

Synthesis of Alumina Fibre by Annealing Method Using Coir Fibre

Alveera Khan^{1*}, Mohammad Ayaz Ahmad², Shirish Joshi¹
and Vyacheslav Lyashenko³

¹Department of Physics, Motilal Vigyan Mahavidyalaya, Bhopal, India.

²Department of Physics, Faculty of Science, University of Tabuk, Tabuk 71491, P.O.Box 741, Saudi Arabia.

³Laboratory "Transfer of Information Technologies in the Risk Reduction Systems", Kharkov National University of Radioelectronics, Kharkov, Ukraine.

Authors' contributions

This work was carried out in collaboration between all authors. Author AK designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author MAA did the Scanning Electron Microscopy (SEM) analysis and share the common ideology and the idea of present work. Author SJ gave a justification for the present research objectives and has managed the analyses of the study. Author VL managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

The present study reports the synthesis of coir fibre treated with alumina salt by using of the annealing method. 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 and has been characterized by X-ray diffraction for the determination of peaks of composites, Scanning Electron Microscope (SEM) and FTIR technique. The successful synthesis of alumina fibre has been confirmed that chemically treated fibre is not fully bonded but is in poor contact with the matrix. Chemically treated fibre could not adhere with the epoxy matrix and therefore interfacial bonding is poor.

*Corresponding author: E-mail: khanalveera@gmail.com, Co-author: E-mail: mayaz.alig@gmail.com;

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1. 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]. The natural fibres have 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].

It was stated by various workers in the field of natural polymers that the natural fibres are having low density, easily available, renewable and biodegradable in nature [10]. 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 [11]. Chemical treatments have 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 deals with the chemical treatment of coir fibre at higher temperature by nitro compounds and the synthesis of alumina fibre (i.e. coir fibre treated with alumina salt) after the treatment of coir fibre has been done using coir fibre.

2. MATERIALS AND METHODS

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.

2.1 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 [12].

2.2 Chemical Treatment of Coir Fibre

2.2.1 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 (NH_4Cl) 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 in the said volume concentration of 500 ml. 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. This muffle furnace [13] is heavy duty vacuum molding ceramic fiber insulation designed to supports heavy load, high temperature and repeated use. And it is used to exposed Kanthal-A1 heating elements offers maximum heat transfer and thermal uniformity, long service life, protection from harmful materials and mechanical shock at maximum temperature up to 1000°C . The fixed mixture was kept for a repeating time interval of 15 minutes at temperature of 700°C [12].

2.2.2 Scanning electron microscopy analysis

The scanning electron microscopy of the untreated coir fibre and 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. The Figs. 1 (a-b) shows 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.

2.2.3 X-ray diffraction analysis

The XRD measurements were done with the help of Bruker D8 Advance X-ray diffractometer [14] a laboratory manual [Bruker D8 Discover User Manual (Version: 2012.08.08)]. The X-rays used had wavelength of 0.154 nm (Cu-K α), 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 θ) at a scanning rate of 1° / min. From Fig. 2. it is clear that after the chemical treatment of coir fibre by aluminium nitrate salt, the XRD peaks were obtained at 2 θ 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 Al₂O₃ in the composites. The peaks corresponds to 20.06°, 37.00°, 45.72° and 67.04° resembled the peaks of γ -alumina as Sivadasan et al. [15] reported similar peaks of γ -alumina in their work of synthesis of γ -alumina. They inferred from the studies done by microwave assisted hydrolysis of aluminum metal. While the peaks at 39.48° resembled the peak of α -alumina as is reported by T. Hong et al. [16] 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 [16]. Similar peaks of α -alumina and γ -alumina found by Benitez-Guerrero et al. [17] fired at 1000°C in their work in which synthesis of γ -alumina fibre has been done using Sisal fibre as template. Similar results have been found by other scholars too [18,19]. As the composite material consists of phases of α -alumina and γ -alumina so the resultant composite may be considered as alumina [15-19].

The XRD peaks of composites with raw coir are shown in Fig. 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 crystallization [20] of composites made from coir fibre is affected on chemical treatment. It may thus be said that presence of coir fibre along with the synthesis of alumina affects the crystallization of the final composites [20]. The change in the *d* value in composites with coir fibre after chemical treatment may be due to the carbonization [21] of coir fibre.

2.2.4 FT-IR analysis

The FT-IR analysis was done for our samples. The results are shown in Fig. 4. The 3446 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 cm⁻¹ in raw coir fibre

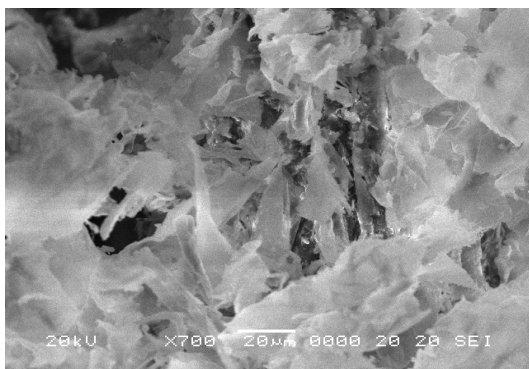
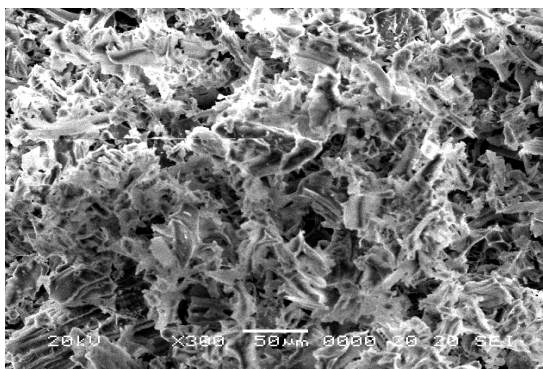


Fig. 1(a). Crushed raw coir fibre



(b) Synthesized alumina fibre

used sample may be due to the presence of hemi-celluloses. It can be assign to the group of C = O stretching bonds. However, the broad intense peak at 1049 cm⁻¹ is the absorption peak of (C-OH) bonds. The peak at 2928 cm⁻¹ refers to alkyl C-H group. The peak at 2359 cm⁻¹ refers to the presence of water. The peak at 1633 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 [22,23,24].

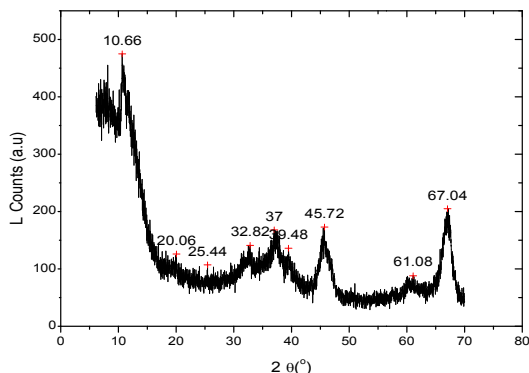


Fig. 2. Xrd pattern of Alumina (chemical treatment done by aluminum salt)

Further, the FT-IR response is as shown in Fig. 5. The FT-IR study reveals that the peak 3446 cm⁻¹ shifts to 3517 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 [25,26]. After the chemical treatment the peak at

1633 cm⁻¹ in composite formed from raw coir fibre, shifts to 1636 cm⁻¹. Hence chemical treatment on fibre can be assumed to be responsible to this kind shift due to structural change.

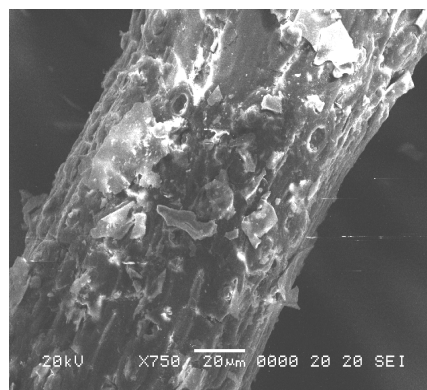


Fig. 3. Xrd of Raw Coir Fibre

The other cause can be removal of lignin on chemical treatment. However, the broad intense peak at 1049 cm⁻¹ shifts to 1084 cm⁻¹ and becomes narrower due to absorption of (C-OH) bond. However, the peak at 1418 cm⁻¹ refers to Al-OH bonding mode [18,24,27-28], while the peak at 620 cm⁻¹ and at 861 cm⁻¹, suggests the presence of stretching mode of Al-O-Al [27,28].

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.

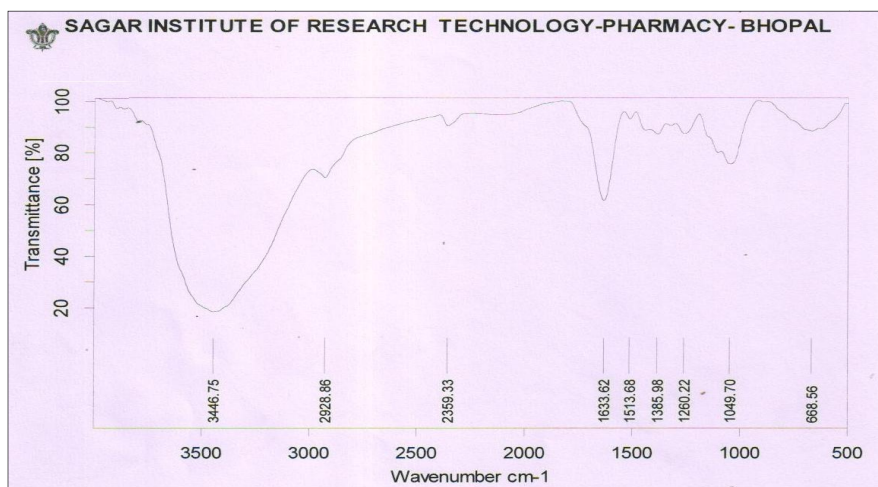


Fig. 4. FT-IR of raw coir

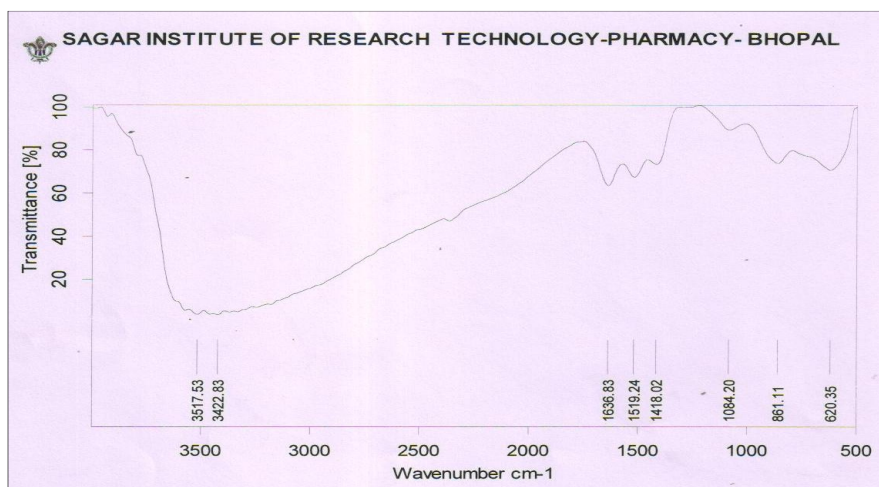


Fig. 5. FT-IR of chemically treated coir fibre

3. RESULTS AND DISCUSSION

The 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. 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.

The crystalline nature [20,21] of the newly synthesized composites by chemically treated coir fibre may help in synthesizing of different composite by different permutations in further studies.

4. CONCLUSIONS

However in the present research work one can find that the peaks at 1418 cm^{-1} , 620 cm^{-1} and at 861 cm^{-1} suggest probable presence of Al_2O_3 in the sample [17-24]. So, it can be inferred that

there has been successful synthesis of Alumina, Al_2O_3 fibre by annealing method.

Chemical treatment prior to composite synthesis is an effective method which is very likely to change the morphology of coir fibre [29-35]. 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 crystallization of the resulting composites. The composite formed by the treatment of aluminium nitrate salt is crystalline in nature. Similar peaks of α -alumina and γ -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. The sample in which there was progressive removal of impurities with each successive treatment on the fibre.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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