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Mubarak Khan¹, Sushanta Ghoshal², Ruhul Khan¹, Shamim—Ara Pervin³ and Ahmed Mustafa²

PREPARATION AND CHARACTERIZATION OF JUTE FIBER REINFORCED SHELLAC BIOCOMPOSITES: EFFECT OF ADDITIVE

¹ Radiation and Polymer Chemistry Laboratory, Institute of Nuclear Science and Technology, Bangladesh Atomic Energy Commission, GPO Box: 3787, Dhaka-1000, Bangladesh, makhan.inst@gmail.com

² Department of Applied Chemistry and Chemical Technology, University of Dhaka, Bangladesh.
 ³ Department of Materials and Metallurgical Engineering, Bangladesh University of Engineering and Technology, Dhaka-1000, Bangladesh.

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Abstract: Hessian cloth (jute fabrics) samples were soaked in the alcoholic shellac solution and dried at 333 K for 4 hours. Six layers of shellac treated hessian cloth were heat pressed (373 K for 10 min at 5 MT pressure) to fabricate biocomposite and then its mechanical properties were evaluated. To improve the mechanical properties of the biocomposite, a series of formulations was prepared using varying percentages of urea (0.25 to 30 %) with shellac in methanol; then the composite was fabricated using same parameters. Mechanical properties such as tensile strength (TS), bending strength (BS), tensile modulus (TM), bending modulus (BM), elongation at break (Eb), impact strength (IS) of the biocomposite were determined. Percentage of urea and soaking time of hessian cloth for the composites were optimized over mechanical properties. The biocomposite prepared with 0.5 % urea at 2 min soaking time showed the highest mechanical properties (TS – 79 MPa, BS - 74 MPa, BM - 7 GPa, IS - 13 kJ/m² and Eb -11.3 %). Scanning electron microscopic images of the fracture surface of the composites were suggested.

Keywords: jute, shellac, biocomposite, mechanical properties, urea.

1. Introduction

Over the last few years, several production technologies have been developed for fiber reinforced polymer composite processing. Lignocellulosic fibers have been identified as a potential substitute for commonly applied man-made synthetic fibers in the preparation of composites due to their lightweight, low cost, no hazardousness and above all environmental friendly characteristics [1]. However, some of the infirmities of natural fiber such as wettability, non-compatibility with

some polymeric matrices and high moisture absorption have prevented complete replacement of synthetic fiber [2-3]. Jute, being one of the most important lignocellulosic agro fibers cultivated in the South Asian region, has attracted worldwide attention as a potential reinforcement of polymer composite because of its inborn properties such as low density, high tensile modulus and low elongation at break [4]. Jute can be used as reinforcing agent to prepare fully biodegradable composite. Biocomposite developments are promising in view of environmental protection. World is moving forward to develop ecofriendly and biodegradable composites which can replace the synthetic ones [5]. There are some biodegradable thermoplastic natural resins which can be used as matrix material with the natural fiber reinforcing agent to prepare biocomposites. Shellac is one of such types of resins. It is a natural, biodegradable, renewable resource product made by bugs. It is derived from the hardened secretion of the lac insect, known as Kerria lacca (Kerr) and cultivated mainly in South Asia. These are scale-like insects, which are parasitic on some trees and bushes cultivated in India, Bangladesh, Burma, Thailand, Laos, Cambodia and Vietnam. Shellac is a hard, tough, amorphous resin, which is nontoxic and produces films of good water resistance and exceptional gloss [6]. Many researchers in different laboratories have been working on jute reinforced polymer composites [7-10]. Khan et al. have prepared degradable jute reinforced polymer composites using different matrix materials [1, 4, 11-13]. Several reports have also documented where the jute fibers are used as reinforcement in thermoplastics like polyethylene and polypropylene and thermo sets like unsaturated polyester and epoxy resin [14].

The aim of the study was to prepare biocomposites made of natural fiber (jute) with naturally occurring resin (shellac) using biodegradable coupling agent (urea) ensuring superior mechanical properties.

2. Experimental

2.1. Materials

Bleached hessian cloth (jute fabric) was supplied by Bangladesh Jute Research Institute (BJRI), Dhaka, Bangladesh. Shellac (raw seedlac) was supplied by Lac Research Station, Bangladesh Agricultural Research Institute (Chapai Nawabgonj, Bangladesh). Urea was collected from BDH, UK. Methanol was used as solvent and obtained from Merck, Germany.

2.2. Methods

The hessian cloth was dried in an oven at 378 K for about 20 h to remove moisture and stored in the desiccators. Seedlac was dissolved in methanol to extract pure shellac. The solution was then filtered and clear filtrate was kept in a beaker to make it dry at room temperature. Nine different shellac formulations in methanol were developed with varying urea percent and are given in the Table 1. Moisture free Hessian cloths were soaked into shellac formulations for different soaking times. The treated cloths were then dried at 333 K for 4 h. Six layers of the treated hessian cloth were then pressed using a heat press (Carver, USA) at 373 K, 6 tons pressure for 10 min. The samples were cooled for 5 min using another press. Following that the samples were packaged in a polythene bag and kept in the desiccators.

Table 1
Percentage of shellac and urea (w/w) in methanol to prepare formulations

Formulations	F1	F2	F3	F4	F5	F6	F7	F8	F9
Shellac, %	100	99.75	99.5	99	98	97	95	85	70
Urea, %	0	0.25	0.50	1	2	3	5	15	30

2.3. Characterizations

The matrix (shellac) material loading (ML) of the composite was determined from the difference in weight between final weight of the composite and the weight of the dried hessian cloth. Percentage of jute content in each prepared biocomposite was also measured. The tensile properties were determined for the composites according to DIN 53455 and DIN 53452 standard methods by a Universal Testing Machine (Hounsfield Series S, UK). Three point bending test was carried out using UTM testing machine (model 1011, UK) in accordance with ASTM D-790. The thermo-mechanical properties of the composites were determined using TMA (LINSEIS TMA, L-77, USA). The fracture surfaces of the composites were studied by Scanning Electron Microscope (Philips, UK).

3. Results and Discussion

3.1. Optimization of the percent of urea in the biocomposite

The matrix (shellac) material loading (ML) by the Hessian cloth during soaking are presented in the Fig.1 as a function of urea content. It was found that with the increase of urea, the ML was also increased and at 30 % urea content, the ML values decreased slightly. The highest value is obtained for 15 % urea content (F8 formulation).

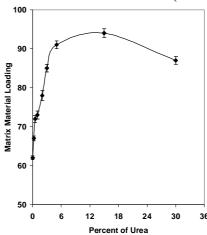


Fig.1. The matrix material loading by the Hessian cloth as a function of urea content

Jute content (wt %) in the composite was varied and is given in the Table 2. The TS values of the biocomposite are presented in Fig. 2 against different urea content. TS values were found to increase with the increase of the percent of urea up to 0.5 % urea (F3) and then decreased. The composite (0.5 % urea) produced the highest TS value 78.9 MPa, which is 48.86 % higher than that the of the control samples.

Table 2
Percentage of jute in jute-shellac biocomposite with respect to formulations

Formu- lations	F1	F2	F3	F4	F5	F6	F7	F8	F9
Jute, wt %	61.6	59.7	58.2	58.1	56.6	54.0	52.3	51.4	53.3

The Eb of biocomposite are presented in Fig. 3 against various percantage of urea content. Eb values decreased with the increase of formulation up to 3 % urea concentration and then increased up to 30 % of urea content (F9). The highest Eb value is obtained at F1 formulation containing no urea.

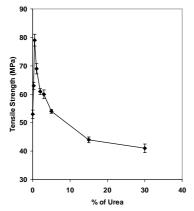


Fig 2. The tensile strength of the composite

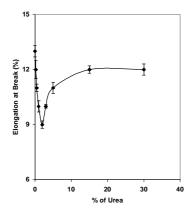


Fig. 3. The elongation at break of the composite

3.2 Optimization of soaking time

Dried Hessian cloth layers were soaked into 0.5 % urea (F3) containing solution (optimized with respect to tensile strength as described earlier) for 1-5 min. The ML of Hessian cloth during soaking are presented in Fig. 4 against various soaking times. The ML found the lowest value when Hessian cloth samples were soaked in F3 formulation for 1 min. It increased with the increase of soaking time up to 2 min in the formulation and then decreased as soaking times increased up to 5 min.

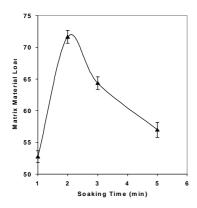


Fig.4. The matrix material loading on Hessian cloth

The TS values of the composite are presented in Fig. 5 against various soaking times. The TS values were found to increase with the increase of soaking time up to 2 min and then decreased. The composites prepared using F3 formulation at 2 min soaking time demonstrated the highest TS value 78.9 MPa. The Eb of composite are presented in Fig. 6 against various soaking times. Eb values increased with the increase of soaking time up to 2 min and then decreased up to 5 min soaking time. The highest Eb value is obtained at F3 formulation at 2 min soaking time.

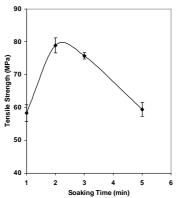


Fig. 5. The TS of the composite against various formulation

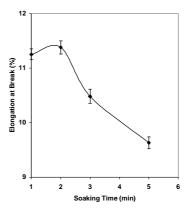


Fig. 6. The Eb of the composite

3.3 Thermal properties

The thermal properties (on set, glass point and off set of melting points) of the matrix and the composites were evaluated using thermo-mechanical analyzer. The on set, glass point and the off set of melting points of the pure shellac and the composites (F3 formulation at 2 min soaking time) are given in Fig. 7. It was found that the thermal properties of the composites improved significantly compared to pure shellac. The on set of melting temperatures for the pure shellac and the composite was found to be 316.3 K and 345.3 K, respectively. An important thermal property – glass point temperature for the pure shellac and the composite - was found to be 320.9 K and 349.1 K, respectively. From this investigation it is clear that the thermal stability of the composites improved dramatically when urea and jute are incorporated into the matrix. Probably, higher thermal properties are attributed to the complex reaction between hydroxyl group of jute, shellac and urea.

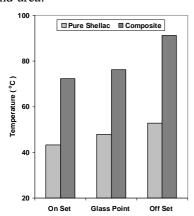
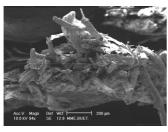


Fig. 7. Thermal properties of the composites

3.4 Fractography of the composites measured by SEM

The fracture surface of the control and urea containing composites (F3 formulation at 2 min soaking



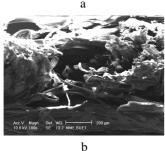


Fig 8. The fracture surfaces of the untreated (a) and urea treated (b) composites

time) are given in Fig. 8. It is found that the fiber pull out is higher in control sample (Fig. 8a) compared to that of the urea containing composite (Fig. 8b). As was reported above urea containing composites showed better mechanical properties over the untreated one. The SEM images supported the findings mentioned here. It is possible that urea acted as a good stiffening agent in the composites promoting better fiber matrix adhesion and demonstrating better thermal and mechanical properties.

4. Conclusions

Totally degradable biocomposite was prepared by using Hessian cloth and shellac. The amount of additive in the composite and soaking time were optimized. The best tensile strength of the composite was obtained when 0.5 % urea was used at 2 min soaking time. TMA suggested that the melting temperature of the composite improved in contrast to the matrix. The prepared composites possessed excellent mechanical properties and is suitable for general construction materials.

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ПРИГОТУВАННЯ І ХАРАКТЕРИСТИКА ЗМІЦНЕНИХ ШЕЛАКОВИХ БІОКОМПОЗИТІВ З ДЖУТОВОГО ВОЛОКНА: ВПЛИВ ДОДАТКІВ

Анотація. Вивчено зразки джутової мішковини, вимоченої у шелаковому спиртовому розчині і висушеної при 333 $\,K\,$ протягом 4 годин. Оцінено механічні властивості біокомпозиту, приготовленого із шести шарів обробленої мішковини під тиском та температурі 373 К протягом 10 хв. Вивчено вплив низки додатків з різним вмістом сечовини (від 0,25 до 30 %мас.) у розчині шелаку в метанолі на такі механічні властивості біокомпозиту, як межа міцності при розриванні та згинанні (ММР і ММЗ), модуль розривання і згинання (МР і МЗ), подовження при розриванні (ПР) та ударну в'язкість (УВ). Встановлено, що найкращі механічні властивості (ММР-79 МПа, ММЗ-74МПа, MP- $7\Gamma\Pi a$, ΠP -11,3 %, VB-13 кДж/м 2) проявля ϵ біокомпозит із вмістом сечовини 0,5 % при тривалості томлення 2 хв. 3 використанням скануючої електронної мікроскопії наведені зображення поверхні розривання композитів.

Ключові слова: джут, шелак, біокомпозит, механічні властивості, сечовина.