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The effect of alkalization and bleaching treatment of Sorghum fibre on the crystallinity index of PP composite

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Abstract. Natural fibres are increasingly being employed as the reinforcing material for polypropylene (PP). However, the fibre surface needs to be modified with particular treatment to improve the interfacial adhesion and thus mechanical properties of the composite. The aim of this work was to study the effect of alkalization and bleaching treatment to the structure of sorghum fibre and how it affects the crystallinity of polypropylene (PP)-sorghum composite. For the alkalization treatment, the fibre was soaked in NaOH solution 10% v/v while for the bleaching treatment NaClO₂ solution 1.7% v/v was used. The FTIR and SEM analysis of the sorghum fibres indicated that the alkalization and bleaching process eroded some part of amorphous structure in the fibre. It was found that using the treated fibre as the reinforcing agent in PP matrix could increase the crystallinity of the resulting composite compared to the crystallinity of pure PP.

Keywords: sorghum fibre, alkalization, bleaching, crystallinity

1. Introduction

Polypropylene (PP) has several attractive features that promote it to be one of the most used polymers for wide range of applications. However, it has low impact strength which can be improved by reinforcing it with fibres. Natural fibres are increasingly being employed as the reinforcing material for PP in place of glass fibre due to its lower price, better recyclability, hygienic harmless and a carbon dioxide neutral combustion [1]. Bamboo, sugar cane bagasse, jute, kenaf, coir, flax, silk, hemp and grass are some of the most common natural fibres that have been utilized commercially [2-4]. The hygroscopic nature of the natural fibres, however, is the biggest challenge for their use as reinforcement in polymers. During lifetime, polymers are exposed to various climatic conditions, including unsteady hygroscopic environments. The absorption of water by these fibres will result in volume increase of the composite and could lead to internal stress. On the other hand, during polymerization at temperature higher than 100°C, water molecules that are trapped inside fibres will evaporate thus results in composite shrinkage [1]. Petro based polymers, such as PP, are hydrophobic in nature, making them incompatible with natural fibres. This incompatibility results in poor adhesion between the two materials hence failing the stress transfer from the polymer matrix to the reinforcing fibre [5].



Enhancing interface quality between natural fibres and the petro-polymer matrix is one of the largest areas of current research [6]. Fibre surface needs to be modified with particular treatment to improve the interfacial adhesion and accordingly the mechanical properties of composites [7]. Several treatments have been investigated to reduce the hydrophilicity of natural fibres [8]. Chemical treatment by dissolving natural fibre in alkaline solution, e.g. NaOH, is said to be one of the most used method for this purpose [3, 5]. The aim of this treatment is to remove lignin and impurities covering the surface of the fibre cell wall [9]. Alkalization by NaOH solution could also disrupts the hydrogen bonding in hemicelluloses and amorphous regions of the cellulose which are generally responsible for the hydrophilic nature of the fibres [10]. The interfacial adhesion between polymers and natural fibres can be further improved, and at the same time enhance the physical appearance of the composite, by bleaching treatment using sodium chlorite (NaClO_2) solution [11].

This work aims to study how the alkalization and bleaching process will affect the surface properties of sorghum (*Sorghum bicolor* L.) and how it will improve the crystallinity of PP based composite. There are few similar studies that have been conducted previously [11-15]. Ismojo *et al.* [11] used 4% v/v NaOH solution and 1.7% v/v NaClO_2 solution for the alkalization and bleaching process respectively. However, in that study the modified sorghum fibre was not applied as the reinforcing material in any polymer matrix.

In this present work the alkalization will be executed for 2 hours using 10% v/v NaOH solution, followed by bleaching treatment for 4 hours using 1.7% v/v NaClO_2 solution buffered with acetic acid. Afterward the treated sorghum substrate was blended with PP matrix. The analysis results show that this chemical treatment sequence could enhance the crystallinity of the PP-sorghum composite.

2. Materials and Methods

2.1. Material

The sorghum fibre was obtained from a traditional market in Bogor, West Java. Sodium hydroxide (NaOH) in pellets form, CH_3COOH 0.2 M and 1.7% v/v NaClO_2 solution was purchased from Merck. Experimental

2.2. Sample preparations

The sorghum fibres were washed and dried in open air. The size of the sorghum fibres was reduced using crushing machine and thus sieved to obtain 40 mesh fibres.

2.3. Chemical treatments

In alkalization treatment the sorghum fibres were soaked in 10% (v/v) NaOH solution in agitated condition for 2 h at 70°C in one cycle. The treatment was continued with bleaching which was carried out by soaking the alkalized fibres in 1.7% (v/v) NaClO_2 solution, buffered with CH_3COOH 0.2 M (pH 4.5) solution in agitated condition for 4 h. After the alkalization and bleaching treatment were completed, the substrates were washed three times with tap water and then dried at room temperature for 3 days. The substrates were carefully kept in a dry container prior to characterizations.

2.4. Preparing the PP-sorghum composite

The PP-sorghum composite was prepared using hot melt mixing method. The mass ratio of chemically treated sorghum/PP was 0.0 (pure PP), and 0.05. The PP was first melted at 165°C for 5 min and then mixed with the fibre. The mixture was further heated at same temperature and stirred for another 10 min. This cycle was repeated twice. The composite was cut into small pieces for differential scanning calorimetry (DSC) analysis.

2.5. Characterizations

There are three instruments used for characterizations in this study namely Scanning Electron Microscopy (SEM) for studying the morphology of PP-sorghum composite, Fourier-Transform Infrared

(FT-IR) for analysing the chemical composition of the untreated and treated sorghum fibre, and Shimadzu XRD-7000 X-Ray Diffraction for analysing the crystal structure of the fibre qualitatively. Results from XRD analysis were processed for quantifying the crystallinity of the sorghum fibre using the following equation [11].

$$CI(\%) = \frac{I_{(002)} - I_{(amp)}}{I_{(002)}} = 100$$

CI is Crystallinity Index while $I_{(002)}$ is the maximum intensity located at angle around $2\theta = 22^\circ$. $I_{(amp)}$ is the intensity scattered by amorphous part of the sample at the diffraction angle around $2\theta = 18^\circ$.

3. Results and Discussion

3.1. Functional group analysis

FTIR analysis was conducted to compare the characteristics of the sorghum fibres, before and after receiving chemical treatments. As presented in Fig. 1, in the virgin fibre spectrum there are four distinguished peaks detected at relatively high transmittance. After receiving alkalization treatment, those peaks are shifted to lower transmittance while at the same time new peaks are discovered. Bleaching treatment however shifted back the spectrum to higher transmittance but the peaks are remained. Table 1 lists all peaks shown in Fig. 1 with the corresponding functional groups.

The alkalization treatment removed wax, impurities lignin and small part of hemicellulose. Hence some functional groups that exist in hemicellulose, pectin and cellulose, such as C-H, O-H carboxylic acid, and C-O aromatic ester, are more exposed to the light. The lower transmittance in ‘‘Alkali’’ spectrum confirms that there was an increase in the concentration of these groups which consequently rising the amount of light absorbed.

The bleaching treatment eroded most part of lignin, hemicellulose, and pectin. There are two peaks that is no longer visible after this treatment, i.e. O-H carboxylic acid and O-H phenol. These two groups belong to pectin and lignin respectively. The other peaks that were revealed after alkalization treatment are retained in the ‘Bleach’ spectrum but at higher transmittance. It shows that there was a decline in the concentration of these groups thus the amount of light absorbed is decreasing.

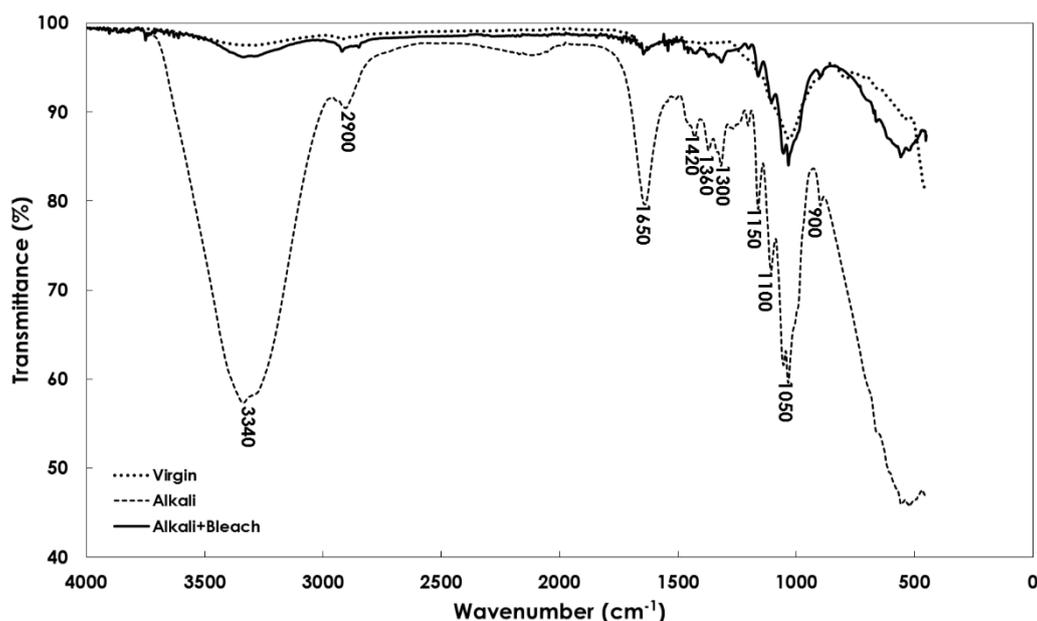


Figure 1. FTIR spectra for untreated (virgin) and treated sorghum fibre

Table 1. Existing peaks in FTIR spectra for untreated and treated sorghum fibre

Wavenumber (cm ⁻¹)			Functional Group
Virgin	Alkali	Alkali + Bleach	
3300	3340	3340	O-H, stretching, alcohol
2920	2900	2920	C-H, stretching, alkane
1650	1650	1650	C=C, stretch, cyclic alkene
	1420		O-H, bending, carboxylic acid
	1360		O-H, bending, phenol
	1300	1300	C-O, stretching, aromatic ester
	1150	1150	C-O, stretching, aliphatic ether
	1100	1100	C-O, stretching, aliphatic ether
1050	1050	1050	C-O, stretching, primary alcohol
	900	900	C=C, bending, alkene

3.2. Morphology analysis

The physical changes of the sorghum fibre after the chemical treatment sequence was observed using scanning electron microscopy (SEM) analysis. Figure 2 clearly shows the significant change in the physical appearance of the fibre surface. SEM image in Figure 2(a) reveals that the surface of the untreated sorghum fibre is relatively rough because it is still covered by wax and impurities. Another point of observation is that the surface is still containing layers of lignin that bind the cellulosic fibres creating somewhat larger bundles.

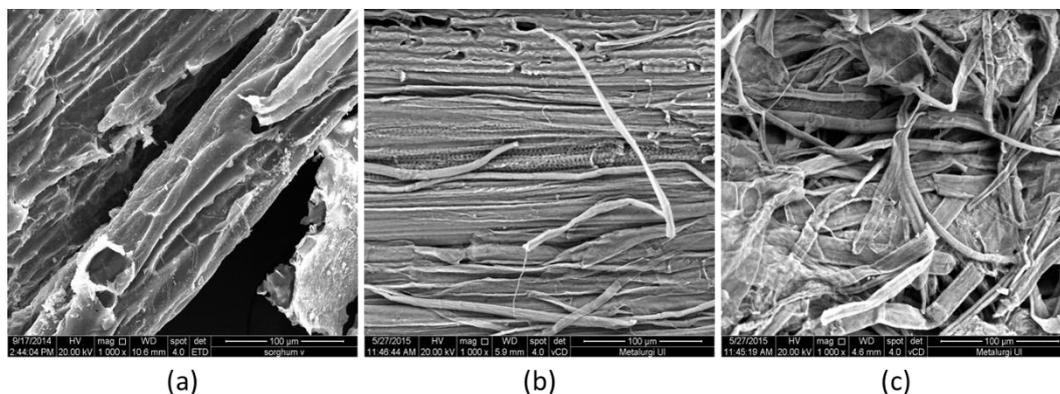


Figure 2. SEM Images of Sorghum Fibre with (a) No Treatment, (b) Alkalization, and (c) Alkalization and Bleaching Treatment at 1000x magnification

The 10% (v/v) NaOH washed the impurities, lignin and some of hemicellulose in the alkalization treatment which transformed the fibre surface into a cleaner look (Fig. 2(b)). In the figure it can be observed that single fibres are now visible but still attached to each other in a larger bundle. It means that the hemicelluloses are not fully removed in the alkalization treatment, which is also supported by result from FTIR analysis. Further treatment using NaOCl₂ 1.7% (v/v) in the bleaching treatment was able to separate those fibres, as presented in Fig. 2(c). Defibrillation shown in the figure indicates that this treatment has effectively reduced most part of amorphous part e.g. lignin and pectin that tied the fibres. This hypothesis is confirmed by the absence of particular peaks that belong to this component.

3.3. PP composite crystallinity

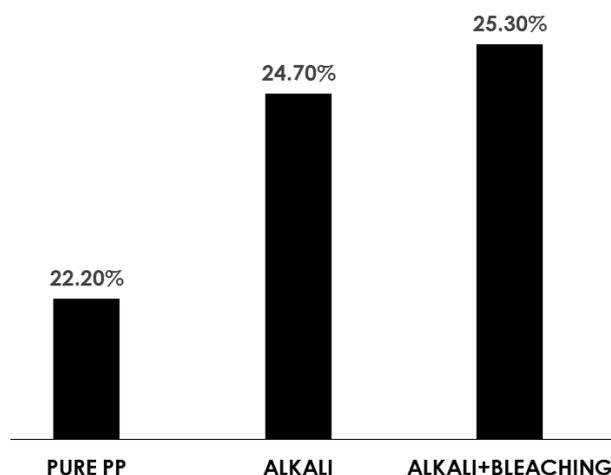


Figure 3. Crystallinity index comparison between pure PP and PP-sorghum composite

The crystallinity indices of PP-sorghum composites produced from different treatment are in agreement with FTIR and SEM analyses. Figure 3 compares the crystallinity index between pure PP and PP composites in which the sorghum fibre had received alkalization and alkalization-bleaching treatment. NaOH solution removed lignin and small part of hemicellulose, which are the building block of amorphous region in the fibre. Therefore, when this treated fibre was used as the reinforcing agent in PP matrix, the crystallinity of the resulting composite is increasing compared to the crystallinity of pure PP. Adding bleaching treatment slightly increase the crystallinity of the composite compared to the result from alkalization treatment. This could mean that the operating condition in bleaching treatment was not optimal enough to remove most part of amorphous region in the fibre. Another possible explanation is that the ratio between chemically treated fibres to PP was not sufficient.

4. Conclusions

The morphological analysis of the sorghum fibres indicated that the alkalization and bleaching process removed wax, impurities, most part of lignin, hemicellulose and pectin from the surface of the fibre. Using FTIR analysis, we found that some particular peaks are absent from the spectrum confirming that the treatment eroded some part of those components. This is also supported by SEM images that expose the clean surface of defibrillated cellulose fibres, indicating that most of amorphous regions have been removed. The composites were obtained by incorporating the treated fibres into the PP matrix in a hot melt mixing process. It was found that using the treated fibre as the reinforcing agent in PP matrix could increase the crystallinity of the resulting composite compared to the crystallinity of pure PP.

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