Comparison of chemically surface treated Luffa cylindrica using scanning electron microscopy (SEM)

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Abstract. The world as we probably know is currently facing a big difficulty referred as environmental pollution. Researchers from around the globe have been working on biodegradable materials in order to reduce the overall consequences due to environmental pollution. Furthermore, biodegradable composites also known as green composites made using natural fibers are highly considered over non-green synthetic fiber composites. Additionally, natural fibers are low in cost, have good mechanical properties, biodegradability and require less production energy. Focus of this research paper was on one of the natural fibers, Luffa cylindrica (LC). There are plenty of surface treatments available including chemical and mechanical surface treatments. However, the focus of this paper was on chemical surface treatments. Sodium hydroxide, silane and acetylation chemical surface treatments were utilized. Chemically surface treated LC was compared with untreated LC with the help of scanning electron microscopy (SEM). With the help of SEM, it was observed that all of the chemical surface treatments were effective. Furthermore, it was also noticed that each of the chemical surface treatment affected LC differently. However, sodium hydroxide surface treated LC samples showed the best outcome by removing waxy impurities as well as voids from the surface of LC.

1. Introduction

For decades, composites have been made using synthetic fibers. Some of the commonly used synthetic fibers are glass fiber and carbon fiber. These synthetic fibers have been used due to their mechanical properties thus, their composites are well favoured over steel and aluminium. Although the total emissions are lesser than steel and aluminium, recycling or biodegradability problems arise [1]. Synthetic fibers are not entirely biodegradable. They are flammable and emit huge quantities of smoke and toxic fumes. However, natural fibers on the other hand have decent mechanical properties, good soundproofing, high impact strength and are good fire retardants therefore, natural fibers can be regarded as multipurpose materials [2, 3]. Additionally, natural fibers are abundant, recyclable and biodegradable [4]. Plenty of natural fibers are present however, focus of this research paper is on Luffa cylindrica (LC) which is commonly known as a sponge gourd as seen in figure 1. The LC plant is abundantly available in China, India, and Japan. Furthermore, LC is one of the eight species present in a family of vegetable sponges. The plant itself has a dense outer skin layer which is peeled in order to expose the
fibrous mat of LC [5]. As the cells present inside LC expands, which then leads to elongation of cells which then develops a fibrous structure. Dry fibrous structure of LC mainly consists of cellulose, hemicellulose and lignin [6]. As it has been described, LC is fibrous and spongy therefore, LC has great mechanical properties such as high strength, stiffness and energy absorption [7]. Microstructure of LC is mainly made of cellulose, hemicellulose and lignin which make it fall under the category of lignocellulose natural fibers as seen in figure 2. Many researchers have utilized LC to produce composites with various types of polymers, given the mechanical properties of LC [8].

As recorded by other researchers, LC has a high cellulose content of 55–70%, 8–22% hemicellulose, 10–23% lignin, 3.2% extractive and 0.4% ash as seen in Table 1 [7]. LC density is between the range of 0.82–0.92 g/cm³, which is not so high compared to other widely used natural fibers such as sisal or hemp [10, 11]. Natural fibers such as LC has a higher fiber content than synthetic fiber, and small amounts can provide comparable efficiency, reducing the overall fiber consumption [12].

![Figure 1. Luffa fiber](image1.png)

![Figure 2. Classification of natural fibers](image2.png)
Table 1. Shows mechanical properties and chemical composition of luffa fiber [7, 13].

| Mechanical Properties          | Luffa fiber | Chemical Composition | Luffa fiber |
|--------------------------------|-------------|----------------------|-------------|
| Tensile Strength (MPa)         | 202.3       | Cellulose (%)        | 55–70       |
| Young’s Modulus (GPa)          | 4.5         | Hemicellulose (%)    | 8–22        |
| Elongation at break (%)        | 2.5         | Lignin (%)           | 10–23       |
| Density (g/cm³)                | 1.2         | Extractives (%)      | 3.2         |

Scanning electron microscopy (SEM) is widely used technique which was first introduced 80 years ago. SEM analysis has been used as a key process for characterization of the sample. With the help of SEM various researchers were able to notice microscopic changes occurring on the surface of the fiber due to chemical surface treatments. Furthermore, this technique of analysing the samples is preferred by researchers due to images being in high resolution with an average magnification range of 10–500,000 times [14]. Previously researchers were able to notice changes in terms of fiber surface, removal of impurities, waxy impurities and voids [15–18].

In this research paper the focus was on analysing the effects on LC surface after chemical surface treatments. Sodium hydroxide, silane and acetylation surface treatments were utilized. Chemically surface treated LC was compared with untreated LC with the help of SEM analysis. Changes were noticed in terms of LC surface, impurities and voids were analysed before and after chemical surface treatments.

2. Methodology

2.1. Preparation of samples

LC utilized in this research was bought from a local dealer. In order to produce composites using natural fiber, surface treatment is a necessary step. In order to enhance the compatibility between LC and any polymer matrix, surface treatments are necessary. In addition to that chemical fiber surface treatments have shown successful removal of naturally occurring waxy impurities and hydroxyl groups which contribute to ineffective bonding between a fiber and a polymer. In this specific research LC fibers were chopped using a chopper and with the help of a sieve, 3mm chopped LC samples were separated from the batch.

Removal of surface impurities like dust is a crucial step before performing any type of chemical surface treatment. LC samples with a length of 3mm were soaked for 2 hours in a solution which was made of detergent and distilled water at 60 °C. This step as mentioned earlier would help in removing any kind of impurities present on the surface such as dust. Furthermore, LC samples were removed from the solution and were rinsed thoroughly using distilled water. Clean and rinsed LC samples were left for drying for 3 days.

2.2. Fiber chemical surface treatment

2.2.1. Sodium hydroxide (NaOH) chemical surface treatment. Dried 3 mm in length LC samples were soaked for an hour at room temperature. Sodium hydroxide (1 N) solution with distilled water was utilized in this research. LC samples were then removed and cleaned thoroughly using distilled water containing a few drops of acetic acid to neutralize the LC samples. Furthermore, LC was rinsed
thoroughly under a steady stream of distilled water to ensure sodium hydroxide was eliminated entirely from the surface. Rinsed LC samples were left for drying at room temperature for 24 hours. Furthermore, samples were also dried for 12 hours at 80 °C in the oven. This approach to prepare sodium hydroxide surface treated samples for SEM testing has been standardised by various other researchers in their research using different parameters [19–21].

2.2.2. Silane chemical surface treatment. Dried LC chopped 3 mm samples were soaked for an hour at room temperature in a (60:40) solution made from ethanol and water. Furthermore, 5wt% silane was added in the solution. Silane coupling agents known as Bis-(3-triethoxysilylpropyl) and 3-amino propyltriethoxysilane (APS) were also added in the solution. In order to maintain the pH value at 4, acetic acid was utilized. After an hour of immersion LC samples were removed from the solution and were washed thoroughly with the help of distilled water to remove any remaining solution. Rinsed LC samples were left for drying at room temperature for 24 hours. Furthermore, samples were also dried for 12 hours at 80 °C in the oven. This approach to prepare silane surface treated samples for SEM testing has been standardised by various other researchers in their research using different parameter [21, 22].

2.2.3. Acetylation surface treatment. Dried LC chopped 3 mm samples were soaked for an hour. The solution consisted of acetic acid and acetic anhydride with. Ratio of 1:1 was utilized furthermore, concentrated sulphuric acid (1ml) was added in the solution as well. Concentrated sulphuric acid acted as a catalyst in the solution. LC samples were removed from the solution after an hour of soaking time. Distilled water was utilized to thoroughly clean the samples until the solution was completely removed from the surface of LC. Rinsed LC samples were left for drying at room temperature for 24 hours. Furthermore, samples were also dried for 12 hours at 80 °C in the oven. This technique to perform acetylation surface treatment on the fiber samples for SEM testing has been standardised by various other researchers in their research using different parameter [21, 23].

2.3. Scanning electron microscopy (SEM) SEM analysis is one of the most commonly used electron microscopies to understand the surface composition of the fibers. Images are produced with the help of electrons from the beam and electrons from the sample combining to create signals. Various researchers have used SEM to study various chemical surface treatments and their effects on the surface of natural fibers. SEM was performed using JOEL (JSM-6300F). Whereas, accelerating voltage was set to 20 KV and working distance of 20mm was used as mentioned in the research. Magnifications of 100x, 500x and 800x were utilized to analyse the surface of the fiber thoroughly. Furthermore, SEM was used to compare treated and untreated fiber samples. Similar standardised approach regarding SEM process has been used by various researchers with different parameters [7, 24, 25].

3. Results and discussion Process of scanning electron microscopy (SEM) as mentioned above provided images at magnification of 100x, 500x and 800x for untreated and chemically surface treated LC samples. Untreated samples of LC can be seen in figure 3. Smooth fiber surface can be seen due to the waxy impurities present on the surface shown in figure 3 (a). Furthermore, figure 3 (b) confirms the presence of voids on the surface which can be seen as black dots. Furthermore, impurities can be seen in white dots. similar results can also be seen in the research shown by various researchers regarding fiber surface treatments [15, 16]. Due to the presence of waxy impurities, impurities and voids, adhesion between polymer and the LC will be poor, which leads to poor mechanical properties.
Figure 3. Shows untreated luffa fiber samples, (a) 100x magnification and (b) 800x magnification.

Sodium hydroxide surface treated samples can be seen in figure 4 shown underneath. Using the methodology explained earlier, 1 N of sodium hydroxide solution was used to chemically surface treat LC. Rougher fiber surface can be seen which is due to the removal of waxy impurities from the surface of LC. Removal of waxy impurities was achieved due to the impurities being soluble in sodium hydroxide solution [26]. However, some of the impurities can still be seen on the surface of LC as shown in figure 4 (a) and (b). This can be due to the immersion time being only one hour. Furthermore, there is no sign of voids present on the surface of LC. Removal of waxy impurities and voids from the surface of LC can be seen in other research papers as well shown by other various researchers [17, 27]. Better adhesion between LC and polymers can be expected due to surface being rough and without voids. Due to better adhesion, better mechanical properties can be expected from LC and polymer composites.

Figure 4. Shows sodium hydroxide surface treated luffa fiber samples, (a) 100x magnification and (b) 500x magnification.

Silane surface treated samples can be seen in figure 5 shown underneath. Using the methodology explained earlier, 5wt% silane solution with ethanol was used. Bis-(3-triethoxysilylpropyl) and 3-amino propyltriethoxysilane (APS) coupling agents were utilized. It can be seen in figure 5 (a) and (b) that there is not much difference on the fiber surface. Surface of LC is still smooth due to silane sticking on the surface of the fiber [23]. However, figure 5 (b) shows removal of surface impurities but
some voids on the surface are still present [28]. This result can be due to immersion time being only one hour. Furthermore, better adhesion can be expected between the fiber and polymer due to removal of waxy impurities and impurities from LC surface. However, due to the voids present on the surface of the fiber, this may affect the overall adhesion between LC and polymer.

![Smooth surface](image1.png) ![Voids](image2.png)

**Figure 5.** Shows silane surface treated luffa fiber samples, (a) 100x magnification and (b) 500x magnification.

Acetylated surface treated samples can be seen in figure 6 shown underneath. Using the methodology explained earlier, 1:1 mixing was used between acetic acid and acetic anhydride to chemically surface treat LC samples. It can be seen in figure 6 (a) and (b) that fiber surface is still smooth after the surface treatment. This is due to the acetyl functional groups replacing hydroxyl functional groups from the surface of LC [23]. Furthermore, impurities from the surface are removed to some extent. However, figure 6 (b) shows voids present on the surface of luffa fiber samples. This result can be expected due to immersion time being only one hour. Better adhesion can be expected between the fiber surface and polymer due to removal of waxy impurities and impurities to some extent. However, presence of voids may not give the best mechanical properties.

![Smooth surface](image3.png) ![Voids](image4.png)

**Figure 6.** Shows acetylated surface treated luffa fiber samples, (a) 100x magnification and (b) 500x magnification.
4. Conclusion
Focus of this research was on chemically surface treated LC. Sodium hydroxide, silane and acetylation chemical surface treatments were utilized. Untreated LC was compared with chemically surface treated LC with the help of SEM. From the results shown in the previous section, it can be concluded that untreated LC surface consists of waxy impurities which causes smooth surface, other impurities, and voids. All these factors play a huge role in decreasing the adhesion between the fiber and the polymer hence, leading to poor mechanical properties. However, by utilizing chemical surface treatments, these factors can be tackled. Sodium hydroxide surface treatment was able to remove waxy impurities and voids from the surface of LC but, struggled to remove other impurities completely. On the other hand, silane surface treatment showed smoother surface due to silane being deposited on the surface of LC and the impurities were removed significantly. However, voids were not removed completely. Acetylation surface treatment showed smoother surface as well because the acetyl groups replaced the hydroxyl groups in the chemical structure of LC. Furthermore, impurities were removed to some extent; yet voids were still present on the surface after the surface treatment. Overall, with the help of SEM analysis it was predicted that each of the chemical surface treatment utilized in this research according to the methodology was successful. However, each of the chemical surface treatment affected LC differently.

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