Evaluation of the Performance of Bagasse, Paddy Straw, and Banana Stem Fibers Pretreated by Alkaline Hydrogen Peroxide Pretreatment for Development of Cellulose-based Composite

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Abstract

In present, cellulose fibers are used to reinforce sustainable composite materials which are used in building constructions. Cellulose is a natural long chain polymer which can be extracted from lignocellulose materials. In this study, Alkaline Hydrogen Peroxide (AHP) pretreatment was used to extraction of cellulose from three available lignocellulose materials (Bagasse, Paddy Straw, Banana Stem) in Sri Lanka. Three pre-treatment solutions were prepared by mixing three different concentrations of NaOH with equal volume (10ml) and an equal volume (0.5 ml) of Hydrogen Peroxide(H_2O_2). The experiment consists of 27 samples combination of the above mentioned different NaOH and H_2O_2 concentrations and replicating each as three times. After AHP Pre- treatment, percentage of weight loss, percentage of cellulose content, fiber diameter, tensile strength and morphological characteristics were tested. Additionally, Fourier transform infrared spectroscopy (FTIR) was done for the confirmation of extracted fibers were cellulose. Holocellulose content and weight loss percentage were 86.63%-95.90% and 49.99%-74.32% respectively in pre-treated fibers. The tensile strength of banana stem fiber is highest (171.40Mpa) and paddy straw fiber is lowest (18.34Mpa) those fibers' diameters were varying between 134.0-268.1 µm. According to the above test results, optimum pre-treatment solution concentration was separately selected by those lignocellulose materials.

Keywords: lignocellulosic, pretreatment, cellulose, alkaline hydrogen peroxide

Introduction

Most of the cellulose in nature is lignocellulosic compound, containing cellulose, hemicellulose, and lignin. The Structure of lignocellulosic compound consists of hemicelluloses which are wrapping around the long chains of cellulose. Thus, cellulose and hemicellulose are encased by lignin [1]. Lignocellulosic waste is produced in large quantities all around the world and some examples of lignocellulosic materials are cornhusks, corn stoves, bagasse, sugar cane leaves, guinea grass, banana stem, paddy straw, and wood fibers etc. Cellulose is a natural long chain polymer having the chemical formula of $(C6H10O5)n$ [2], which has low density, biodegradability, renewability, and satisfactory level of tensile strength and water absorption [3]. Lignocellulosic waste is used to process cellulose fibers for food packing applications, sustainable cement based products (sustainable martials). mortar, reinforcement of brittle building materials, and tunable filters[4]. Several pretreatment methods such as biological, chemical, and physical pre-treatments are available to remove lignin from lignocellulosic materials [2]. Generally, in industrial processing, some chemical pretreatments such as alkaline, microwave-assisted alkaline, and Alkaline Hydrogen Peroxide (AHP) pretreatments are applied. According to the research conducted by Fernando, et al [1], AHP pretreatment is an effective, simple, and low-cost method, which is performed at room temperature and atmospheric pressure and used in industry to remove lignin from lignocellulosic materials. In the last few years, the construction industry focused on increasing the reinforcement of composite mixtures by using cellulose fibers from agro plants waste to development of healthy, environmentally friendly, and commercially viable. Although Cotton is naturally available cellulose, it is not suitable as it is not abundantly available in Sri Lanka [5]. Therefore, In the present study bagasse, paddy straw, and banana stem those are widely available agro plant residues in Sri Lanka were selected for extraction of cellulose fibers and which cellulose contents are respectively 32-34%, 32% and 47% [1]. The objective of the present study is to evaluate the performance of lignocellulosic fibers of Bagasse, Paddy straw, and Banana stem pretreated by alkaline hydrogen peroxide pretreatment, for development of cellulose-based composites. In addition, the best pretreatment solution was evaluated based on the extraction of maximum cellulose percentages with minimum weight losses of lignocellulosic materials.

Methodology

A. Materials

Industrial grade Sodium hydroxide (NaOH), Hydrogen peroxide (H_2O_2) and Hydrochloric acid (HCl) were purchased. Bagasse sample was collected from Palwattha Sugar Industry and other lignocellulose materials were collected from Hambantota District.

B. Alkaline Peroxide Pretreatment

Pre-treatment solutions were prepared by mixing three different concentrations (C1, C2 & C3) of NaOH with equal volume (10ml) and an equal volume (0.5 ml) of hydrogen peroxide(H_2O_2) as giving Table I.

Selected three lignocellulose materials were named as follow, Bagasse (B), Paddy straw (PS), and Banana stem (BS). The experimental design was a two-factor factorial. Two variable factors were three plant residues and three pretreatment solutions. The experiment consists of nine treatments as giving table II, Further, replicating of each treatment three times, the experiment consisted of 27 samples. Ten milliliters from each prepared pretreatment solutions were added to 1 g of lignocellulosic materials. A total of 5 g of biomass with 50 ml of pretreatment solutions were added to the 500 ml beakers. Then, all beakers of the mixture were mixed every 4 hours and kept for 24 hours for further degradation. Then, each samples were washed three times with water. Afterward, mixtures were immersed in 10% HCl for 2 hours and washed with normal water once. Finally, pre- treated fibers were heated at 60 ^{0}C for 4 hours and kept in a desiccator until reached a constant weight.

The holocellulose percentage of pretreated fibres and percentage of weight loss were tested using chlorination method as described by Fernando et al [2]. In addition, variations of weight losses and holocellulose content of pre-treated fibers were analyzed using Minitab version 20.

Table 01: Concentration of Pre-treatment Solutions

Pre-treatment solutions	**NaOH Concentration**	**99% H_2O_2 volume**
C1	10 ml of 1,0 mol/l	0.5 ml
C2	10 ml of 1.5 mol/l	0.5 ml
C3	10 ml of 2.0 mol/l	0.5 ml

C. Fourier Transform Infra-Red Spectroscopy Analysis

Raw lignocellulosic materials of bagasse, paddy straw, banana stem, and pretreated fibers were analyzed using Fourier Transform Infra-Red spectroscopy to conform the extracted fibers were cellulose. The spectra were collected between 4000 – 600 cm-1 range at 4 cm-1 resolution with an accumulation of 24 scans in transmittance mode. The testing was performed at ambient conditions on a Bruker ALPHA spectrometer (Bruker Corporation, Billerica, MA).

D. Scanning Electron Microscopy (SEM)

The selected three raw lignocellulosic materials and pretreated fibers from the AHP pretreatment were observed the surface morphology using Scanning Electron Microscopy (SEM). The Samples were gold sputter coated prior to observation and examined with an accelerating voltage of 10 kV. Morphological analysis of fibers were performed on EVO 18, Carl Zeiss AG, Germany.

E. The Tensile Strength and Diameter

According to the ASTM D3822 – 07, tensile strength of pretreated fibers was measured using Instron Universal Testing Machine. The diameter of pretreated fibers was measured by Projectina Microscope.

Table 02: The Nine Treatments

Treatment No	**Treatments**
1	B+C1
2	PS+C1
3	BS+C1
4	B+C2
5	PS+C2
6	BS+C2
7	P+C3
8	PS+C2
9	BS+C3

Results and Discussions

A. Weight Loss Percentage and Holocellulose Content

Fig. 1 shows the visual inspection of extracted fibers. The colour of pre-treated fibers ranged from white to light yellowish, and wool formations were observed on the surface. Fig. 2 and Fig. 3 show the variation in weight loss percentages and holocellulose content percentages of pre- treated fibers, respectively. All extracted fiber samples contain more than 80% holocellulose. The weight loss percentage depends on the concentration of pretreatment solutions and the chemical composition of the raw lignocellulosic materials. As the concentration of pretreatment solutions increases from C1 to C3, the weight loss percentage rises. This increase in weight loss is due to the removal of lignin, holocellulose, and other components from the raw materials. According to the chemical composition of raw lignocellulosic materials, bagasse has the highest lignin content (25.33%) [4], while banana stem has the lowest lignin content (13%) [6]. Consequently, bagasse exhibits the lowest weight loss percentage, whereas banana stem shows the highest. The chemical composition of raw plant residues may influence the weight loss percentage of lignocellulosic materials during pre-treatment

The results of the chlorination method used to determine holocellulose percentage indicate that holocellulose content is highest in pre-treated bagasse samples and lowest in pre-treated banana stem samples. That indicates the concentration of the pretreatment solutions are affected by the percentage of cellulose percentage of pretreated fibers. Therefore, fibers pre-treated with C3 concentrations had a higher amount of holocellulose content, and the lowest consisted of fibers, which were extracted from C1 pre-treatment. Due to increasing the NaOH in pretreatment solutions, its ability to remove a large portion of lignin from the lignocellulosic materials [1].

Figure 01: Visual inspection of extracted fibers

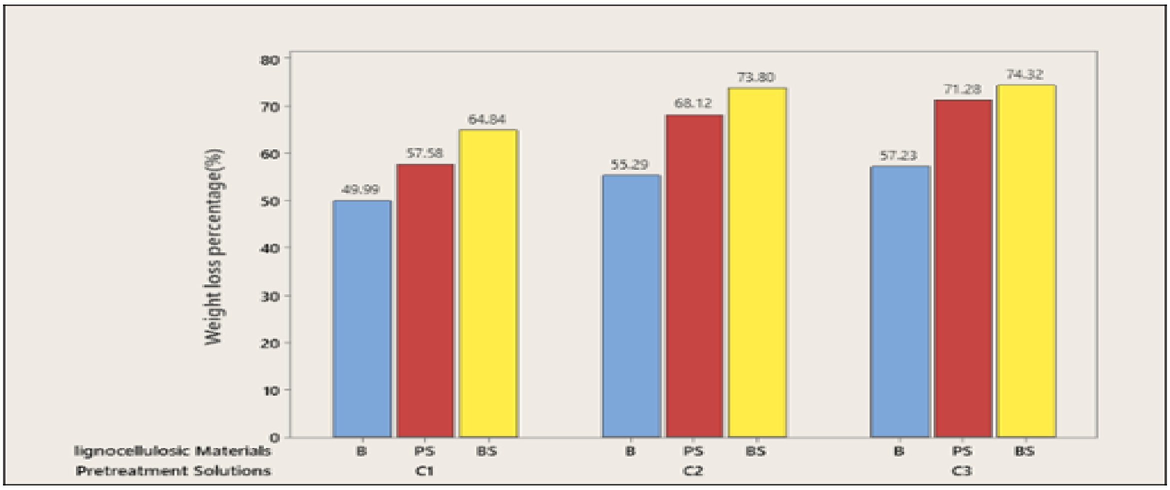

Figure 02: Variation of percentage of weight loss

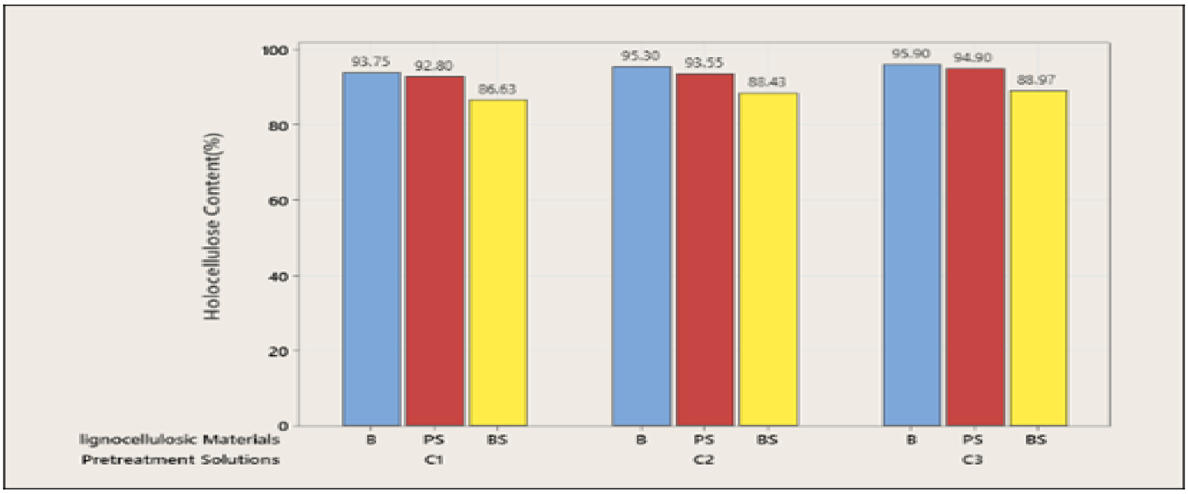

Figure 03: Variation of percentage of holocellulose content

A. Structural Characteristics

The comparison of FTIR results of raw bagasse, raw paddy straw, raw banana stem fibers, and pre- treated fibers from the AHP pretreatment with three different pretreatment solutions (C1, C2 & C3) of lignocellulosic materials have confirmed that the extracted fibers were cellulose as a comparison the wave number. Fig4, Fig 5, and Fig 6 shows the FTIR spectra of raw and pretreated fibers from three different pretreatments of bagasse, paddy straw, and banana stem. The peak at 1732 cm-1 in raw fibers are assigned to the characteristic of aliphatic ester in lignin and/or hemicellulose [7]. Disappearing this pick in the treated fibers is due to the removal of most of the lignin and hemicellulose from the pretreated fibers.

The peak at 893 cm-1 represents the glycosidic C–H deformation with ring vibration contribution and OH bending in the treated fibers, which indicates the typical structure of cellulose [3]. The broad peak was observed around 3445 cm-1 in to -OH stretching vibrations and a small sharp peak that appeared at 2922 cm-1 is attributed to the C-H stretching vibration of cellulose. Therefore, the comparison of the wavenumbers of raw fibers and each pre-treated fiber with the FTIR results of original cellulose fibers reported in [2] indicates that the pre-treated fibers were cellulose.

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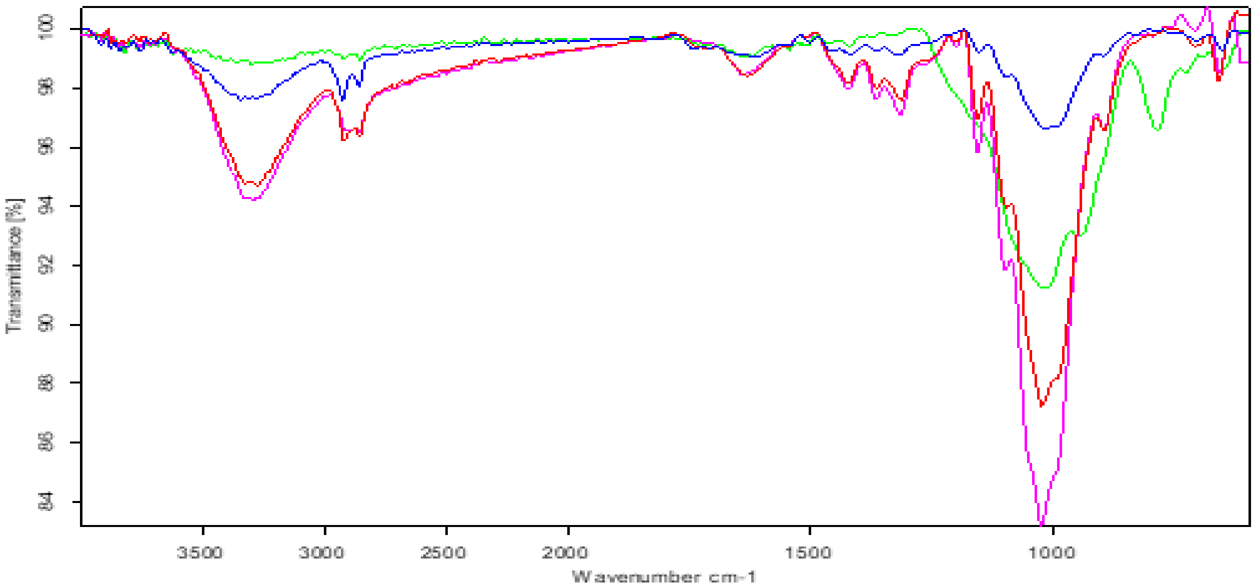

Figure 04: FTIR spectra of bagasse fibers (blue colour-raw fibers, red colour- C1 solution treatment, green colour- C2 solution treatment, pink colour – C3 solution treatment)

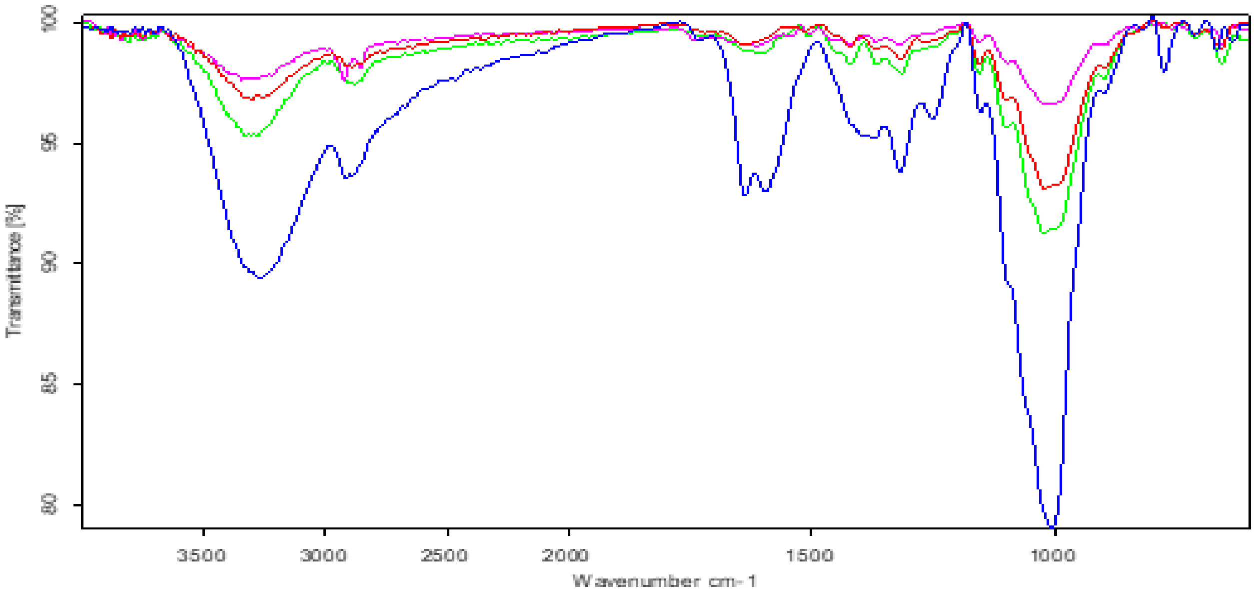

Figure 05: FTIR spectra of paddy straw fibers (blue colour-raw fibers, red colour- C1 solution treatment, green colour- C2 solution treatment, pink colour – C3 solution treatment)

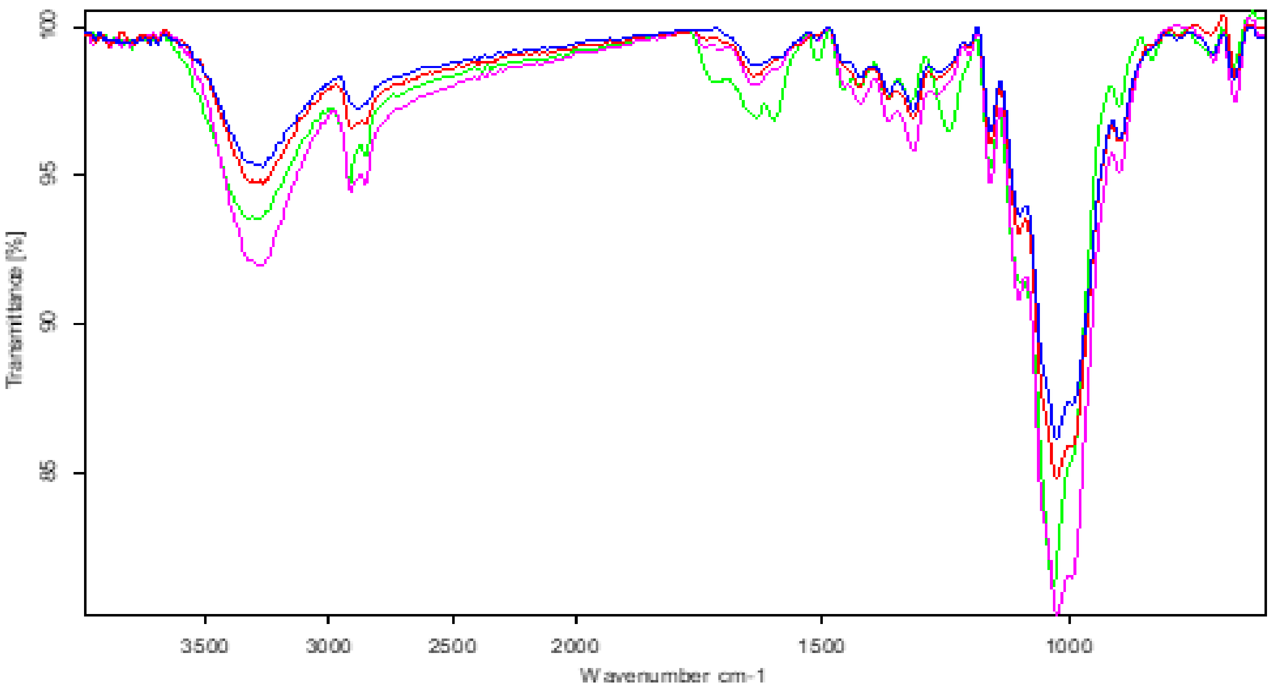

Figure 06: FTIR spectra of banana stem fibers (blue colour-raw fibers, red colour- C1 solution treatment, green colour- C2 solution treatment, pink colour – C3 solution treatment)

A. Morphological Analysis

The morphological features of raw lignocellulosic and pretreated three sampled of bagasse, paddy straw, and banana stem from prepared three different AHP pretreatment solutions Samples were analyzed by applying the Image J software for SEM images Fig 07 (a, b, c d), Fig 08 (a, b, c d) and Fig 09 (a, b, c d) respectively. Visual observation of Image J picture shows comparatively smooth surface in raw fibers and all pre-treated fibers show rough surface. Figure 04 shows tensile strength of pretreated fibers and diameter. Paddy straw fibers have a lower tensile strength. This may be due to the presence of significant amounts of silica particles, those particles reduce the hydrogen bond between the nanofibers [8]. Although each lignocellulosic material was pre-treated with three different solutions, there were no huge differences in the measured tensile strength and diameter (shown in Table 03).

Table 03: Pre-treated Fiber Diameter and Tensile Strength

Pre-treatment fibers	**Fiber diameter (μm)**	**Tensile Strength (single fiber/ Mpa)**
BC1	238.4	43.12
BC2	238.01	42.20
BC3	238.0	40.20
PSC1	268.1	20.00
PSC2	268.0	18.50
PSC3	265.0	18.34
BSC1	136.4	171.40
BSC2	136.1	169.69
BSC3	134.0	143.14

A. Comparison of Characteristics of pre-treated fibers with existed natural fibers used in reinforcement applications

According to Humphrey Danso, soil building blocks can be reinforced with natural fibers (Coconut, oil palm, Bagasse). The tensile strength and diameter of these fibers vary certain range. Bagasse fiber exhibit tensile strength of 25-62 Mpa and a diameter of 310-1190 μm. Oil palm fibers have a tensile strength of 65-141 Mpa and a diameter of 190-820 μm. Coconut fibers exhibit tensile strength of 83-222 Mpa and a diameter of 180-1010 μm o [10].

Moreover, these pre-treated fibers (bagasse, paddy straw, banana stem) also can be effectively used to reinforce soil building blocks because these experimental pre-treated fibers' tensile strength and diameter meet requirements for developing soil building blocks. A study by Elsaid reported the use of natural kenaf fibers to reinforce concrete with the fiber tensile strength is nearly 160 Mpa [11]. BSC1 and BSC2 pre-treated fibers tensile strength align with kenaf fiber tensile strength. Acidic pretreatment and ionic liquid both pretreatment methods can be used to extraction of fibers from lignocellulosic materials. However, reactions were happed some elevated temperature and pressure condition [12]. Comparing AHP pre-treatment can be performed at room temperature and atmospheric pressure [1]. It is a low-cost and simple method to extraction of fibers from lignocellulosic materials (bagasse, paddy straw, banana stem).

Cement mortar composite mixer can be reinforced with several fibers such as asbestos, PET, PP, PE, carbon fiber, etc. However chemical instability and higher cost are several disadvantages of those composites, further asbestos fibers are harmful to human health [9]. There is the possibility of overcoming those disadvantages by cellulose reinforcement in cement mortar.

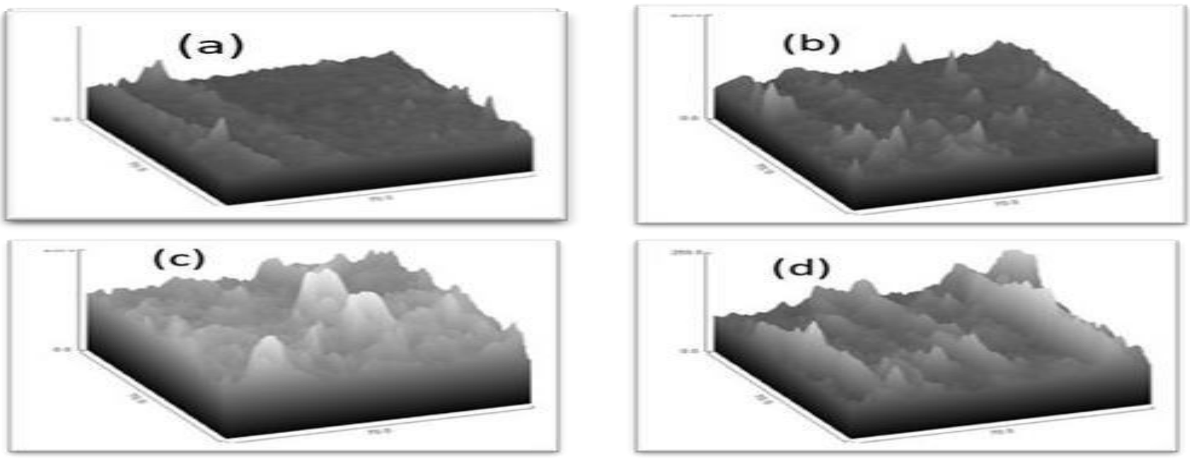

Figure 07: Analysis of surface roughness of bagasse raw fiber and pre-treated fibers- Application of Image J software for SEM images surfaces (a-Raw, b-C1 Concentration, c-C2 concentration, d- C3 concentration)

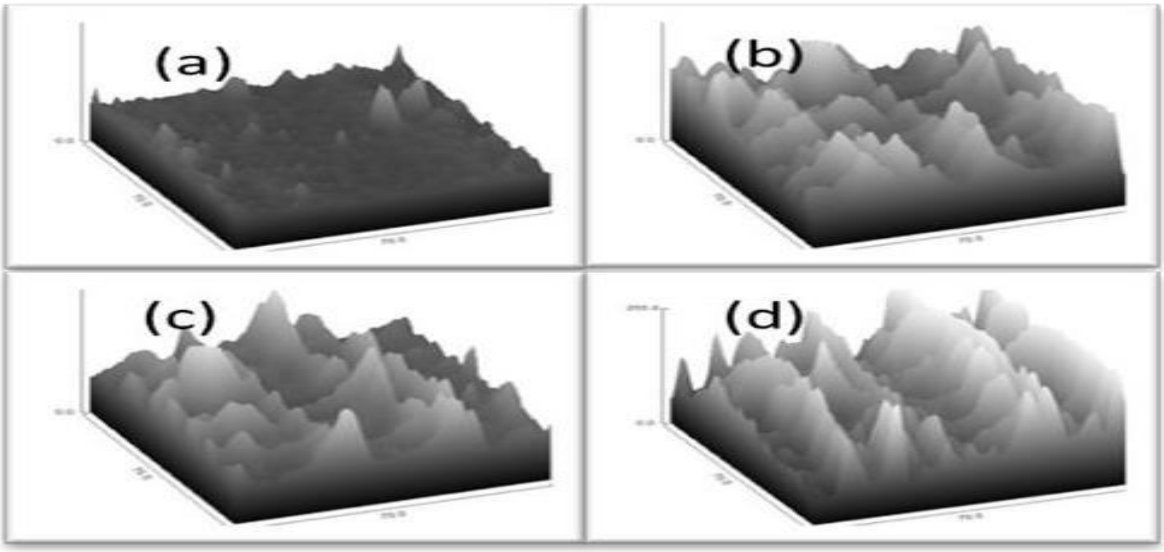

Figure 08: Analysis of surface roughness of paddy straw raw fiber and pre-treated fibers-Application of Image J software for SEM images surfaces (a-Raw, b-C1 Concentration, c-C2 concentration, d- C3 concentration)

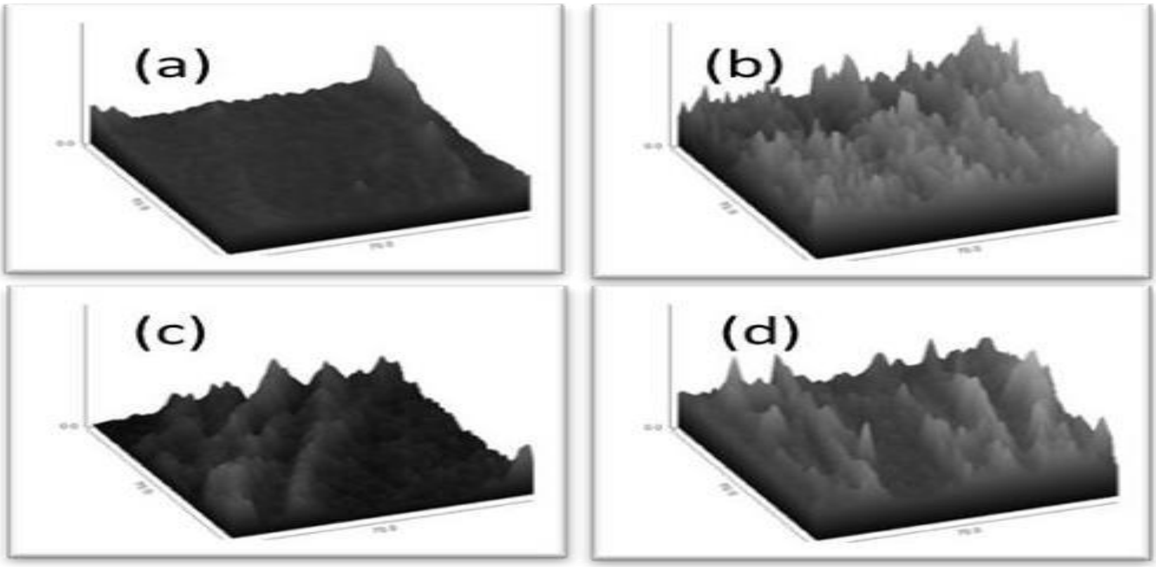

Figure 09: Analysis of surface roughness of banana stem raw fiber and pre-treated fibers-Application of Image J software for SEM images surfaces (a-Raw, b-C1 Concentration, c-C2 concentration, d- C3 concentration)

Conclusion

When increasing the concentration of pretreatment solutions, weight loss percentage and holocellulose content increase. Pretreatment fiber surface roughness increases with the concentration of pretreatment solutions. Bagasse fibers have a lower weight loss (49.99%) than Paddy straw (57.58%) and Banana stem (64.84%). Bagasse fibers has a higher holocellulose content (93.75%). According to the results, C1 solution (10 ml of 1,0 mol/l NaOH and 0.5 ml 99% H_2O_2) was the optimum pretreatment solution for extracting cellulose fibers with maximum holocellulose content and minimum weight lose percentage from selected Lignocellulose.

However, since AHP pretreatment can be performed at room temperature and atmospheric pressure, there is no need for additional energy to extract fibers from lignocellulosic materials. Additionally, natural fibers are not as harmful as asbestos fibers. Therefore, the pre-treated fibers prepared in the present study could be applied as reinforcements.

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