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Research paper

The Physical, Chemical Properties of untreated and chemically treated Palmyra Palm Leaf Fibres

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Abstract

Palmyra palm leaf fibre a natural fibre extracted from palmyra palm tree was treated with sodium hydroxide, benzoylchloride and potassium permanganate. Effects of these treatments on the changes in physical, chemical properties were determined experimentally. The density of the fibres was found to increase, while the diameter of fibres reduced due to treatments. The morphology of untreated and treated fibres were examined using Scanning electron microscope.

Keywords: *Natural fibre; pre-treatments; physical properties; chemical properties; SEM*

1. Introduction

Natural fibres are used as reinforcements in plastics because of their high specific strength, resistance to impact, modulus and stiffness [21]. Most of the natural fibres are renewable, biodegradable, carbon dioxide neutral, also the total energy involved in producing natural fibre is only 2% in comparison to production of glass fibres [18] [25]. [11] Reviewed bio fibres and bio composites and stated that bio based composites are finding wide range of applications due to the reason that the specific properties are comparable with synthetic fibres like glass fibres. Enviornment consciousness among end users and society at large has accelerated the application of natural fibre reinforced composites. Natural fibres possess properties that are comparable with synthetic fibres but the hydrophilic nature of natural fibres limits their use. Hydrophilicity results in increased moisture absorption which induces swelling of fibres and degradation of fibres. This results in poor fibre matrix adhesion which is considered to be a common failure in composites. Pre-treatment on natural fibres is an alternative method to overcome the above limitation. Pre-treatments result in reducing the hydrophilicity of fibres, improved wetting of matrix at the fibre surface and improved mechanical properties [22].

Palmyra palm (Borassus) is a genus of six species of fan palms. It is grown in tropical regions and is a native of Africa and Asia. One of the spices of palmyra palm is Borassus flabefllier which is grown in southern parts of Tamil Nadu, Asia. The tree has a fan shaped leaf with a stem called leaf stalks which is approximately 1m in length. The leaf stalks have fibre which is suitable for manufacturing ropes, broom sticks, painting brushes. Only few studies on the properties like chemical contents, mechanical, thermal properties of Palmyra fibre and composites fabricated by using Palmyra fibre are found in literatures. In the current investigation, the procedure for extraction of the Palmyra fibre and the effect of treating the fibres with alkali (Mercerization), benzoylchloride and potassium permanganate on the changes in chemical and physical properties are reported.

2. Pre-treatment methods

Mercerization (Alkali treatment)

[22] Investigated the mechanical properties and moisture absorption behaviour of sisal fibres before and after alkali treatment. It was found by them that the mechanical properties of the composites improved and moisture absorption reduced than in the case of untreated fibre composites. It was found after treatment, there was a decline in the tensile strength, but tensile and flexural sterength of the composites increased. This is due to the improved fibre -matrix adhesion [22] [25]. [21] Experimented with kenaf, flax and hemp fibres for their changes in morphology of the surface after alkali treatment and they found that natural fibres tend to absorb more moisture when compared to glass fibres. According to [1] the hybridization of sisal and roselle fibres after alkali treatment had the improved mechanical properties of the composites. Treatment duration of the fibres with NaOH affected various mechanical properties of the composites. It was also reported by them that water absorption characteristics of the composites changed with fibre weight percentage and fibre length. The flexural properties of alfa fibres treated with alkali - polyester composites was found to have enhanced in comparison to untreated afla fibre composites, due to good fibre/matrix adhesion [15]. The effect of treating date palm fibres with alkali under different processing conditions on their properties were studied using Single fibre tensile test, TGA , FTIR and SEM and have reported that concentration of NaOH for treatments needs to be optimized for achieving enhancement of properties [4] [16]. [20] studied the young's modulus and tensile strength of sisal fibre after alkali treatment. They have reported that alkalization modified the internal structure of sisal fibres and found that the specific stiffness that was almost same as that of steel. Various surface treatments like alkaline, admicellar, aetylation, silane treatment, and preimpergnation with polyethylene solution

resulted in the improvement of strength due to increase in mechanical interlocking [13] [11] [20] [7] [27] and [2].

Benzoylchloride treatment

Benzoyl chloride is used to decrease the hydrophilicity of the fibre [24]. Benzoylchloride treatments leads to the formation of benzoyl rings on the cellulose structure which decreased the hydrophilicity of the fibres and improved fibre and matrix adhesion [13]. Sisal fibres were subjected to benzoylchloride treatment and the composites fabricated out of benzoylchloride treated fibres exhibited improved mechanical properties [22]. [14] investigated the thermal stability of sisal fibres after benzoylchloride treatment and reported improved properties in comparison to untreated fibres composites. Improved interfacial adhesion was observed for composites fabricated out of benzoylchloride treated flax fibres which had resulted in enhanced mechanical properties. Similar improvements in mechanical properties were observed for palmyra fibres and sansevieria fibres which were treated by benzolychloride [26] [23].

Potassium permanganate treatment

Hydrophilicity of plant fibres can be reduced by treating with potassium permanganate treatment [13]. [22] Studied the effect of permanganate treatment on mechanical and moisture absorption characteristics of sisal - polyester composites. Their results indicated that there was reduction in moisture absorption and increase in the mechanical properties and after permanganate treatment. Similar results were observed for permanganate treated PPLSF/polyester composite [26]. Similar results were reported by [23], when sansevieria fibres were treated with potassium permanganate. Potassium permanganate treatment results in uneven surface on the fibres due to which fibre- matrix adhesion improved [18].

3. Materials and Methods

Palmyra Fibres (PPLSF) were extracted from the leaf base of plamyra palm tree. The thorns at the edges and skin of the leaf base were removed. These leaf base was then immersed in water for 40 days followed by soft hammering with a wooden hammer. The fibres in the stalks and the waxy substance sticking to the fibre surface are removed manually. The fibres are then washed with water to remove any impurities followed by drying them in sun light in order to remove the moisture in the fibre. The cleaned fibres were then subjected to pre-treatments as discussed in the following sections. Chemicals for treatments such as sodium hydroxide (NaOH), benzoylchloride, acetone, ethanol and potassium permanganate ($KMnO_4$) of AR grade were obtained from acclaimed suppliers in the region and the chemicals were used as such.

Pre treatments on natural fibres

Alkali, Benzoylchloride and permanganate treatment were carried out according to the procedure stated in [26].

Physiacl, chemical testing of fibres

Fibre diameter distribution

A sample of 50 fibres were randomly selected and the diameter measured at the top, bottom and middle sections using air wedge method and the average diameter of fibre was calculated.
Chemical contents in the fibre
Chemical contents are estimated at South Indian Textile Research Association (SITRA), Coimbatore, India using the procedures stated by [16].

FTIR spectroscopy

FTIR spectra of the raw and treated PPLSF were recorded using Perkin Elmer Spectrum RXI FTIR spectrometer in a KBr matrix with a scan rate of 32 scans per min and a resolution of 2 cm^{-1} in the wave number region from 400–4000 cm^{-1}. The fibres were chopped into powders, mixed with KBr and pelletised for FTIR spectra analysis.

Surface morphology analysis

The surfaces morphology of the untreated and treated PPLSF was examined by using Scanning electron microscope (SEM) JEOL JSM 6390 model at an accelerating voltage of 10KV.

4. Results and Discussions

Fibre diameter distribution

Natural fibres do not have same diameter throughout its length. The length of PPLSF was found to vary between 1m to 1.5m. The diameter and length of natural fibres varies due to varying soil condition, availability of water and age of the plant [16]. The average diameter of untreated PPLSF was found to be 320 μm with standard deviation of ± 16 μm. The diameter of the PPLSF was found to vary between 190 μm to 240 μm. The removal of lignin, wax and other cementing substances available on the fibre surfaces resulted in the decreased diameter of fibres after treatments.

Chemical contents of untreated and treated fibres:

The chemical tests showed that the fibres have lignin, cellulose, hemicellouse, wax etc. The presence of hemicelluloses and amorphous domains of the cellulose are bound to absorb moisture because of their hydrophilic nature [11] and it is likely that their removal will greatly reduce the moisture absorption.
The chemical constituents of untreated and treated PPLSF are shown in Table 1. It can be noted that hemicellouse was completely removed and all the other constituents reduced partially after alkali, benzoylchloride and permanganate treatment. [3] reported that the alkali treatment resulted in removal of hemicelluloses, lignin, wax and oil on the fibre surface due to the ionization of hydroxyl groups, which will result in depolymerization of cellulose and exposing of crystallites. The fibre -OH ($-CH_2OH$) reacts with NaOH, which is a strong alkali and will produce alkoxide ion ($-CH_2O^- Na^+$) resulting in the reduction of chemical contents. The possible reaction during alkali treatment is shown in equation 1.

$$\text{Fiber} - OH + NaOH \longrightarrow \text{Fiber } O^- Na^+ + H_2O \quad (1)$$

During benzoylchloride treatment, the alkoxide ion ($-CH_2O^- Na^+$) produced during alkali treatment reacts with benzoylchloride and will result in ester formation. The possible reaction mechanism during benzoylchloride treatments is shown in equation 2. In the case of permanganate treatment, the permanganate group MnO^{-4} leads to the formation of cellulose radical by formation of MnO^{-3} ion, these highly reactive permanganate ions are responsible for initiating graft co-polymerization [12]. The reaction during permanganate treatments is shown in equation 3 .

$$\text{Fiber } O^- Na^+ + Cl - \overset{\overset{\displaystyle O}{\|}}{C} - Ph \rightarrow \text{Fiber} - O - \overset{\overset{\displaystyle O}{\|}}{C} - Ph + NaCl \quad (2)$$

$$\text{Cellulose} - H + KMnO_4 \rightarrow \text{Cellulose} - H - O - \underset{\underset{\displaystyle O}{\|}}{\overset{\overset{\displaystyle O}{\|}}{Mn}} - OK^+ \quad (3)$$

Alkali treatment is performed before benzoylchloride and permanganate treatment, in order to remove the extractable impurities like hemcielluose, lignin and wax and also to change the orientation of cellulose further exposing the hydroxyl (OH) groups which may in turn react with benzoyl and potassium permanganate [17]. The highest reduction in cellulose content was 10% which was observed for alkali treated fibres and the highest reduction in lignin and wax was found to be 33% and 68% respectively due to permanganate treatment on fibres in comparison to untreated fibres. Chemical treatments also had an influence of the weight and density of fibres. The highest weight loss (29%) was found for benzoylchloride treated fibres while permanganate and alkali treatments resulted in 26% and 23% reduction in weight of fibres (Figure 1). Benzoylchloride has no restriction to react with Fibre O-Na+ which resulted in ester formation, due to which molecular weight increases and resulted in increased fibre density after treatment. Oxidization of –OH groups of the fibre in the case of permanganate treatment and formation of alkoxide ion ($-CH_2O^-Na^+$) due to alkali treatment resulted in increased density of fibre.

Table 1: Chemical contents of the untreated and treated Palmyra fibres

	Cellulose %	Hemi cellulose %	Lignin %	Wax %	Density (g/cc)
Untreated	58.58	22.8	13.48	0.53	0.456
Alkali Treated	52.56	Nil	10.23	0.16	1.152
Benzoyl chloride treated	52.55	Nil	10.36	0.15	1.194
Permanganate treated	51.98	Nil	8.92	0.16	1.186

Table 2: Comparison of Chemical and physical properties of natural fibres with Palmyra fibres

	Cellulose	Hemi Cellulose	Lignin	Wax	density	Dia-meter
PPLSF	58.58	22.8	13.48	0.35	0.456	320
Jute	58.63	-	12-14	--	1.3	10-40
Sisal	66-78	10-14	10-14	0.3	1.5	50-200
Cotton	82.7	5.7	-	0.6	1.6	--
Flax	64.1	16.7	2	1.5	1.5	--
Ramie	68.6	13.1	0.6	0.3	1.5	--
Coir	--	16.8	32.1	--	1.2	100-450

FTIR Analysis

The effects of chemical treatments on the fibres were analyzed using FTIR. Figure 2 shows the FTIR peaks of untreated, alkali, benzoyl chloride and potassium permanganate treated fibres.

The absorption band around 3500 cm^{-1} could be due to the hydrogen bonded O–H stretching and it is available in all the fibres [4]. This band is available at around 3400 cm^{-1} and for the treated fibres this band was found to have slight shift and variation in intensity. This indicates the reduced hydrogen bonding in cellulosic hydroxyl groups resulting in reduced hydrophilic nature and exposing the –OH groups to interact with matrix during reinforcement [12]. The thealiphaic C -H stretch at 2900cm^{-1} [21], [19] could be seen in all the fibres both untreated and treated and it is found to decrease after chemical treatments. The carbonyl peak around 1730 cm^{-1} could be attributed to the C=O stretching of the acetyl groups of the hemicelluloses and this could be observed in the untreated fibre and is not present in all the other fibres which have underwent treatment, this confirms that the hemicelluloses were removed during the treatments [4]. A small shift in the peak observed around 1632 cm^{-1} with reference to 1730cm^{-1} is not evident in alkali, benzoylchloride and permanganate treated fibre compared to untreated fibre .This peak represented the C=O bonds on hemicelluloses and was further evident that hemicelluloses were removed from fibre surfaces due to alkali treatment. The intensity observed around about 1375 cm^{-1} band is attributed to bending vibration of CH_3 group. The peak at 1263 cm^{-1} corresponds to C=0 and C-O stretching of lignin. In the case of alkali, benzoyl and permanganate treated fibres a small shift in band could be observed, also the intensity reduced indicating the removal of lignin form the fibres after treatments [21].

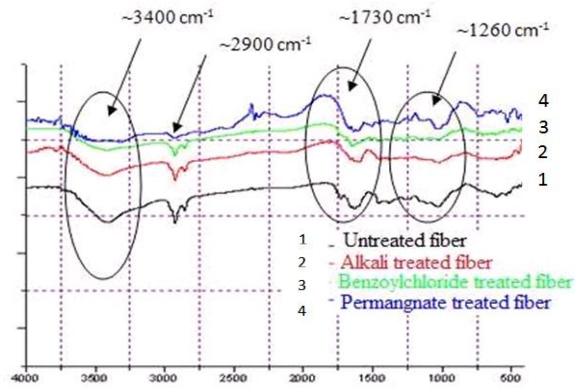

Fig.1: FTIR spectra of untreated and treated fibres.

5. Conclusion

PPLSF extracted from the leaf stalks of palmyra palm tree were subjected to alkali, benzoylation and permanganate pre-treatments. The changes in chemical, physical, mechanical and morphology of the untreated and treated fibres were analyzed and following were concluded.

1. Diameter of fibres is not found to be uniform throughout the length of the fibre. Untreated fibres had an average diameter of 320µm. The diameter of fibres were found to reduce due to treatments by 25%, 34% and 41% for alkali, benzoyl and permanganate treated fibre respectively in comparison to untreated fibre.
2. Chemical analysis ndicated the removal of hemicelluloses and partial removal of cellulose, lignin and wax.

The density of fibres increased due to chemical pre-treatments. Highest density was observed in the case of benzoylchloride treatment compared to other treatments.

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