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RESEARCH ARTICLE

PREPARATION OF ENVIRONMENT FRIENDLY COMPOSITES FROM EFFLUENTS OF ALEURITIC ACID INDUSTRY AND MODIFIED BETEL-NUT FIBER

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Abstract

The synthesis of aleuritic acid from shellac is associated with generation of huge quantity of waste whose disposal leads to clogging of the sewage system and contaminates the water bodies into which the drain leads. The shellac waste, which was collected from the factory site and betel-nut fiber has been used to form a sustainable, eco-efficient and economical composite. Mechanical properties of the composites were measured. The morphology of the composite was studied with the help of Scanning Electron Microscopy (SEM).
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Keywords: shellac waste, betel-nut fiber, composite, characterization.

Introduction

Shellac in its refined form is a polyester type of resin consisting of inter-and intra-esters of polyhydroxy carboxylic acids formed from certain hydroxy acids and sesquiterpene acids. It is believed to have five hydroxyl groups including vicinal hydroxyl group, one carboxyl group, in free state, three ester groups, one double bond and one partly masked aldehyde group, and the probable linkages are ester, acylal, acetal and ether in an average molecule. [Sharma et al (1983)]

From a study of the composition of total shellac acids [Khurana et al (1964)], it was concluded that the chief building blocks of shellac are aleuritic acid and jalaric acid-A (a tricyclic sesquiterpene) connected by lactide and ester linkages. Aleuritic acid, which is mainly used in the perfumery industry, as a starting material for the preparation of isoambritolite a major constituent of synthetic "musk" aroma compounds as well as medicinal and bioactive compounds, is isolated from the resin by saponification. The synthesis of aleuritic acid is associated with generation of huge quantity of waste amounting to almost ten times the weight of the produced aleuritic acid. The discharge of these wastes creates a huge problem as gallons of waste are released from a single shellac factory each day. This waste disposal leads to clogging of the sewage system and contaminates the water bodies into which the drain leads. The Pollution Control Board has expressed serious concern about waste disposal of this nature. Work on utilization of shellac industry effluents has also been reported from IIT, Delhi and All-India Inst. Hyg. Public Health, Calcutta. Several methods have been reported on utilization of lac waste [Chawla (1999); Oki et al (1970); Seth et al (1955); Bhowmik (1980); Ajore et al (1973); Goswami et al (2004); Sao et al (2009)]. These methods mainly deal with process for development of anti-fungal coating from shellac waste [Chawla (1999)], preparation of organic fertilizers [Oki et al (1970)], preparation of laccoated urea [Bhowmik (1980)], utilization of lac mud for the reclamation of alkali soils [Ajore et al (1973)], preparation of high thermal resistant baking type insulating varnishes [Goswami et al (2004)], preparation of medium density particle board in conjunction with lac resin [Sao et al (2009)].

Natural / biofiber composites based on annually renewable agricultural and biomass feedstock can form the basis for a portfolio of sustainable, eco-efficient products and are emerging as a viable alternative to glass fiber reinforced composites [Mohanty et al. (2002); Satyanarayan et.al. (2006)].

The present work targets at preparation of economically viable composite from effluents of shellac industry and betel-nut fiber. This work will also provide a feasible and economically viable solution to waste disposal from the shellac industry.

Experimental

Raw materials

Shellac waste, which is the effluent of the factory after extraction of aleuritic acid, was collected from the factory site. The solvents and chemicals used were of laboratory grade.

Preparation of resin

The aqueous waste liquor as obtained from shellac industry (after isolation of alueritic acid from shellac) was washed repeatedly with cold water and then with hot water to free from NaCl. The waste liquor was then treated with dilute HCl to precipitate out. The precipitated sticky mass was washed repeatedly with cold water and then with hot water to free it from NaCl. It was then heated to 120 °C and 11% by weight of maleic anhydride was added for esterification. Subsequently, it was heated to 140°C for half an hour. 5% Oxalic acid was then added as catalyst and then heated at 160°C for fifteen minutes. Then it was poured into a plate and allowed to solidify. [Biswas et al (2013)]

Preparation of composite

The prepared resin was heated to about 160 °C and then 10 % by weight of dolomite was added and mixed thoroughly. Mercerization was applied to betel-nut fibres for improving the surface topology and improving surface contact of the fibres within the matrix. The treatment was carried out by boiling the betel-nut fibres in 1 % Sodium Hydroxide (NaOH) solution for 1 h. After mercerization, the fibres were washed for several times with water and finally washed in very dilute acetic acid solution and then rinsed in de-ionized water until residual sodium hydroxide was removed completely as examined by pH paper. The fibre was then dried at hot air oven for 24 h at 70^0 C and stored in a sealed plastic bag before use.

Dried betel nut fibres were arranged into uniform mats of thickness 3-5 mm. These fibre mats and the mold measuring 120 × 120 × 3 mm were p reheated at 80 ^{0}C for 5 min and then cooled. Silicon oil, a mold releasing agent was applied on the surface of the mold to facilitate easy removal of the composite after curing. The resin was degassed before pouring into the mold. The resin- dolomite blend was spread at the bottom of the mold and one betel nut fibre mat was placed and dipped on it. Again a thin layer of resin was spread on it and another fibre mat was placed over it. The process was repeated to get the proper thickness and in each step the air bubbles which were entrapped inside the mat were released by pressing with a steel roller. Some resin was poured on the top. Mold was closed and kept under pressure (1 ton) at ambient temperature for few hours to effect curing.

Characterization of the composite

The density of the composite was found to be 1.18 g ml^{-1}. The moisture absorption was almost negligible. The mechanical properties for the composite were studied for different fibre lengths and weights. Characterization of the composite by SEM (Fig-3, 4) was carried out.

Results and Discussion

The shellac waste collected from the factory site contains some leftover aleuritic acid linked to the terpenic acid in alkali stable linkage. On addition of maleic anhydride the free hydroxyl groups gets esterified. The resin thereby formed is mixed with 10 % dolomite. The presence of dolomite reduces the moisture absorptivity of the composite. Greater amount of dolomite is avoided keeping in mind the ultimate cost of production. It was found that the addition of mercerized betel-nut fiber as filler bestows good mechanical strength to the composite. The chemical reaction between lignocellulosic fibre and NaOH can be shown in the following way:

$$\text{Cellu–OH} + \text{NaOH} \longrightarrow \text{Cellu–O}^-\text{Na}^+ + H_2O + \text{impurity}$$

Figure 1. Variation of tensile strength with percentage weight of fibre for different lengths of betel nut fibre

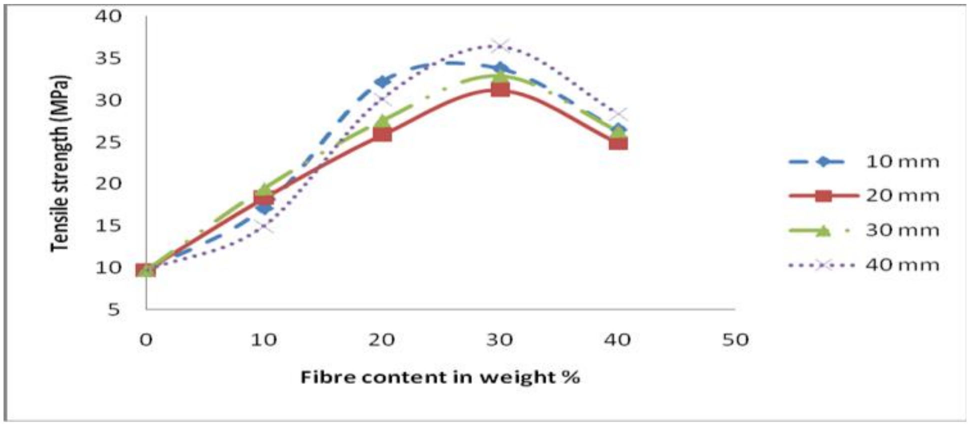


Figure-2: Variation of flexural strength with percentage weight of fibre for different lengths of betel nut fibre

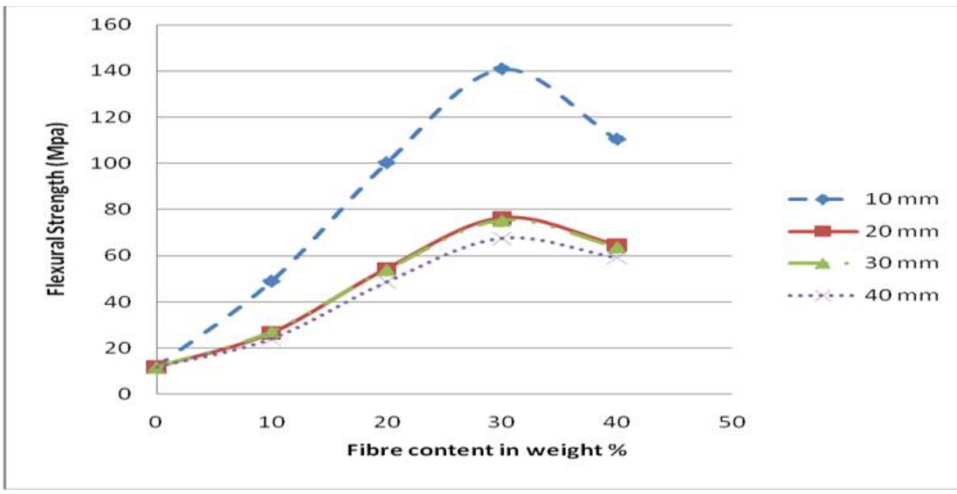
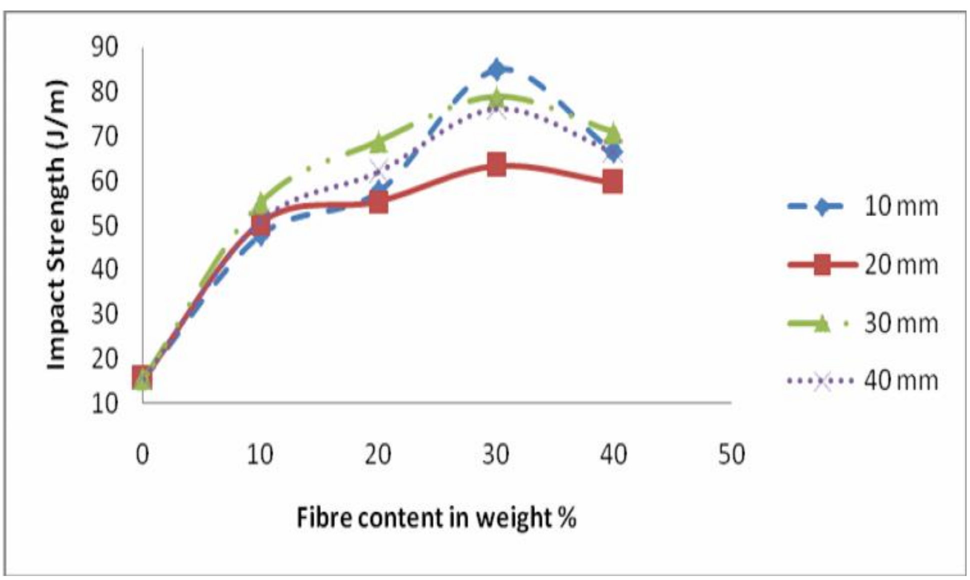


Figure-3: Variation of Impact strength with percentage weight of fibre for different lengths of betel nut fibre



Natural fibre is hydrophilic due to a large number of hydroxyl groups in their structure which makes it incompatible with polymer matrix that is hydrophobic in nature. In this reaction, a number of hydroxyl groups can be eliminated, which decreases the tendency of water absorption and improves the compatibility between natural fibre and polymer matrix.

Mechanical Properties

The mechanical properties of betel nut fibre reinforced composites (BFRC) were measured.

Tensile Strength

It is observed that the tensile strength of BFRC increases significantly with fibre loading from 10-30% but decreases with fibre loading 40% (Figure-1). The highest value of mechanical properties is exhibited by the composite containing 30% fibre loading. It can be explained in terms of orientation and homogeneity of fibres within the matrix. At this stage fibres get maximum level of orientation and mix homogeneously within the matrix and actively participate in stress transfer. When load is applied, stress is uniformly distributed among the fibres. As a result, mechanical properties of the composite achieve maximum values. At low levels of fibre loading, the orientation of fibres was poor, the fibres were not capable of transferring load to one another and stress accumulated at certain points of the composites. This has led to decrease in the mechanical strength. On the other hand, at higher level of fibre content (>30%) agglomeration of fibres within the matrix takes place, which produces non uniform stress transfer capacity. Hence stress transfer was partially blocked resulting in lowering of mechanical properties above 30% loading.

Flexural and Impact Strength

Similarly, the values of flexural strength also increase significantly with fibre content from 10 - 30 % (Figure- 2). The most obvious reason for the identical results of tensile and flexural strength is due to increase of betel nut fibre content in different length. Further, it is seen that better results of flexural strength is obtained for composite with 10mm fibre length. This may be due to short fibre end interaction with matrix.Like tensile and flexural strength, impact strength also increases with increasing fibre loading (Figure- 3).

SEM

SEM images (Figure- 4 & 5) of a single untreated and treated betel nut fibre are presented respectively. A lot of foreign substances are trapped on untreated fibres which are removed due to alkali treatment. This makes the fibre surface rough. Alkali treatment also eliminates hemicelluloses and lignin for which, trichomes, the tiny, hairy, fine outgrowths protrude out from the fibre surface.

Figure 4. SEM of single untreated betel nut fibre

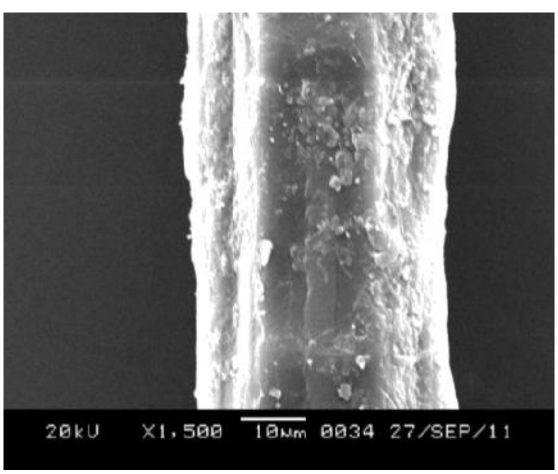


Figure 5. SEM of single treated betel nut fibre

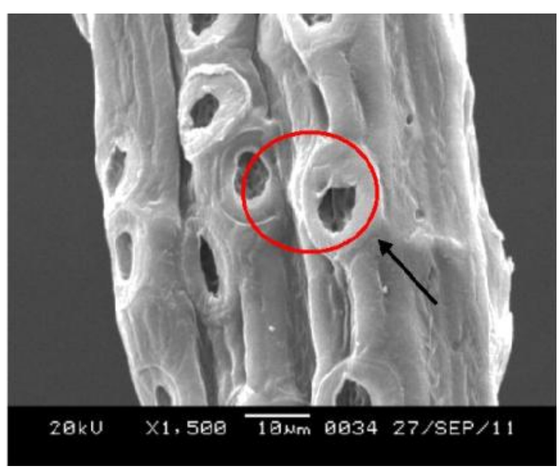


Figure 6. Fracture surface of 10% BFRC

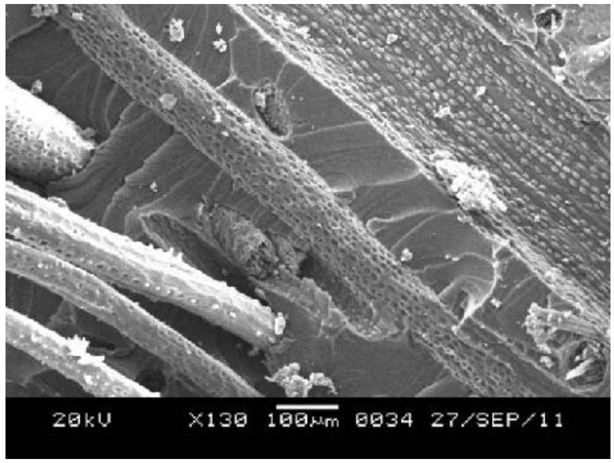
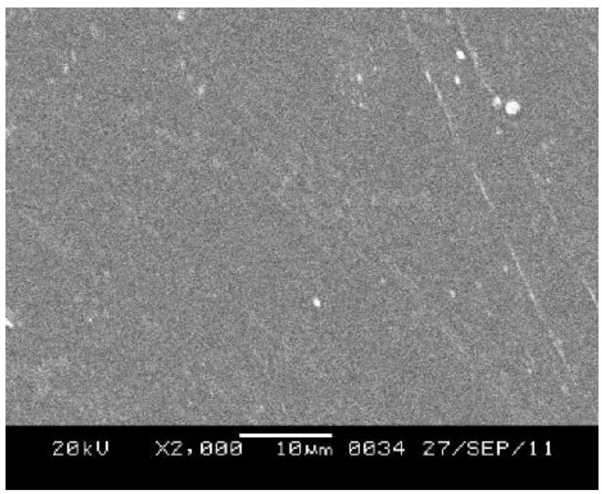


Figure 7. Surface morphology of 10% BFRC



The trichomes give mechanical gripping with fibre and matrix by nature and thus resisting interfacial failure between them. The trichomes make betel nut fibre unique as it has not been evidenced in other natural fibres. The SEM micrograph (Figure- 6 & 7) gives visual information about the dispersion of fibres in the matrix system.

Application

The composite thus formed has very good mechanical properties Most of the cost of production for the preparation of composites of this type is due to t he high cost of the resin. The present method will be using discharged industrial waste for the preparation of resin, thereby reducing the cost of production of the composite.

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