Improvised hand layup fabrication of alkali treated jute epoxy composites: A comparative study of positive and vacuum-assisted compaction

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Abstract. There lie several benefits of using fiber composites which have increased the desire for using these materials in various higher-level applications. They have been widely used in automobile sector, aerospace, sports industry, medical field, and so on. This has created a demand for better manufacturing techniques with cost-effectiveness. This work has been focused on improvising the hand layup procedure. To enhance the properties of the samples prepared by this conventional method, surface treatment was incorporated. Woven jute fiber was chemically treated with KOH under various sizing conditions. Hand layup was carried out for the samples followed by applying pressure considering two different methods; vacuum-assisted compaction and positive compaction. The jute composites prepared by the positive compaction hand layup technique were found to be better than the vacuum-assisted or negative compaction composites for the same set of sizing samples. There is a maximum increase of 32.4% in the tensile strength of treated composites prepared by positive compaction in comparison to untreated samples. On the other hand, the values of all the treated samples showed a reduction in tensile strength with a maximum decrease of 50% than the untreated sample for the negative compaction technique.

1 Introduction

The hand layup method is a conventional process of layering up fabrics manually. The cost incurred in infrastructure for hand layup is comparatively marginal [1-3]. The steps involved in the process are simpler. Initially, a release agent is used over the surface of the mold surface to prevent the resin from adhering to the surface. The first layer of woven fabric or chopped fiber as per specified size is placed over the mold.

The woven reinforcements can be plied at four different orientations; 90°, +45°, -45°, and 0°. This process of layering up is followed by pouring the resin uniformly upon the first layer. The subsequent layer of the reinforcement is then placed. Rollers or brushes are used to evenly spread the resin. Gentle pressure is used while using a roller to eliminate any excess

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entrapped resin and also to remove the air within the layers. The primary purpose of this step is to eradicate any amount of voids that may arise due to the air trapped inside. Then the process is repeated for the next set of reinforcement layers. After stacking the layers, curing is executed. Curing can be employed by simply laying the setup at room temperature or selecting a specific temperature for the process. According to the examination made by Cooley [4], this conventional method of curing the material in the open may lead to excess resin and voids entrapped inside.

Mohammad et al. [5] mentioned that improper mixing of resin and the presence of voids are the main limitations observed while using hand layup as a manufacturing method. Wanberg [6] suggested that an extended method of vacuum infusion with hand layup can reduce the percentage of void formation. The main idea behind the process is to apply external pressure for consolidation. This will enhance the mechanical properties of the composites. Composites are composed of reinforcement and polymer. Both the fabric and polymer must be bonded well to impart better properties to the composite material [7-12]. The fiber which is used to improve the bonding after being chemically analyzed changes the surface and additionally, helps in enhancing the strength for better joining of the filament with the matrix system. This can also lead to lesser absorption of water by composites while upgrading their properties; mechanical and thermal. The mechanical properties of short jute fiber-reinforced epoxy composites after silanization are examined by Basak et al. [13]. It was determined that the treated composites prepared at the upper-temperature range indicated better mechanical properties. Willy et al. [14] investigated waru bark fiber (Hibiscus tiliaceus) by adding methacryloxy propyl trimethoxy silane-coupling agent (0.75% conc.) for 4h. There was an improvement in tensile strength recorded. In the present work, the hand layup method was modified and used to fabricate both untreated and KOH treated and jute epoxy composites.

2 Materials and methodology

2.1 Sizing of fiber

Woven jute fabric of 300GSM was used as reinforcement after blow drying it (LB700 7Amp). The fabric was cut into size 275mm x 200mm with the help of electric scissors, including the anomaly area from all the edges. Potassium hydroxide was considered for the chemical modification of jute. Samples were prepared considering 5% and 15% KOH under varying mediums [15]. Table 1 gives the list of the different types of samples. The process of KOH application is given in Figures 1(A) and 1(B).

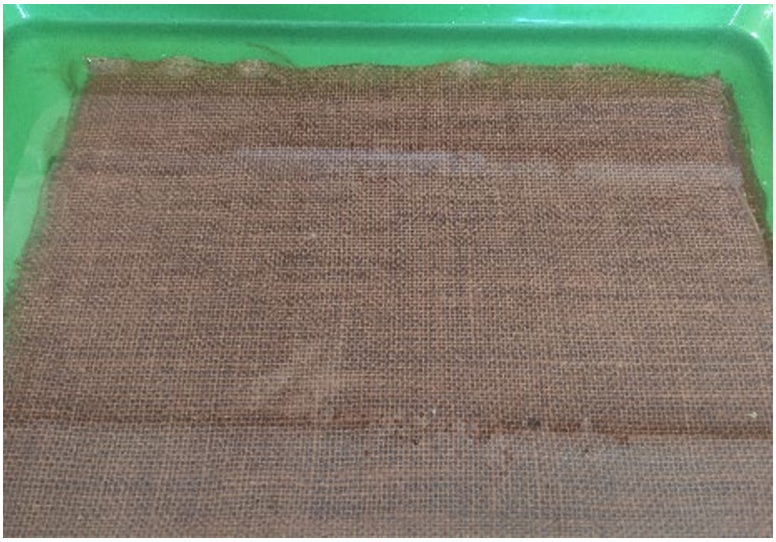
2.2 Fabrication of composite

Resin CT/E - 556 with a density of 1.15-1.20 g/ and hardener CT/AH –951 of density: 0.97-0.99 g/cc were used in 100:10-12 ratio by weight. The products were acquired from Composites Tomorrow, Vadodara. The jute epoxy composites were prepared by two improvised hand layup processes. The first method selected was positive compaction hand layup. It involved mixing resin physically onto the reinforcement surface. To avoid any damage caused due to the sticking of resin onto the mold surface, a release film was used.

After placing the first layer of fabric, the resin was evenly distributed using a roller to exert moderate pressure to remove extra resin and entrapped air. After repetition of the same method for each layer of fabric, the mold was kept within a compaction chamber. The positive compaction setup consisted of a hydraulic press (Refer to Figure 2). A flat or shaped mold surface where the fiber reinforcement is laid up to provide dimensional accuracy to the

laminate, mold release agent to prevent sticking of the laminate to the mold and makes demolding easier and fiber reinforcement layers placed in sequence as per the stacking design. A pressure of 5 bar was applied for 24h. The composite sample under the positive compaction chamber. Preparation of samples A1, B1, C1, D1, E1, and U1 were equipped with the positive compaction hand layup method.

(a)



(b)

Fig. 1. Samples under (a) Wet apply, and (b) Soaking.

Table 1. List of samples prepared under different sizing methods.

Positive Compaction Samples	Negative Compaction Factor	%conc. Of KOH	Method of treatment	Parameters of Curing
A1	A2	5%	Wet apply	80°C, 2h
B1	B2	15%	Wet apply	80°C, 2h
C1	C2	5%	Wet Apply	Room temperature, 24h
D1	D2	5%	Soaking, 1h and washing with 5% acetic acid	Room temperature, 24h
E1	E2	5% with acetone	Wet apply	56°C,10min
U1	U2	Untreated	-	-

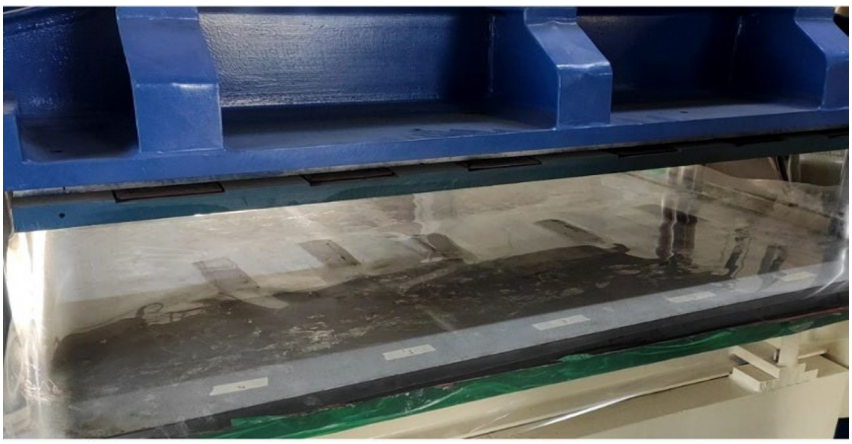


Fig. 2. Samples under positive compaction hand layup.

The second improvised method incorporated was vacuum-assisted compaction hand layup (Refer to Figure 3(a-d). This method is primarily different from the former process in terms of the pressure applied. A release film was placed over the mold's surface, followed by the stacking of layers of jute manually. The resin was carefully spread over' mechanical properties and the first layer of jute using rollers, followed by subsequent plying of reinforcement layers and resin pouring. Vacuum bagging was implemented after placing a breather cloth over the samples. The breather fabric helped to provide conformity to the composite under vacuum. Besides, it facilitated the exchange of airflow and bleeding out of excess entrapped resin material. Sealant tape was used further to ensure the airtight sealing for providing an efficient vacuum ambiance. The vacuum supply was provided under negative pressure of -0.8 bar. Preparation of samples A2, B2, C2, D2, E2, and U2 was done using the vacuum-assisted hand layup metshod, with a **holding time under vacuum for** 3–4 hours during resin curing, **leakage tolerance** < 2% drop in vacuum level over 30 minutes and **resin bleed control** achieved through breather fabric and controlled vacuum suction.

Fig. 3 (a). Preparation of mold.

Fig. 3 (b). Peel ply on fabric layer.

Fig. 3 (c). Placement of distribution medium.

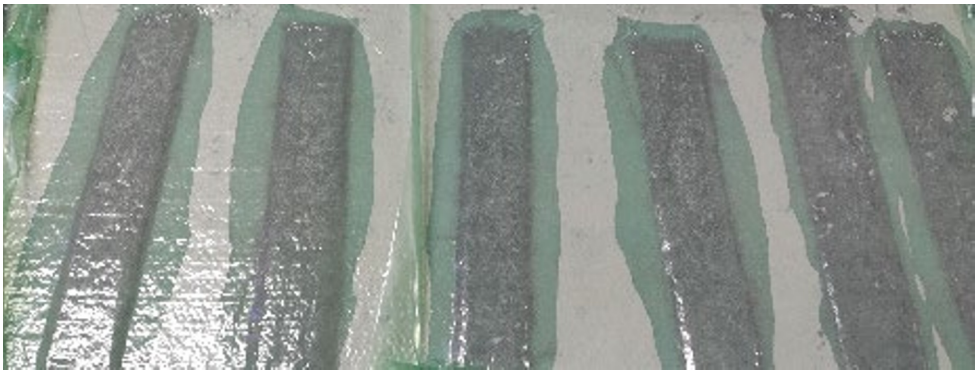


Fig. 3 (d). Samples under negative compaction hand layup.

2.3 Tensile test

Tensile testing of both types of prepared samples was carried out following ASTM D3039 using SHIMADZU UTM with 20KN frame. This test aimed to evaluate the effect of improvisation of manufacturing techniques on the tensile strength of the composite. 2mm/minute was the speed considered for the testing. Each sample was prepared with a dimension of 250 mm × 25 mm. Figure 4(a-b) depicts one of the samples under the tensile test.

3 Results and discussion

3.1 Evaluation of sizing

Natural jute fibers contain cellulose and lignin. Their surface is not modified, leading to poor adhesion with polymers in composites. When KOH + water treatment iss used, KOH helps in removing lignin and impurities from the fiber surface, cleaning it. Some hydroxyl groups (–OH) were exposed.

Fiber surface becomes rougher for better mechanical interlocking. Also, there is mproved wettability for later polymer coating. When acetone is used as medium for modifying the surface with Epichlorohydrin sizing agents, Strong chemical bonding between the fiber and polymer matrix is seen. Surface becomes chemically active and more compatible with the polymer matrix. There is improved interfacial adhesion, leading to higher tensile strength and better composite performance (Refer to Figure 5).

Fig. 4 (a). Sample after fabrication.

Fig. 4 (b). Sample under tensile testing.

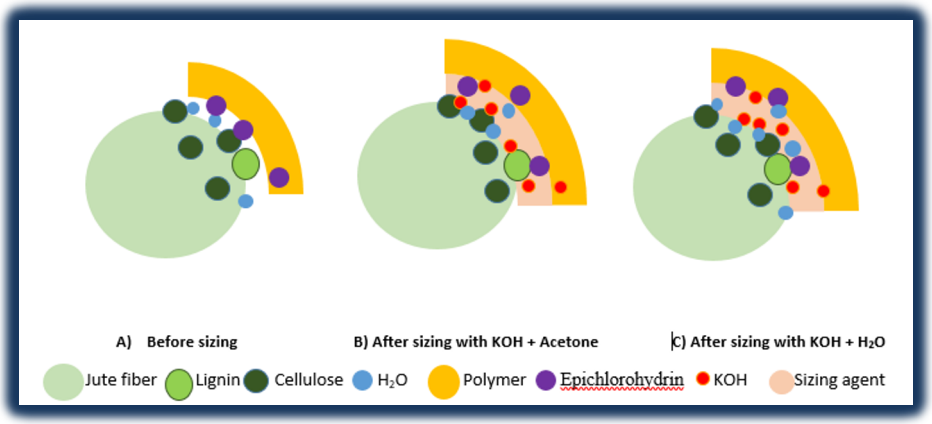


Fig. 5. Schematic illustration of jute fiber surface treatment: A) Untreated fiber B) Further treatment with KOH + Acetone C) Alkali treatment with KOH + H_2O.

Table. 2 Tabular list of values obtained after tensile test.

Samples	Tensile Strength (MPa)	Sample	Tensile Strength (MPa)
A1	44.57 ± 0.51	A2	30.40 ± 0.53
B1	33.80 ± 0.26	B2	21.50 ± 0.20
C1	49.80 ± 1.06	C2	30.47 ± 0.42
D1	48.20 ± 0.26	D2	23.30 ± 0.26
E1	62.07 ± 0.40	E2	42.40 ± 0.36
U1	46.27 ± 0.31	U2	42.50 ± 0.20

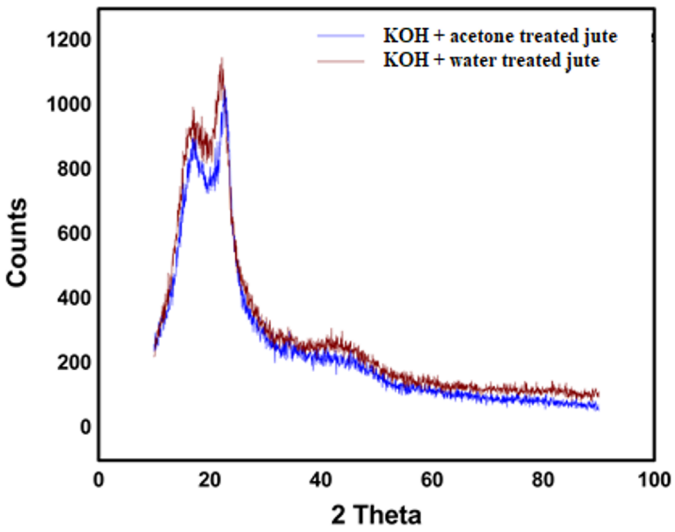


Fig. 6. XRD Analysis for identifying acetone as a potential medium for KOH treatment on jute fibers.

The results after tensile testing are listed in Table 2. The highest tensile value was indicated by sample E1 where KOH treatment was implemented in a medium of acetone: water in a ratio of 90:10 by weight. This can be attributed to the better surface modification

of jute in presence of evaporative acetone medium. A higher concentration of KOH led to the failure of the internal structure. As there was a 27.2% and 50% decrease in the results of samples B1 and B2 from their respective untreated composite samples U1 and U2. Room temperature curing was efficient when compared to curing at 80°C. The values of tensile strength reduced to 44.7MPa from 46.6MPa for sample A1 and reduced to 30.2MPa from 42.7MPa for sample A2. Both indicated that fabrics became unstable at a higher temperature of 80°C. The process of wet application of KOH had a positive impact on the strength of the composites in comparison to the soaking process. This can be highlighted due to the deterioration of the fibers under prolonged exposure to the KOH-water medium for samples D1 and D2. The wet application of KOH to the fabric detached any extra wetness, which attributed to the better fibre K+ bond formation.

XRD analysis for both acetone and water as solvents displayed a broad peak around $2\theta \approx 20°$**,** typical for cellulose crystalline regions in natural fiber composites (Refer to Figure 6). The KOH + water treated composite showed a slightly higher peak intensity compared to the KOH + acetone treated composite. The latter showed a small reduction in peak intensity and a broader peak. The higher peak intensity in the water solvent composite suggested that it retained more of the natural cellulose crystalline structure. The acetone solvent treatment partially removed lignin and other amorphous components, but also disrupted some cellulose crystalline regions. This led to a slight reduction in crystallinity composite, reflected by the lower and broader peak. Thus KOH+ acetone treatment improved fiber-matrix interaction by making the fiber surface more reactive, even though it slightly reduced crystallinity. The reduced crystallinity in the treated composite improved chemical bonding with the polymer matrix, enhancing composite performance like better tensile strength, and improved adhesion

3.2 Evaluation of improvised fabrication

The results indicate that there was a negative impact on the values of the tensile strength of the composites prepared by the vacuum-assisted hand layup technique. All tensile strength values are presented as mean ± standard deviation, calculated from three replicates (n = 3) for each sample. There was an increase of 32% in the tensile strength value of the treated jute composite sample E1 when compared to the untreated composite. The best value was obtained for KOH treated jute composite under acetone + water medium; sample E. The increment was accredited to the high pressure of 5 bar applied during the positive compaction hand layup method**.**

On the contrary, for the negative compaction method, tensile strength reduced from 42.7MPa to 42MPa for the sample E2 after treatment under the same KOH+water medium. Moreover, there was no significant increase in the samples after treatment, rather all the treated composites indicated lower strength values than the untreated jute epoxy composite. The reason behind the decrease in the value hinted at the influence of negative pressure of –0.8 bar provided. The entrapped resin could not be bled out, thereby deteriorating the strength of the composite. There were 32.4% (A2), 37.1% (B2), 38% (C2), 51.1% (D2), 31% (E2), and 8.3% (U2) decrement in the values of tensile strength of the samples respectively when fabricated under negative compaction. A curve depicting the values of the tensile test for both sets of composites is shown in Figure 7 and Figure 8.

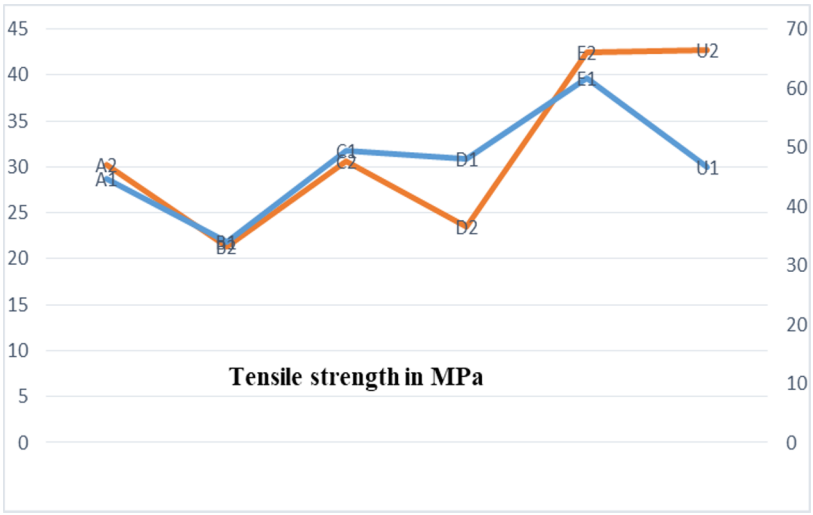


Fig. 7. Graphical representation to compare the values of tensile Strength of jute epoxy composites prepared by both positive and negative compaction methods.

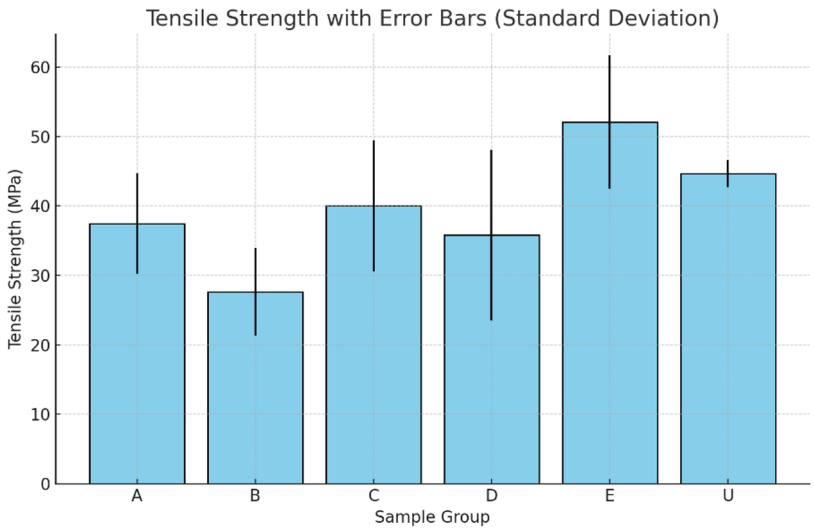


Fig. 8. Graphical representation with standard deviation values.

4 Conclusion

The main objective behind the research was to determine the impact of improvising the traditional hand layup technique. The two methods followed were positive compaction under a pressure of 5 bar and negative compaction under -0.8 bar. The tensile testing was carried out for both sets of samples. Prior to this, surface treatment of the jute fabric was initiated to improve the adhesion properties of the fabric and polymer. On evaluating the sizing process, sample E1 with KOH treatment under water + acetone medium provided the best results with an increase of 32.4% from the untreated composite. This confirms the positive role of chemical treatment combined with positive compaction in enhancing interfacial bonding and

mechanical performance. The negative compaction method under vacuum ambiance had a negative impact on all the types of treated and untreated samples. The value was reduced majorly in the case of samples A2, B2, C2, and D2. A maximum reduction of 50% from the untreated sample for the negative compaction technique was noted. This was followed due to the negative pressure considered during fabrication. This performance drop is attributed to insufficient matrix consolidation caused by vacuum-induced negative pressure, which hindered proper adhesion and fiber wetting during fabrication. Thus, the improvised hand layup process under positive compaction can be used to provide better strength properties to the composites. Further research could focus on long-term performance evaluations such as fatigue testing, impact resistance studies, and durability under environmental aging. These investigations will help establish the broader applicability of the positive compaction method in industrial composite manufacturing, especially in applications requiring high reliability under dynamic loading.

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The authors declare no dataset generated during the current study
SR contributed to the data compilation. RB contributed to the findings and manuscript preparation.

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