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Examining the Surface Effect on *Areca Catechu* by Different Retting Methods and the Effect of Enzyme and Alkali Treatment with the Help of SEM Analysis

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Abstract
Natural fibers like coir, sisal, jute, cotton and others have attracted the attention of various researchers for applications in civil engineering works because they are cheap, locally available, biodegradable, eco-friendly and their inclusion significantly increases the engineering properties of soil. Among all the natural fibers Arecanut fiber appears to be one of the promising fiber because it is inexpensive, available in abundant and it is a very high potential perennial crop. The present study gives an attempt to study the effect of retting process and pre-treatments on the fiber surface through SEM analysis, where the Areca nut fruit is retted with like stagnant water, running water and salt water for 5 days. After 5 days it is to be dried in the shade for 2 to 3days. Areca nut dried well the fiber were extracted manually and the extracted fiber were treated with alkali and enzyme. The structure and properties of the fiber and SEM (scanning electron microscope) images of untreated and enzyme treated fiber were analysed. Also the length and burning properties are also examined. The SEM images show a significant change is found on the surface of the fiber due to retting and finishing process. Whereas the length of the fiber is not influenced by any of the process.

Keywords: Eco-friendly, alkali, enzyme, retting, surface morphology

1. Introduction

Similar to agriculture, textiles have been an essential part of human life since the origin of civilization. Fibers such as jute and coir have been in cultivated since antiquity. As new methods and innovation have grown largely in the extraction and production of textile materials with natural fibers, in spite of the end use have been limited to making of cloth and containers and to insulate, soften and decorate our living space. The traditional textiles are being used extensively for industrial purpose as well as in components of composite materials, in medical implants, and geo and agro-textiles [(1)].

The thrust towards environmental awareness and consciousness all around the globe has developed an interest in natural fibers and its application in various fields [(2)]. Many efforts has been put forward in promoting ‘green buildings’ and ‘sustainable environments’, engineers have been push into the ‘natural materials’ spotlight. The natural fibers are either altered or combination as natural synthetic blends/mixture to fulfill the need in many applications [(3)]. Lignocellulosic fibers are being utilized in various areas such as automotive, building and packaging application to replace conventional man-made fibers. Various types of natural fibers have been subject to intense research interest such as flax, sisal, jute, kenaf and hemp [(4-7)]. This leaves another source of hemicellulose fibers, such as agriculture byproducts, to be further investigated [(4, 7, 8)]. A similar agriculture by product is *areca catechu* which is found to be wasted as landfills or burning.

India has a presence of a number of other natural fibers however the same have not been fully commercially exploited. India does not have a significant presence in other natural fibers, though ramie, flax, linen are used by Indian Textile Industry. Leaf fibers such as agava (sisal) and fruit fibers such as coconut and palm and banana and pineapple fibers are yet another group of natural fibers that have huge potential [(15)]. The natural fiber market is growing in a fast manner due to awareness on environment aspects and increase in demand of such products. The National Fiber Policy has been designed with a decadal perspective of 2010-20 and seeks to the World Fiber map by strengthening the existing policy framework and providing institutional and technological support for rapid fiber growth in the country in the coming decade [15].

The year 2009 has been assigned by UN to be the international year of natural fibers. Natural fiber industry employs millions of people all over the world, especially in the developing countries. As a major non-food commodity natural fibers and their products are processed in many small and large industries and consumers all over the world profit from the provided products. The transition towards a bio-based economy and sustainable developments as a consequence of the Kyoto protocols on greenhouse gas reduction and CO_2 neutral production offers high perspectives for natural fiber market [16]. New nonwoven materials made entirely from Plant-Based Materials wins 2015 INDA RISE durable product award [17].

The interest in natural fiber-reinforced polymer composite material is rapidly growing both in terms of their industrial application and fundamental research. They are renewable, cheap, completely or partially recyclable and biodegradable. Plants such as arecanut, flax, cotton, hemp, jute, sisal, kenaf, pineapple, ramie, bamboo, banana, etc., as well as wood, used from time immemorial as a source of lingocellulosic fibers, are more and more often applied as the reinforcements of composited[11]. Demand for natural fibers will be increased day by day in the years to come [12]. Quest for new, cheaper and sustainable sources of fibers that can replace or work against with fibers viz., jute, sisal, cotton, flax, ramie, polyolefin, polyester, polyamides and polyacrylics in terms of economics viability and eco-friendly properties. In the recent past, agro-based waste products have been utilized as natural sources to obtain to suit subjected to given conditions for agro-tech protective clothing [13, 14]. The researchers are in keen concern to the maximum utilization of resources in the best possible way.

Intense research of these agriculture byproducts will not only support the rural community by adding value to their products, but also will protect the environment by prevention of burning these fields leftovers [9]. Few studies have been carried out to indulge natural fibers in every field of textile usage due to various reasons like surplus availability, environmental issues, exploitation of available resources etc., one such less explored narural fiber is arecanut husk fiber. Arecanut, also known as beetlenut is the kernel obtained from the fruit of arecanut (areca catechu L.)[10].An area of concentration into arecanut fiber is been taken as a key objective and to develop textile material with the fiber can be an alternative or supporting material for industrial applications. Understanding the fibers for its characters, behavior and response towards various agents is important to engage the fiber in suitable end use.

2. Materials and Methods

2.1. Material

Areca nuts were sourced in the areas around Coimbatore, Tamilnadu, India. Well ripen fruit are chosen for the sampling. The nuts inside are used in the factories for the production of supari, medicine, colouring and many other. The epidermis of the fruit is thrown out as an agro waste or been used as a material for burning. This outer husk is a rich source of cellulose which is been used as a raw material for this study.

2.2. Fiber Retting Process

Retting is the process carried out to loosen the fibers from the shell. There various methods of retting namely water retting, dew retting, chemical retting, enzymatic retting, and physical retting.

The most widely practiced method of retting is called water retting, by submerging bundles of stalks in water. The water, penetrating to the central stalk portion, swells the inner cells, bursting the outermost layer, thus increasing absorption of both moisture and decay-producing bacteria. Retting time must be carefully judged; under-retting makes separation difficult, and over-retting weakens the fiber [18].

The water retting method is chosen for the study since it is simple and less expensive method. For this study water retting is carried out in three methods as follows:

- Stagnant water retting
- Running water retting
- Salt water retting

The process is carried out with 9 kilograms of well ripen fruit where the inner nuts are removed separated into three equal portions for the above methods. It is carried out for 5 days till the bacteria react to loosen the fibers and remove the impurities.

2.3. Fiber Extraction

The retted fibers are made to dry in shade to remove excess water content and the extraction is done manually by removing the loosen fibers from the shell. After removing the fibers it is washed with excess water to make sure the extracted fiber are free from impurities, and then it is shade dryed to remove the moisture present in the fiber. Now the fiber it ready for further processing.

2.4. Fiber Treatment

Simple alkali and enzymatic treatments are selected for the study because these are mostly followed to converting the surface of the fiber and further product developments.

2.4.1. Alkali Treatment

The extracted areca fibre from the areca husk was alkali treated with potassium hydroxide (KOH) to obtain better interfacial bounding.

Recipe

- Water used: 7 litres
- Potassium hydroxide (KOH):10%
- Acetic acid: 2%
- Fibres taken: 1kg

First, the extracted areca fibre from the three modules of water were taken and the areca fibres were soaked in a solution of 10% KOH (Potassium Hydroxide) they were soaked in the solution for 24hrs in shade. It was then thoroughly washed thoroughly in water and then neutralized with a 2% Acetic Acid solution. Lastly, it was again washed in water to remove the last traces of acid sticking to it, so that the pH of the fibres is approximately 7(neutral). Then, they were dried at shade for 48 hrs to get alkali treated fibres.

2.4.2. Enzymatic Treatment
The extracted areca fibre from the areca husk was treated with pectinase enzyme to obtain better softness to the fiber.

Recipe
- Water used: 7 litres
- Phosphate buffer :0.05m
- Pectinase Enzyme: 2%
- Fibres taken: 1kg

Take the husk and immerse in the 2% enzyme solution (pectinase) with 0.05M phosphate buffer at 55 C, M: L ratio of 1:50 for 1 hour. After treatment, raise the temperature to 100°C for 10 mins to stop the enzyme action. Wash the husk in hot water followed by cold water and finally dry.

2.5. Testing and Analysing
These are the testing to analyse the effect of changes in the untreated and treated fibres. The following tests are done to analyse the fibre:
- Length
- Burning
- Scanning Electron Microscope (SEM)

2.5.1. Length
For, any structural material the length must be confirmed in order to satisfy the requirement of application. Here, the length of the areca fibre is determine and compared with the other well-known natural coir fibres. Selected areca fibre husk were used to prepare the fibre as explained in the earlier section.
The length of the areca fiber is tested manually by using the hand. First, the fibres are combed by comber by hand manually and they are places on the plates. Then it is measured by scale using the manually process. After, that it is analysed by using the mean formula.

2.5.2. Burning
Performing a burn test is a simple and relatively accurate way to identify the fiber content on which we are working on. Performing a burn test takes a bit of practice but is simple, effective and can be done easily. We will need holder and a candle and the fibre should not be taken by hand.
First, the fibre is taken with the help of a holder and then it is shown on the candle. After, that the flammability, smell and ash of the fibre are identified to analyse the difference between the treated and untreated fibre.

2.5.3. Scanning Electron Microspoce
Scanning Electron Microscopy (SEM), also known as SEM analysis or SEM microscopy, is used very effectively in microanalysis and failure analysis of solid materials. Scanning electron microscopy is performed at high magnifications, generates high-resolution images and precisely measures very small features and objects.
The treated and untreated fibres are cut according to the size of the holder and it is kept inside the Scanning Electron Microscope. Then the magnification kept for fibre is 300 and 350. Lastly the difference between longitudinal view of treated and untreated fibre are analysed.

3. Results and Discussion
3.1. Length
The length of the sample is analysed and the results are:

Table 1: Fiber length of areca husk at different stages.

	Units in Centimeters		
	Untreated	**Alkali Treated**	**Enzymatic Treated**
Stagnant Water	4.35	4.2	4.13
Running Water	4.26	4.16	4.2
Salt water	4.66	4.53	4.4

The above results show there is not big change observed in length in concern to the retting methods and the treatment given to the fiber.

3.2. Burning Test of Alkali Treated Sample
3.2.1. Stagnant Water

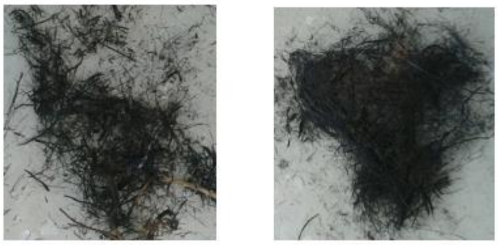


PLATE 1: A) Untreated **B)** Treated

The fibre catches flames immediately and it burns of within in 4 seconds and the ash of the treated fibre is like crusty irregular black when compared to treated fibre.

3.2.2. Running Water

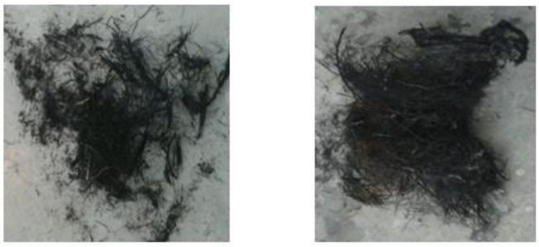


PLATE 2: A) Untreated **B)** Treated

The fibre catches flames immediately and it burns of within 3.5 seconds and the ash of treated fibre is in the cluster from when compared to untreated fibre..

3.2.3. Salt Water

PLATE 3: A) Untreated **B)** Treated

The fibre catches flames immediately and it burns of within 5.1 second and the ash of treated fibre forms a white ash in it when compared to the untreated fibre. When compared to all the fibres it takes more time to burn.

3.3. Burning Test of Enzymatic Treated Sample

3.3.1. Stagnant Water

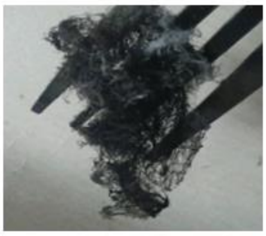


PLATE 4: A) Untreated **B)** Treated

The untreated fiber catches fire immediately and burns evenly with the flame in yellowish orange color the ash is crusty irregular black. Whereas the treated sample burns solely with a whitish black ash.

3.3.2. Running Water

PLATE 5: A) Untreated **B)** Treated

The untreated sample has the sil=milar effect as stagnant water retted sample with the treated sample shows a very slow burning with orange yellow color.

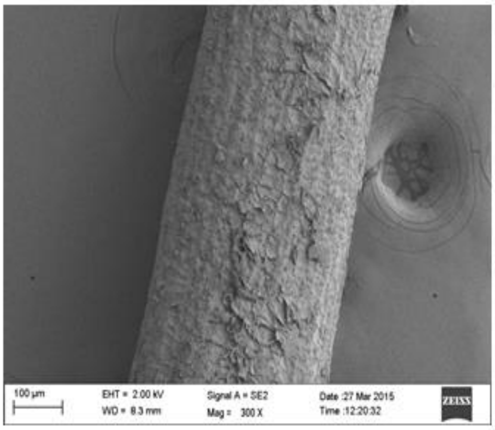
3.3.3. Salt Water

PLATE 6: A) Untreated **B)** Treated

The untreated and treated sample burns in orange flame with some sputtering effect. The residue of treated sample forms ash like threads.

3.4. Scanning Electron Microscope (SEM)

3.4.1. Alkali Treated
Stagnant Water



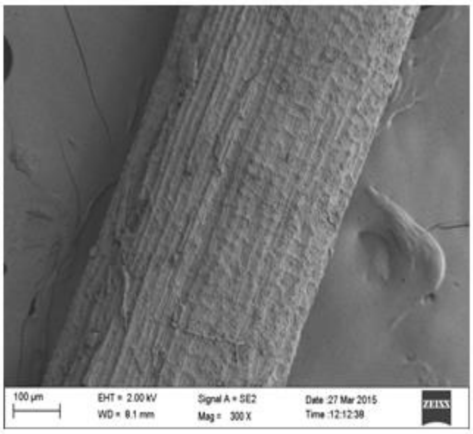
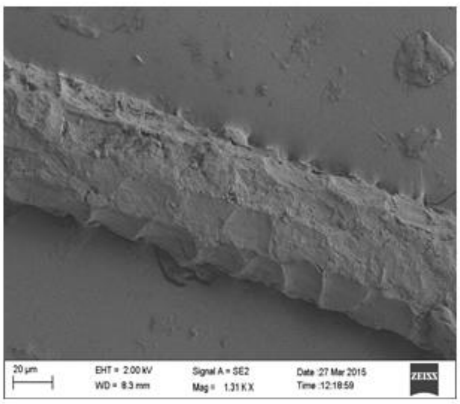


PLATE 7: A) Untreated **B)** Treated

The magnification kept to identify the fibre is 300. It is identified that the appearances of the treated distilled fibre are softer than the untreated fibre. When distilled water fibre treated with alkali it becomes soft.

Running Water



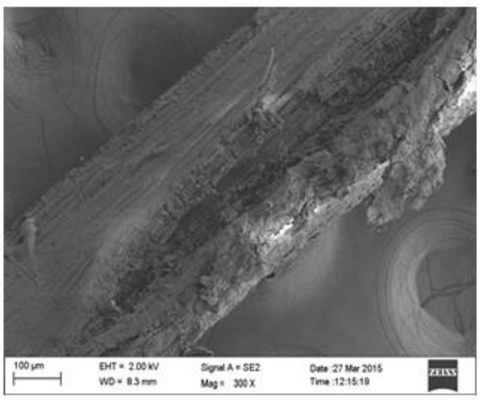


PLATE 8: A) Untreated **B**) Treated

The magnification kept to identify the fibre is 300. It is then identified that the appearances of the treated running water fibre are plead when compared to the untreated fibre. The untreated fibre gives a scaly appearance to the surface of the fibre when the running water fibre treated with alkali it pleas the surface of the fibre.

Salt Water

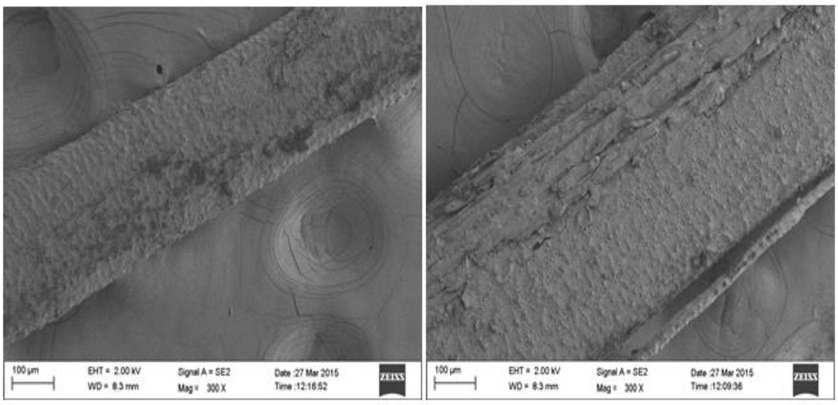


PLATE 9: A) Untreated **B)** Treated

The magnification kept to identify the fibre is 300. It is then identified that the appearance of the treated salt water fibre slightly scaly and breaks the surface of the fibre when compared to untreated fibre. When the salt water fibre treated with alkali it slightly breaks the surface of the fibre.

3.4.2. Enzymatic Treated

Stagnant Water

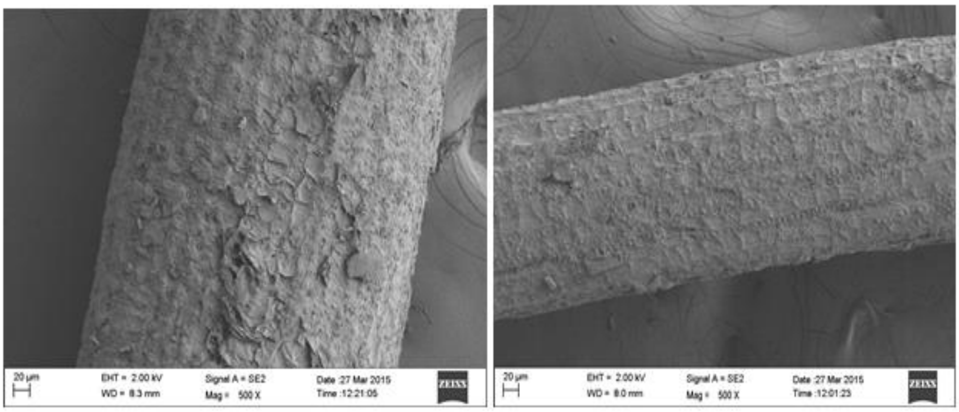


PLATE10: A) Untreated **B)** Treated

The SEM image of untreated areca fiber (A): on surface of the fiber there are some damages were formed. The SEM image of untreated areca fiber(B): on surface if the fiber compare with untreated fiber the damages were repaired due to the enzyme treatment.

Running Water

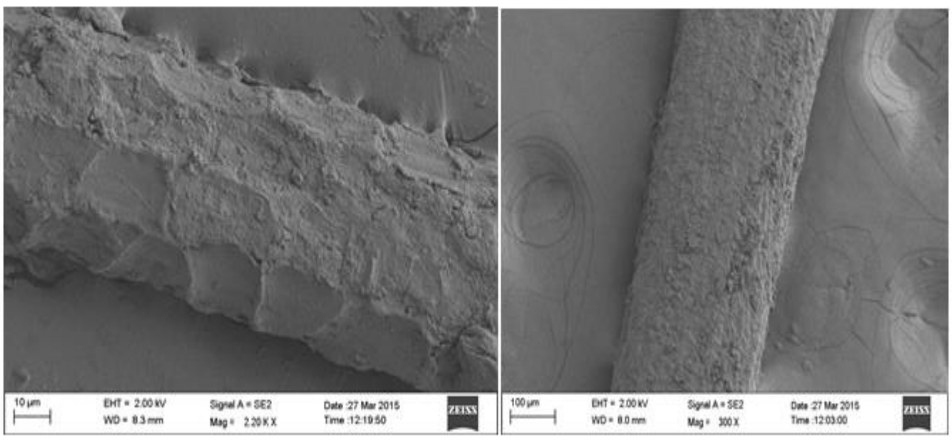


PLATE11: A) Untreated **B)** Treated

The SEM image of untreated areca fiber(A): on surface of the fiber some scaly surface are formed. The SEM image of treated areca fiber(B): on surface of the fiber were changed into cylindrical shape due to the enzyme treatment.

Salt Water

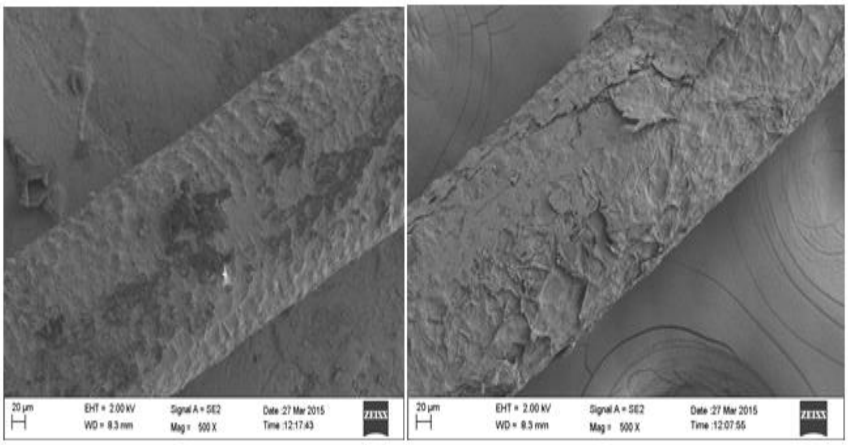


PLATE 12: A) Untreated **B**) Treated

The SEM image of untreated areca fiber (A): on surface of the fiber some black colour scratches were formed. The SEM image of treated areca fiber (B): on surface of the fiber due to the enzyme treatment the surface were damaged.

Conclusion

Based on the present investigation a broad study on retting and treatment of fiber is done. Identical samples of Arecanut were treated with alkali and enzyme under various retting conditions and the properties were studied. The retting process and the treatments given to the fiber change the surface morphology and it is proven with the help of SEM analysis. In the burning test when compared the untreated fiber with untreated fiber the ash and the colour were changed. No significant change in the fiber length between untreated and treated fiber were observed. The areca husk fiber is an good input for the textile industry with further research in the processing and product development.

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