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EFFECT OF ALKALI TREATMENT ON THE WATER ABSORPTION OF PINEAPPLE LEAF FIBER

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Abstract- To develop a high strength biocomposite material, it is highly required to make a good interfacial adhesion between the natural fiber and polymeric resin. Due to the presence of a hydroxyl (OH) and other polar groups in various constituents of cellulosic fibers, the biocomposite materials absorb moisture in humid environment condition which leads to poor interfacial adhesion, dimensional instability and loss of mechanical properties. Therefore, it becomes requisite to impart hydrophobicity in cellulosic fibers by employing an appropriate chemical treatment process. In this work, experiments were performed to analyze the effect of an alkali treatment of various concentrations (2%, 4%, 6%, 8%, and 10%) on the water absorption behavior of pineapple leaf fiber. Characterization of fibers (both untreated and alkali treated) have done by using FTIR and FE-SEM techniques. The results show that an alkali treated fibers look cleaner, and fiber bundles are more separated with a highly serrated surface due to the removal of wax, adhesives, hemicellulose, and gummy substances. Changes in the peak at 1730, 1525, and 1244 cm^{-1} in FTIR spectra corresponds to the partial removal of hemicellulose and lignin after alkaline treatment. The results indicate that the 8% NaOH treated pineapple leaf fiber absorbs maximum water in distilled and river water and fibers treated with 10% NaOH shows maximum water absorption in hand pump water. This was due to the presence of cavity, holes, and micro-pores on the surface of PALF after alkali treatment.

Keywords- Pineapple Leaf Fiber (PALF), Alkali Treatment, Water absorption, Hydrophilic nature, Natural fiber

I. INTRODUCTION

Lignocellulosic materials such as agricultural waste, wood, grasses, and plant waste fibers are extensively used in textile, paper manufacturing, and packaging industries since they exhibit low cost; lightweight; good electrical, thermal, chemical, and acoustic properties; and high resistance to fracture. Among different types of plant fiber, the hard cellulosic fibers such as jute, flax, banana, and pineapple leaf are the first choice of engineers and technologists for making biocomposites because of their unique chemical composition, structure, and good mechanical properties such as high tensile strength & modulus. Pineapple leaf fiber appears to be a promising material because it is abundantly available and inexpensive. Pineapple (*Ananas comosus*) is abundantly grown in Thailand, Philippines, Costa Rica, China, and India. The total production in the world is around 19412.91 thousand tons (Indian horticulture database-2015). Commercially, pineapple fruit is very important and its leaves can be used for obtaining cellulosic fibers. Pineapple leaf fiber has a significant potential to supplant expensive and non-renewable synthetic fibers. In the tropical region, pineapple plant (fibrous plant) is copiously available. The chemical composition of pineapple leaf fiber (PALF) is reported in Table 1. Due to the presence of high cellulosic content in PALF, it has marvelous mechanical properties and can be exploited as reinforcement in polymeric materials. Instead of the wide varieties of advantageous properties, it shows some problems such as low thermal resistance, poor adhesion with synthetic polymers, and hydrophilic nature which leads to prevent their use at large scale (Srinivasa *et al.* 2012) [1]. The hydrophilic characteristics of pineapple leaf fiber results in ineffective stress transfer from the matrix to fiber, poor dimensional stability, development of internal stress, plasticization of the fiber, and the decrease of Young's modulus. Lee *et al.* (2010) studied the hygroscopic deformation of natural fibers by AFM. It was found that there is a delay time of swelling after water uptake [2]. A lot of researches have already conducted to reduce the hydrophilicity of natural fibers by chemical treatments (permanganate, peroxide, maleic anhydride, sodium hydroxide, and organosilanes) but very few works have done on the alkali treatment of pineapple leaf fiber. Among the methods, an alkaline treatment of natural fiber is the most economical, simplest, and easiest technique to ameliorate the thermal stability, dimensional stability, and mechanical properties. Moreover, alkali treatment roughens the fiber surface which leads to a better interfacial adhesion between fiber and matrix. Rong *et al.* (2001) reported that the alkaline treatment removes hemicellulose and lignin component from natural fiber surface which results in the production of better-purified cellulose [3]. From the past studies, we can conclude that the effect of alkali treatment on water absorption of pineapple leaf fiber has not been addressed so far. Therefore in this work, we have investigated the effect of alkali treatment on surface morphology, surface chemistry, and water absorption of pineapple leaf fiber.

Table 1 Chemical composition of pineapple leaf fiber

Fiber	Cellulose %	Hemicellulose %	Lignin %	Pectin %
PALF	70-82	18.8	5-12.7	1.1

A. Moisture Sorption Mechanism

The moisture sorption by natural fibers could be due to both diffusion phenomena and capillary action. According to Carter *et al.* (1978), the absorbed moisture consists of both mobile and bound phases. Diffusion of water molecules in cellulosic materials depends on the diffusion coefficient D_γ and probability of water absorbed (bound) per unit time γ at certain sites such as void within the polymer [4]. Many authors proposed a classical Fick model to delineate diffusion kinetics of water in cellulosic material (Gouanve *et al.* 2007) [5]. Recently, Hill and Xie *et al.* (2011) reported the Parallel Exponential Kinetics model (PEK) to analyze the absorption and desorption curves of different cellulosic fibers [6]. Robert Kohler *et al.* (2006) studied the sorption and desorption isotherm of viscose and scotched flax by PEK model. They found that the viscose has higher water uptake capacity than the scotched flax [7].

The PEK model equation for the mass gain is

$$M_t = M_{\infty 1}\left(1 - e^{-1/\tau_1}\right) + M_{\infty 2}(1 - e^{-1/\tau_2})$$

Where $M_t =$ mass at any time t, M_∞= mass at equilibrium and τ = time constant

According to Langmuir's model, the natural fibers absorb water onto the specific sites; i.e. hydroxyl function of hemicellulose and carboxylic function of pectin by hydrogen bonding. At high relative humidity, the water molecules yoke together to form clusters which result in the increase of water concentration by a power function (Celino *et al.* 2014) [8].

II. EXPERIMENTAL

A. Materials

Raw pineapple leaf fiber (R-PALF) was purchased from M/s Go Green Products, Chennai, India. The average density and moisture content of PALF was equal to 0.98 g/cm^3 and 10-11% respectively. A sodium hydroxide (NaOH) was used for alkaline treatment and obtained from the local supplier.

B. Fiber Treatment

Initially, the pineapple leaf fibers were washed in tap water and dried in sunlight for three days to remove the surface impurities and absorbed water molecules. The cleaned and dried fibers were immersed in various concentrations (2%, 4%, 6%, 8% and 10 wt%) of alkali (NaOH) solution for 24 hr at room temperature, followed by the treatment with 2 wt% acetic acid (CH_3COOH) solution and then rinsed in distilled water to control the pH level at 7. The neutralized wet fibers were dried again at room temperature and then followed by oven drying at 70^{0}C for 24 hr (until the constant weight was obtained).

C. Method

1) Fourier-Transform Infrared Spectroscopy (FTIR): Fourier-transform infrared spectroscopy (model Perkin Elmer 2000) was used to analyze the effect of alkali treatment of different concentrations (2%, 4%, 6%, 8%, and 10 wt%) on chemical bonding exist in PALF. FTIR spectra were analyzed with an infrared spectrophotometer in the range 4000 cm^{-1} to 400 cm^{-1}. FTIR of PALF had done in Analytical Instrumentation Laboratory, CSIR-CSIO (Chandigarh).

2) Morphological Study: The morphology of untreated and alkali treated pineapple leaf fibers were studied by using a scanning electron microscope (FE-SEM, Hitachi 4300) at the accelerating voltage of 10 kV in Analytical Instrumentation Laboratory, CSIR-CSIO (Chandigarh).

3) Water Absorption Measurement: Water absorption measurements on untreated and alkali-treated fibers were done in various water samples such as distilled water, river water, and hand-pump water. The pH of water samples was distilled water: 7.0, river water: 7.67, hand-pump water: 7.42. The samples were prepared from a bundle of individual fibers (4g) bound together and kept inside the beaker containing a various source of water at room temperature (25^{0}C). The weight of the fibers was measured after every 24 hr and the moisture content was calculated by weight difference,

$$\mathbf{MC} = \frac{m_a - m_d}{m_a} \times 100$$

Where **MC** is the moisture content, m_a is the mass of the sample after exposure to moisture, and m_d is the mass of the dry sample.

III. RESULTS AND DISCUSSION

D. Fourier-Transform Infrared Spectroscopy (FTIR)

Figure 1 shows the IR spectra of untreated and alkali treated pineapple leaf fiber (PALF) that was obtained by using an FTIR spectrometer. The peak at 3332 cm^{-1} is linked to -OH stretching vibration present in amorphous and crystalline cellulose. The carbonyl stretching (C=O) peak (1730 cm^{-1}) which was related to the hemicellulose component can be seen in untreated PALF but not present in alkali treated pineapple leaf fibers. This may be due to the removal of hemicellulose component after alkali treatment. The CH_2 bending peaks at 1432 cm^{-1} are present in both untreated and alkali treated fibers. The small peak at 1525 cm^{-1} is related to the lignin component. This peak is not present in alkali treated samples. It could be due to the partial removal of lignin after alkali treatment. It can be observed that the peak at 662 cm^{-1} (corresponds to COOH bending) are present in both untreated and alkali treated fibers. An alkali treatment removes wax, adhesives, hemicellulose, and gummy substances that bind elementary fibers in plant fiber yarns. The peak at 1244 cm^{-1} is much smaller in alkali treated PALF than the untreated one. This peak corresponds to C=O stretch of the acetyl group of lignin.

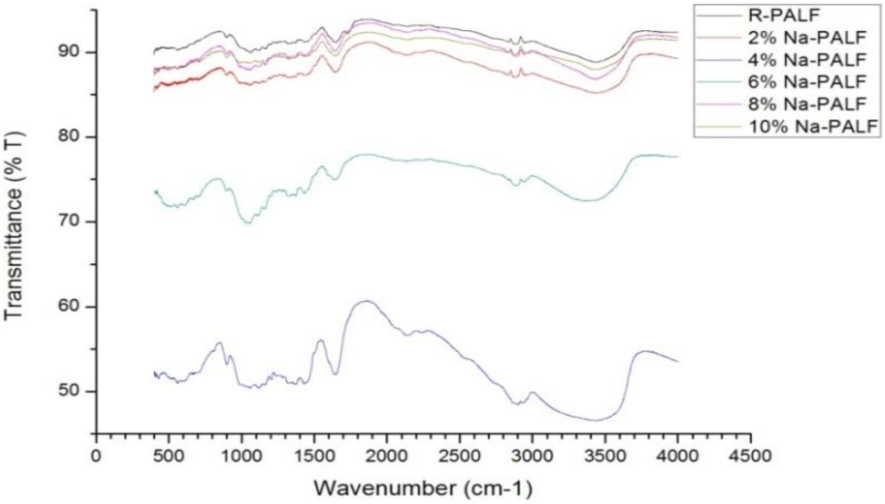

Figure 1 IR spectra of untreated and alkali treated pineapple leaf fiber

E. Fiber Surface Morphology: SEM Analysis

The SEM micrographs of untreated and alkali treated pineapple leaf fiber are shown in Figures 2 and 3. It can be observed from Figure 2 that the untreated pineapple leaf fiber (PALF) has a network structure in which the microfibrils are bound together by amorphous cellulose, hemicellulose, and lignin. The SEM of untreated PALF clearly depicts the presence of longitudinally oriented unit cells. These unit cells are held together due to the presence of binder lignin and waxy substance in between the intercellular space. N. Sgriccia *et al.* (2008) also reported that the intercellular space in hemp fiber is filled up by fatty substance and hemicellulose [9]. The surface morphology of alkaline treated PALF shows the presence of cavity and micro-pores which are due to the removal of binding material, fatty deposits, and cuticle in the surface. Bismarck *et al.* (2001) studied the effect of alkaline treatment on surface morphology of cellulosic fibers. They found that fibrillation increased and cracks were developing in the fibers due to the removal of binding material [10]. The alkali-treatment of pineapple leaf fiber results higher degree of roughness. This was because of partial removal of hemicellulose.

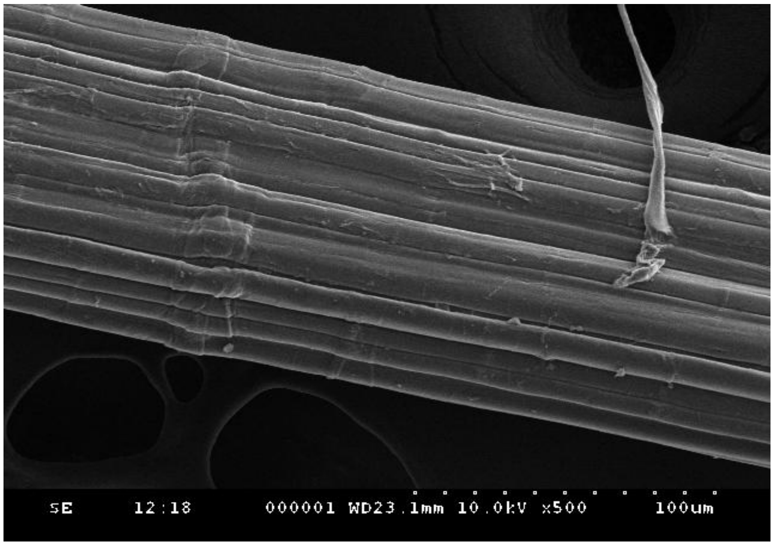

Figure 2 SEM micrograph of untreated pineapple leaf fiber

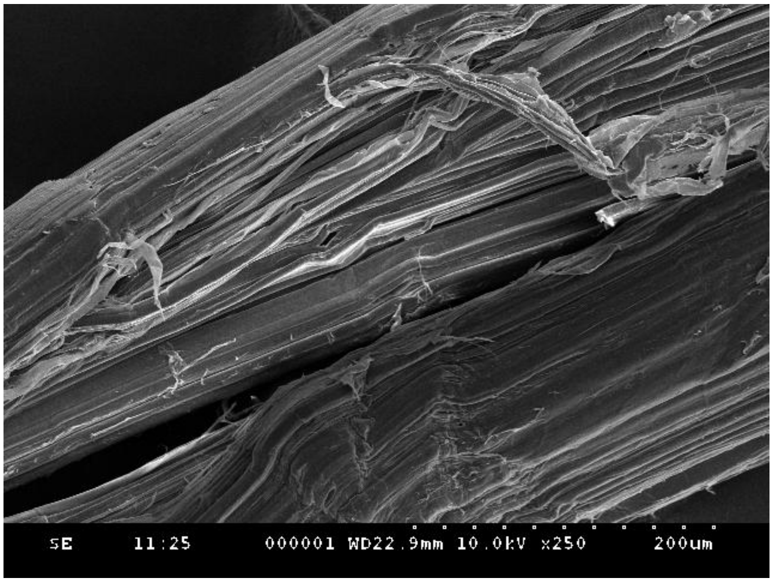

Figure 3 SEM micrograph of 8% alkali treated pineapple leaf fiber

F. Water Absorption

The water absorption of PALF was done by immersing them in three different water samples (distilled, river, and hand pump water). After every 24 hr, the fibers were taken out and the excess water was mopped with filter paper before weighing. Four replicates were tested and their average value was considered as a result. The experiment results showed that the untreated PALF absorbs more water in distilled water as compared to the other water samples. The water sorption is primarily due to the presence of hemicellulose, non-crystalline cellulose, and lignin. Bessadok *et al.* (2007) reported that when water concentration exceeds threshold limit, a significant swelling of natural fibers takes place. This was due to the linkage of water molecules to the network of the polymer via hydrogen bonds [11]. The maximum water absorption by untreated PALF was found to be 69.56%, 67.45%, and 65.62% in the distilled, river, and hand-pump water respectively. Sampathkumar Dhanlakshmi *et al.* (2012) studied the water absorption behavior of untreated areca fiber and concluded that they absorb maximum water (78.5%) in the borewell water sample. It was due to the presence of hemicellulose and hollow cavity (lumen) which provides an easy pathway for water diffusion into the fiber [12].

1) Effect of Alkali Treatment on Water Absorption: Figure 4 illustrates the effect of alkali treatment on water absorption of pineapple leaf fiber. The alkaline treatment has improved the sorption ability of pineapple leaf fiber. This result was due to the removal of protective waxy and gummy substances at the fiber surface. Alexander *et al.* (2001) also reported the de-waxing and higher accessibility of active functional group in sisal fiber surface after an alkali treatment [10]. Hatakeyama *et al.* (1998) studied the interaction of water with hydrophilic polymers and found that the amount of bound and free water in fiber depends mainly on the chemical structure of the material [13]. It was observed that alkaline treatment leads to the removal of hydrophobic compounds and uncovering the fiber fibrils which results in an increment in the specific area. Rong *et al.* (2001) stated the partial dissolution of hemicellulose and accessibility of hydroxyl and carbonyl groups in the pectin component of fiber after an alkaline treatment [4]. The 8% and 10% NaOH fiber revealed the maximum water absorption in distilled and hand pump water respectively. This improvement may be attributed due to the change in surface morphology of pineapple leaf fiber after an alkaline treatment (Figure 3).

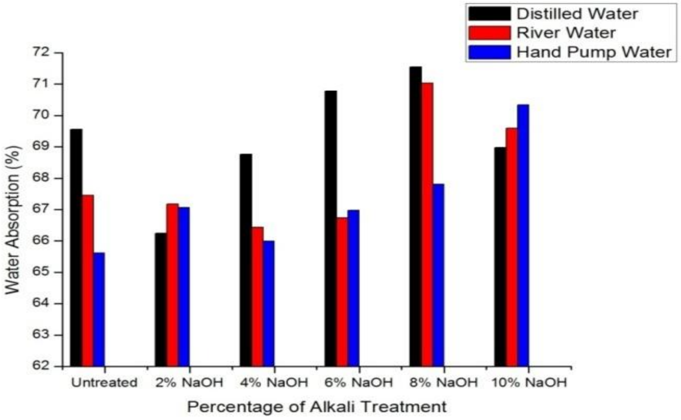

Figure 4 Effect of alkali-treatment on water absorption of pineapple leaf fiber

Figures 5 to 7 show the effect of immersion time **t**, on water absorption of untreated and alkaline treated pineapple leaf fibers. All samples of fiber absorb water initially with immersion time **t** up to saturation level. This absorption is mainly due to both diffusion and capillary action through the existing voids in the structure which leads to a significant swelling of the material. Celino *et al.* (2014) described that heterogeneous morphologies, hydrogen bonding, and voids within the polymer are certain sites for diffusion of water [8]. When the immersion time becomes significant, a decrease in water absorption is due to the hydroelastic characteristic of natural fibers.

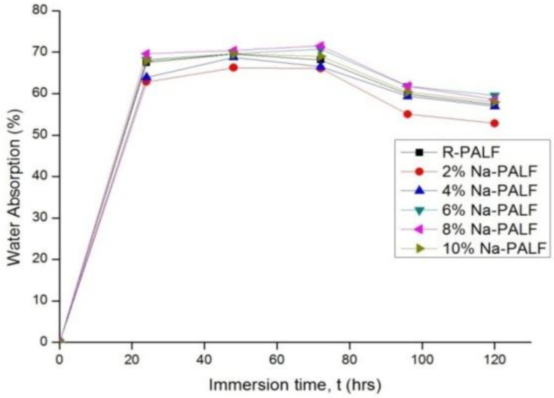

Figure 5 Effect of immersion time on water absorption of untreated and alkali-treated PALF in distilled water

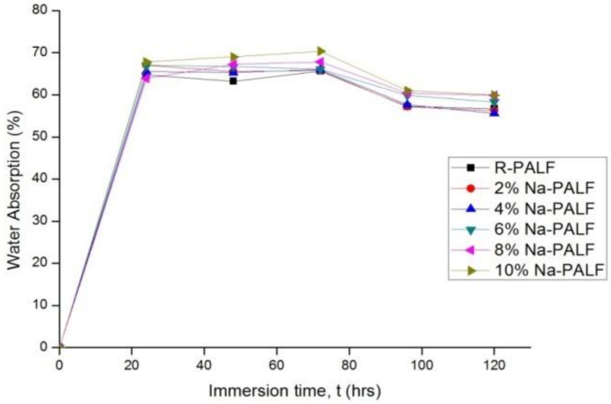

Figure 6 Effect of immersion time on water absorption of untreated and alkali-treated PALF in hand pump water

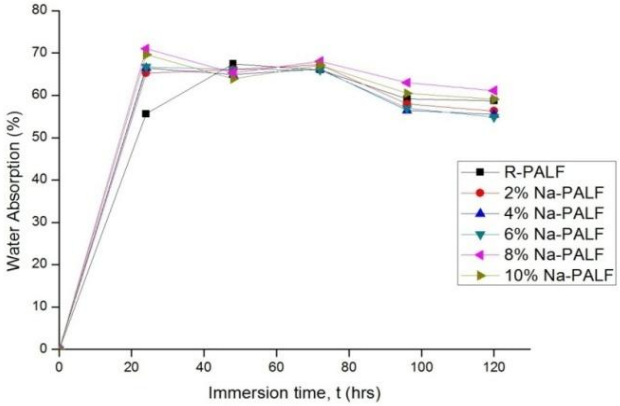

Figure 7 Effect of immersion time on water absorption of untreated and alkali-treated PALF in river water

IV. **CONCLUSIONS**

Experiments were conducted to study the water absorption behavior of pineapple leaf fiber and to analyze the effect of alkaline treatment on the moisture sorption of PALF. From the above study, it can be concluded that the maximum water absorption of untreated pineapple leaf fiber was 69.56% in distilled water. It was due to the presence of hemicellulose and hollow cavity (lumen) which provides an easy pathway for water diffusion into the fiber. It was observed that the alkali treated pineapple leaf fibers exhibit higher water absorption than the untreated fiber sample. This was due to the presence of holes, cavity, and micro-pores on the fiber surface after an alkaline treatment of PALF. The 8% NaOH pineapple leaf fiber revealed the maximum water absorption in distilled water. It was due to the removal of protecting waxy and gummy substance at the fiber surface.

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