

Fabrication of transesterified cured coir reinforced composites and evaluation of their properties

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ABSTRACT

This article describes the fabrication of simple and hybrid composites of parent, alkali treated, transesterified and benzoyl peroxide (BPO) cured transesterified coir fibers. Simple and hybrid composites of coir fiber (20-35%) in chopped mat and glass fabrics (11%) along with unsaturated polyester resins (USP) were fabricated using catalyst (methyl ethyl ketone peroxide) and accelerator (cobalt naphthenate) under appropriate reaction conditions. Similarly, USP resin sheets (cured polyester sheets) were fabricated using USP, catalyst, and accelerator. Resin content, density and mechanical properties such as tensile strength, flexural strength and flexural modulus of fabricated composites and cured polyester sheets were determined. The density data was directly related to the nature of chemical modification imparted onto coir fiber. Tensile strength, flexural strength and flexural modulus of composites fabricated with transesterified and BPO cured transesterified coir along with matrix resin (USP) have higher values in comparison to the composites fabricated with using parent coir along with matrix resin (USP). The data of the mechanical properties are self-explanatory on the basis of the interfacial bond strength between the coir fibers and the matrix resins. Such bond strength between coir fiber reinforcing candidate and the matrix resins as predicted be attributed to the nature of chemical modification imparted onto fiber and hydrophobicity of modified coir.

1. Introduction

A fiber reinforced resin system is a composite material comprising a network of reinforcing fibers embedded in a matrix of polymeric resin. Other materials such as fillers and pigments may also be present, although they are not an essential part of the composite. In general the resin used consists of a syrupy liquid which when combined with a suitable catalyst or hardener can be cross-linked into a hard infusible solid. By impregnating the fibrous material, frequently in the form of a cloth or mat, with the catalyzed resin and allowing the resin to cure, the composite material or laminate is formed. This process is carried out in a mould which may be open or closed. The resultant product is a fully cured moulding which is an exact replica in reverse of the original mould surface.

Reinforced plastics have gained popularity over conventional materials because of high strength to weight ratio and rigidity, ease of fabrication, unlimited moulding size, wide range of manufacturing techniques, low cost, good water resistance, resistance to a variety of chemical reagents and resistance to weathering/corrosion. Good degree of transparency may be achieved if needed and can be made fire-retardant and electrically/thermally resistant. These overwhelming advantages of composites have been prime criteria for their wide industrial applications [1-11] and one would feel as if all requisites for human living can be made with FRP.

Reinforcements ordinarily are natural or synthetic fibers [12-21] that are chosen to impart specific physical, chemical or thermal properties to the reinforced plastics. The

reinforcements may be used as continuous filaments or as chopped fibers. Reinforcing fibers are selected on the basis of their physical properties and thermal stability depending on the desired end use of the final composites. Many different fibers and wires are used as reinforcements. The choice of reinforcements depends on the required properties of composites. The strength to weight ratio of reinforced plastics is attributed largely to the nature of the reinforcements. The matrix material or resin though not in itself providing much strength, is essential as it serves to bond the reinforcements together and transmits the load to the reinforcing fibers.

Catalysts or initiators are chemical agents that initiate curing or cross-linking of the matrix resins during the fabrication of composites and at times they need the help of an accelerator (or promoter) that makes the curing to occur at much ease rate [22-26]. The type of catalyst to be used in the fabrication of composite mostly depends on the nature of the matrix resins. For unsaturated polyester resins the catalysts are organic peroxides and hydroperoxides such as benzoyl peroxide, cumene hydroperoxide, Methyl Ethyl Ketone Peroxide (MEKP) etc. Accelerators and Promoters are materials which when used in conjunction with the initiators make the curing reaction facile [22-26] to occur at ambient temperature. For polyester resins they are mostly metal compounds and tertiary amines, such as cobalt naphthenate, siccatoate or octoate, dimethyl aniline, diethyl aniline, dimethyl p-toluidine etc. Lignocellulosic fibers or Natural fibers are materials rich in lignin, hemicellulose, cellulose, pectin and some water soluble materials. Coir is a naturally occurring fiber and is available plenty in the coastal belt of India. Coir is a multi-component fiber whose chemical constituents are cellulose (36-43%), lignin (41-

45%), hemicellulose (0.15-0.25%) and pectin together with some water soluble material [27].

A wide range of possible moulding process can be undertaken with the use of a suitable mould [9, 28-30]. The selection of a suitable mould is one of the most important steps since it affects the quality of the moulding and ease of production. Various types of moulding process such as Pressure bag moulding, Vacuum bag moulding, Contact moulding (wet-layup), Pressure injection moulding, Foam reservermoulding, Cold press moulding, Hot press/matched metal moulding, Vacuum-impregnation and injection, Resin injection or Resin transfer moulding, Transfer moulding, Centrifugal moulding, Injection moulding, Filament binding and Continuous sheet manufacture used for the fabrications of reinforced composites. In the present investigation the simple hand lay-up technique has been adopted for the fabrication of the composites in open air.

This work has got diverse benefits. First, the agro waste fibers are abundantly available free of cost and have the distinctive properties of converted to more hydrophobic fiber after transesterification and curing. Secondly, the technique adopted here is very simple and user-friendly. Transesterification is a technique for reducing the hygroscopicity of the natural coir fiber. In this case transesterification is carried out in basic medium using catalyst sodium hydroxide or pyridine. The hydroxyl group of the cell wall of coir fiber reacts with acrylate moiety of butyl acrylate and methyl acrylate to yield acrylate incorporated fiber having ester linkages, so that fiber becomes more hydrophobic in nature. Further on curing of these transesterified fibers with BPO, phenyl and benzyl groups are introduced in the vinyl part of acrylate moiety of transesterified fibers leading to hydrophobicity and rigidity due to interlocking. Hydrophobicity of coir lead to good solubility and interfacial bond strength with the matrix resin, and the resulting composite is likely to exhibit improved properties and performance in comparison to parent coir composite with the same matrix resins. These fabricated composite material is used as window frames, window guides, flooring for toilets, roofing sheets and other multifarious applications.

2. Experimental

2.1 Materials and Methods

Coir fibers were purified by soxhlation with 1:1 benzene-ethanol mixture for 72 hours, followed by washing with acetone and ethanol and air-drying. Butylacrylate and methylacrylate were purified as reported earlier [31]. Other chemicals like sodium hydroxide, acetic acid, acetone, ethanol, sulphuric acid, hydrochloric acid, sodium chloride, benzoyl peroxide, etc., were of analytical grade (BDH chemicals) and were used after purification by standard methods.

2.2 Methods of Chemical Modification

2.2.1 Alkali Treatment of Coir

The purified coir was treated with alkali (NaOH, w/v) solution of varying concentrations (2–10%) for 1 h at 35 °C with occasional shaking followed by washing with dilute acetic acid

and distilled water for a week to leach out the absorbed alkali. The materials were then air-dried.

2.2.2 Transesterification of Parent and Alkali-treated Coir with Butylacrylate

Transesterification of 4% NaOH alkali-treated coir (1g) was done under reflux with butylacrylate (20 mL) at 50 °C for 6 hours. While transesterification of the parent coir (1 g) was done under reflux with butylacrylate (20 mL) at 50 °C for 6 hours using 0.4% NaOH (20 mL) solution as a catalyst. Gel-like material was observed during the reflux, which has been identified to be polyacrylic acid formed by thermal polymerization of acrylic acid resulting from the alkaline hydrolysis of butylacrylate. This viscous liquid was removed by washing the material several times with warm methanol. The transesterified fibers were then washed with acetic acid, followed by acetone, and with distilled water, then air-dried.

Direct transesterification of coir was also affected by refluxing the coir (1 gm) with butylacrylate (20 mL) in acetone (20 mL) using pyridine (4 mL) as catalyst at 50 °C for 6 hours. The second set of transesterification was done without adding acetone, while keeping the rest of the conditions unchanged. The reacted fibers were washed perfectly with dilute acetic acid and acetone, followed by methanol and distilled water, then finally air-dried.

2.2.3 Transesterification of Parent Coir with Methylacrylate

Transesterification of parent coir (1 gm) was done under reflux with methylacrylate (20 mL) at 50 °C for 6 hours using pyridine (4 mL) as a catalyst. The reacted coirs were washed successively with dilute acetic acid, acetone, and distilled water and dried.

2.2.4 Curing of Transesterified Coir with Benzoylperoxide

Curing of butyl acrylate and methylacrylate transesterified coir (0.4 gm) was done under reflux with 10 mL of 0.1 MBPO in acetone (40 mL) at 50–60 °C for 2 h, washed with fresh acetone and dilute acetic acid, followed by distilled water then air-dried.

2.2.5 Glass fabric

Polyester compatible glass fabric (Fiber glass Pilkington, weighing 360 gm/m²) was obtained from Acropolymers Pvt. Ltd., Faridabad, U.P., India, and was used as such with the unsaturated polyester resin for the fabrication of composites.

2.2.6 Unsaturated polyester resins, initiators and accelerators

Unsaturated polyester resin instyrene monomer, Methyl ethyl ketone peroxide (initiator) and cobalt naphthenate (Accelerator) of commercial grade were supplied by Ruia Chemicals Pvt. Ltd., Calcutta, India, and were used as such.

3. Methods

3.1 Composite Fabrication

Simple and hybrid composites of parent and chemically modified coir (20-35%) in chopped mat and glass fabrics (11%) along with unsaturated polyester resins were fabricated by hand lay-up technique in open air process. The mould surface

used were either sunmica base and/or steel mould surface. Prior to fabrication of composites the mould surface were thoroughly cleaned and dried. The mould surface is coated with a silicon free wax, which is used to give a glossy smooth finish after the application of wax. A layer of polyvinyl alcohol (PVA) is applied with a sponge as the releasing agent. The film assists the ready removal of the moulding from the mould. The resin formulation consisting 100 parts of unsaturated polyester resins, 1-2 parts of catalyst (methyl ethyl ketone peroxide) and 2 parts of accelerator (cobalt naphthenate) is chosen in composite fabrication. The resin and the accelerator after being thoroughly mixed were added to the mould. This is then followed by addition of the catalyst and the components were thoroughly mixed. Within a couple of minutes the required fibers were spread in loose form over the resin and were draped by means of a brush, such that the fibers sink well into the viscous components in the mould. The contents were then cured for 4-6 hours. Composites in the form of transparent hard sheets were then ejected carefully from the mould and then subjected to various tests.

Composites were also fabricated using chopped mat of the fibers. In this case a stainless steel mould using teflon as a spacer of desired area was taken and cleaned perfectly. The fiber mat was cut in exact shape and size to fit the moulding area and required number of layers of the mat were taken according to the composite thickness. The fiber mats are placed over the mould surface. Formulated resin (USP - 100 parts, MEKP - 1-2 parts, cobalt naphthenate - 1 part) is distributed uniformly over the mat surface. Bristling is then followed for complete absorption. The upper layer is subsequently covered with a polyester (Miller) sheet over the total moulding area. The air voids are extracted out from the system by using a rubber pad. The removal of air voids became possible because the composite components are visible through miller sheet. Finally the system is allowed to cure for 4-6 hours. With similar method hybrid composites with varying percentage of coir to glass and resin were fabricated. The fabricated composite is taken out and subjected to usual tests.

3.2 Casting of USP resin sheets

Unsaturated polyester resins with the formulation of 100 parts, Methyl-Ethyl ketone peroxide (2 part) and cobalt naphthenate (1 part) were stirred well and then poured into a sunmicamould or stainless steel mould using teflon as a spacer and Silicon/ PVA mould release agent were allowed to cure in open air for 4-6 hours. The cured polyester sheets were then wiped with filter paper to remove the mould release agent. The cured sheets were transparent and were of the desired size of the mould specifications as that of the composites.

4. Evaluation of Properties

4.1 Resin Content

The resin content of the composites with parent and modified coir was determined from the differential weight between the composite laminate and the plane polyester sheets of the same size and shape. On the other hand resin content of the hybrid composites with combined glass-coir reinforcement was determined by a pyrolysis in a furnace. For

this a known weight (0.4-0.5 gms.) of the laminate was heated in a silica glass crucible for four and half hours. From the differential weights and its comparison with plane polyester sheets and polyester coir composites of similar size and shape, the resin content were evaluated. For both types of composites the results of the resin content are recorded in tables 1-3.

4.2 Density

The density of the composites and the unsaturated polyester resins sheets were determined according to ASTM D 1475 60 (1974) using xylene and carbontetrachloride mixture in a specific gravity bottle.

4.3 Tensile Strength

Tensile strength is a measure of the ability of a material to withstand forces that tend to pull it apart and to determine what extent of the material stretches before breaking. The tensile strength of coir-unsaturated polyester composites were determined according to ISO-R-3268, 1978 type-II and ASTM D 638-77a.

Specimen: - Both the end of the specimen are firmly clamped in the jaws of an universal tensile testing machine "Instron", U.K. The jaws may move apart at rates of 0.2, 0.5, 2 or 20 inches a minute, pulling the sample from both ends. The stress is automatically plotted against strain. Thus the strength are determined.

4.4 Flexural Properties

Flexural strength or modulus is the ability of the material to withstand bending force applied perpendicular to its longitudinal axis. Flexural strength and modulus were determined according to ASTM D790-71 using Instron, (Model No.1122).

Specimen: Rectangular specimens of 80mm × 10 ± 0.2 mm × 3.8 mm were machined from the composite sheet.

Procedure: - The specimen were placed on the two supports spaced 16h (where h = thickness) apart. A span-to-depth ratio of 32:1, a cross head speed of 5mm/min and a chart speed of 50mm/min were used while applying the load at the centre of the specimen. The flexural strength and flexural modulus were calculated using the relationships[9].

$$\text{Flexural strength} = 3PL/2bd^2$$

$$\text{Flexural modulus} = L^3m/4bd^3$$

Where P = Maximum Load at rupture N (lbf)

L = Span length m (in)

b = depth of specimen m (in)

m = Slope of the tangent to the initial straight line portion of load deflection curve N/m (lbf/in).

5. Results and Discussion

5.1 Density

As may be seen in table 1-3, the density of the composite with parent and alkali treated coir are higher than the simple cured USP resin laminate and composites fabricated with chemically modified coir. The density data is directly related to the nature of chemical modification imparted onto fiber and hydrophobicity of modified coir.

5.2 Mechanical Properties

The mechanical properties such as tensile strength, flexural strength and flexural modulus of unsaturated polyester resin sheet (100% USP), and the composites with 65% resin to 35% of parent and modified coir, and the hybrid composite of modified coir (24%) and glass (11%) with 65% USP content are presented in table 1-3. The data of the mechanical properties are self-explanatory on the basis of the interfacial bond strength between the fibers and the matrix resins. Such bond strength between the natural fiber reinforcing candidate and the matrix resins as predicted be attributed to the nature of chemical modification and hydrophobicity of modified fiber.

From table 1 and 2 it is found that, the density of the composite with parent and alkali treated coir (Coir-ONa) are higher than the simple cured USP resin laminate and composites fabricated with using transesterified and BPO cured transesterified coir respectively. The density data is directly related to the nature of chemical modification imparted

onto fiber. Further the tensile strength, flexural strength and flexural modulus have less values for the composites fabricated with using Coir-ONa in comparison to the composites fabricated with using parent coir. But tensile strength, flexural strength and flexural modulus of composites fabricated with using all the transesterified and BPO cured transesterified fiber have higher values in comparison to the composites fabricated with using parent coir. It is found that among all transesterified and BPO cured transesterified fiber, very good mechanical properties are shown by the composites fabricated with using Coir-MA/Py and Coir-MA(Py)-Cu-BPO respectively. From table 3, the density of hybrid composites containing Coir-BA/Py-Acetone and Coir-BA(Py-Acetone)-Cu-BPO are found to be higher than USP laminates, which arises due to better reinforcing ability of Coir-BA/Py-Acetone, Coir-BA(Py-Acetone)-Cu-BPO and glass with USP resin. Both tensile strength and flexural strength increases two times for hybrid composites containing Coir-BA/Py-Acetone and Coir-BA(Py-Acetone)-Cu-BPO respectively, in comparison to USP laminates. Similarly flexural modulus increases three times for hybrid composites containing Coir-BA/Py-Acetone and Coir-BA(Py-Acetone)-Cu-BPO respectively, in comparison to USP laminates. These increase in mechanical properties arises due to better reinforcing properties of modified coir and glass in hybrid composites containing Coir-BA/Py-Acetone and Coir-BA(Py-Acetone)-Cu-BPO respectively.

Test Results

Table 1 Properties of virgin unsaturated polyester Resin (USP) sheet and composites comprising USP Reinforced with parent and chemically modified (alkali and transesterified) Coir.

| PROPERTIES TESTED | SAMPLES | | | | | | | |
|---|----------------|------------------|------------------|---------------------|------------------|------------------|-------------------|------------------|
| | USP | Parent Coir | Coir-ONa | Coir-BA/ Py-Acetone | Coir-BA/Py | Coir-BA/ 4% NaOH | Coir-BA/0.4% NaOH | Coir-MA/Py |
| Resin Content (w/w %) | 100 | 65 | 65 | 65 | 65 | 65 | 65 | 65 |
| Coir Content (w/w %) | - | 35 | 35 | 35 | 35 | 35 | 35 | 35 |
| Glass Content (w/w %) | - | - | - | - | - | - | - | - |
| Density (gm/cm ³) | 1.12 | 1.31 | 1.33 | 1.26 | 1.24 | 1.28 | 1.27 | 1.28 |
| Tensile Strength MPa Psi x 10 ³ | 30.4 (4.4) | 48.9 (7.09) | 47.5 (6.88) | 52.2 (7.57) | 53.9 (7.81) | 50.9 (7.38) | 51.8 (7.51) | 54.5 (7.9) |
| Flexural Strength MPa Psi x 10 ³ | 60.7 (8.8) | 106.3 (15.41) | 102.6 (14.88) | 107.5 (15.59) | 114.0 (16.53) | 112.0 (16.24) | 110.8 (16.0) | 115.3 (16.72) |
| Flexural Modulus GPa Psi x 10 ⁵ | 2.12 (3.07) | 4.1 (5.94) | 4.0 (5.8) | 5.3 (7.68) | 5.1 (7.39) | 5.2 (7.54) | 5.3 (7.68) | 5.5 (7.97) |

Table 2 Properties of virgin unsaturated polyester Resin (USP) sheet and composites comprising USP Reinforced with parent and chemically modified (alkali and BPO cured transesterified) Coir

| PROPERTIES | SAMPLES | | | | | | | |
|--|---------------|----------------|----------------|----------------------------|--------------------|--------------------------|----------------------------|--------------------|
| | USP | Parent Coir | Coir-ONa | Coir-BA(Py-Acetone)-Cu-BPO | Coir-BA(Py)-Cu-BPO | Coir-BA (4% NaOH)-Cu-BPO | Coir-BA (0.4% NaOH)-Cu-BPO | Coir-MA(Py)-Cu-BPO |
| Resin Content (w/w%) | 100 | 65 | 65 | 65 | 65 | 65 | 65 | 65 |
| Coir Content (w/w%) | - | 35 | 35 | 35 | 35 | 35 | 35 | 35 |
| Glass Content (w/w%) | - | - | - | - | - | - | - | - |
| Density (gm/cm ³) | 1.12 | 1.31 | 1.33 | 1.25 | 1.23 | 1.25 | 1.26 | 1.26 |
| Tensile Strength MPa Psi x 10 ³ | 30.4 (4.4) | 48.9 (7.09) | 47.5 (6.88) | 57.5 (8.33) | 55.9 (8.1) | 56.3 (8.16) | 56.9 (8.25) | 60.0 (8.7) |

| | | | | | | | | |
|--|----------------|------------------|------------------|-----------------|------------------|------------------|------------------|------------------|
| Flexural Strength MPa Psi x10 ³ | 60.7 (8.8) | 106.3 (15.41) | 102.6 (14.88) | 125.6 (18.2) | 115.4 (16.73) | 118.5 (17.18) | 122.0 (17.69) | 130.2 (18.88) |
| Flexural Modulus GPa Psi x 10 ⁵ | 2.12 (3.07) | 4.1 (5.94) | 4.0 (5.8) | 5.4 (7.83) | 5.2 (7.54) | 5.3 (7.68) | 5.4 (7.83) | 5.5 (7.97) |

Table3 Properties of Hybrid composites fabricated using chemically modified coir/glass as reinforcement with unsaturated polyester Resin (USP) as matrix materials

| PROPERTIES | SAMPLES | | |
|--|----------------|--------------------|----------------------------|
| | USP | Coir-BA/Py-Acetone | Coir-BA(Py-Acetone)-Cu-BPO |
| Resin Content (w/w %) | 100 | 65 | 65 |
| Coir Content (w/w %) | - | 24 | 24 |
| Glass Content (w/w %) | - | 11 | 11 |
| Density (gm/cm ³) | 1.12 | 1.26 | 1.25 |
| Tensile Strength MPa Psi x 10 ³ | 30.4 (4.4) | 60.2 (8.73) | 64.9 (9.41) |
| Flexural Strength MPa Psi x10 ³ | 60.7 (8.8) | 127.6 (18.5) | 128.2 (18.59) |
| Flexural Modulus GPa Psi x 10 ⁵ | 2.12 (3.07) | 6.0 (8.7) | 6.0 (8.7) |

Analysis of the data of the mechanical properties clearly indicates that the magnitude of tensile strength, flexural strength and modulus are related to the nature of the reagent used in chemical modification and so also the extent of hydrophobicity. Comparison of the nature of reagents used for modification and the mechanical properties as noted in table 1-3, yield a good correlation between the two types of parameters. Transesterification of coir and subsequent curing of the resulting vinyllog material, impart good reinforcing ability to coir and as such the composites with these materials and the USP matrix resin yield good mechanical properties.

From the results and discussions it is concluded that chemical modification like transesterification and subsequent curing with BPO enhance the interfacial bond strength between the fiber and matrix resin (USP) and this in turn provides good mechanical strength to the composites, which is similar to other reports [32-37]. However much thorough and deep studies need further be carried out in evaluating other important properties like impact strength, hardness, flammability, weathering/corrosion resistance, water resistance, chemical inertness etc., before recommending the composites for commercial utility. This is aimed at developing composites at varying parameters in the fiber, such as its percentage, length etc., with varying nature of the matrix resins for commercial exploration of the method to find multifarious application of the materials. It will be beneficial economically for a country as ours and others which are rich in coir.

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