Chemsearch Journal 4(1): 42 – 46, June, 2013
Publication of Chemical Society of Nigeria, Kano Chapter

Received: 23/05/2013 Accepted: 28/06/2013

Effects of Treated Wood Flour on Physico-Mechanical Properties of Filled Natural Rubber

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ABSTRACT
Wood flour was crushed in to particle size and given two surface treatments each with alkali and 3-chloro-2-hydroxylpropyltrimethylammoniumchloride. The raw, alkali-treated and bonding agent treated fibers were used as natural rubber composites. The samples were used to produce fiber-reinforced natural rubber composite at varying filler loadings. Properties such as tensile strength, hardness and impact resistance of the composites were investigated. The tensile strength of the composites varied such that both the alkali-treated and cationized fillers recorded higher values than the untreated fillers. The impact strength and hardness properties were also found to be better in the modified than the untreated ones. This work has shown some general improvements arising from causticization and cationization of cellulosic filler as reinforcing material for natural rubber.

Keywords: Cationization, Causticization, Mechanical properties, Natural rubber, Wood flour.

INTRODUCTION
Natural rubber, also been called 'the supreme agricultural colonist of all times', is originally indigenous from the Amazon Valley forest. It has been cultivated principally in Southeast Asia, especially for countries like Malaysia and Indonesia, where more than fourteen million acres of land have been cleared and planted with rubber trees (Allen and Mullins, 1967). A considerable amount of research has been done in the field of fiber reinforced elastomer composites. Researchers have studied the effect of different fibers in natural and synthetic rubber, for most of these researches petroleum-based resources have been used and which are however, non-biodegradable and their disposal contributes to many environmental problems and this prompt researchers to develop biodegradable materials (Abdulhamid *et al.,* 2010). The investigation of physico-mechanical properties carried out on natural rubber-coconut fiber composites, proved that coconut fiber is potential reinforcing filler for natural rubber compounds (Akinlabi *et al.,* 2011). Palm kernel, wheat husk were also established to be good reinforcing fillers for natural rubber compounds (Egwaikhide, 2007).

Previous work also indicated that the use of lignocellulosic fibers as fillers can improve the properties of polymers (Abdulhamid *et al.,* 2010), and this research work therefore work intends to explore the possibilities of using wood flour cellulose fibers as potential reinforcement in natural rubber. The most important parameters that affect the fiber-reinforcement are fiber loading, fiber dispersion, fiber orientation and adhesion between the fiber and the matrix (Greethamma and Thomas, 1996).

Natural cellulose fiber has the potential to be an attractive alternative to synthetic fiber and is currently being explored in sectors such as the automobile and building industries. Natural fibers have advantages over synthetic fibers because of their renewable nature, low cost, biodegradability and ease of chemical modification (Kalia *et al.,* 2011). Effects of various fibers in both natural and synthetic rubbers were studied. And for most of these studies, petroleum-based resources have been used, which are however, non-degradable and whose disposal contributes to many environmental problems. This prompted researchers to developing biodegradable materials, as opined by Abdulhamid *et al.,* (2010) as a better alternative.

MATERIALS AND METHODS
The reagents used were prepared using standard analytical methods of preparation. The cationizing agent (3-chloro-2-hydroxylpropyltrimethylammoniumchloride) used was obtained from Sigma Aldrich and used as received.

Materials
Crumb natural rubber which conforms to technically specified rubber (TRS) was obtained from Rubber Research Institute of Nigeria (RRIN),

Ayonomo, Benin City Edo State. The wood flour was obtained from Madobi, Madobi Local Government Area, Kano State.

Sample preparation

The samples were prepared by grinding using a cleaned and dried grinding machine followed by sieving to the particle size of 1mm.

Moisture content determination

One gram of each sample was put in a watch glass and kept at room temperature for 48hours. The watch glass containing the sample was recorded as initial weight (W_1). They were placed in an oven and maintained 100^0C and continued to be weighed at regular intervals of 30minutes till a constant weight was recorded. The weight was recorded as the final weight (W_2). The moisture content was determined using the relation:

$$\% \, Moisture = \frac{W_1 - W_2}{W_1} \text{ x } 100$$

Where W_1 = initial weight, and W = final weight (Yakubu *et al.,* 2010).

Causticization (Alkali treatment)

Five hundred gram of 1mm size sample was caustisized by the use of 20% (owf) sodiumhydroxide solution for 24hours at room temperature after dilution with 7 liters of distilled water. The wetted fibers were squeezed to remove excess water and then washed repeatedly to remove excess NaOH. It was finally dried in an oven at 80^0C for 5hours, and pounded to 120µm particle size (Yakubu *et al.,* 2010).

Cationization

Five hundred gram of the caustisized fiber were cationized by the bonding agent 65% 3-chloro-2-hydroxylpropyltrimethylammoniumchloride at 10% on weight of the fibre in the presence of 10.92% NaOH after dilution with 7.8 liters distilled water. The mixture was kept at room temperature for 24hours maintaining the pH of 12 by periodic addition of the 10.92% NaOH and agitated. The cationized fiber was washed repeatedly to remove the excess NaOH and the bonding agent. The wet fibers were put in an oven at 80^0C for 5hours to dry and pounded to 120µm particle size (Yakubu *et al.*, 2010).

Compounding

The recipe used in the formulation of the natural rubber composites is given in the table below. Mixing was carried out on a laboratory two-roll-mill in accordance with the method described in the American Society for Testing and Materials (ASTM-D3184-80). Cured samples produced on electrically heated press at 150^0C for 40minutes under a pressure of 7torrs.

Table 1: Typical formulation of rubber compounds

Ingredients	Quantity (pph)
Natural rubber	100
Filler	0-50
Zinc oxide	5.0
Stearic acid	1.5
Sulphur	2.5
MBTS	0.5

MBTS = Mercaptobenzthiazoledisulide

Determination of Physico-mechanical Properties

Tensile Strength

Tensile properties of the control and cured composites were measured with Instron universal tester at cross-head speed of 60mmmin^{-1}using dumb bell shaped test specimens as contained in ASTM-D412-87

Impact Test

The impact test was carried out using Resil compactor, model 16650 type 6957 in accordance with the British standard BSEN 6603-2. The total impactor mass used is 1.00kg.

Hardness

Hardness of the cured composites was measured in shore A, by Durometer instrument, model 5019. The measurement was in accordance with ASTM-D2240.

Fourier Transform Infra red Spectroscopy (FTIR)

FTIIR spectra of the compounded Wood flour-Natural rubber were recorded on 8400s Fourier transform infrared spectrophotometer using KBr pellet technique in the range 4500 – 400cm^{-1} with a resolution of 2cm -^{1}.

RESULTS AND DISCUSSION

The alkali treatment (causticization) removed pectin and other soluble carbohydrates like hemi-cellulose, leaving behind the alkali resistance cellulose. This exposes the hydroxyl group of the fiber, cause removal of lignin, and increase bonding sites in the fiber interface thereby making the surface of the fibre more reactive.

The mechanism of the cationization is as shown in Figure 1.

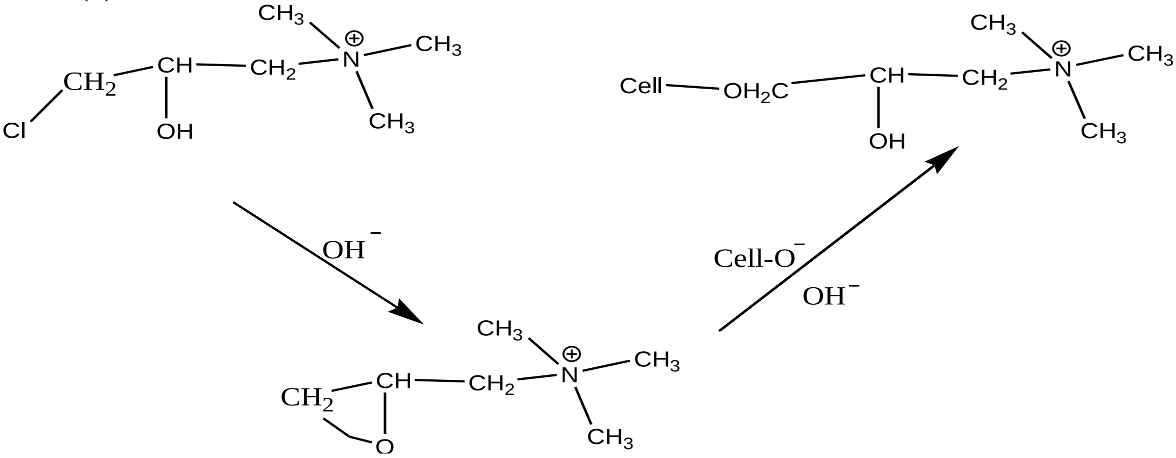

Figure 1: Mechanism of Cationization

The moisture content of the dry fiber, and cationized fibers show the values of about 8%, 7.2% and 5.6% respectively. This gives a good dispersion of the fibers in the natural rubber matrix. Similar report was given by Okieimen and Imanah (2003).

Broad absorption band of hydroxyl group around 3790 – 3169 cm^{-1} is attributed to the OH stretching vibrations of cellulose, hemicelluloses and water absorbed constituents of the fiber. The OH stretching decreased with causticization, and cationization. The C=O stretching vibration of carbonyl groups in hemicelluloses in the fiber can be seen at peak near 1733 cm^{-1}. Those of the causticized and cationized samples have been shifted to 1603cm^{-1} and 1722cm^{-1} respectively. The presence of aromatic ring in the sample can be seen at 1600 – 1400cm^{-1}. The absorption band near 1400 – 1300cm^{-1} may be attributed to aliphatic C-H in the plane deformation vibration of methyl or methylene groups. The C-O stretching vibration of aliphatic primary and secondary alcohols in cellulose can be seen in band region 1300 – 1000 cm^{-1} (Abdulhamid *et al*., 2010).

The tensile strength of the composites shown in Figure 2 indicated strength decrease with increase in fiber loading. The rubber molecules are themselves internally cross-linked, possessing rather high tensile strength as a result of the so called strain-induced crystallization (Yakubu *et al.*, 2010). However, the addition of the fiber interfered with this natural tendency leading to disruption of the regular arrangement of the molecules, resulting in the loss of ability to crystallize and hence the observed decrease in tensile strength with increase in fiber loading. The results showed that the tensile strength of the composite filled with causticized and cationized fibers were higher compared to composites made with untreated fibers at most loadings especially of 20 to 30 phr. From the result, it is clear that the alkali and bonding agent treated cellulosic fibers gave higher adhesion to rubber matrix. This is in agreement with the work of Lovely and Rani (2006). Who opined that the effect was more pronounced in the cationized fiber composites.

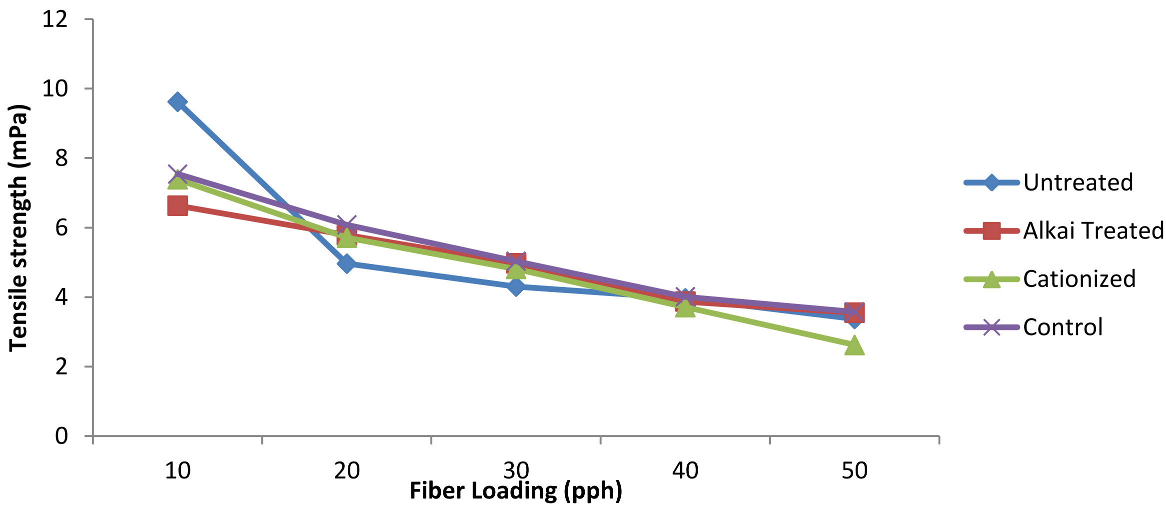

Figure 2: Effect of fiber loading on tensile strength of natural rubber-wood flour composites

From Figure 3 it can be observed that cationized fibers show better impact properties than the alkali-treated and untreated fibers. The insensitivity of surface treatment with alkali on impact strength was reported by Chuai and his co-workers (Chuai *et al.*, 1998). Generally, the cationized fibers show better impact properties than the alkali-treated and untreated fibers.

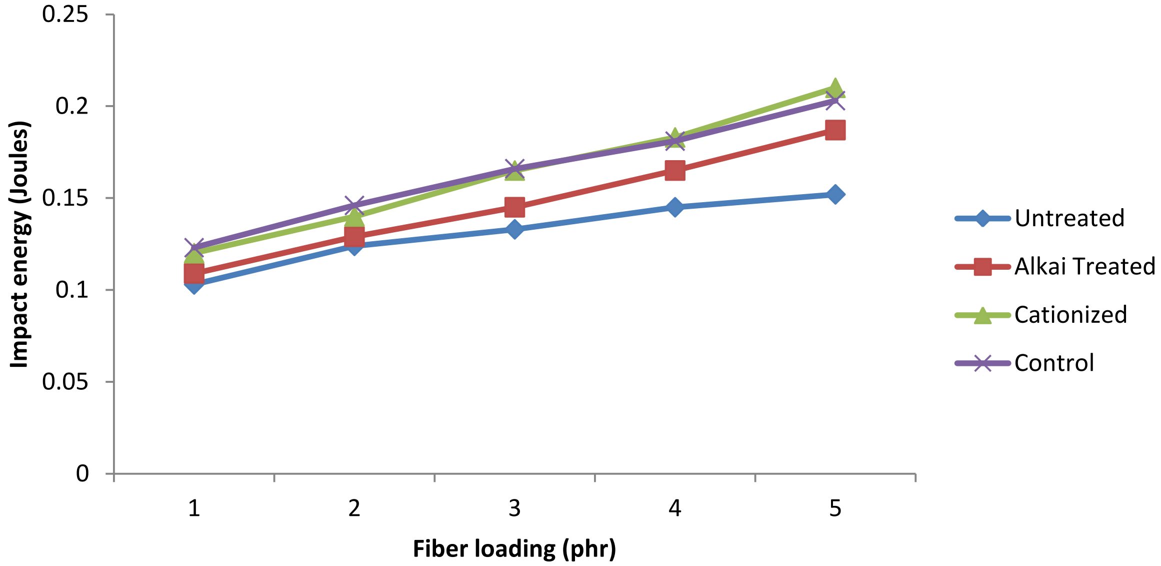

Figure 3: Effect of fiber loading on impact strength of natural rubber-wood flour composites

From Figure 4 it can be seen that the hardness results of the different samples increased with increasing fiber content. This is in line with the reduced elasticity as a result of the reinforcement in the rubber molecules. (Abdulhamid *et al.*, 2010), credited this increase in hardness with fiber loading to the increase in rigidity as the elasticity of the virgin rubber decreases. In the case of untreated wood flour fiber, hardness increased with increase in fiber content of the composite of up to 20% weight fraction. This is because there is decrease in coalescence of the fiber particles which also reduces the rate of getting in of the fiber in to rubber, hence reduced the hardness property (Egwaikhide *et al*, 2007). In each case, the cationized fiber-reinforced samples were observed to give a higher hardness ratings while causticized sample is intermediate between the untreated and cationized samples.

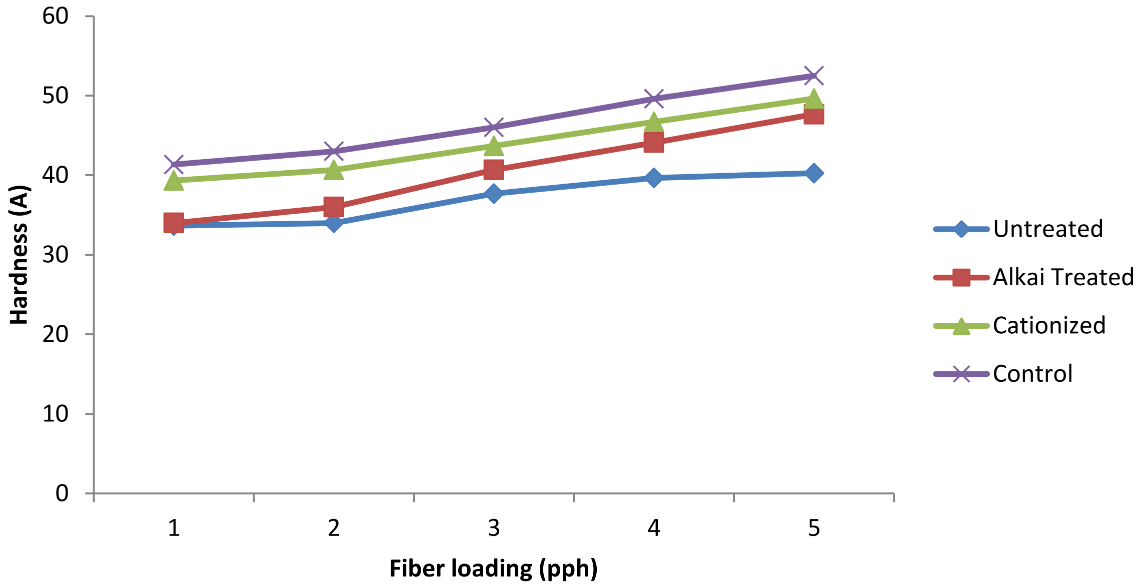

Figure 4: Effect of fiber loading on hardness of natural rubber-wood flour composites

CONCLUSION

Two different treatments (causticization and cationization were carried out on wood flour cellulosic fiber and the treated as well as the untreated fibers used for the compounding of natural rubber. The Mechanical properties such as impact strength and hardness of both fibers were found to increase with increase in fiber loading. While tensile strength decreases with increase in fiber content of the composites. Therefore the use of wood flour fibers as filler for natural rubber is of economical value and their consumption as filler would help in reducing environmental waste.

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