Retraction

Retracted: Effect of Aluminium Tetrahydrate Nanofiller Addition on the Mechanical and Thermal Behaviour of Luffa Fibre-Based Polyester Composites under Cryogenic Environment

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This article has been retracted by Hindawi following an investigation undertaken by the publisher [1]. This investigation has uncovered evidence of one or more of the following indicators of systematic manipulation of the publication process:

1. Discrepancies in scope
2. Discrepancies in the description of the research reported
3. Discrepancies between the availability of data and the research described
4. Inappropriate citations
5. Incoherent, meaningless and/or irrelevant content included in the article
6. Peer-review manipulation

The presence of these indicators undermines our confidence in the integrity of the article’s content and we cannot, therefore, vouch for its reliability. Please note that this notice is intended solely to alert readers that the content of this article is unreliable. We have not investigated whether authors were aware of or involved in the systematic manipulation of the publication process.

Wiley and Hindawi regrets that the usual quality checks did not identify these issues before publication and have since put additional measures in place to safeguard research integrity. We wish to credit our own Research Integrity and Research Publishing teams and anonymous and named external researchers and research integrity experts for contributing to this investigation.

The corresponding author, as the representative of all authors, has been given the opportunity to register their agreement or disagreement to this retraction. We have kept a record of any response received.

References

Effect of Aluminium Tetrhydrate Nanofiller Addition on the Mechanical and Thermal Behaviour of Luffa Fibre-Based Polyester Composites under Cryogenic Environment


1Institute of Agricultural Engineering, Saveetha School of Engineering, SIMATS, 602 105, Chennai, Tamilnadu, India
2Department of Mechanical Engineering, Velammal Institute of Technology, Chennai, 601204 Tamil Nadu, India
3Department of Mechanical Engineering, Rajalakshmi Engineering College, Rajalakshmi Nagar, Thandlam, Chennai, 602 105 Tamil Nadu, India
4Department of Mechanical Engineering, Graphic Era Deemed to Be University, Bell Road, Clement Town, 248002 Dehradun, Uttarakhand, India
5School of Mechanical Engineering, Vellore Institute of Technology, Vellore, Tamil Nadu, India
6Department of Mechanical Engineering, Saveetha School of Engineering, SIMATS, Chennai, Tamil Nadu 602105, India
7Department of Mechanical Engineering, Ambo University, Ambo, Ethiopia

Correspondence should be addressed to G. Velmurugan; velresearch032@gmail.com, L. Natrayan; natrayanmech007@gmail.com, and Ketema Bobe; ketema.bobe@ambou.edu.et

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In recent times, research has shifted away from conventional materials and alloys and more towards nanocomposites to create lighter, more efficient materials for specific applications. The major goal of this research is to see how successful adding aluminium tetrahydride (ATH) filler to a luffa fibre/polyester-based hybrid composite is. The compression moulding process was used to create the nanocomposite. The following limitations were used to achieve the goals mentioned above: (i) weight percent of ATH, (ii) weight percent of luffa fibres, and (iii) cryogenic treatment hours. The mechanical properties of the materials, such as flexural, tensile, and impact, were examined. The scanning electron microscope observed the morphology pictures, revealing flaws such as interface behaviour, fibre pullouts, voids, and interior cracks. As a result, the current study found that adding nanofiller to a natural fibre composite can improve its mechanical properties, because it established a strong link between the matrix and its reinforcements, which would aid in the effective transmission of stress in the hybrid system. It also improved moisture resistance, which might be useful in construction and commercial industries. The composite with 1 wt.% of ATH, 24 wt.% of luffa fibres, and 30 minutes of cryogenic treatment showed better mechanical strength. Cryogenic treatment reduces compressive interface stresses, which helps maintain fibre and matrix in contact and improve adhesion, resulting in superior results. TGA analysis was used to confirm it.

1. Introduction

Glass fibre-reinforced (GFR) composites currently come in a wide range of prices and mechanical properties. Shipbuilding and car manufacture, as well as turbine making, are all instances of industrial purposes. GFR composites, on the other hand, are made of nonrenewable resources and are known to use a significant amount of energy during manufacturing. Aside from that, GFR recycling is not easy [1, 2]. Environmental concerns are growing in importance, prompting the development of more environmentally friendly composite materials. Natural fibres are frequently regarded as being more
Natural fibre materials have poor adherence to the matrix due to their moisture absorption characteristics and the hydrophobic character of the matrix. As an outcome, poor fibre-matrix interaction happens, decreasing the reinforcing effect of the fibre and impeding stress transfer from the matrix to the load-carrying fibres. The level of contact between the reinforcement and the matrix determines the features of natural fibre polymeric materials [11, 12]. Researchers have looked at several ways of enhancing fibre properties and boosting fibre matrix contact to solve the concerns above. Chemical treatments are commonly used to change the characteristics of fibres. Alkaline, silane, peroxidase, permanganate, isocyanate treatments, and polymer coupling agents are widely used chemical treatments [13]. According to Devarajan et al. [14], fibres treated with 5% NaOH showed optimal mechanical properties and elastic modulus compared to nontreated fibres and good surface fracture toughness with a polyester compared to the untreated fibre surface. In this investigation, luffa fibres were chemically treated with a 5 wt. percent NaOH solution for 4 hours to enhance the interaction of the luffa fibre with the matrices in this investigation.

The inclusion of fillers into a matrix was another method for improving the qualities of natural fibre-reinforced composites. It has proven to be a viable alternative for improving biocomposites’ mechanical behaviour. The right combination of matrices, fillers, and reinforcements can result in a composite material with similar or superior qualities to traditional metallic materials [15]. However, in terms of enhancing mechanical properties, the influence of mixing fibres in matrices has reached its limit. As a result, using nanoparticles in polyester is becoming increasingly popular. The nanoparticles are used to improve the strength of the oath between the matrix and the fibre, which improves the properties even further. This research was aimed at figuring out how to make industrial aluminium trihydrate laden polymer composite waste powder more usable. The ATH might be utilized as fresh material for novel goods since its worldwide output is anticipated to be around 86,000 tonnes each year [16]. Cryogenic behaviour can increase the mechanical characteristics of fibre-reinforced composites. For instance, resources used in aeroplane construction should be able to withstand severe temperatures of up to 200°C. Composites and plastics that have been cryogenically treated mean they show high strength, are tougher, and have improved rigidity and wear resistance [17]. As a result, liquid nitrogen treating of composites can become an important aspect of ongoing investigation and growth to enhance natural composite material behaviour.

The primary goal of this study is to create and test hybrid natural nanocomposites for mechanical characteristics. The ATH and luffa fibre combined polyester composites were fabricated in compression moulding. The mechanical characteristics of the laminated composites were measured after they were submerged in liquid N2 at 77 K for varying durations.

2. Investigational Resources and Methods

2.1. Resources. The fruits Luffa cylindrica belong to the Cucurbitaceae family. Compared to other natural fibres, such as jute and pineapple fibres, Luffa cylindrica fibre has a thick skin on the fruit’s exterior and a spongy gourd on the inside. The inner sponge gourd’s fibres are organized into a multidimensional array that simulates a natural matrix. To eliminate the waxes and lignin content from the luffa fibres, they were rinsed with distilled water and dried in a hot air oven for two days. The dried fibre is shown in Figure 1(a). The fibre was cut into the mat-like shape illustrated in Figure 1(b) after curing. The present luffa fibre is hydrophilic, so an alkali treatment on the fibre’s surface
changed its nature from hydrophilic to hydrophobic. The water content and waxy particles were removed from the fibre using an alkali treatment with 5% NaOH. Throughout this operation, the fibre was initially submerged in a small beaker of a 5 percent alkaline solution in the shape of a mat-like layer. The submerged fibre was rinsed with water after 4 hours to remove the adhering sodium hydroxide from the fibre’s exterior. It was also neutralized with weak acetic acid, and the fibre was rinsed with water once more. The treated fibre was dried at a temperature of 353 K in an oven. The filler material HN361 type of aluminium tetrahydrate with 50 m was supplied by Naga Chemicals, Chennai, Tamilnadu, India. The filler was then enlarged to 20 nm in the ceramic consultant’s laboratory in Chennai, Tamilnadu, India, using the ball milling procedure. GVR industry, Madurai, Tamilnadu, India, provided the unsaturated polyester resin, cobalt naphthenate (accelerator), and methyl ethyl ketone peroxide (catalysts). Figures 2(a) and 2(b) show the chemical structure and the photographic image of ATH.

2.2. Alkaline Process. To remove impurities, the luffa fibres were cleaned in 1 to 2 percent washing liquids for 1 hour at 60 to 70°C then rinsed with distilled water before drying in a high-dry oven at 60°C for 1 hour and 30 minutes. "Dry fibres" were characterized as untreated fibres. The strands were cleaned by soaking them in a 2:1 mixture of benzene and ethanol for 70 to 72 hours at 50°C, after which they were thoroughly rinsed and sun-dried for one day. Following that, the fibres were immersed in a 5% NaOH solution at 20°C for 4 hours.

2.3. Lamination of Hybrid Composites. The hybrid nanocomposites were made using a traditional hand layup approach before compression moulding. The weight compositions of fibre, ATH filler, and matrix components are reported in Table 1. The hybrid composite laminates were manufactured with dimensions of 300 × 300 × 3 mm based on the compositions in Table 1. To achieve a homogenous mixture, both matrix materials and the ATH nanofiller were completely mixed with a stirrer in this study. To create a varied composite laminate with various filler concentrations, the weight percentage of the polyester matrix was altered continually. Finally, with 10 bar pressure and temperatures ranging from 48 to 50 degrees Celsius, the mould cavity is inserted between the top and bottom plungers of the compression moulding machine. Figure 3 is a picture of the compression moulding process.

2.4. Cryogenic Treatment. After manufacturing, the cured laminates were removed from the mould and trimmed to ASTM standard dimensions. The sample size for tensile testing is 250 × 25 × 3 mm; for flexural testing, it was cut to the dimensions of 127 × 12.7 × 3 mm; and for impact testing, it was cut to the dimensions of 63.5 × 12.5 × 3 mm. A temperature-controlled cryogenic chamber with programmable controls is used for the cryogenic implementation. The temperature was lowered to 196°C using a controlled cooling rate (3°C/min). After that, the samples were

Table 1: Design and compositions of hybrid nanocomposites.

<table>
<thead>
<tr>
<th>Trail no</th>
<th>Symbols</th>
<th>Compositions of nanocomposites</th>
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<tbody>
<tr>
<td>Trail 1</td>
<td>S1</td>
<td>Polyester+ luffa fibre (25 wt.%)</td>
</tr>
<tr>
<td>Trail 2</td>
<td>S2</td>
<td>Polyester+ luffa fibre (24 wt./%)+ATH filler (1 wt.%)</td>
</tr>
<tr>
<td>Trail 3</td>
<td>S3</td>
<td>Polyester+ luffa fibre (23 wt./%)+ATH filler (2 wt.%)</td>
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<tr>
<td>Trail 4</td>
<td>S4</td>
<td>Polyester+ luffa fibre (22 wt./%)+ATH filler (3 wt.%)</td>
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<tr>
<td>Trail 5</td>
<td>S5</td>
<td>Polyester+ luffa fibre (21 wt./%)+ATH filler (4 wt.%)</td>
</tr>
<tr>
<td>Trail 6</td>
<td>S6</td>
<td>Polyester+ luffa fibre (20 wt./%)+ATH filler (5 wt.%)</td>
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immersed in liquid N2 at 77 K for 15 minutes, 30 minutes, and 45 minutes, respectively, for cryogenic treatment. The nanocomposites were finally brought back to normal temperatures. The cryogenic processing of a prepared sample in a cryogenic chamber is shown in Figure 4.

2.5. Microstructural Study. SEM was utilized to conduct microscopic investigations into fractured composite samples. The specimens were laved, dehydrated, and surface coated with 10 nm of gold before SEM clarity to increase the composites' electrical conductivity. The following equations were used for calculating the mechanical properties.

\[
\text{Tensile Strength} = \frac{P}{b \times t}, \quad (1)
\]

where \( P = \) tensile force in N, \( b = \) specimen width in mm, and \( t = \) thickness in mm.

\[
\text{Flexural Strength} = \frac{3PL}{2bd^2}, \quad (2)
\]

where \( P = \) flexural force in N, \( b = \) specimen width in mm, and \( d = \) thickness in mm.

3. Result and Discussions

3.1. Tensile Behaviour of Nanocomposites. The largest force a material can sustain without cracking when expanded is divided by its original cross-sectional region and its tensile strength. The tensile behaviour of luffa fibre-based hybrid composites with and without ATH nanofiller is shown in Figure 5. The results showed that a mixture of 1 wt.% ATH nanofiller and 24 wt.% luffa fibre had a maximum tensile strength of 18.28 MPa. Compared to empty luffa fibre composites (11.46 MPa), ATH nanofiller-based luffa fibre composites with 1 wt. percent ATH nanofiller show a 62.68 percent improvement in tensile strength.

As a result of these findings, it was evident that increasing the fibre load content reduced composite tensile strength. This discovery highlighted the matrix’s and reinforcements’ weak interfacial adhesion because of the excellent dispersion of ATH nanofiller in the matrix. The inclusion of an ATH nanofiller affects the differences in tensile strength in the manufactured composites. It established a strong link between the matrix and its reinforcements, which would aid in the effective transmission of stress in the composite system. By lowering the void content, the ATH nanofiller’s inclusion enhanced the composites’ rigidity. The ATH filler is used as a key link in this study to transfer load or stress between the composite laminates. When the filler addition exceeded the limitations above 1 wt.%, the tensile strength of the nanocomposites was lowered. This might be due to increased filler additions, resulting in more particle-particle interaction than fibre-matrix interaction. The unequal dispersion of the nanofiller was causing more voids in the laminated composites [18].

The ATH nanopowder-filled and NaOH-treated luffa fibre-based composites are shown in Figures 6(a) and 6(b). Figure 6 depicts the cracked surface of a tensile specimen. Figures 6(c) and 6(d) depict the cracked surface of ATH filler-added hybrid composites ranging from 0% to 5% by weight. The strength of the composites is primarily determined by the interfacial bonding strength between the reinforcements and the matrix. In the initial conditions (i.e., 0 wt. percent ATH filler), Figure 6(c) shows a greater pullout of fibre in the composite. A superior fibre and matrix contact was generated when ATH filler was added to luffa fibres. As
a result of these factors, the number of fibres extracted from the fabric was reduced [19]. Figure 6(d) shows this very clearly. Adding 1% ATH filler to the luffa composites improved fibre and matrix adhesions. The tensile strength of nanocomposites is reduced as the weight percent of the ATH nanofiller increases, owing to the composite’s amalgamation of particle-rich areas. It demonstrates the SEM, Figure 6(e).

3.2. Flexural Behaviour of Nanocomposites. The laminated samples were evaluated for flexural strength (also known as bending strength). Figure 7 depicts the flexural strength fluctuations of the laminated samples. A flexural test was
conducted to evaluate the flexural strength of the luffa fibre composite and ATH-filled luffa fibre composite samples and explore the influence of nanofillers. The results demonstrate that a polyester composite with luffa fibre as reinforcement with 1 wt.% ATH added had a maximum flexural strength of 28.91 MPa. At the same time, compared to the luffa fibre-reinforced composites without nanofiller ATH addition, the percentage increase in flexural strength with 1 wt. percent ATH was 53.72 percent (21.26 MPa). The flexural strength of the reinforcement luffa fibre decreases when the polyester resin is applied, which might be due to the natural fibre’s weak adhesive property with the matrix due to the hydrophilic and hydrophobic properties and matrix. A poor fibre-matrix interaction was produced due to this aspect, which reduced the fibre’s reinforcing action and prevented stress transmission from the matrices to the load-carrying fibres. To address this issue, the filler ATH was mixed with the matrix at various weight percentages, and the composite laminate S2 was found to have the highest flexural strength of 28.96 MPa. The increase in strength could be due to the filler ATH nanoparticles’ fine dispersion in the matrix, which causes good interfacial adhesion between the matrix and reinforcement. This could efficiently distribute the load from the matrix to the fibre, delaying crack growth [20].

The interface interaction of the fibre-matrix was evaluated using a scanning electron microscope on the broken surface of the flexural testing samples. Figures 8(a) and 8(b) show a micrograph image of the cracked surface of the composite samples combined with the nanofiller ATH to a matrix ranging from 0 wt. percent to 5 wt., and the fibre-matrix interface bonding mostly determines the percent characteristics of the polymer composite. Figure 8(a) shows that the fibre-matrix contact for luffa fibre polyester composites with 0% ATH filler has significant pull-outs and holes, resulting in poor bonding, low strength, and fibre-matrix interaction [21]. Figure 8(b) shows that the fibre-matrix interaction has improved with the addition of nanofiller ATH to the matrices, the presence of 1 wt. percent ATH in the matrix. Sample S2 had the least fibre pullout, resulting in improved interfacial adhesion between the fibre and the matrix, which enhances the flexural strength of the specimens.

3.3. Impact Strength of Nanocomposites. Figure 9 depicts the impact strength fluctuations. An impact test has been carried out on the luffa fibre composite and nano-ATH-filled luffa fibre composite samples to determine the impact strength and the involvement of ATH nanoparticles. The results reveal that a polyester composite with luffa fibre as reinforcement with 1 wt. percent ATH added provided maximum impact strength values of 14.89 KJ/m². When luffa fibre-reinforced materials with 1 wt. percent ATH were compared to luffa fibre-reinforced composites without nanofiller ATH addition (12.56 KJ/m²), the percentage improvement in impact strength was 5.59 percent. The addition of nano-ATH fillers to the matrices enhances the rigidity of the manufactured composites, which may arise due to greater adherence between the fibre and the matrix, resulting in enhanced impact resistance [22].

A scanning electron microscope was used to examine the fibre pullouts and interfacial adhesion between the fibre-matrix and the shattered regions of the impact-tested samples. The micrograph picture of the cracked surface of the composite samples treated with the nanofiller ATH, ranging from 0 wt. percent to 5 wt. percent, is shown in Figures 10(a) and 10(b). The impact strength of the polymer composite is mostly determined by the fibre-matrix interface bonding, as per the microscopy pictures. Figure 10(a) illustrates that the fibre-matrix interaction of the composite sample S1 without filler has significant fibre pullouts, fibre breakage, and voids, resulting in poor bonding, minimal strength, and a poor fibre-matrix interaction. Figure 10(b) depicts a microscope image of a composite sample.
Figure 11: Microstructural analysis of nanocomposites at cryogenic durations of (a) 15 min, (b) 30 min, and (c) 45 min.

Figure 12: TGA curves of untreated and cryogenic treated luffa fibre composites.
containing 1% filler; the addition of nanofiller ATH to the matrix improves the fibre-matrix interaction. Sample S2 had the least amount of fibre pullout, resulting in greater interfacial adhesion between the fibre and the matrix and improved impact strength [23].

3.4. Effect of Cryogenic Treatment. The composite plate was exposed to liquid N2 at -196°C and thermal cycling during cryogenic processing. The use of cryogenic treatment to improve the mechanical properties of polymer-based composites is a unique technique. Figure 11 shows the effect of cryogenic treatment on the mechanical properties of polymer-based hybrid composites. The morphological pictures of 15, 30, and 45 min of cryogenic treatments are shown in Figures 11(a)–11(c). The majority of the composites are visible, with 15 to 30 minutes of cryogenic treatment demonstrating good mechanical results. It might be due to the residual stress created by the compression interface due to the cryogenic straining of composite materials, because of the changing matrix and fibre shrinkage create residual stresses at low temperatures [3].

Because the fibre has a lower thermal expansivity than the polymer matrix, the resultant stresses are compressive in the fibre and tensile in the matrix [24]. These compressive interface stresses assist in keeping fibre and matrix in contact and enhance adhesion, resulting in better outcomes [25]. The cryogenic embrittlement of fabricated composites causes the components to be stiffer at low temperatures. As the stiffness of the sample decreases, the elasticity of the sample also decreases, resulting in a reduced deflection [26]. After cryogenic treatments, the debonding resilience of ATH and luffa hybrid composites was enhanced due to the expansion of compressive shrinkage stresses at the border. When composite materials are exposed for more than 45 minutes, their mechanical properties deteriorate. Longer liquid nitrogen cooling times may increase thermal stress due to the increasing quantity of fibre-resin imbalance [27]. The delamination behaviours are more destructive to the ATH and luffa hybrid-polyester structures caused by lower contact. It might be because it has a very high reduc- tion in the time it takes to cure polyester resin [28]. Low-bonded composites include large interface debonding regions, which exacerbate various risk factors that might lead to failure. For composites processed for longer than 45 minutes, internal forces are created by changes in the thermal expansion coefficient (TEC) between the matrices and the fibres, released by physical phenomena including potholing and matrix/fibre interaction debonding and splitting [13, 24]. The findings above are readily visible in SEM pictures.

3.5. TGA Analysis. Figure 12 depicts the remarkable enhancement in heat stabilization of luffa fibre-based polyester composites in a cryogenic atmosphere. Untreated luffa fibre composites have a secondary phase derivatives weight of 33.24 percent, greater than the 15-minute, 30-minute, and 45-minute cryogenic processed luffa fibre composites, which had secondary phase derivative weights of 29.31 percent, 26.13 percent, and 27.15 percent, correspondingly [29]. The third phase mass loss of untreated luffa fibre is 52.12 percent, which is lower than the mass loss of 15-minute, 30-minute, and 45-minute cryogenic treated luffa fibre composites, which are 54.12 percent, 54.13 percent, and 53.89 percent, correspondingly. The cryogenic treatment on luffa fibre composites reflects a reduction in lignocellulosic materials and lignin concentration with an increase in the cellulose of the fibres [30]. Untreated luffa fibre reaches a third phase high of 439.4 degrees Celsius, while cryogenically treated fabric reaches third phase maxima of 459 degrees Celsius, 455.8 degrees Celsius, and 457 degrees Celsius for 15, 30, and 45 minutes, correspondingly, as illustrated in Figure 12. Because of the cryogenic treatment on the material, the second phase peak has also grown substantially [31]. These two stages aid in understanding a material’s significant thermal characteristics [32]. In these phases, the natural fabric’s fundamental elements (hemicellulose, cellulose, lignin, and so on) are decomposed [33]. Because the decomposition temperature of cryogenic treated fabric is greater than that of an untreated sample, cryogenic treatment at 77 K has increased the thermal properties of luffa composite materials [34].

4. Conclusion

The mechanical characteristics of ATH nanopowder and woven luffa fibre-based polyester hybrid composites were examined in this study, and the following observations were obtained:

(i) Nanocomposites with increased mechanical characteristics come from the combination of numerous factors. The suggested levels of controlled design parameters for ATH and luffa-based nanocomposites are 1 wt. percent ATH powder, 24 wt. percent luffa fibre, and 30 minutes of cryogenic treatment

(ii) Due to the impact of voids countered by the inclusion of nanomaterials, the S2 specimen (1 wt. percent of ATH) resulted in high strength of hybrid composites compared to the S1 specimen (0 wt. percent of ATH). It established a strong link between the matrix and its reinforcements, which would aid in the effective transmission of stress in the composite system

(iii) The maximum stress generated at the interfaces during the cryogenic treatment was compression in character, assisting in greater matrix-fibre adhesion but only for the first 30 minutes of processing. Because the fibre has a lower thermal expansivity than the polymer matrix, the resultant stresses are compressive in the fibre and tensile in the matrix. These compressive interface stresses assist in keeping fibre and matrix in contact and enhance adhesion, resulting in better outcomes. It was confirmed through TGA analysis

(iv) In SEM photographs, lower permeability levels were visible. This could lead to a greater degree of ATH powder particles in the luffa fibre and the matrix
Data Availability
The data used to support the findings of this study are included within the article. Should further data or information be required, these are available from the corresponding author upon request.

Conflicts of Interest
The authors declare that there are no conflicts of interest regarding the publication of this paper.

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