HYDROPHOBIC AND ANTIBACTERIAL TREATMENT OF JUTE FIBERS AND STUDY THEIR APPLICATION IN BIO COMPOSITES DEVELOPMENT

MAHVISH, FATIMA[1] AND WASEEM, IJAZ[2*]

[1] Department of Physics, College of Science, Qassim University, Buraydah 51452, Qassim, Saudiarabia.
[2] Department of Chemistry, Riphah International University, Faisalabad, Pakistan.

ABSTRACT

Bio-composites refers to composite materials made from sustainable materials. Jute fiber reinforced composites have inherent problem due to moisture and bacterial attack. The developed green composites are having resistance against environmental factors. At first, waste of jute fibres was pre-treated. Then two different approaches were adopted to enhance ageing factors of developed green composites. This research was proposed to go through methyltrimethoxysilane (MTMS), hydrophobic treatments, and for antipathogenic effect with ZnO nanoparticles were used. Morphological effects of chemical treatments on the jute fibers was analysed by scanning electron microscope. A significant decrease in moisture regain, increase in antibacterial zone of treated and untreated reinforcement samples was observed, when the concentration of chemical finish (methyltrimethoxysilane (MTMS) was 30 g/L and ZnO NPs also 30g/L was used. Subsequently the effects on the both mechanical properties and regain of moisture of composites reinforced with jute fiber was observed. At the concentration of 30 g/L a notable difference was spotted in moisture regain values of both treated and control (untreated) samples of reinforcement. Treated based composites regain less content of moisture and presents better mechanical properties (tensile strength and flexibility).

KEYWORDS

Antibacterial; Bio-composites; Chemical treatment; Moisture regain; ZnO nanoparticles; Degradation.

INTRODUCTION

The word "bio-composites" refers to composite materials that use biopolymers as the embedding matrix or natural reinforcing fibers [1]. Jute, flax, hemp, kenaf, and sisal are among the natural fibers that are most frequently utilized as reinforcement in bio-composites [2]. Because of its many unique qualities, affordability, ease of access, and environmental friendliness, jute is a highly valued degradable natural fiber in composites. The jute fiber has drawn the consideration as support for composite materials due to its biodegradability, quality to weight proportion and great insulation properties. In any case, higher dampness assimilation of these strands prevents the utilize of this fiber in composites. The dampness retention may cause the swelling and maceration of the strands, hence essentially diminishing its mechanical properties. So, the jute fiber got to be altered either physically or chemically to make strides the compatibility between the fiber and the polymer matrix. The surface of common filaments is ordinarily chemically altered to minimize the wetting of strands as well as to move forward the interface between the matrix and the reinforcement. A few chemical surface adjustment methods incorporate treatment with sodium chlorite[3], methaacrylate [4], isocyanate [5], silane treatment [6], acetylation [7], , mercerization [9] [10] etherification [11], enzymatic treatment [12] , peroxide medications [10], benzoylation [9], dicumyl peroxide treatment [5], plasma treatment [13], ozone medications [14] [15] and joining [16] [17]. The oxidation of polyolefins [18], [19] has moreover been detailed to move forward the contradiction between the surfaces of characteristic fiber and polymer network. All these medications are pointed to decrease the dampness recapture of the characteristic strands and their resulting composites. It is well-documented that materials with lower surface free energy exhibit reduced moisture regain [20]. Methyltrimethoxysilane (MTMS) surface free energy compared to most compounds used in previous studies. Despite their lower surface tension, methyltrimethoxysilane (MTMS are not commonly utilized in treating jute fibers for composite applications. Jute fibers are less immune towards microorganisms, like synthetic fibers which give an excellent environment to microorganisms for growth due to their moisture-retaining property Different

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[*] **Corresponding author:** Waseem I., e-mail: *ijazw4357@gmail.com*

types of bioactive nanoparticles and materials have been used to enhance the antipathogenic effect in reinforcements and composites [20].

Therefore, this study aimed to decrease the moisture absorption of composite materials made from jute reinforcement treated with methyltrimethoxysilane (MTMS) (silane finshes) to investigate the properties of these composite materials, and to validate our assumptions regarding MTMS. Moreover, second objective was to eliminate the ageing effect that comes due to the bacterial attack. The jute fiber reinforcements were further treated with ZnO NPs to gain the antipathogenic properties. Subsequently, Composites were fabricated by hand layup technique. The composites can be used in a number of application fields such as door panels, auto structure building materials, and furniture etc.

MATERIALS AND METHOD

Plain woven fabric with a density of 194 ± 2 g/m^2 was used to reinforce the bio expoxy resin matrix were purchased from resin research, and 1% cobalt naphthenate and methyl ethyl ketone were used as an accelerator and hardener. The chemical used in this study, methyltrimethoxysilane (MTMS), and ZnO nanoparticles were purchased from HUNTSMAN (Pvt).

Pre-treatment of Jute fibres

Initially, jute fibers were pre-treated (scourged) before performing various chemical treatments. For scrubbing, jute cloth was dipped in a pot of water and boiled. 10 g/L of NaOH, 2 g/L of detergent (Teepol), 2 g/L of wetting agent, and 2 g/L of Teepol detergent, two grams per liter of wetting agent, and two grams per liter of sequestering agent had been added to the pot. In order to ensure proper fabric penetration, the wetting agent has been applied. Sequestering agents have been used to extract minerals and heavy particles. At 70 °C, the jute cloth was stirred periodically for 60 minutes.

Hydrophobic treatment of Jute fibers

Jute fibers were hydrophobically treated with methyltrimethoxysilane (MTMS). The critical surface tension of methyltrimethoxysilane (MTMS) is 20–25 mN/m, which is extremely low [21]. Distilled water was used to dissolve methyltrimethoxysilane (MTMS) at three distinct concentrations (10, 20, and 30 g/L). With the aid of acetic acid, the pH of the solution was kept at 6. After dipping jute fabric in the prepared solution for half an hour at room temperature, the surplus liquid was squeezed out using a padder. Fibers were then dried for five minutes at 100 °C.

Antibacterial treatment of Jute fibers

Jute fibers were treated antibacterially using ZnO nanoparticles. ZnO nanoparticles dissolve in distilled water at three different concentrations (10, 20, and 30 g/L). After that, 2 g/L of the binder MTEX was added to the solution. The fabric was submerged in these solutions for 30 min. The cloth was crushed with a padder to remove any leftover moisture, and it was then dried at 100 °C for 5 minutes. Table 1 illustrates the reinforcement experiment design.

Composite fabrication

The hand lay-up approach was used to produce the composites. With a composite plate size of 200×200 mm^2 and a fiber volume proportion of 33%, they utilized eight layers of woven jute cloth as reinforcement. After an hour of initial treating at ambient temperature, three hours of post-curing were conducted at 120°C. Untreated jute reinforcement and jute reinforcement treated with methyltrimethoxysilane (MTMS) hydrophobic compound was used to produce composites. Table 2 provides specifics about the composite samples developed for the investigation.

Table 1. The design of experiments for reinforcements treated with different chemicals.

Sr. #	Reinforcement samples	Chemicals	Concentrations [g/L]
1	R1	No treated	-
2	R2	Hydrophobic agent (methyltrimethoxysilane (MTMS)	10
3	R3	Hydrophobic agent (methyltrimethoxysilane (MTMS)	20
4	R4	Hydrophobic agent (methyltrimethoxysilane (MTMS)	30
5	R5	ZnO nanoparticles	10
6	R6	ZnO nanoparticles	20
7	R7	ZnO nanoparticles	30

Table 2. Experimental design for composites samples.

Sr. #	Samples ID	Reinforcement ID	Chemicals
1	S1	R1	No
2	S2	R4	methyltrimethoxysilane (MTMS)
3	S3	R7	ZnO particles
4	S4	Simple resin	No

Surface Characterization

The morphological characteristics of treated reinforcements and ZnO nanoparticles have been evaluated by particle size analysis (Malvern Zetasizer) and scanning electron microscopy (Zeiss). SEM was used to study each variation in the shape of the ZnO particles. This can be achieved by using the 15K accelerated voltage.

Moisture regain of reinforcement

The moisture recovery test of a chosen jute cloth was examined using the standard test method known as ASTM D2495. Both treated and untreated jute fabrics were utilized in the tests. A well-known oven dry method was used to determine how much moisture the material had absorbed in a controlled environment. Additionally, composite samples were tested according to the standard test procedure known as ASTPMD5229 in order to examine the amount of moisture exchange.

Mechanical properties

Universal tensile strength tester measured the ultimate tensile strength using the standard testing approach [22]. Moreover, a standardized testing method named ASTM D7264 was applied to explore flexural properties via three-point bending test.

Antibacterial assessment

The standard AATCC-147disc diffusion method was followed to provide a qualitative assessment of all reinforcement and composite samples.

RESULTS AND DISCUSSION

Surface morphology

The surface morphologies of jute fiber, both treated and untreated, were examined using a scanning electron microscope. Figure 1 displays images of the untreated and nanoparticles treated (ZnO particle) fibers. In fact, a network of smaller particles can develop. The constant connection and impact coating between the tiny ZnO nanoparticles were noted. The deposition of ZnO nanoparticles were combined and evenly distributed throughout the whole surface of fiber substrate. It was discovered that the coated surface of fibers was entirely covered. It was found that particles deposition was more consistent and dense. The prepared coating sample was found to have the potential to produce a thicker layer over the fiber surface.

Moisture regain of reinforcement

The moisture regains %age of untreated fabric sample and those samples treated with methyltrimethoxysilane (MTMS) (silane finishes) as function of their concentrations is shown in Figure 2. The percentage of humidity regain of fibers treated with methyltrimethoxysilane (MTMS) of R4 (30g/L) is the lowest when compared to all other samples. The supplier of the chemicals states that they include hydrophobic reactive groups as required by the technical applications for which they are desired. In fact, the mechanism involved the cross-linking during curing, their interaction with the -OH groups in cellulose, and the development of films on fiber. Regarding methyltrimethoxysilane (MTMS), films formed on fibers primarily display hydrophobic chains, whereas films formed using methyltrimethoxysilane (MTMS) show both hydrophobic and hydrophilic chains. Figure 2 further, indicates clearly that the treated sample has a moisture regain of 3.83–7.38%, while the untreated sample has a moisture regain of roughly 12%. Furthermore, at a concentration of 30g/l, treated R4 reinforcement shows a lower moisture recovery value of 2.71%. In contrast, at a concentration of 30g/l, the moisture regain values of R2 and R3 are roughly 3.10% and 3.05%, respectively.

Moisture regain of composite

In the equilibrium condition, Figure 3 displays the moisture regain %age of composite samples made from both untreated and treated jute cloth. The plain resin sample had a moisture regain %age of 0.29%. In contrast to the 12% moisture regain of jute cloth, the untreated jute composite sample absorbed 4% moisture.

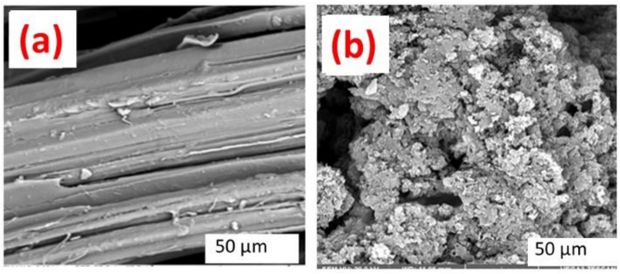

Figure 1. The Surface morphologies of (a) Untreated jute, (b) ZnO nanoparticles deposited.

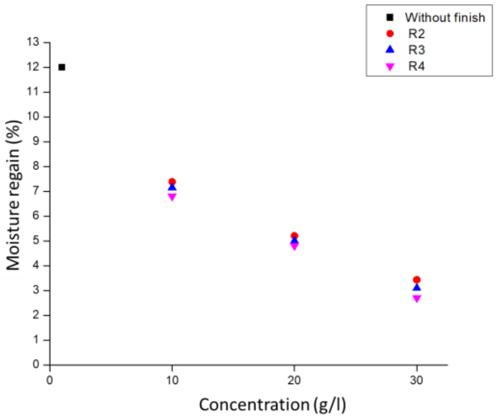

Figure 2. Moisture regain of untreated and treated fabric as function of chemical concentration.

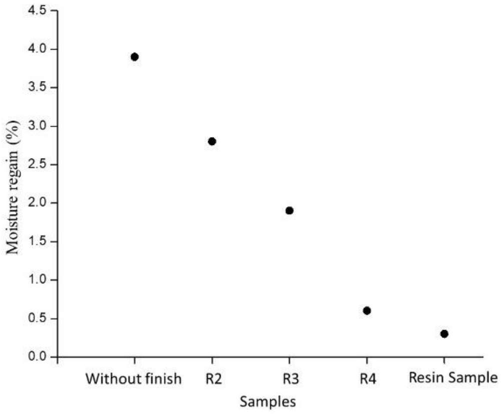

Figure 3. Moisture regain of resin and composite samples in equilibrium state.

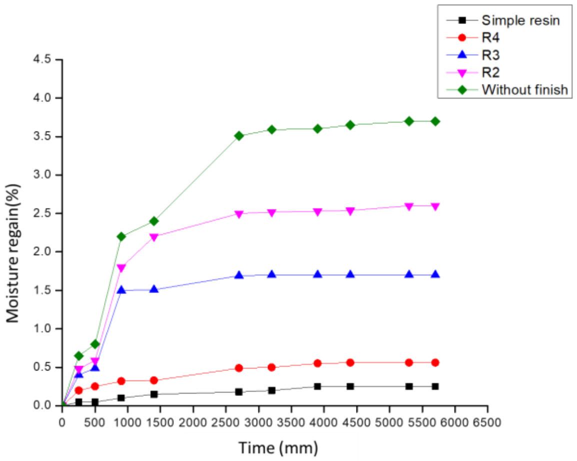

Figure 4. Moisture regains of composite samples.

The regain of moisture in composites and samples of clear resins verses the square root of time is shown in figure 4. All of the composite samples showed a consistent increase in moisture intake from the start to the 1000-minute mark. This makes sense because all test specimens were completely dried. Generally, figure 4 depicts the moist after 1000 minutes, the saturation of specimen began, and the rate of moisture recovery slowed. Almost every sample got saturated after 3000 minutes. To ensure that samples recovered the maximum amount of moisture and reached equilibrium, the test duration was extended.

Mechanical Properties

Analysis of the mechanical properties was performed on each composite sample. Following the ASTM D3039 standard, the tensile strength tester calculated the strength at break. The largest load that composites can withstand before failing was used to determine the maximum tensile strength of the manufactured composites. The tensile strength of every composite sample generated is shown in Figure 5. The tensile and flexural characteristics of composite materials were shown in table 3. S2 and S3 are composite sample created with treated reinforcement and S1, untreated reinforcement, While S4 only resin sample (having no reinforcement. Each sample is different in strength, because the strength of reinforcement has a significant impact on the strength of composite. The tensile strength of composites made with R7 (S3), reinforcement is greater than that of composites made with R1 and R7 reinforcement. Composites with treated reinforcing have varying tensile strengths. It is due to that R7 has

Table 3. Mechanical properties of composite materials.

Sr. #	Samples ID	Reinforcement ID	Chemicals	Tensile Strength [MPa]	Flexural Strength [MPa]
1	S1	R1	No	32.34	38.71
2	S2	R4	methyltrimethoxysilane (MTMS)	42.56	67.11
3	S3	R7	ZnO particles	48.72	81.73
4	S4	Simple resin	No	43.23	56.39

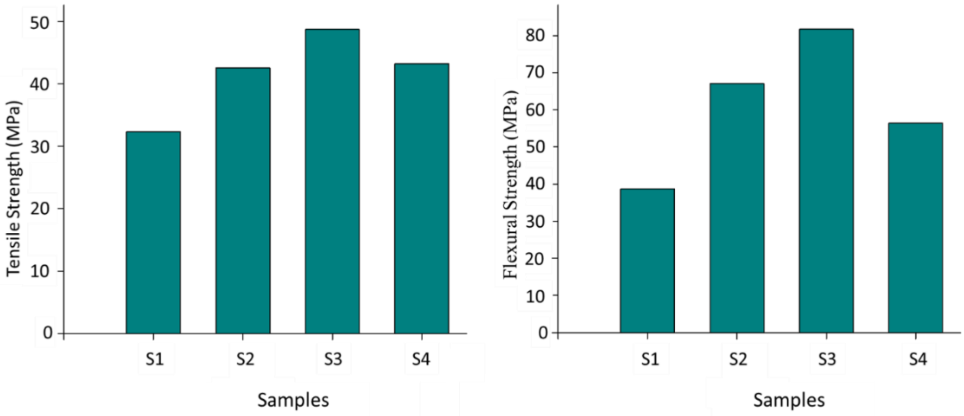

Figure 5. Mechanical properties of composite samples, (a) Tensile strength and (b) Flexural Flexural strength.

Table 4. Antibacterial properties of composite material.

Sr. #	Samples ID	Reinforcement ID	Chemicals	S. Aureus ZOI [mm]	E. Coli ZOI [mm]
1	S1	R1	No	0	0
2	S2	R4	methyltrimethoxysilane (MTMS)	0	0
3	S3	R7	ZnO particles	4	3
4	S4	Simple resin	No	0	0

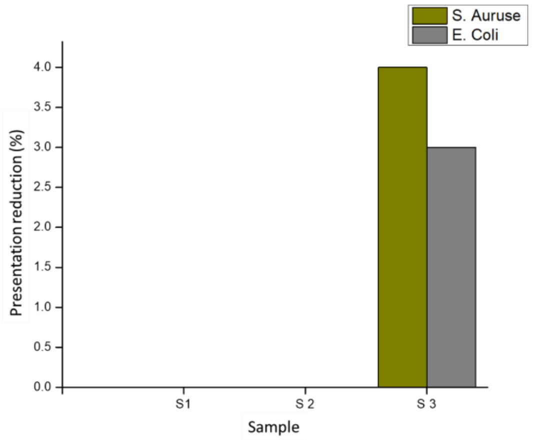

Figure 6. Antibacterial properties of composite samples against *E. coli* and *S. Aureus*.

higher concentration of ZnONPs. The provided data makes it evident that the treated reinforcement flexural strength of composite is greater than that of the sample of untreated reinforcement composites. This can be explained by the fact that the treated reinforcement is more powerful than the untreated one. Because of its improved resin-matrix interaction, the R2 treatment produced the biggest deflection.

Antibacterial Properties

The antibacterial properties of composite materials are shown in table 4. The antibacterial activity was tested against Gram-negative *Escherichia coli* (*E. coli*) and Gram-positive *Staphylococcus aureus* (*S. aureus*). S1 sample, which was not treated with any chemical showed no zone of inhibition against both bacteria (*E. coli* and *S. aureus*). Sample S2, treated with methyltrimethoxysilane (MTMS), also showed no antibacterial activity as MTMS is not an antibacterial agent. Sample S3 is treated with ZnO nanoparticles (antibacterial agent) displayed significant antibacterial properties. These results suggest that the sample S3 has moderate antibacterial properties, whereas S1 and S2 lack efficacy against the tested bacteria. Two nano ZnO composite films, ZnO/PC and ZnO/LLDPE, were made with a low doping of 0.2%. Compared to the ZnO/PC film, the ZnO/LLDPE film had a greater antibacterial rate (99.3% vs. 55.4%). ZnO/PC and ZnO/LLDPE had a 99.9% antibacterial rate when the nano ZnO concentration was quadrupled to 0.2%. Both composites can therefore attain a high degree of antibacterial activity [24].

CONCLUSIONS

This study concludes that there is a notable difference in the moisture regain between untreated and treated reinforcements. The reinforcement treated with methyltrimethoxysilane (MTMS) exhibited the lowest moisture regain value of 2.71% at a concentration of 30 g/L, in contrast to the other treatments. Reinforcement in the composite absorbs more moisture, as the moisture regain % for the pure resin sample was only 0. undefined Untreated jute composite sample has a moisture regain of 4% whereas for the composites prepared by using the methyltrimethoxysilane (MTMS) treated reinforcement has the lowest moisture regain of 0. 79% thus conforming the moisture regain of the corresponding reinforcement. Mechanical result comparison between the composite sample with untreated reinforcement and treated reinforcement showed the difference exists. ZnO nanoparticle-treated composites showed the greatest tensile strength (48.72 MPa) and flexural strength (81.73 MPa) because of improved resin-fiber bonding and consistent nanoparticle deposition. Zones of inhibition (ZOI) for *S. aureus* and *E. coli* were 4 mm and 3 mm, respectively, indicating considerable antibacterial efficacy in ZnO nanoparticle-treated composites, but MTMS-treated composites showed no antibacterial activity. When bio-epoxy resin is combined with hydrophobic and antibacterial treatments, it may find use in settings that need bacterial protection, mechanical endurance, and moisture resistance.

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