

## Synthesis of Alumina Fibre by Annealing Method using Coir Fibre

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### Abstract

The present study report the synthesis of alumina fibre by annealing method using coir fibre. In this study chemical treatment of coir fibre has been done using a nitro compounds at higher temperature. The composite formed after the chemical treatment has been characterized by Xrd, SEM and FTIR technique. Successful synthesis of alumina fibre has been confirmed after the chemical treatment of coir fibre.

**Keywords:** Coir; Annealing; Nitro compound; Alumina fibre

### Introduction

Currently scientist are showing ardent attention in synthesizing composites from recycled waste specially by means of the natural environment-friendly fibres as reinforcing fillers and thermosetting polymers as matrix [1]. Natural fibres play a vital role as fillers in the fabrication of composites. The use of these fibres is beneficial not only as biodegradable substance but also it affects the cost of composite and also boost life of the composites. Natural fibres are being used as a reinforcing agent in place of synthetic fillers such as glass, aramid, talc, and silica; as they are having low density and they do not leave any by-product at the time of manufacture of composites as they are biodegradable in nature. Also, processing of these fibres can be done at low temperature and thus may play an essential role in energy consumption for the production of composites [2-9]. Natural fibres has been used as reinforcing agent in polymer composites and becomes the point of attention and attraction among several researchers during last many years owing to their easy accessibility and biodegradable nature [3,4].

As natural fibres are having low density so they can be used, easily available, renewable and biodegradable in nature. Natural fibres (NFs) have provided raw materials to meet the human requirements of fibres in their life. The first utilization of natural fibre composite (NFC), made with clay in Egypt, can be dated back to 3000 years ago. With the high-tech developments of man-made fibres, NF lost much of its interest and many of the ancient natural fibres are no longer in use. However, as a result of a growing awareness of the interconnectivity of global environmental factors, the principles of sustainability, industrial ecology, eco-efficiency, and green chemistry and engineering are being integrated into the development of the next generation of materials, products, and processes. Though their use as a reinforcing agent is strongly reduced because of their hydrophobic nature, their tendency to amass at the time of processing and their poor resistance to moisture [10]. Chemical treatments has been done for treating natural fibres as the bonding between the fibre and matrix can be improved by physical and chemical modification of fibre surface. A study has been done to show the effect of chemical treatment on the structure and morphology of coir fibre [5]. The present study shows some important effects of chemical treatment on the structure and morphology of coir fibre. The objective of the present study is to optimise overall properties of coir fibre so as to use coir fibre as a reinforcing agent in thermoplastic and thermosetting polymers. In the present study, coir fibre is treated with ferric nitrate salt. A thermal treatment has been done at temperature of 1000°C by using annealing method. X-ray diffraction of the treated coir fibre reveals the crystalline nature of the fibre. Change in morphology

has been found in coir fibre when subjected to scanning electron microscopy. Finally, the Fourier transform and infrared spectrographs show the presence of traces of iron oxide:fibre in the prepared composite. The present study deals with the chemical treatment of coir fibre at higher temperature by nitro compounds and the synthesis of alumina fibre after the treatment of coir fibre has been done using coir fibre.

### Materials and Method

In the present study chemical treatment of coir fibre has been done with aluminum nitrate salt in order to find the change in the structure and morphology of fibre embedded composites.

### Processing of coir fibre

The fibre used for the experimental studies was kept dipped in water for 24 hours and then washed so as to remove impurities like dust etc. On drying in direct sunlight chemical treatment as under was given to it [11].

### Chemical treatment of coir fibre

**Chemical treatment done by aluminum nitrate salt:** Aluminium nitrate [Nonahydrate, Extra pure,  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ] and ammonium chloride ( $\text{NH}_4\text{Cl}$ ) salts were used for the chemical treatment of the fibre. The coir fibre used was collected from the Temples of Bhopal city.

**Process -** In 500 ml of distilled water dissolved aluminum nitrate salt and ammonium chloride salt in the proportion of 10:4 respectively. 100 g of fibre was kept in this solution and then 100 drops of ammonia was poured to it and left it for one hour. After one hour the mixture was dried for 48 hours at room temperature and then annealed the mixture in a muffle furnace at 1000°C. The fired mixture was kept for 15 minutes at that temperature [11].

The scanning electron microscopy of the untreated coir fibre and

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chemically treated coir fibre was carried out by JSM 6390A (JEOL Japan) at different magnifications. Gold coating was done on the samples by gold coating unit before taking it for examination. Figures 1a-1b show the pictorial view of crushed raw coir fibre and chemically treated coir fibre. One can notice from the figures that there is change in the morphology of coir fibre embedded composites by chemical treatment done on it. The Scanning Electron pictographs of crushed to powder form of raw coir fibre shows needle shaped structure at small intervals. It is clear that after chemical treatment of coir fibre done by aluminum nitrate salt the composite material appeared to be in fibrous form and the composite is said to be synthesized alumina fibre as the presence of alumina has been confirmed by Xrd of the composite using coir fibre as template. Similar work has been done by Khan et al., [12] in which synthesis of gamma alumina fibre has been done by bio-replica technique using sisal fibre as template.

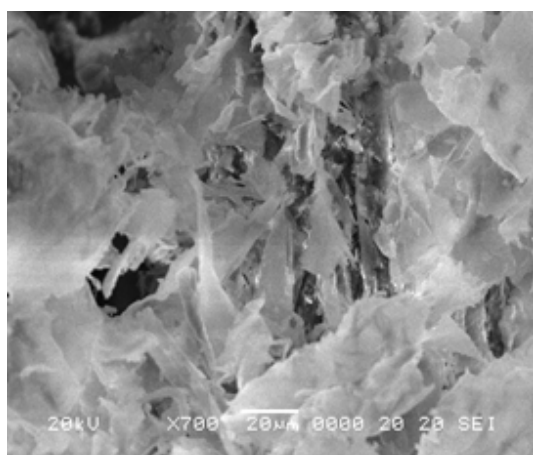
### X-Ray Diffraction Analysis

The XRD measurements were done with the help of Bruker D8 Advance X-ray diffractometer. The X-rays used had wavelength of 0.154 nm (Cu-K $\alpha$ ), produced in a sealed steel tube.

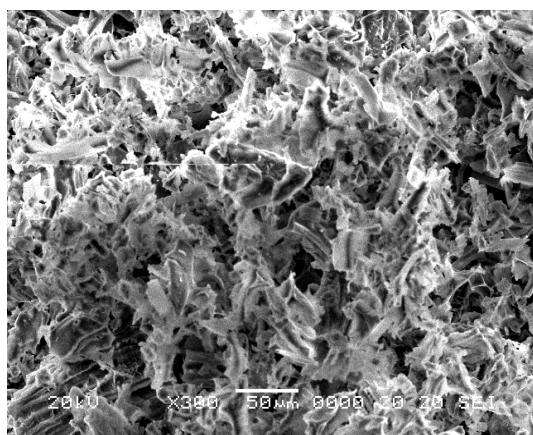
The diffracted X-rays were detected by a fast counting detector based on silicon strip technology (Bruker Lynx Eye Detector). Measurements of diffraction parameters of the samples were carried

out in the range from 5° to 70° (of 2 $\theta$ ) at a scanning rate of 1°/min from Figure 2. It is clear that after the chemical treatment of coir fibre by aluminium nitrate salt, the XRD peaks were obtained at 2 $\theta$  values of 10.60°, 20.06°, 25.44°, 32.82°, 37.00°, 39.48°, 45.72°, 61.08° and at 67.04°. These indicated the extent of crystallinity of the composite. The XRD peaks resembled with the peaks obtained for alumina. Hence we can expect the presence of Al<sub>2</sub>O<sub>3</sub> in the composites. The peaks corresponds to 20.06°, 37.00°, 45.72° and 67.04° resembled the peaks of  $\gamma$ -alumina as Sivadasan et al., [13] reported similar peaks of  $\gamma$ -alumina in their work of synthesis of  $\gamma$ -alumina. They inferred from the studies done by microwave assisted hydrolysis of aluminum metal. While the peaks at 39.48° resembled the peak of  $\alpha$ -alumina as is reported by Hong et al., [14] in their work in which they prepared the precursor sol of alumina by sol gel method by taking aluminum nitrate and malic acid as raw materials [14]. Similar peaks of  $\alpha$ -alumina and  $\gamma$ -alumina found by Guerro et al., [12] fired at 1000°C in their work in which synthesis of  $\gamma$ -alumina fibre has been done using sisal fibre as template [12]. Similar results have been found by other scholars too [15,16]. As the composite material consists of phases of  $\alpha$ -alumina and  $\gamma$ -alumina so the resultant composite may be considered as alumina [12-14].

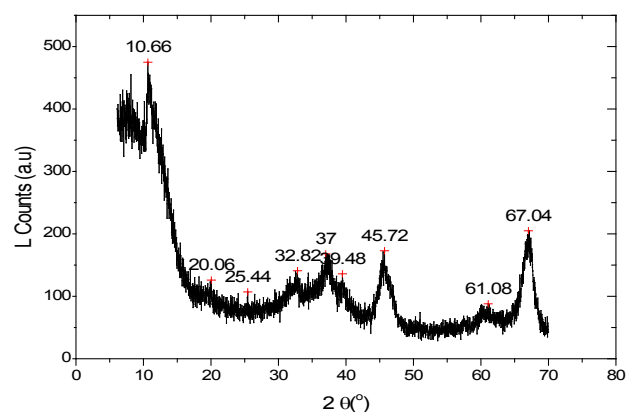
The XRD peaks of composites with raw coir are shown in Figure 3. It shows a peak at 12.9° while after chemical treatment of coir fibre by aluminium nitrate, there is a shift in the peak of coir fibre which is seen at 10.66° after the treatment. Thus, it is obvious from the above discussion that the crystallinity of composites made from coir fibre are affected on chemical treatment. It may thus be said that presence of coir fibre along with the synthesis of alumina affects the crystallinity



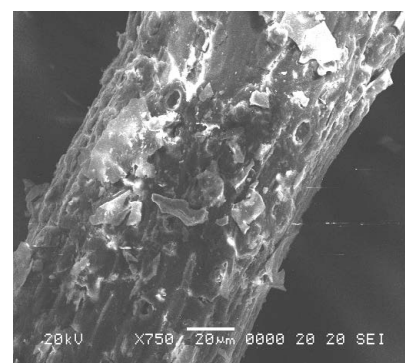
**Figure 1a:** Crushed Raw Coir Fibre.



**Figure 1b:** Synthesized Alumina Fibre.



**Figure 2:** XRD pattern of Alumina (chemical treatment done by aluminum salt).



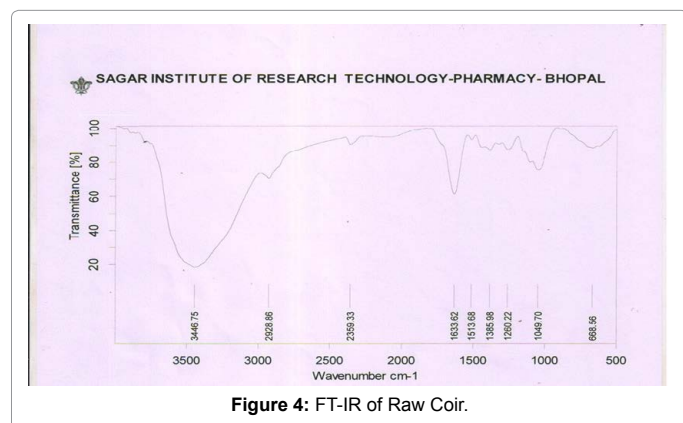
**Figure 3:** XRD of Raw Coir Fibre.

of the final composites. The change in the  $d$  value in composites with coir fibre after chemical treatment may be due to the carbonization of coir fibre.

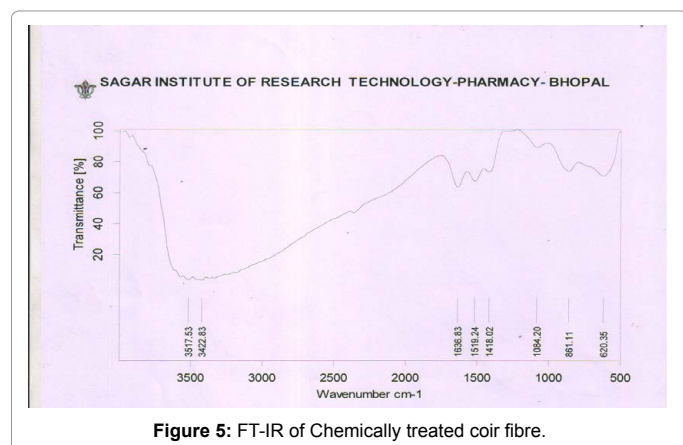
FT-IR analysis was done for our samples. The results are shown in Figure 4. The  $3446\text{ cm}^{-1}$  broad intense peak, in raw coir fibre included sample is due to the O-H stretching for hydrogen-bonded hydroxyl group present in polysaccharides. The broad, strong band emanates from cellulose, hemi-celluloses and lignin present in coir fibre. The weak intensity peak occurring at  $1386\text{ cm}^{-1}$  in raw coir fibre used sample may be due to the presence of hemi-celluloses. It can be assigned to the group of C = O stretching bonds. However, the broad intense peak at  $1049\text{ cm}^{-1}$  is the absorption peak of (C-OH) bonds. The peak at  $2928\text{ cm}^{-1}$  refers to alkyl C-H group. The peak at  $2359\text{ cm}^{-1}$  refers to the presence of water. The peak at  $1633\text{ cm}^{-1}$  in composites with raw coir is due to the C = C aromatic skeletal ring-vibration, which is most likely due to the presence of lignin [17-19].

FT-IR response is as shown in Figure 5. The FT-IR study reveals that the peak  $3446\text{ cm}^{-1}$  shifts to  $3517\text{ cm}^{-1}$  and becomes narrower. It suggests that after the chemical treatment to the fibre, the peak gets narrower due to the reduction of O-H bond, and is shifted due to partial removal of lignin, cellulose and hemi-cellulose. Similar results have been reported by one of the authors of our laboratory [18]. After the chemical treatment the peak at  $1633\text{ cm}^{-1}$  in composite formed from raw coir fibre, shifts to  $1636\text{ cm}^{-1}$ . Hence chemical treatment on fibre can be assumed to be responsible to this kind shift due to structural change.

The other cause can be removal of lignin on chemical treatment.



**Figure 4:** FT-IR of Raw Coir.



**Figure 5:** FT-IR of Chemically treated coir fibre.

However, the broad intense peak at  $1049\text{ cm}^{-1}$  shifts to  $1084\text{ cm}^{-1}$  and becomes narrower due to absorption of (C-OH) bond. However, the peak at  $1418\text{ cm}^{-1}$  refers to Al-OH bonding mode [15,20,21], while the peak at  $620\text{ cm}^{-1}$  and at  $861\text{ cm}^{-1}$ , suggests the presence of stretching mode of Al-O-Al [21,22].

From the above study it is clear that there can be a trace of coir fibre in the sample (may be in oxidized form), which might be carbonized coir fibre, that causes succinct removal of impurities present in the fibre. However the peaks at  $1418\text{ cm}^{-1}$ ,  $620\text{ cm}^{-1}$  and at  $861\text{ cm}^{-1}$  suggest probable presence of  $\text{Al}_2\text{O}_3$  in the sample. So, it can be inferred that there has been successful synthesis of Alumina,  $\text{Al}_2\text{O}_3$  fibre by annealing method.

## Results and Discussions

Present study was carried out with a primary objective of undertaking chemical treatment to coir fibre, to use a waste product by nitro-composites in order to synthesize metal-oxides and to convert a non-degradable waste product into useful product for the welfare of the society. It was specifically done in order to improve the structure and morphology of coir fibre composites. It, in future, can be used as a good replacement of synthetic fibre in polymers. Some more details are given in our recent publications [23-28]. The important findings of the present investigation are as follows:

Satisfactory modification in the morphology of the coir fibre was noticed. The newly formed composites results in the form of alumina fibre may be used in polymers as fillers in place of synthetic fibres so as to monitor the properties of polymers to our advantage. Crystalline nature of the newly synthesized composites by chemically treated coir fibre may help in synthesizing of different composite by different permutations in further studies.

## Conclusions

Chemical treatment prior to composite synthesis is an effective method which is very likely to change the morphology of coir fibre. The treatment done by nitro compounds improves the surface coarseness of the surface of coir fibre in contrast to untreated coir fibre composites. It is clear that after chemical treatment of coir fibre done by aluminum nitrate salt the composite material appeared to be in fibrous form while powdered form of raw coir fibre shows needle shaped structure at small intervals

The XRD studies reveal the crystallinity of the resulting composites. The composite formed by the treatment of aluminium nitrate salt is crystalline in nature. Similar peaks of  $\alpha$ -alumina and  $\gamma$ -gamma alumina was found in the composite material so the resultant composite may be said to be alumina. So, it can be concluded that when coir fibre is treated with aluminum nitrate, alumina fibre is found after the treatment.

FT-IR study done on chemically treated coir fibre (treatment was done by aluminum nitrate) showed traces of coir fibre which may be in the form of carbonized coir fibre. There are various samples in which there was progressive removal of impurities with each successive treatment on the fibre. However, the peaks obtained at  $1418\text{ cm}^{-1}$ ,  $620\text{ cm}^{-1}$  and  $861\text{ cm}^{-1}$  suggest the presence of  $\text{Al}_2\text{O}_3$  in the sample. As a result it can be inferred that the synthesis of alumina ( $\text{Al}_2\text{O}_3$ ) was successful by annealing.

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