

Original Article

Experimental Investigation on Morphology of Untreated and Alkaline Treated Phoenix Dactylifera through Scanning Electron Microscopy (SEM)

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Abstract

Scanning Electron Microscopy (SEM) has been proved as a powerful tool for investigating surface morphology of untreated and chemically modified fibers. In this paper SEM (Scanning Electron Microscopy) analysis of untreated and alkaline treated Phoenix Dactylifera fibers has been used to investigate the degree of surface roughness, agglomeration of impurities, fiber pull out, fiber breakage etc To uncover the morphology of natural Phoenix Dactylifera fiber, FE SEM from Zeiss (ULTRA plus) at CIF, IISER Bhopal is used for getting high resolution images. This research work may lead to better understanding of the fiber surface morphology and it may be helpful in fabricating Phoenix Dactylifera fibers reinforced polymer composites. Such biocomposite could reveal a better world with bio waste utilisation by making useful products.

Keywords: Scanning Electron Microscopy (SEM), alkaline treatment, fiber morphology, untreated and treated Date Palm fibers, bio waste

Introduction

With the growing environment concerns, a lot of research is being carried out in the direction of producing environment friendly materials. In the same line, fiber reinforced polymer composite is gaining huge attention these days. Being environment friendly, natural fibers enjoy a better place than synthetic fibers. The scientific community, these days, is focused on utilising bio waste which are thrown either way. By studying morphology of the natural fibers through different highly efficient tools, with great resolving power, they are poking into the fiber structure. A lot of research works these days are focused on preparing bio composite so that environment sustainability could be achieved. And thus not only fresh natural fibers from the source is being utilised but also waste or used up natural fibers like banana peels, coconut shell powder, sugarcane fibers, walnut residues etc. are being utilised in preparing composite for diversified uses. But, before making use of any natural fibers as reinforcing material, it is necessary to understand its physics, morphological, chemical, electrical, thermal, tribological etc features so that desired properties could be attained. Thus it is a vast area and requires great attention and analysis of the properties of natural fibers. How these could be altered or enhanced? How this hydrophilic nature of these fibers could be overcome? How can be better bonding between fiber and resin be supported? And many more. One of the answers which can help researchers is surface modification techniques which can improve the bonding between fiber and resin. Surface Modification [1] can be chemically (alkaline, silane, Acetylation), physically (plasma treatment etc.), coating etc. (Pickering et al. 2016; Pokhriyal et al. 2024). Alkaline treatment has been proved its efficacy [3,4,6,7] in this regard. Previously it can be realised in the work of Rao et al. (2025) Pokhriyal et al. (2023), Rafiqah et al. (2025); Pickering et al 2016; Kabir et al. (2012, Mishra et al. (2003) etc. In this article, Alkaline treatment is employed to modify the morphology of date palm fiber waste so that some useful end products could be attained by improving natural fibers / matrix interlocking and thus increasing strength with fiber reinforcement. Since this Phoenix Dactylifera fiber waste is thrown in open or burnt in open causing environmental pollution so this work is indeed a great attempt in conserving the environment and moving towards a sustainable world.

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Fig. 1 Phoenix Dactylifera tree

Materials and Methods

Materials [2-3]

Phoenix Dactylifera leaflet used as reinforcing material in the current work which belongs to the species Phoenix Dactylifera. As per literature available. It is composed of (47.14%) of cellulose, 16.13% of hemicelluloses and 36.73% of lignin. (Ramadan 2016). Sodium hydroxide is used in the form of pallets purchased by researchers from Qualikems Pvt Ltd. Vadodara, Gujrat. Phenolic Resin and hardeners are provided by the Permal Wallase Ltd. Bhopal.

Methods

Fiber Preparation Method used in this work is as follows:

Raw Material

Raw samples from the leaflet of Phoenix Dactylifera tree were collected from the local broom makers. Fibers were extracted and cleaned manually and then washed thoroughly in running water. Washed samples are then allowed to dry. After dried properly, raw fiber samples are packed.

Alkaline Treatment

Alkaline treatment is used here to understand the effect of changing alkaline concentration on Phoenix Dactylifera fibers. Thus extracted raw fibers are soaked in Alkaline solution for different immersion time and then washed with running water. These samples are dried properly. Finally, the treated fiber samples were preserved in airtight bags (to resist moisture).



Fig. 2 Alkaline Treatment of fiber

Fiber Characterization

For MORPHOLOGICAL studies fiber samples both treated and untreated are sent to Indian Institute of Science Education and Research IISER Bhopal, and images are produced by the ULTRA plus model (Zeiss) which were analysed later.

Scanning Electron Microscopy (SEM) of Natural Fiber

In SEM physical image of the fiber sample is obtained by focussing the fine beam of electrons on samples and analyzing backscattered electrons. SEM micrographs can be obtained at different magnifications so that topography of surface could be

analysed properly. This analytical technique is being widely used in morphological analysis of natural fibers. SEM of fibers can be done in three ways: Imaging, Mapping and Energy dispersive X-ray spectroscopy (EDAX). EDAX is used for chemical characterization of materials

Present work is focussed on study of changes in fiber morphology with or without treatments so SEM imaging is done here.

Research Design

Work flow of present investigation is as follows:

1. Review of Literature
2. Gap Findings
3. Sample collection
4. Sample treatment
5. Fiber characterization
6. Data analysis, Results & Discussion

Results

Sem Analysis of Phoenix Dactylifera Fibers

Figures 3 to figure 7 shows scanning electron micrographs of untreated and treated Phoenix Dactylifera fibers with sodium hydroxide with different concentrations and fixed soaking time.

From figure 3, it can be seen that the fiber surface is not smooth, it is uneven and Impurities are stuck over it. Such uneven surface represents the possibilities of presence of dirt, waxes lignin, hemicellulose. For making useful end product such Impurities need to be removed as they hinders the proper binding of fiber with resin during production of Composites.

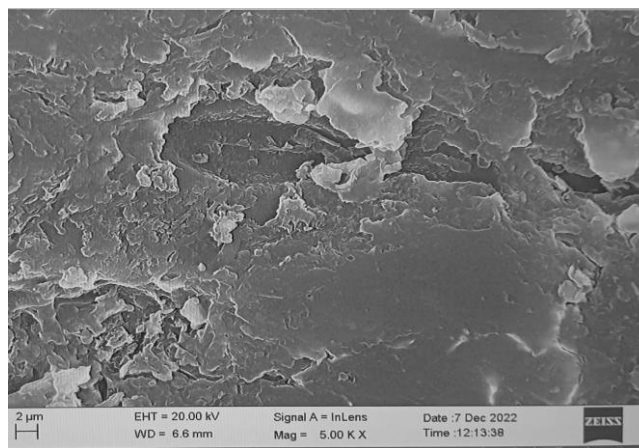


Fig. 3 Untreated Date Palm Fibre

As alkaline treatment is found useful in improving fiber morphology, thus in this experimental work, aforementioned fiber is treated by 2wt%, 3wt%, 5wt% and 6wt% NaOH for fixed 12 hours immersion time by keeping room temperature constant and treated fibers micrographs are shown in fig 4, fig.5, fig.6, fig.7 respectively. Here, impurities and waxes seen on the surface of micrographs earlier are altered by alkaline treatment.

Effect on surface roughness and impurities [7]

It is evident from SEM micrographs that Phoenix Dactylifera fiber is cylindrical in shape for both treated and untreated fiber. Phoenix Dactylifera fiber is cylindrical in shape for both treated and untreated fiber. Diameter of fiber shrinks after treatment, it may be due to alkalization which removes impurities, hemicellulose and lignin content. (Rao et al.2025) On comparing surface micrographs, it can be inferred from that untreated fiber shows a rougher surface with impurities like hemicellulose, wax, lignin, pectin, dirt etc.(fig.3)

While fibers treated with 2% NaOH show hollow cavities and vascular bundles. (fig. 4)

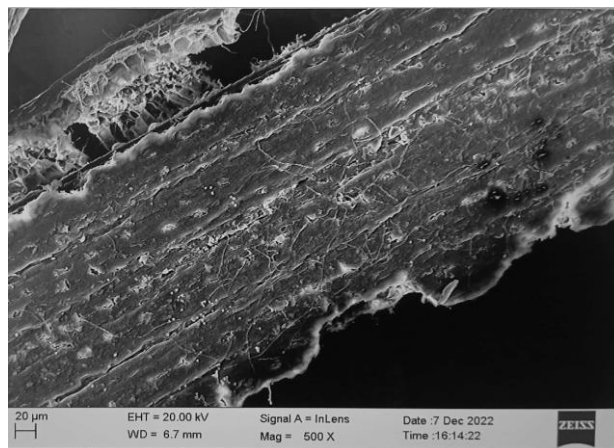


Fig. 4 SEM Micrograph of 2% NaOH treated for 12 hour at room temperature

On further increasing the alkaline concentration to 3%, then more hollow cavities and vascular bundles can be seen easily. (fig.5)

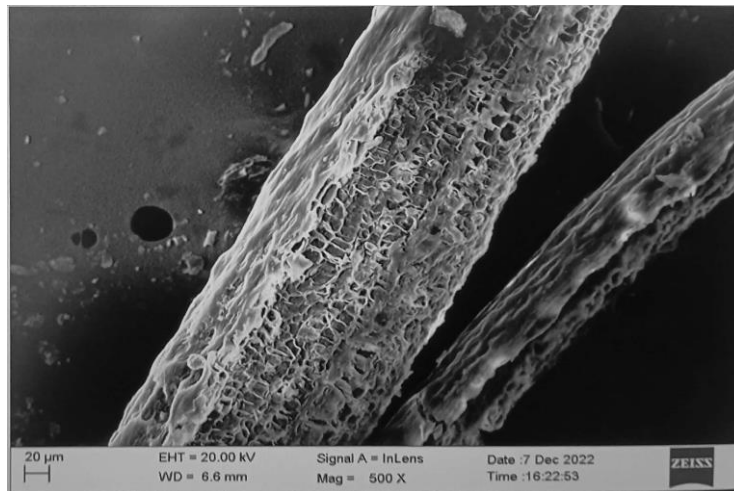


Fig. 5 SEM Micrograph of 3%NaOH treated for 12 hour at room temperature

When alkaline concentration reaches to 4% the damaged hollow cavities and fiber breakage are evident.(fig.6)

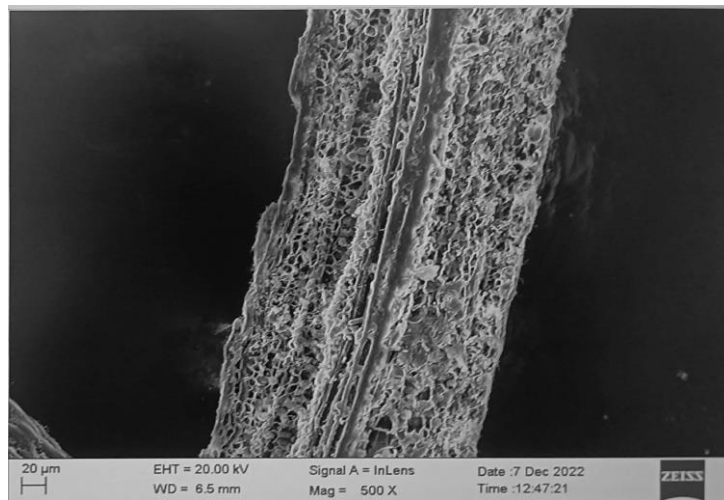


fig. 6 SEM Micrograph of 4%NaOH treated for 12 hour at room temperature

At higher concentration (6% Alkaline treatment) damaged the figure structure, fiber agglomeration and damaged Vascular bundles are evident through SEM Micrographs. (fig.7) This may be due to removal of natural cementing material.

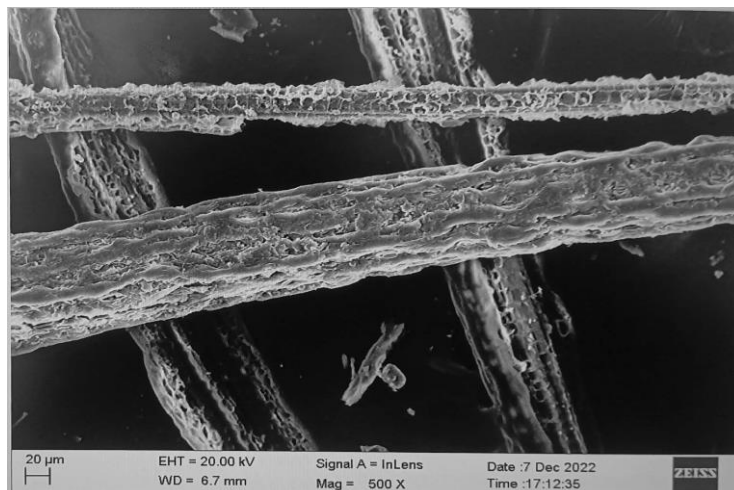


Fig. 7 SEM Micrograph of 6%NaOH treated for 12 hour at room temperature

Alkaline treatment modifies the surface [3,4,7] by removing impurities and waxy layers. On the same time it improves the number of pores and fibrils of the treated fiber as compared to untreated fibers. This may be helpful in proper bonding between fiber and matrix while preparing NFRPC. (Rafiqah et al.2025; Kabir et al. 2012, Mishra et al.2003)

Conclusion

Phoenix Dactylifera fibre is found abundantly in nature and due to ignorance of the society it has been thrown as a waste. There are lots of possibilities with this fiber. Proper scientific investigation can provide a raw material which can be used as a

reinforcement while making composites. This work uncovers the possibilities of scanning electron micrographs in removal of impurities and the other substances after alkali treated. The presence of fibrillar structure is noticed in alkali treated Phoenix Dactylifera fibre which makes it as a suitable candidate for NFRPC. Other features like water absorption need to be addressed to make use of its potential. Such investigation may open a new era of utilisation of bio waste for creating useful products. It seeks further work on mechanical, thermal, electrical properties of the aforementioned fiber.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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