treatment on jute surface and use of hazardous chemical solvent. Simple water based soy resin (soy milk) were used instead of SPC and SPI for fabricating jute composite. Composite prepared with 60 wt% jute felt showed highest tensile strength of 37.1 MPa and tensile modulus of 1040 MPa. Water absorption of these composites is moderately high, which can be reduced by using different resin modifier. Weight loss, FTIR and optical photograph of composites after soil burial degradation revealed that composites are biodegradable in nature. The basic advantage of this composite is its biodegradability that ensures safe disposal of waste material to the environment. The probable end applications of these composites are in automotive sector, indoor furniture, packaging, sapling pots, computer cabinets etc. as a replacement of eco-hazardous plastic and plastic reinforced composites. Developed jute composites also may be applicable as decorating materials, false sealing, and workplace cubicles. Since these composites are biodegradable and leave safe carbon residue after complete biodegradation, so they can be dumped to form effective organic manure and improve fertility of soil without harming environment.

Acknowledgement

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