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The Influence of Alkali Treatment on the Chemical, Crystalline, and Thermal Characterization of Burmese Silk Orchid Fiber

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ABSTRACT

The chemical properties of the fiber have an important role in predicting composite performance. So, the objective of this research was to extract Burmese silk orchid naturalfibers and determine the influence of alkali treatment on chemical, thermal and crystalline properties. Standard testing procedures were employed to determine the chemical attributes of Burmese silk orchid fiber, such as wax and ash content, and the results were compared to those of other plant fibers. In this research, Burmese silk orchid fibers were treated with 5% and 10% alkali (NaOH) solutions, and their effect on fiber characteristics was examined. Alkali-treated fibers have a higher content of ash (3.69%) and lower amount of wax (0.43%) as compared to raw Burmese silk orchid fiber. Thermo-gravimetric analysis (TGA) was used to evaluate the thermal stability of fiber. The percentage of crystallinity and crystalline index of the fiber was determined using X-ray Diffraction Test (XRD). Finally, the chemical characteristics, TGA, and XRD characterization of Burmese silk orchid fiber significantly support its use as a reinforcing material in polymer composites. The morphology and impurities of the fiber were examined using a Scanning Electron Microscope (SEM). The novelty of this work was to investigate the characteristics of natural fiber materials for sustainable development, as well as their usage as reinforcement with resins in the fabrication of composite materials for relevant applications.

Keywords: Chemical properties, Burmese silk orchid fiber, TGA, XRD, Alkali treatment.

INTRODUCTION

Many nations have put strong rules and regulations in place to minimize solid waste in material producing industries like construction, packing, and automobiles in order to safeguard the environment. Bio-renewability, light weight, environmental friendliness, plentiful availability, affordable price, and moderate strength make natural fibers a viable substitute for synthetic and conventional materials[1]. Usage of natural fibers would help in lessening of pollution-concerns such as solid waste dumping, land and marine filling, toxic waste, and greenhouse gas emissions[2]. Due to their distinct

chemical structures, these fibers have drawbacks including hydrophilicity, rapid moisture absorption, and difficulty in being compatible with polymers. To achieve appropriate use, issues can be addressed using different fiber surface modifications and chemical treatment techniques. Various researchers studied the Physical and chemical properties of newly identified fibers such as Balanites aegyptiaca, Careya Arborea, kapok fibre, Burmese silk orchid fiber, and Himalayacalamus falconeri culms[3-7].

The word "natural fibers" describes a type of fiber produced by geological processes and derived from various plant or animal organs. These natural fibers possess threads like appearance and are long, thin, and flexible enough to take on whatever shape is needed. These can be spun into yarns or threads, which can subsequently be woven or stitched into fabrics[8]. Fibers are harvested from various parts of the plant; including (i) bast fibers are collected from the stem, (ii) leaf fibers from the peduncle, (iii) wood fiber from the trunk, and (iv) fruit fiber from the fruit of the plant. Recently, authors used bark fibers such as Hibiscus, jute, flax, cannabis, ramie, and kenaf for their study[35]. Plant fibers primarily consist of cellulose, hemi-cellulose, lignin, and wax[9]. Plant fibers are classified as lignocellulosic fibers because they contain a higher percentage of cellulose as well as lignin. These elements aid in the growth of the plant[10,11]. Several authors have recently identified the new plant fibers and investigated their chemical components[36]. Nesrine *et al.,*[12] conducted a detailed examination on the chemical properties of *Pergularia Tomentosa* L fiber extracted from the seed of the plant. In contrast to other fibers, these fibers have 2.74% ash content and 1.88% wax content.

The hydrophilicity of natural fibers is the primary disadvantage when utilizing them in polymer composites. Natural fibers take in moisture or water from the surroundings, which cause the matrix and fiber to not adhere well, leading to composite fracture[13]. Due to their low manufacturing and maintenance costs, certain natural fibers like sisal, flax, jute, hemp have been identified and tested in a variety of industries, including sports, infrastructure, automobiles, and packaging[14,15]. Scientists, academicians, and researchers are encouraged to look for substitute plant fibers with comparable characteristics in order to meet today's standards. This can lead to a good matrix in a composite that is less hydrophilic[16]. Boopathi *et al.,*[17] investigated the physical, FTIR, SEM, mechanical, and chemical characterization of raw borassus fiber before and after NaOH treatments from 5% to 15%. The spectrum obtained by FT-IR confirms the amputation of impurities and remainingelements from the fiber after increasing the NaOH treatments from 5% to 15% on the fiber. De Andrade Silva *et al.,*[18] testified that after alkali treatment, hemicellulose was reducedfrom the fiber, this finding was supported through the FT-IR results. Kar *et al.,*[19] extracted raw Calamustenuis cane fibers, treated them with alkali, and subjected to physiochemical, mechanical, and morphological properties to evaluate their suitability as a polymer composite reinforcement.

The choice of Burmese silk orchid fiber is well foundedsince it is derivative from a renewable source, making it eco-friendly, inexpensive, sustainable and low-weight. It is widely available in India. Examining their chemical and thermal properties was important observation from the literature. The aim of this study thus is to investigate the chemical properties such as ash and wax of raw Burmese silk orchid fibers and fibers treated with NaOH at 5% to 10% (alkali treatment). The results were compared with other types of natural fibers reported in the literature.In order to have a better understanding of the behavior of Burmese silk orchid fibers, TGA, XRD and SEM characterization techniques were employed. These characteristics aid in expanding the use of fiber for structural and thermal applications.

MATERIALS AND METHODS

The materials and techniques utilized in the current work are listed and explained in this section.

Materials

The natural fibers originate from the Burmese silk orchid tree. The chemicals needed for characterization such as sodium hydroxide and petroleum benzene were obtained from the chemistry laboratory at G. Pulla Reddy Engineering College: Kurnool. Our chemistry lab purchases many chemicals for its daily functioning and the above chemicals are part of the purchases from their reliable suppliers.

Extraction of fibers

Fresh stems of Burmese silk orchid fiberstrands were harvested from Dorigallu village in the district of Sri Sathya sai, Andhra Pradesh, India. After being submerged in water for 15 days, the fiber bundles were manually rasped and rinsed under running water to remove any leftover greasy substance and extract the fine fiber. To take out any moisture or wetness, the fibers were taken away and dried in the sun for seven days. The fibers were beaten up smoothly to remove any undesirable short fibers or dry flush particles.

Alkali treatment

Dry Burmese silk orchid fibers were treated distinctly for two hours at room temperature with 5%, and 10% NaOH solution. To get rid of any NaOH residue that had adhered to the surface, all of these fibers were then washed with clean water. Nevertheless, any NaOH that remained in the fiber was neutralized at room temperature using a 2.5% HCl solution. Fig. 1(a)-(c) shows the raw, 5% and 10% NaOH modified Burmese silk orchid fibers.

Fig. 1(a). Raw, (b) 5% NaOH modified (c) 10% NaOH modified Burmese silk orchid fibers

Chemical properties

Wax content

The soxhlet extractor was extensively used to (separate and analyze the components in natural products) measures the wax content of the Burmese silk orchidfiber. Petroleum benzene solvent was introduced into the flask and heated up to 70°C and a weighted quantity of Burmese silk orchid fiber was put into the extractor. Then allowed to run for 1 h of reflux time and the fiber samples were dried after the 1 h of reflux time. The fiber was weighed after drying, and the weight difference revealed the presence of wax[17].

Ash content

First, the crucible was dried at 105°C for 20 min before cooling it in the desiccator. The weight of the blank crucible (W_1), and the weight (2 to 6 g) of Burmese silk orchid fiber sample (Ws) in the crucible were noted. The crucible containing thesamplewas placedinside the furnace and the temperature was set at 700°C for 2 hours. The sample was subsequently burned, and the final weight of the crucible containing the ash is determined (W2). Using the formula, calculate the ash content[17].

$$\text{Total ash content} = \frac{\text{Weight of the ash content}}{\text{Weight of the original sample}} \times 100$$

$$\text{Ash (\%)} = \frac{W2-W1}{Ws} \times 100$$

Thermal stability analysis (TG Analysis)

The NETZSCH STA 2500 TGA instrument was utilized to assess the thermal steadiness of both raw and treated fibers. The measured quantity (1.82 mg) was added to the Al_2O_3 crucible and heated at a rate of 10°C/min in the heating chamber between 25°C and 850°C. To ensure controlled heating, nitrogen gas was constantly circulated through the furnace at a rate of 20 mL/min to avoid the oxidation effect.

X-ray diffraction analysis (XRD)

With an X-ray diffractometer model: Smart lab; Rigakumake, the XRD examinations of the raw and treated powder samples were carried out. The percentage of crystallinity and crystallinity index were determined for the selected samples.

Morphological Analysis (SEM)

The raw and alkali-treated fibers' morphology and impurities were scanned using a JEOL/MP SEM machine (Model: JSM-IT500). To enhance the conductivity and scan quality of the fibers, they were washed, dried, and covered with a thin layer of gold (3 µm).

RESULTS AND DISCUSSION

The data acquired from the materials and methods section was displayed as figures or tables in the Results section, and the results were interpreted in the Discussion section.

Chemical properties of Burmese silk orchid fiber

Table 1 shows the results of the raw fiber and alkali treatment of Burmese silk orchid fibers with 5%, and 10% NaOH solutions. The fiber characteristics changed as a result of the alkali treatment. The raw

Burmese silk orchid fiber consisted of 0.59% wax and 2.26% ash content. The treatment with alkali resulted the cellulosic fiber to expand and dissolve the hemicellulose and other contaminants from the fiber surface. The alkali treatment did not affect the cellulose micro-fibrils. Improved mechanical qualities resulted from the removal of contaminants. Burmese silk orchid fiberwas compared to a variety of other natural fibers based on chemical composition and displayed in Table 2.

Table 1: Chemical properties of Burmese silk orchid fibers

Sr. No	Fiber type	Wax Content (wt. %)	Ash Content (wt. %)
1	Raw fiber	0.59	2.26
2	5% NaOH treated fiber	0.43	2.68
3	10% NaOH treated fiber	0.16	3.02

Table 2: Chemical compositions of Burmese silk orchid fibercompared with other natural fibers

Fiber Name	Wax (Wt.%)	Ash (Wt.%)	Reference
Burmese silk orchid	0.59	2.26	Present Paper
Acacia planifrons	0.57	4.06	[15]
Prosopis Juliflora	0.61	5.2	[20]
Acacia Arabica	0.49	-	[21]
Sansevieria cylindrica	0.09	-	[24]
Cyperus pangorei	0.17	-	[23]
Oil palm empty fruit bunch	4		[24]
Agave Americana	0.26	-	[25]
Henequen	0.5	-	[26]
Acacia pennata fiber	0.12	5.13	[22]

Wax content

The wax content in the Burmese silk orchid fiber was determined using a Soxhlet extractor, and the findings are shown in Table 1. In Table 2, the wax content of raw Burmese silk orchid fiber was compared to that of different other raw natural fibers. According to Table 2, Prosopis juliflora fiber[20] had the highest wax content when compared to Burmese orchid fiber, and the remaining fibers, which include Acacia planifrons[15], Acacia pennata[22], Acacia Arabica[21], Cyperuspangorei[23], Oil palm empty fruit bunch[24], Agave Americana[25], and Henequen fibers[26], had the lowest wax content. Raw Burmese silk orchid fiber contains 0.59% wax; alkali treatment reduced wax content to 0.43% and 0.16%, respectively, for 5% and 10% NaOH. The properties of the composite materials were enhanced by the reduced wax content.

Ash content

The ash content of the Burmese silk orchid fiber was measured using the standard procedure mentioned in the materials and methods section and the findings are shown in Table 1. Table 2 compares the ash content of various other raw natural fibers with that of raw Burmese silk orchid fiber. Ash content of raw, 5%, and 10% NaOH treated fiber was found to be 2.26%, 2.68%, and 3.02%, respectively. The raw fiber had higher ash content than the other NaOH-treated fibers, due to the presence of amorphous hemicelluloses, lignin, and other impurities on the fiber surface. After being treated with 5%, and 10% NaOH, raw fiber ash content increased from 2.26% to 3.02%. Table 2 shows that Acacia planifrons[15], Acacia pennata[22], and Prosopis juliflora[20] have the highest ash content when compared to Burmese silk orchid.

Thermo-gravimetric Analysis (TGA)

Using this analysis, the degradation of the fibers in the temperature environment was evaluated. The fibers thermal degradation was assessed using its rate of mass loss as a function of temperature. Fig. 2 and Table 3 shows the thermographs of raw, 5%, and 10% treated Burmese silk orchid fibers that underwent TG analysis. The first stage of thermal deterioration began at a temperature of 135°C, and at this temperature, 11.8% mass loss was noticed, which denotes the removal of water vapor, moisture content, and wax material from the fiber[27]. A similar tendency of thermal deterioration was noticed in the treated fibers also, but the mass loss was less compared to raw fibers. Moreover, in the next stage, mass loss started in the middle of 100 and 300°C (around 220°C) for treated fiber, indicating the termination of hemicellulose, lignin, and inadequate cellulose from the fiber. For raw fibers, deterioration began early (around 175°C)[18,28]. However, mass loss was nearly the same among all fibers (raw, 5%, and 10%). In the third stage, deterioration took place in the temperature range of 300 to 400°C (around 380°C). Significant mass loss (50%) was noticed at this temperature, which was the result of the decomposition of cellulose and lignin content[29]. The fourth stage of deterioration occurred in the temperature ranges of 400 to 600°C for all the fibers, with 10% NaOH treated fibers having higher thermal stability, up to 565°C[28]. This indicates the decomposition of lignin and wax in the fibers.The final stage of deterioration happens at temperatures rangingfrom 500 to 700°C. This shows that lignin and wax contents were decomposed in the fibers[27].

Table 3: Thermal degradation stages for raw, 5%, and 10% NaOH fibers

Sr. No	Thermal Degradation (°C)				
	1st stage	2nd stage	3rd stage	4th stage	5th stage
Raw	135	175	370	520	665
5% NaOH	140	218	382	520	685
10% NaOH	140	220	382	565	685

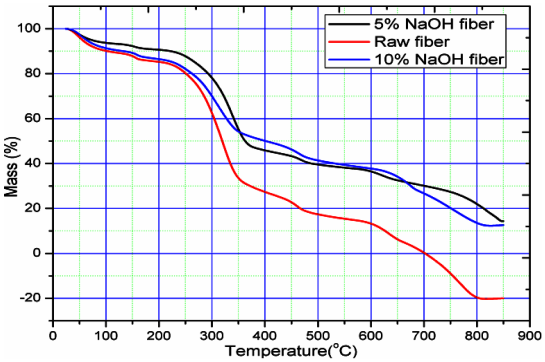

Fig. 2. Thermograph of raw, 5%, and 10% treated Burmese silk orchid fibers

X-ray Diffraction (XRD) Analysis

Figure 3 indicates the XRD patterns of raw and alkali-treated Burmese silk orchid fibers. The fiber's amorphous components were shown by the first peak, which was seen at 2θ = 15.17 and represented the planes of (011). The plane of (201) depicts the crystalline constituents of the fibers represented by the second peak, which ensued at 2θ = 22.81 and had a higher intensity. The third peak which ensued at 2θ = 24.94 was allocated to the (211) crystallographic plane. The plane of (301), which depicts the amorphous components of the fibers, corresponds to the fourth peak, which was less intense and occurred at 2θ = 30.34. The final, less intense peak was found at 2θ = 38.44, which is the plane of (231) and indicates the fibers' amorphous components. The raw Burmese silk orchid fiber had a crystallinity index of 46.39%, which increased to 55.29% after treatment with 5% NaOH. The increased crystallinity after 5% NaOH treatment was caused by the reaction of the Burmese silk orchid fiber's hydroxyl group with NaOH solution, which resulted in the discard of constituents from the fiber's external substrate. A reduction in the crystallinity index to 54.02% occurs from a further increase in NaOH solution concentration to 10%. Increased NaOH concentration affected the quality of the fiber, as also perceived in the earlier research[30,31]. The crystallinity index attained by raw and 5% and 10% NaOH treated specimens was greater than that of the treated and untreated date palm fiber in earlier studies[32]. Those values were 31% and 41%, respectively. Nevertheless, Benchouia et al., 33 found that untreated and 10% NaOH-treated date palm leaflet fiber exhibited higher percentage crystallinity after 12 h of treatment, at 68.42% and 70.06%, respectively. This fiber exhibits monoclinic cellulose I structure.

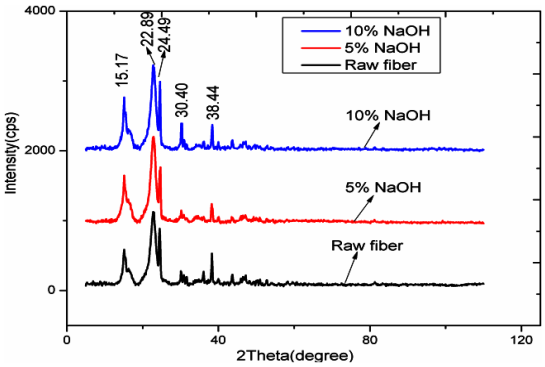

Fig. 3. XRD patterns of raw and NaOH-treated Burmese silk orchid fibers

Morphological Analysis (SEM Analysis)

SEM scans of raw, 5%, and 10% alkali-treated Burmese silk orchid fibers were examined; Fig. 4(a)–(c) shows these scans. Fig. 4(a) shows the SEM scan of raw fiber containing surface impurities and fiber constituents. Fig. 4(b) shows the SEM scans of 5% NaOH-treated fibers. It confirms that the most of surface impurities were disappeared. The surface of the alkali-treated fibers was rougher and clearer than that of the raw fiber. Its roughness increased the effective surface area that resin could wet and encourages mechanical interlocking. This roughness facilitates the bonding between the fiber and matrix.Nevertheless, a SEM scans of fibers treated with 10% alkali (NaOH) is displayed in Fig. 4 (c). At higher (10%) concentrations, it revealed that the chemical reaction with NaOH caused damage to the fibers[34].

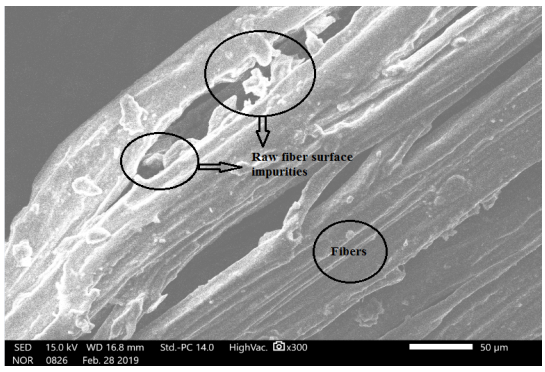

Fig. 4(a). SEM scans of raw Burmese silk orchid fibers

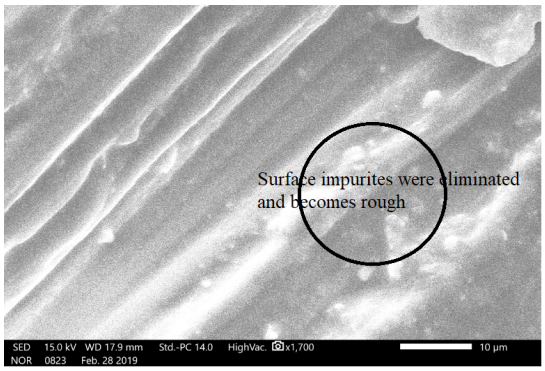

Fig. 4(b). SEM scans of 5% NaOH Burmese silk orchid fiber

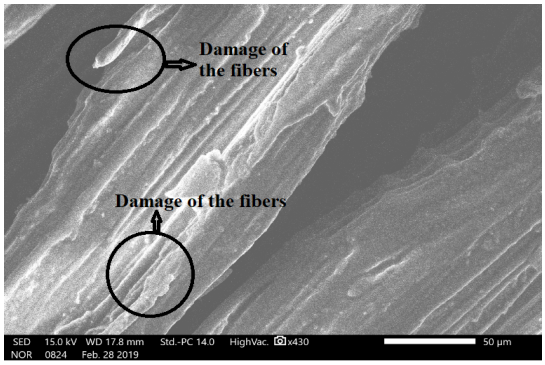

Fig. 4(c). SEM scans of 10% NaOH Burmese silk orchid fiber

CONCLUSION

The chemical characteristics of raw Burmese silk orchid fibers and those treated with 5% and 10% NaOH were contrasted in this study. Thermal and XRDcharacterization was performed on both raw and treated fiber samples. The following conclusions were drawn after an investigation of the extraction, chemical, and thermal characteristics of Burmese silk orchid fiber.

1. A chemical study showed that raw Burmese silk orchid fibers contain 0.59% wax, 2.26% ash. These values were altered after 5% and 10% NaOH treatment.
2. The results of two types of NaOH treatments and their research showed that the properties of the Burmese silk orchid fibers were improved by 5% alkali treatment over raw and 10% alkali-treated fibers. Therefore, the Burmese silk orchid fiber that had been treated with 5% NaOH could be employed as reinforcement in the composites. This fiber is also useful for sustainability development.
3. Based on the results of the TG analysis, Burmese silk orchid fibers remain stable at temperatures as high as 225°C, and when the fibers were treated with NaOH, their thermal stability further increased.
4. The XRD analysis revealed that the crystallinity index and percentage crystallinity of 5% and 10% NaOH-treated Burmese silk orchid fibers increased as compared to raw fibers.
5. SEM scans displayed the morphology, impurities and fiber quality of both raw and chemically (NaOH) treated fibers.
6. The above chemical, crystalline, morphology, and thermal characteristics of Burmese silk orchid fiber strongly suggest the possibility of reinforcement in polymer composites for various industries, such as construction, automobile, aerospace, encouraging sustainability and enhancing product performance.

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Conflicts of Interest

The author declares no conflict of interest.

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