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Significance of Physical Surface Treatment on Areca Fiber – An Ecological and Economical Process

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ABSTRACT: *Control of surface properties is very much significant in case of natural fibers. This will help to get better and proper bonding between the natural fiber and matrix material in order to have improved mechanical and physical properties. In this regard, physical surface modifications of the areca fiber have been carried out by both Atmospheric Low pressure Plasma and UV radiation techniques with different expose time. This paper presents comparative study between the Atmospheric Low Pressure Argon Plasma and UV radiation with aim to identify most suitable and economical physical treatment process to get enhanced areca fiber surface properties. The morphological analysis, chemical composition changes and surface roughness improvement on areca fiber have been studied using SEM. Substantial enhancement in Thermal Stability is also been studied using TGA. From the results it have been noticed that UV treatment leads for the enhanced chemical characteristics in comparison with Ar Plasma and both treatment have showed the equal contribution towards the improvement of surface roughness. This study leads improved surface properties of areca fiber by using physical surface treatment process which will help in developing composite. Hence, there are more opportunities to develop economical and ecologically material by utilizing these areca fibers and its composites for suitable industrial applications.*

Keywords: *Areca Fiber, Surface Treatment, Chemical characteristics, Surface roughness. Thermal Stability.*

I. INTRODUCTION

With the growing environmental awareness and with new rules, scientists and engineers are forced to seek new materials which are more eco-friendly in nature. [1] [2] [3] [4] Natural fiber composites are one such kind of materials. Usage of natural fibers in the composites is well known, because of its inherited qualities such as lignocelluloses, renewable and biodegradability. [5] There are other several reasons that are in favor of using natural fibers instead of any other artificial or synthetic fibers. They are lightweight materials having superior strength, competitive specific mechanical properties, high specific modulus, and reduced energy consumption. Further, they are non-toxic & nonhazardous in nature, naturally recyclable, available in abundance, flexible in usage, less expensive, and that allows clean energy recovery etc. [6] [7] [8]

Studies on most of the natural fibers like cotton, coir, sisal, jute, banana, flex, maize, areca, to list few, has been carried. The main limitations of these natural fibers are: hydrophilicity - most of these are hydrophilic (exhibit high moisture absorption) in nature because of hydroxyl and other polar groups in their constituents. Because of this, these will have limited application. [9] [10]

In order to develop a composite material made from natural fibers with significantly improved strength, stiffness, durability, and reliability, it is important to have better fiber–matrix interfacial bonding. This can be achieved by the surface treatment of the fiber. Generally the surface treatment/modification of natural fibers will not only improve the bonding between the fiber and matrix, but also improve specific aspects such as reducing the moisture absorption sensitivity. Similar polarity between the two materials will be adding / improving the better adhesion strength between the matrix and natural fiber. So the surface modification / treatment will be major criteria to improve properties of fiber. [2] [3] [11]

Different surface modification processes are involved. Majorly four are extensively and exhaustively used to improve the fiber surface properties; chemical, physical, physical – chemical and mechanical. These mentioned treatments are helpful in improving the adhesion property between the fiber and the matrix, by improving the surface roughness on the fiber. This will lead to the significant increase in the strength or other properties of composites. Additional information on different surface treatment/modification of natural fibers may be referred in many references. [3] [9] [11] and [12]

Chemical treatments like alkaline, acetylation, permanganate, acetic anhydride, stearic acid, silane, maleic anhydride, benzoylation are studied and have been used for natural fiber surface treatments by many researchers. Improvement on the Interfacial bonding between the fiber surface and matrix has been displayed by many of these treatments. Along with the advantages there are many disadvantages like higher processing cost, discarding the chemicals after the treatment which leads to environmental pollution problems. [13] In the recent past, among the scientist, researcher and industrialist it's been observed that there is an increased concern towards the environmental pollution because of the wide usage of chemicals as part of chemical surface treatment.

Thus lots of studies are undergoing on the uplifting the other surface treatments like mechanical, physical and physio-chemical. In this research, physical surface treatment has been studied. Among the several physical pretreatment –Atmospheric low temperature plasma (ALTP) and Ultraviolet (UV) technique is being used. These are very gentle but very much significant process for the surface treatment. And also it shows promising approach for surface modifications by increasing the surface energy of artificial as well as natural fibers. [10] [13] [14] [15] and [16]. The pretreatment process involves physical sputtering and chemical etching which leads to cleaning, activation, and surface bonding. These interns influence the quality of the fiber surface. Cleaning is to remove even the finest particles of dust. The physical pretreatment process are being dry in nature, these have numerous advantages over the chemical (wet) process. The Advantages are: No mechanical damage is done to the fiber, thin layer treatment will not affect the bulk property of fiber, no harmful waste/ chemicals emitting therefore it is an environmental friendly process, No wet processes so no wastage of water, very simple process and needs lesser time. Produced surface are much better than any conventional techniques. [10] [14] [17]

Plasma is a partially ionized and these are generated by electrical discharge. In Plasma treatment the inert gases, such as, Argon (Ar), Neon (Ne), Helium (He) etc are being used in a plasma state. Because of treatment there will be some reaction on fiber chemically and physically, which will bring in extremely active species like photons, ions, and free radicals. Many of researchers have studied about the Ultraviolet radiation process for the surface treatment of the natural fiber and other elements. The treatment has been carried out at the frequency ranging from as lower as 184.9 nm and as high as 365 nm, under atmosphere of normal air, with ozone. [15] [16] [18]. These exclusively affect the surface of the fiber without affecting the bulk properties. With these treatment there will be significant improvement on the two aspects; one physical sputtering - this will help to build a strong covalent bonds between the fiber and the matrix. And chemical etch – which improve bonding by roughening the surface of fibers and also increases the mechanical interlocks between the fiber and the matrix. Fiber surface property by plasma treatment depends on the many parameters like nature of the gas, distance of the fiber from the nozzle, applied power for gas discharge and treatment time [10] [13]. And the UV treatment depends on the parameters like frequency in use, distance from fiber to source, with or without ozone medium, treatment time. [15] [16] [18] These process will increase the aldehyde groups. And produceds surfaces with –O=C and O-C=O functionalities with better adhesion properties. Treatments help getting very high quality fibers which can be used in developing composites. This can be used in part development, with still being an environmental friendly. [14] [18]

A lot of literatures have been published especially on Atmospheric Low Temperature Plasma (ALTP) and UV radiation treatment for natural fibers like sisal, jute, pineapple and banana fibers on improving surface energy, mechanical, physical and thermal properties with thermoplastic and thermosetting material. However, not much efforts and attention have been made towards the areca fiber surface treatment with Plasma and UV and literature available is also sparse.

In this study, areca fiber is treated with atmospheric low temperature argon plasma and UV with air to modify the surface. The ultimate goal of the research work is designed to get the cleaned, improved surface roughness and energized surface. In this untreated, Ar Plasma and UV treated areca surfaces are scanned by scanning electron microscopy (SEM) at very high magnifications to understand the surface morphology improvement, analyze particular elements (chemical characteristics) and respective percentage and also the surface roughness on the areca fiber surface. And also substantial enhancement in thermal stability is been studied using TGA. With these improvised properties, the usage of the Ar Plasma and UV treated areca fiber can be used in composite preparation. The application of these composites can be wide spread to; housing construction material, automotive, aerospace and home appliances etc., using the advance manufacturing technologies.

II. EXPERIMENTS

2.2 Material and Extraction

Areca raw fruit is taken from a field near Sagar District, Karnataka. Raw fruit were crushed to remove the seed from the shell. These shells rinsed in running water and soaked for 4 days. This process will loosen the areca fibers from husk and fibers area extracted easily by hand. Finally fibers were washed again in running

water and dried at room temperature for about 10 -15 days. The samples used were having the length 40 mm and Ø 0.60 mm. are noted.

2.3 Sample Preparation

As shown in Fig.-1 around 15 to 20 extracted areca fibers were considered on the single sample. These fibers are stick on the thermal tape, which can withstand higher temperature.

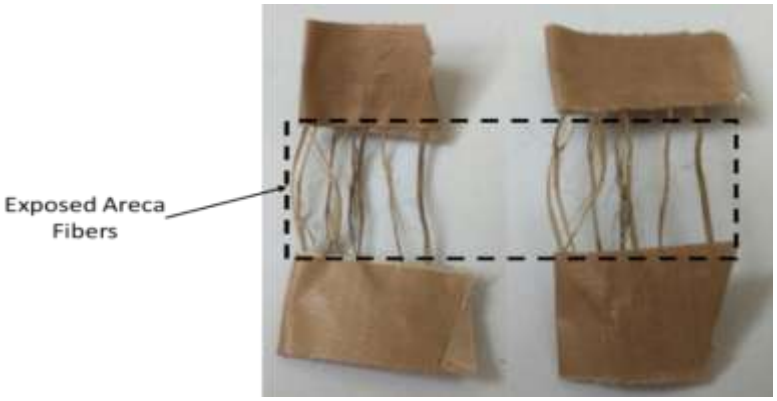


Figure-1: Prepared Sample for Areca Fiber

2.4 Split End of the Areca Fiber

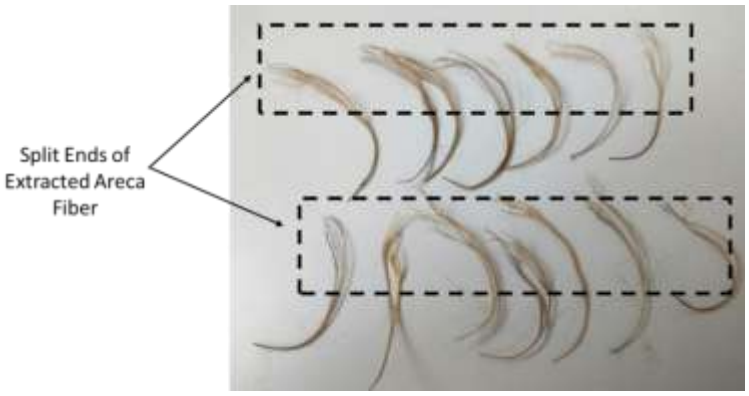


Figure-2: Split End of Areca Fiber

Areca nut strongly attached to the husk. [19] Extraction of the fiber from the nut leads fiber with slit ends as shown in Fig.-2. These split ends are having lesser diameter in comparison with the other part of the fiber. So this portion of the areca fiber will be weak. Indeed the whole areca fiber cannot be utilized as a reinforcing agent. This brings in that, it is better to have short fiber in comparison with full length.

III. TECHNOLOGIES USED FOR SURFACE TREATMENT OF ARECA FIBER

3.1 Atmospheric Low Temperature Argon plasma

Areca fibers are treated by means of atmospheric low temperature Argon plasma. This is been carried at atmospheric air with different exposed time for the 16KW power. The equipment used is for the treatment is with HF plasma from Miller Plasmatran + Co (England) with specification 70V, 500A and 40KW. The samples prepared were held from gun at a distance of 12 inch. Argon was considered as a plasma gas for treating the areca fibre. The surface treatment has been carried for the different exposure time i.e., 30, 90 and 120 sec. This surface treatment will create lots of greater polarity covalent bonds. Considered Plasma system works on the blown ion system. In this, discharge occurs inside the chamber, pressurized argon gas in forced inside chamber. Electron then excited and created positively charged ions. The same is impinged on the areca fiber. Ar gas was introduced into the reaction chamber by a flowmeter which was maintained by a needle valve. The treatment has been carried by having system as shown in the Fig-3.

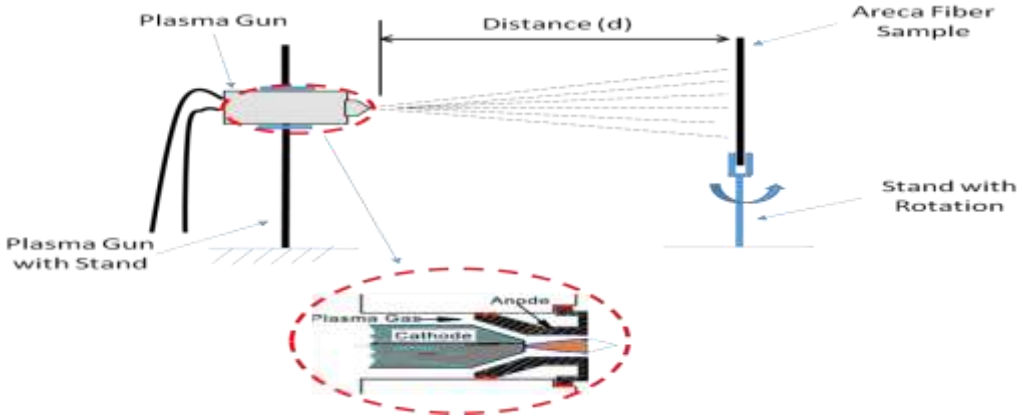


Figure-3: Atmospheric Low Temperature Plasma Surface Treatment Setup

3.2 UV Treatment

The UV radiation treatment was carried out in an atmospheric air. The reactor is equipped with a LED based lamp. The lamp is made of 10 LED (Nichia NC4U133A LED) with input 3.3 V and 1 Amp. This is able to transmit strong line at 365 nm. The intensity of the UV radiation was 13.5–14.5 mW/cm^2 at a distance of 5.25 cm. The treatment has been carried for the different exposure time i.e., 30, 45 and 60 min. Atmospheric temperature and pressure have been maintained during the treatment. To carry out the UV radiation treatment the system is supplemented with fan in order to maintain the cooler LED lamp. Treatment has been carried by having system as shown in the Fig-4.

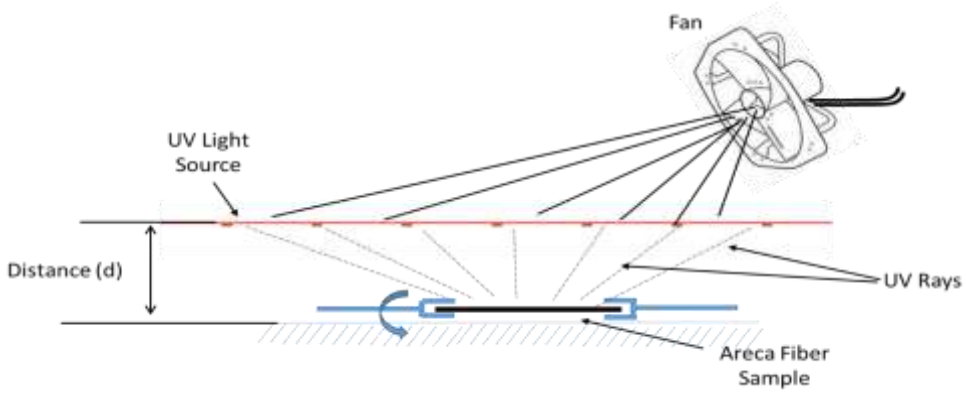


Figure-4: UV Radiation Surface Treatment Setup

3.3 Measurements

3.3.1 Scanning Electron Microscope

The SEM uses a focused beam of high energy electrons to generate a variety of signals at the surface of solid specimens. When beam of electronics strike the surface of the specimen and interact with atoms of the samples, signals in the form of secondary electrons are generated. This will give information about surface topology of the substrate. The surface morphology of untreated, Ar plasma and UV treated areca fiber are investigated by using scanning electron microscopy (SEM). Before examination, the fiber surfaces were prepared on an appropriate disk. Samples were imaged without any coating. SEM was performed on a Zeiss Merlin FE-SEM a voltage of 5 - 10 kV, and 125 pA probe current.

3.3.2 X- Ray Energy Dispersive spectroscopy

Energy Dispersive Spectroscopy (EDS) is used to identify atomic number elements and their proportionate limits (Atomic % for example). This information can be gathered from SEM as well. Different atomic number elements and their distribution images can also be displayed by the SEM backscattered electrons. Entire scan areas of the SEM's X-ray spectrums are utilized by EDS to analyze the elements. Images which are mentioned below are X-ray spectra that were generated from the entire scan area. [21]

3.3.3 Surface Roughness

Surface roughness for natural fiber plays significant role in composite preparation. The preferred surface finish on the natural fiber is usually necessary. Fiber surface roughness influences functional

characteristics such as having proper bonding between the fiber and matrix, wettability. In order to achieve this, selection of the suitable surface treatments are essential. And also the inspection method of the fiber surface is key factor. There are direct and indirect contact methodologies to measure the surface roughness of the fiber. Among these indirect method is used consitently as direct contact method has some limitation over the inderect. In this, SEM being used to get the surface finish by indirect contact method. The samples are prepared on an appropriate disk and imaged without any coating. The range of roughness values were measured for treated and untreated areca fiber samples and then mean results were recorded. This operation is repeated on the same sample for the different locations and the average values results are noted for the five readings. [22]

3.3.4 Thermal Stability

The structural ingredients of natural fibers such as cellulose, hemicellulose, and lignin are very much sensitive towards different range of temperatures. Thus study of thermal stability or degradation of natural fibres becomes important for the development of natural fibers reinforced polymer matrix composites. This is important both in application of the selecting the manufacturing process (extrusion, injection moulding, compression moulding) and product made by composit. Some of the literatures talks about the thermal stability of the areca fiber composites. It has been recorded that thermal stability of the areca fiber and its composites is much improved with surface treatment. [23] [24] NETZSCH STA 409 PC/PG was employed to study the untreated areca fiber thermal degradation. The samples were heated in an alumina crucible at a heating rate of 10°C/min.

IV. RESULTS AND DISCUSSIONS

4.2 SEM images

Fig.-5 shows the areca fiber surface morphology of untreated, with Argon plasma and UV treated. The surface of untreated fiber is covered with dense scales, this will make the areca fiber surface difficult to get wet and have better rough surface. As shown in Fig.-5 (a) the untreated samples have smooth and even surfaces.
However after Ar plasma and UV treatment, it can be seen that the surface scales of the fibers have been removed due to the etching effect caused by plasma and UV treatment. During this active species bombardment will happen on the areca fiber surface which will clean and etch the surface. Thus the treated areca fibers lead to better rough surface of the fibers and helps in building strong covalent bonds between the fiber- matrix. Changes in the surface morphology of Ar plasma and UV treated areca fiber are shown in Fig.- 5 (b) and (c) respectively. The development of some cracks, voids and holes are also observed.

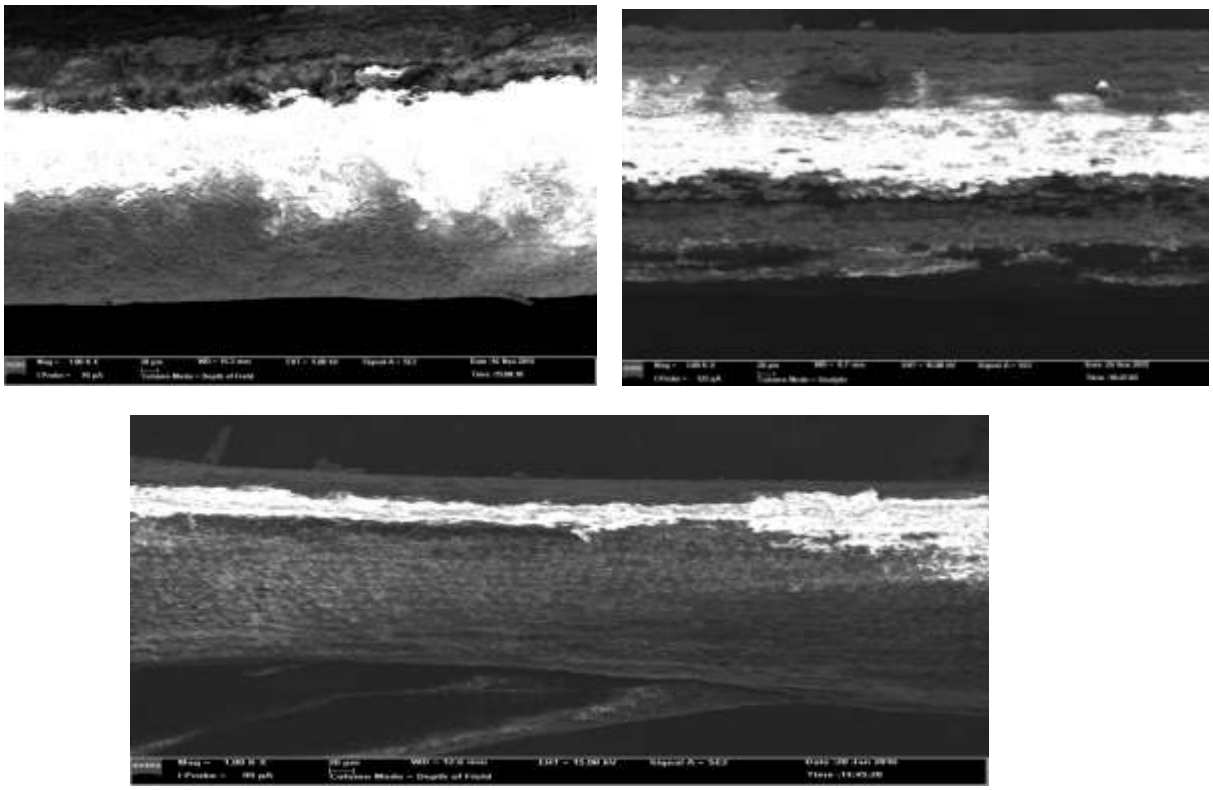
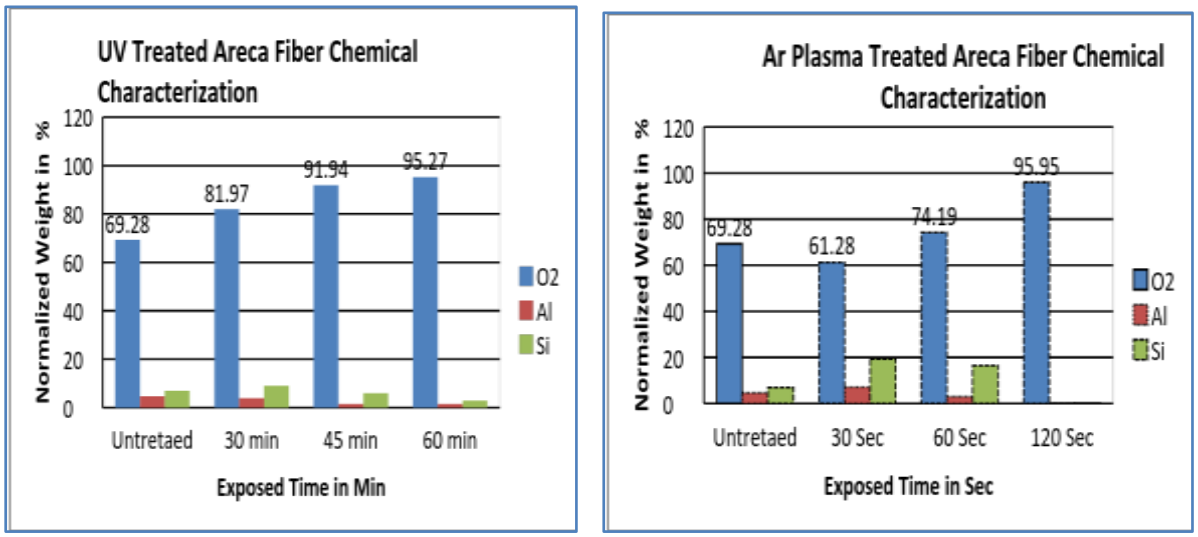


Figure-5: SEM images of (a) untreated, (b) Ar Plasma and (c) UV Radiation Treated Areca Fiber

4.3 Energy dispersive spectroscopy (EDS)

EDS studies were carried out on untreated, Argon plasma treated and UV treated samples to investigate the changes on the surface chemical characteristics. Fig. 6 (a) shows the areca fiber samples treated with Ar Plasma and 6 (b) with UV treated areca fiber with varying exposed time. With the treatment, functional groups were produced on the surface of the areca fibers. This is due to oxidation effect of active species induced by plasma in the gas phase and UV radiation in the light rays on the areca fiber surface. It demonstrates that there are many elements like Al, Si, S, Mg. along with oxygen on the surface of the fiber. In this, oxygen being a major element among the composition. There is increase in % of Oxygen with Ar plasma and UV treatment, which will lead to significant improvement in bonding between the areca fiber and matrix. The increase in oxygen containing surface groups provides a polar surface, which is responsible for better wettability. Fig. - 6 represents along the Y axis elements of normalized weight in % against the untreated and treated fibers for different exposed time along X axis. It has been investigated from the Fig. - 6 that with increased exposed time, better chemical characteristics has been achieved by UV radiation treatment in comparison with Ar plasma.

Figure-6: Chemical Characterization of



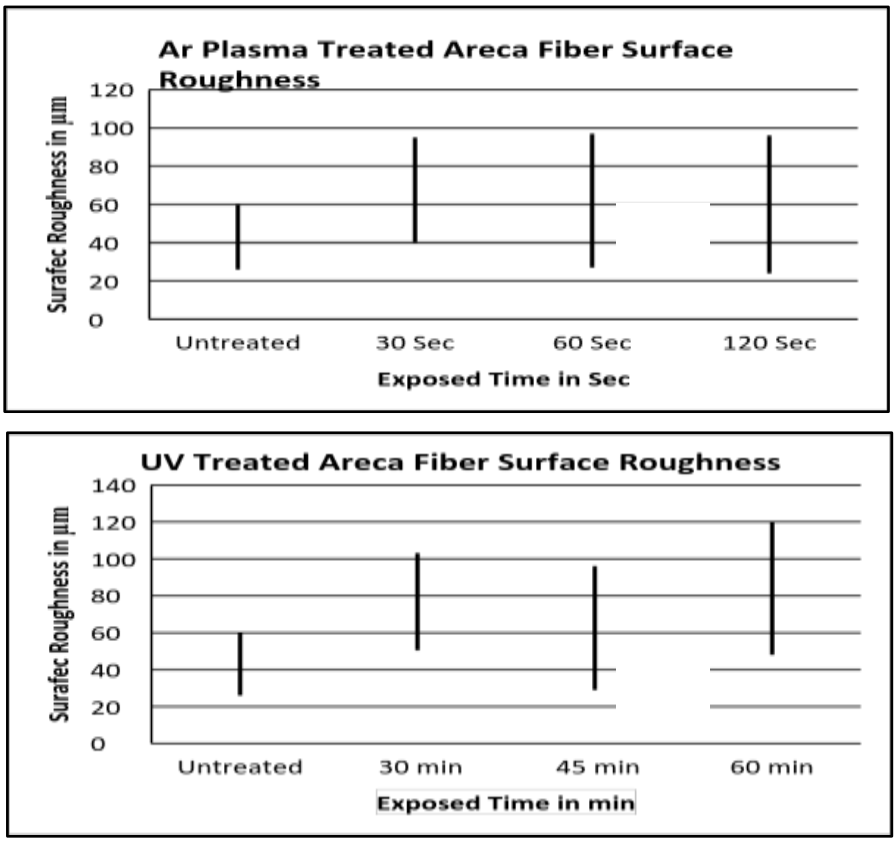
(a) AR Plasma and (b) UV Radiation Treated Areca Fiber at different Exposed time

4.4 Surface Roughness

Fig.-7 represents the surface roughness of the treated and untreated areca fiber. Surface roughnesses of these were measured for fibers which are treated with low pressure Argon plasma and UV. From the plasma and UV treatment it is very much evident that there will be an increase in surface roughness because of the sputtering and etching. It can be noticed that the roughness of treated fiber increases in comparison to the untreated fiber. It has been observed that argon plasma and UV treatment causes etching, which result in a rougher surface which will help to hold matrix properly.

From the Fig. -7 it's been understood that the surface roughness range increases with fiber treatment. And also it has been observed that there is in increase roughness with increase in exposed time. From the results it has been noticed that Ar Plasma and UV treatment has equal contributed towards the areca fiber surface roughness.

Figure-7: Surface Roughness of



(a) Ar Plasma and **(b)** UV Radiation Treated Areca Fiber at different Exposed time

4.5 Thermal Stability

Surface treatment reduces non-cellulosic material and increased crystalline structure of the fibers, which enhanced the thermal stability and lead to the temperature shift. [23] [32]. Fig.- 8 shows the thermal degradation of untreated, Ar plasma and UV Treated areca fiber. From the Thermo gravimetric Analysis spectrum it has been recorded that Ar plasma and UV treated areca fiber showed temperature shift in comparison with untreated. For the analysis degradation has been considered at 10% mass loss of the areca fiber. At 10% mass loss the respective temperature for the untreated, Ar plasma and UV Treated are 120^{o}, 254^{o} and 249^{o} C noticed. Thus it can be noticed that the treated fiber will have better thermal stability than the untreated areca fiber. This will significantly influence the selection of the matrix material and processing technique. It has been noticed thermal degradation temperature is almost same in case of Ar Plasma and UV treated areca fiber.

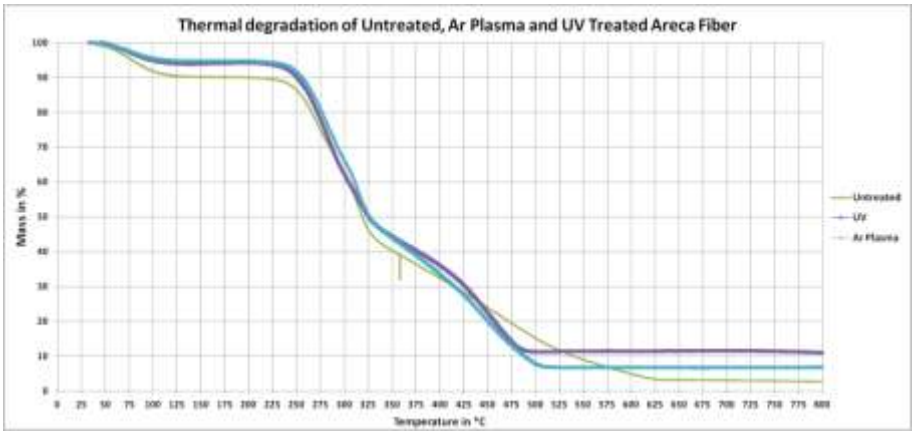


Figure-8: Thermal Degradation of untreated, Ar Plasma and UV Radiation Treated Areca Fiber

V. CONCLUSION

Atmospheric low temperature gaseous Argon plasma and UV radiation was applied on areca fibers are considered for studied. The changes in the surface energy and surface roughness were studied using SEM and EDS. EDS study outcome has confirmed the chemical changes of the areca fibers, while SEM images showed the etching of the surface scales. The plasma and UV treated samples showed better chemical characteristics and surface roughness. The treated areca fiber shows the highly increase in % oxygen content on the surface in comparison with untreated fiber. The results showed that Atmospheric low temperature plasma and UV treatment can be considered as an better pretreatment process, being a dry and clean without the environmental worries related with chemical treatment as well as processing cost is low. These are used to enhance the bonding between the areca fiber and matrix and significantly higher surface roughness range. The thermal stability of the untreated and treated areca fiber is also discussed. TGA showed the increased temperature range with the treatment areca fiber. This will help in selection of the better process as well as matrix material. Thus there is huge opportunity in using these treated fibers in manufacturing methods like injection molding, extrusion using suitable thermoplastic as a matrix to make them more open to industrial applications.

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