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CHARACTERIZATION OF COIR FIBERS AFTER ALKALI AND MICROWAVE TREATMENTS

B. Bakri[1], Naharuddin[1], A. E. E. Putra[2], I. Renreng[2] and H. Arsyad[2]
[1]Mechanical Engineering Department, Tadulako University, Palu, Indonesia
[2]Mechanical Engineering Department, Hasanuddin University, Gowa, Indonesia
E-Mail: bakri@untad.ac.id

ABSTRACT

Coir fibers have been used as reinforcement of composite. To improve adhesion with matrix, modification of surface fibers has been performed with chemical treatment and physical treatment. In this paper, alkali and microwave treatments were implemented to characterize of coir fibers. The characterizations of treated coir fibers by using Fourier transform infrared (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) were studied. Tensile strength of treated coir fibers was also evaluated. The alkali and microwave treatment influenced chemical composition and crystallinity index of coir fiber based on characterization of FTIR and XRD respectively. Mechanical property of coir fiber tends to improve after treatment. Then, the surface roughness of treated coir fiber appeared which may enhance the interfacial adhesion of coir fiber with matrix.

Keywords: coir fiber, alkali treatment, irradiation microwave, tensile strength.

INTRODUCTION

Coir fiber is a natural fiber which has been used as reinforcement of composite. The main components of coir fiber consist of cellulose, hemicellulose and lignin (Abraham *et al*., 2013). Then, Mohanty *et al*., (2000) reported that chemical compositions of coir fiber are 36-43 wt% of cellulose, 0.15-0.25 wt% of hemicellulose and 41-45 wt% of lignin. These components of coir fiber influence its mechanical property (Mohanty *et al*., 2000). Mechanical properties of coir fiber have been studied by some researchers (Kulkarni *et al*., 1981, Silva *et al*., 2000, Bakri and Eichhorn, 2010). Such fiber has low strength and elastic modulus, but it has high elongation. Related to application on composite, it has a disadvantage in compatibility with hydrophobic matrix due to hydrophilic behaviour (Jayavani *et al.*, 2015). Proper surface modification of natural fiber can increase the interfacial fiber-matrix bonding (Borchani *et al*., 2015, Mohanty *et al.*, 2001).

Several processes used to improve adhesion between coir fiber and matrix is chemical and physical treatments. Alkali treatment of coir fibers has been investigated by Nam *et al*., (2011). Its results showed that tensile strength of coir and interfacial shear strength of coir fiber/PBS matrix increased after alkali treatment. In addition, Mohanty *et al*., (2001) has reported that improving interfacial adhesion between fiber-matrix in composite can be achieved by alkali treatment, isocyanate treatment, peroxide treatment, vinyl grafting, bleaching, acetylation, and combination of treatment and coupling agents. Then, plasma treatment is one of physical treatment for surface modification of natural fiber. Praveen *et al*., (2016) has been investigated the effect of plasma treatment on coir fibers. Their results illustrated that after plasma treatment, the surface of coir fiber seem difference of untreated coir fiber and also occurs etching of fiber wall.

Irradiation microwave treatment of natural fiber has been used by Islam *et al*., (2015). Microwave treatment process is claimed as a shorter time treatment than other treatment. It uses radiation energy for exposing substances of natural fiber by heating the environment. This treatment may remove lignin, wax and surface impurities (Islam *et al*., 2015). Then, irradiation microwave has been done for fibrous flax retting (Nair *et al*., 2013) and for extraction fiber (Qu *et al*., 2014). In addition, microwave treatment has been applied in dyeing of wool fabric which can improve its dyeability (Xue, 2015). Irradiation microwave was also used by Islam *et al*., (2015) for surface modification of empty fruit bunch (EFB) fiber. Their results showed that microwave treatment of EFB fiber can get better the mechanical and thermal properties of fiber based composite. The authors also studied irradiation microwave of coir fiber without combination of chemical treatment (Bakri *et al*., 2017a).

This paper focuses on characterization of coir fiber after alkali and microwave treatments. The characterization of coir fiber before and after alkali and microwave treatments was performed by using Fourier transform infrared, X-ray diffraction and scanning electron microscopy. Tensile testing was also used for evaluation of mechanical property of treated coir fiber.

MATERIALS AND METHODS

Coir fibers were extracted from coconut husk. Fibers were washed and cleaned with water to remove surface impurities. Coir fibers then were soaked in sodium hydroxide (NaOH) solution 5 wt% for 24 hours. After soaking, fibers were rinsed and followed by drying in temperature room during 48 hours. In this research, alkali and microwave treatment of coir fibers were divided into three categories. Firstly, treated coir fibers of sodium hydroxide were exposed in the microwave oven with irradiation microwave at different times (10 minute (NM10), 20 minute (NM20) and 30 minute (NM30)) without heating in the oven. Secondly, treated coir fibers of sodium hydroxide were heated in the oven during 2 hours at 100 ºC (2N). Lastly, treated coir fibers of sodium

hydroxide were heated in the oven during 2 hours at 100ºC followed by exposing in the microwave oven with irradiation microwave at different times (10 minute (2NM10), 20 minute (2NM20) and 30 minute (2NM30)). Microwave oven was set with 100% power for all experiments.

Characterizations of coir fiber include Fourier transform infrared (FTIR), X-ray diffraction (XRD), tensile testing and scanning electron microscopy (SEM).

FTIR characterization was performed by using IRPrestige-21 FTIR-8400S SHIMADZU. A small portion of coir fiber powder was taken for FTIR spectroscopy, ground with KBr reagent (KBr: coir fiber powder = 10:1) using mortar and pestle. Then, the mixture was formed into pellet using a dice and hand press machine. Then, the pellet was placed on the sample holder. The IR spectrum was obtained and then analyzed.

XRD analysis was used to determine crystallinity index of raw and treated coir fibers. XRD-7000 X-Ray Diffraction SHIMADZU was used in this analysis. To determine crystallinity index (CI), Segal method was used as in equation 1 (Chandrasekar *et al*., 2017):

$$CI\ (\%) = \frac{I_{002} - I_{am}}{I_{002}} \quad (1)$$

where I_{002} is the maximum intensity diffraction of peak and I_{am} is the minimum intensity diffraction of peak.

Tensile testing of coir fiber was performed to identify mechanical property of coir fiber. Universal Testing Machine – Llyod L10K Plus was used for tensile testing and based on ASTM 3379. The gauge length of fiber specimen was 30 mm and strain rate was set at 2.5 mm/min.

Surface morphology of coir fiber was characterized by scanning electron microscopy. SEM – JEOL JSM 6510 LA type was used in this study.

RESULTS AND DISCUSSIONS

Fourier transforms infrared analysis

FTIR spectra of the treated alkali and microwave samples were shown in Figure-1 and Figure-2.

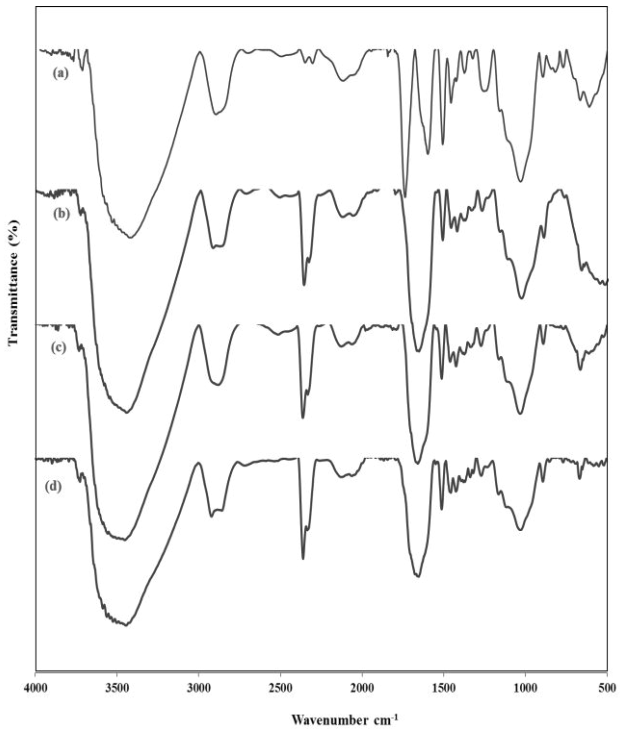

Figure-1. FTIR spectra of coir fibers (a) Raw, (b) NM10, (c) NM20 and (d) NM30.

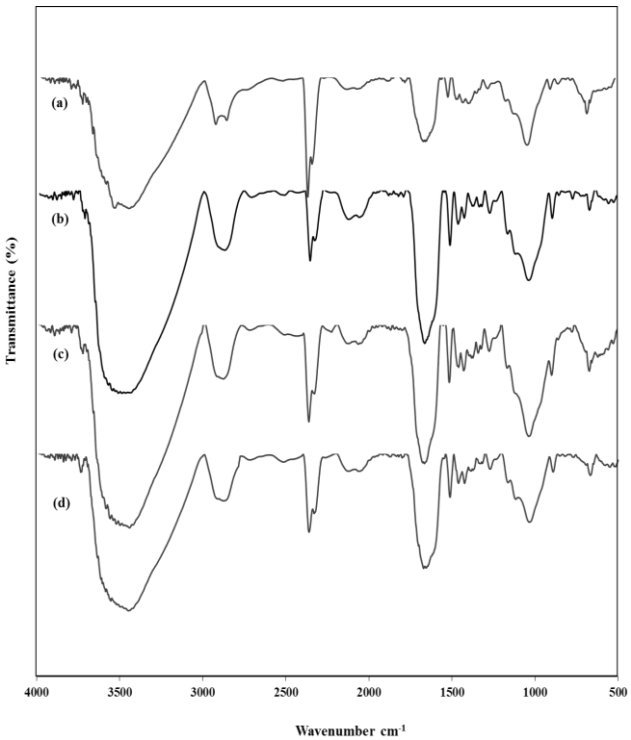

Figure-2. FTIR spectra of coir fibers (a) 2N, (b) 2NM10, (c) 2NM20 and (d) 2NM30.

Raw coir fiber spectra was published by authors (Bakri *et al.*, 2017b). Raw and treated samples spectra seemed shifting of peak intensity from 3442 cm^{-1} to 3450 cm^{-1}. This peak indicated some free OH groups contribution in chemical reaction (Samal *et al*., 1995).

This peak intensity of coir fiber was similar with previous study (Mir *et al.*, 2012). Then, the 2912 cm^{-1} band is a characteristic band for C-H stretching vibration of cellulose/hemicellulose (Samal *et al.*, 1995). This band shift after treatment of alkali and microwave with peak at 2918 cm^{-1} for NM10 and 2N samples, at 2922 cm^{-1} for NM30, and at 2903 cm^{-1} for 2NM10 sample. However, samples of NM20, 2NM20 and 2NM30 shifted to peak at 2879 cm^{-1}, at 2876 cm^{-1} and at 2874 cm^{-1} respectively which indicated OH stretching vibration of inter-and intramolecular hydrogen bonded OH groups (Samal *et al.*, 1995). Peak at 1740 cm^{-1} of coir fiber is corresponding to the aromatic skeletal vibration and carbonyl group where lignin and hemicelluloses probably exist (Mir *et al.*, 2012, Abraham *et al.*, 2013). This peak is related to peak at 1743 cm^{-1} of coir fiber in this research. But, this peak disappears after alkali and microwave treatments.

X-ray diffraction analysis

X-ray diffraction results of raw and treated coir fibers with alkali and microwave treatment without heating in the oven (NM) were showed in Figure-3. In addition, treated coir fibers with alkali treatment followed by heating (2N) and followed by microwave treatment (2NM) were shown in Figure-4. In Table-1, crystallinity index increases after alkali and microwave treatment but it decreases after alkali and microwave treatment with 30 minute exposing with irradiation microwave. Raw coir fiber of crystallinity index has been previous published by authors. Decreasing crystallinity index may cause by destruction to the cell wall (Mwaikambo and Ansell, 2002).

Table-1. Crystallinity index of coir fibers.

Samples	Crystallinity index (%)
Raw	35.02
NM10	43.09
NM20	37.02
NM30	32.05
2N	36.37
2NM10	36.08
2NM20	39.31
2NM30	41.08

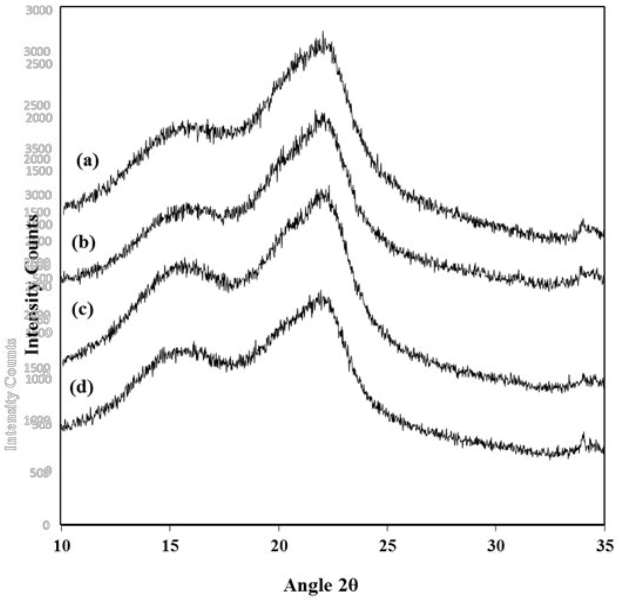

Figure-3. XRD of coir fibers (a) Raw, (b) NM10, (c) NM20 and (d) NM30.

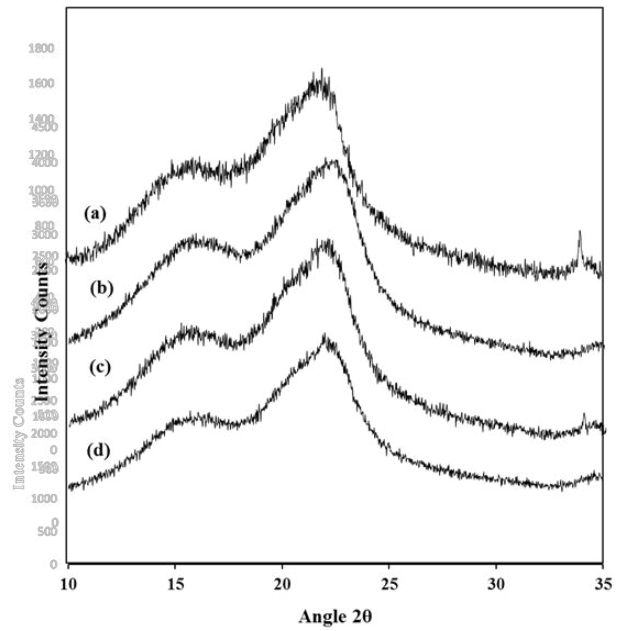

Figure-4. XRD of coir fibers (a) 2N, (b) 2NM10, (c) 2NM20 and (d) 2NM30.

Tensile strength of coir fiber after alkali and microwave treatment

Figure-5 display tensile strength of raw and treated coir fiber. Tensile strength increased significantly after alkali treatment followed by microwave treatment with irradiation microwave during 10 minute (NM10 sample) and after alkali treatment with heating during 2 hours (sample 2N). The increasing percentages of tensile strength from untreated coir fiber (raw) are 62% and 42% respectively. Then, 2NM10 and 2NM20 samples were also increasing compared to raw coir fiber. This results were inverse with alkali treatment of coir fibers which were

reported by Gu (2009) and Karthikeyan *et al*., (2014), whereas Samal *et al*., (1995) and Nam *et al.*, (2011) studied that alkali treatment of coir fiber can increase the tensile strength. Tensile strength of NM20, NM30 and 2NM30 samples reduced compared to raw sample. Long exposure time with irradiation microwave in the microwave oven may also cause reducing of these strengths.

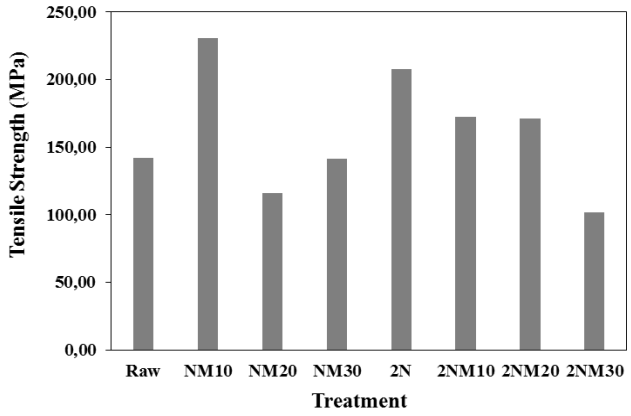

Figure-5. Tensile strength of raw and treated coir fibers.

Scanning electron microscopy of surface coir fibers

Raw of coir fiber has been published by authors (Bakri *et al*., 2017a). Figure-6 and Figure-7 exhibited the SEM images of surface morphology of raw and treated coir.

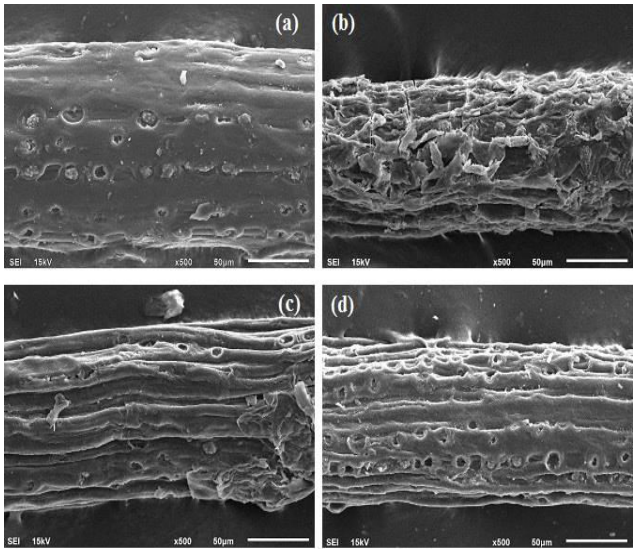

Figure-6. SEM images of coir fibers (a) Raw, (b) NM10, (c) NM20 and (d) NM30.

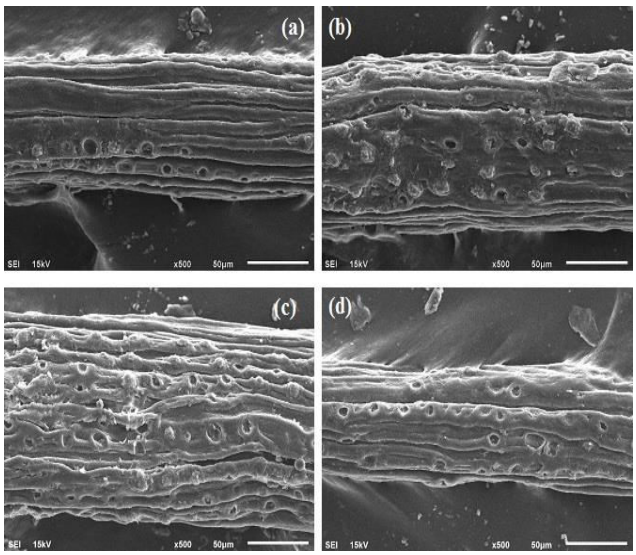

Figure-7. SEM images of coir fibers (a) 2N, (b) 2NM10, (c) 2NM20 and (d) 2NM30.

Coir fiber after alkali and microwave treatment display micropores and more rough compared with raw coir fiber. The surface roughness of coir fiber may induce in better adhesion between fiber and matrix due to mechanical interlocking occurred (Rahman and Khan, 2007; Karthikeyan *et al*., 2014). NM10 sample image was seemed more rough which may correspond to the highest crystallinity index and tensile strength compared with other samples. The surface roughness of treated coir fibers may enhance the interfacial adhesion of coir fiber with matrix.

CONCLUSIONS

The characterization results of raw and treated coir fiber were concluded that alkali and microwave treatment of coir fiber shifted the peak intensities which indicated contribution of chemical reaction in the component of coir fiber. In addition, treated coir fibers tend to increase crystallinity index, whereas crystallinity index decrease after alkali and microwave treatment for 30 minute irradiation microwave of coir fiber. Tensile strength of coir fiber after alkali and microwave treatment with exposing during 10 minute increase significantly about 62% from raw coir fiber. This result was supported by SEM image. Surface morphology of coir fiber after treatment seem rougher than raw fiber. The surface roughness of treated coir fibers may enhance the interfacial adhesion of coir fiber with matrix.

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