Mechanical and Morphological Study of Arecanut Leaf Sheath (ALS), Coconut Leaf Sheath (CLS) and Coconut Stem Fiber (CSF)

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Abstract
A quantitative as well as morphological analysis of three fibers (arecanut leaf sheath, coconut leaf sheath and coconut stem fiber) is showed here. FT-IR analysis of these fibers is also done for finding the difference of functional groups in the frequency domains of 4000-650 cm⁻¹. Chemical analysis of fibers shows a comparative amount of chemical components such as aqueous extract, pectic matters, lignin, α–cellulose, hemicellulose, fatty and waxy matters in percent. Arecanut leaf sheath fiber represents the highest percent of α–cellulose. SEM analysis investigate the characteristics of the surface of the fibers.

Introduction
Agro-fibers represent a significant usage as reinforcements in composites. It has increased dramatically since the last decade of the 20th century [1]. Natural fibers have some advantages over the man-made fibers, including low cost, light weight, renewable character, high specific strength and modulus, and availability in a variety of forms throughout the world [2–4]. The development of natural fiber reinforced biodegradable polymer composites promotes the use of environmental friendly materials. The use of green materials provides alternative way to solve the problems with agriculture residues [5]. Agricultural crop residues such as arecanut leaf sheath, coconut leaf sheath, and coconut stem fiber produced in billion of tons around the world. They can be obtained in abundance, low cost, and they also renewable sources of biomass. Among this large amount of residues, only a small quantity of the residues was applied as household fuel and the major portion of the residues is unused. As a result, for using household fuel it gives a negative effect on the environment due to the air pollution [6]. The vital alternative to solve this problem is to use the agriculture residues as reinforcement in the development of polymer composites [7].

In order to enhance the mechanical and thermal properties of the agro-fiber reinforced matrix based composites it is required to know the chemical composition of the fiber. In our country, Areca catechu and Cocos nucifera trees are available in the coastal area which produces huge leaf-sheath and stem fiber. My vision is to investigate chemical composition and morphological images of the fibers which could be helped to prepare best quality of agro-fiber reinforced matrix based composites.

Experimental
Collection of fibers
Areca nut leaf sheath fibers and coconut stem fibers were prepared from arecanut leaf sheath and coconut stem respectively by soaking the leaf sheath and into water for 15 days. The water loosened the fiber from the resin and waxy materials and then the fibers peeled from the resinous materials, washed with tap water followed by distilled water and air dried properly. Coconut leaf sheath fibers occur in mat form. The leaf sheaths collected from the trees and thoroughly washed with tap water followed by distilled water and air dried properly.

Chemical analysis
The fibers were [8-9] taken uniformly and rejected the hard portion. Then the fibers were mixed thoroughly and washed with caustic soda and soap flake for 1 hour. Finally washed with distilled water and dried at 105°C in an oven. The following steps were followed for the determination of aqueous extract, fatty and waxy matters, pectic matters, lignin, α–cellulose and hemicellulose in the fibers.

The dried fibers were heated with distilled water at 60°C for 2 hours then the fibers were separated by filtration, dried at 105°C for constant weight. The loss of weight gives the amount of aqueous extract in fibers.

For the estimation of fatty and waxy matters the dried fibers were immersed in benzene-alcohol mixture (2:1 by volume) contained in a beaker into the ratio 1 gm dried fiber per 100 ml of the mixture and then allowed to stand for 10 hours with occasional stirring. After 10 hours, the fibers were separated from the mixture by filtration subsequently then washed several time with fresh benzene-alcohol mixture and finally with alcohol and the fibers were dried at 105°C for constant weight.

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weight. The loss of weight on extraction with solvent mixture gives the amount of fatty and waxy matters in the fibers.

The dried de-waxed fibers were taken in a beaker and heated with 0.5% ammonium oxalate solution in the ratio of 1 gm fiber per 100 ml of the solution at 70-80°C for three days in a heating mantle. As evaporation went on the loss of water was compensated by adding hot distilled water to keep the level of the solution constant throughout the process. The fibers were then filtered, washed thoroughly with hot distilled water then dried at 105°C for constant weight. The loss of weight gives the amount of pectic matters in fibers. The dried de-waxed and de-pectinised fibers were treated with 72% $\text{H}_2\text{SO}_4$ (15 ml for 1 gm fiber) and stirring frequently at ordinary temperature. The mixture was allowed to stand for 2 hours and diluted to 3% acid. After refluxing the mixture for 4 hours it was allowed to stand for over-night and filtered through a sintered funnel. Constant weight of the residue in the sintered funnel dried at 105°C gives the amount of lignin content of the fibers.

All non cellulosic matters of the fibers were removed by treatment of bleaching agent, such as sodium chlorite when chlorite holo-cellulose (a combination of $\alpha$-cellulose and hemicellulose) was obtained. For this purpose, the dried de-waxed and de-pectinised fibers were treated with 0.7% sodium chlorite solution buffered at pH 4 (in the ratio, 80 ml liquor for 1 gm fiber) heated at 90-95°C for 90 minutes. 1 ml buffer solution of sodium acetate and acetic acid of pH 4 was added for every 10 ml of chlorite solution to stabilize the constant pH. The fibers were then filtered, washed through with distilled water, then treated with 2% sodium meta-bisulphite solution for 15 minutes and again filtered and washed thoroughly. The cellulosic materials obtained in the sintered funnel were called the chlorite holo-cellulose which was dried at 105°C till constant weight was obtained. The dried chlorite holo-cellulose was treated with 24% KOH solution for 4 hours with occasional stirring. The ratio, 100 ml alkali solution for 1 gm fiber was followed. By this treatment hemicellulose went into solution and $\alpha$-cellulose remained unresolved. The $\alpha$-cellulose was separated by filtration, washed thoroughly with 2% acetic acid solution, finally with distilled water and dried at 105°C for constant weight. The amount of $\alpha$-cellulose thus obtained is deducted from the weight of holo-cellulose taken, gives the amount of hemicellulose.

**FTIR spectral analysis**

The three types of the fibers were cryogenically cooled and powdered separately. These powders were diluted to 1% using potassium bromide (KBr) and pellets were prepared. The FTIR spectra were recorded in the 4,000-650 cm$^{-1}$ region on a Perkin Elmer FTIR instrument.

**Scanning Electron Microscopic (SEM) Investigation**

The surfaces of the fibers were examined using a Hitachi S-4000 field emission scanning electron microscope, operated at 5 kV. Samples were mounted with carbon tape on aluminum stubs and then sputter coated with carbon tape on aluminum stubs and then sputter coated with platinum and palladium to make them conductive prior to SEM observation.

**Results and Discussion**

The chemical composition of the fibers was presented in Table 1. Arecanut leaf sheath fibers contain the highest amount of cellulose than the other fibers. Aqueous extract, fatty and waxy matters, pectic matters and hemicelluloses content of coconut stem fibers are the highest among the three fibers. These properties may enhance the strength of the stem.

**FTIR analysis**

FTIR technique was employed to find out the functional groups of the fibers. The bands at around 1734 cm$^{-1}$ and 1248 cm$^{-1}$ corresponded to hemicelluloses [10]. The bands at around 3435 cm$^{-1}$ and 2930 cm$^{-1}$ corresponded to $\alpha$-cellulose whereas the remaining bands belonged to lignin. From the (figures 1-3), it is found that in case of arecanut leaf sheath fiber the bands at around 1734 cm$^{-1}$ and 1248 cm$^{-1}$ are not present.

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**Table 1. Chemical composition of Arecanut leaf sheath (ALS), Coconut leaf sheath (CLS), and Coconut stem fiber (CSF).**

| Sl. No. | Name                      | ALS  | CLS  | CSF  |
|--------|---------------------------|------|------|------|
| 1      | Aqueous Extract           | 0.72 | 0.53 | 5.52 |
| 2      | Fatty and waxy matters    | 5.06 | 2.86 | 5.91 |
| 3      | Pectic matters            | 1.15 | 0.80 | 1.75 |
| 4      | Lignin                    | 19.59| 27.97| 22.54|
| 5      | $\alpha$-cellulose        | 66.08| 59.39| 46.26|
| 6      | Hemicellulose             | 7.40 | 8.45 | 18.02|
|        | **Total**                 | 100  | 100  | 100  |

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**Figure 1. FTIR of ALS Fiber.**
Figure 2. FTIR of CLS Fiber.

Figure 3. FTIR of CSF.

Figure 4. ALS Fiber.

Figure 5. CLS Fiber.
strongly sharp comparatively with the other fibers. It explains that the hemicelluloses content in the arecanut leaf sheath fiber is lower than the other fibers. On the other hand in case of arecanut leaf sheath fiber the bands at around 3435 cm⁻¹ and 2930 cm⁻¹ are strongly sharp comparatively with the other fibers. It showed that the α-cellulose content in the arecanut leaf sheath fiber is higher than the other fibers.

**SEM analysis**

From the analysis of (figures 4-6), it is found that the image of arecanut leaf sheath fiber is more fibrous than the other fibers which will show better mechanical properties. On the other hand some gaps are investigated in the figure 6 which is responsible for low mechanical properties.

**Conclusion**

Arecanut leaf sheath, coconut leaf sheath and coconut stem fibers were analyzed by chemical, FTIR and SEM techniques. Higher amount of hemicelluloses was found in the fibers of *Cocos nucifera* species. On the other hand the fibers of *Areca catechu* showed the highest amount of α-cellulose than the fibers of *Cocos nucifera* species. From SEM images, it was observed that the arecanut leaf sheath fibers are more fibrous than the other fibers.

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