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Effect of Thermal & Alkali Treatment on Pterocarpus Angolensis (Mukwa) Wood Flour

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Abstract

Environmental friendly pre-treatment of fibre has been lately adopted by many researchers worldwide but not fully understood. Although various chemical modifications of fibre through several chemical treatments have been explored, but less has been done on lower sodium hydroxide (NaOH) concentrations. This paper investigated the effect of thermalization at 115 C and lower NaOH of concentrations (1, 3 and 5) effect on mukwa wood flour as a way of minimizing chemical impact on possible manufacturing of eco-friendly wood polymer composites (WPC).Weight loss analysis was carried out to evaluate the effect of thermalization and alkalization modification. X-ray diffraction (XRD), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM) were used to characterize the untreated and treated mukwa wood flour. The results showed significant surface modification thereby improving fibre characteristics as impurities (hemicellulose, lignin and other non-cellulosic substances) were removed.

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Keywords: Sodium hydroxide; Pterocarpus angolensis; eco-friendly; ligin; hemicellose;

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1. Introduction

Pterocarpus angolensis *(mukwa)* is a valuable hardwood species scattered throughout southern Africa. It has different common names; in Botswana it is *mukwa*, in South Africa widely known as *kiaat* or *morhotso*, and its application is diversified into furniture making, construction and medicine [1-3]. Usage of lignocellulosic materials as fillers in polymer composites manufacturing have recently increased as they enhance properties. Moreover, their environmental friendliness, low density, no abrasion during processing, biodegradability and availability makes them advantageous over mineral fillers [4]. Surface modification of hydrophilic fibre can either be physical or chemical, with the physical thermalization aiding cellulose hydrolysis of fibre [5]. Alkalization has been reported to modify the surface of natural fibers successfully by reducing water absorption, extracting cellulosic material (hemicellulose and lignin), dewaxing and removing oil on fibre surface [6-9].

However, these chemical treatments have a major setback of degrading the environment due to their handling and disposal at larger amounts [10,11]. Therefore, lowering chemical concentration or adopting modifications that are environmental friendly is necessary.

The objective of the present work was to investigate the influence of thermal and sodium hydroxide treatment on pterocarpus angolensis. Lower sodium hydroxide concentrations modifications on *mukwa* were assessed in making the powders suitable for the possibility of green manufacturing and eco-friendly polymers. The effect of thermal and NaOH treatments on mukwa properties such as crystallinity, surface morphology and weight loss were investigated and fully discussed.

2. Experimental procedure

2.1. Materials

Lignocellulosic mukwa wood flour was obtained from Terry Cooney Company, a furniture manufacturing company in Gaborone, Botswana. Sodium hydroxide pellets of 99% concentration were obtained from Rochelle Chemicals, South Africa.

2.2. Mukwa flour treatment

Two kilograms of mukwa was washed with water to remove dust and dirt. It was then sun dried for a week prior to thermal treatment. Less than 100microns sieved fine particles were subjected to hot air oven at 115 C for 24 hours (to reduce moisture content), and then stored in sealed plastic containers for subsequent use. 2.5-g thermalized mukwa wood flour was submerged into three 100 ml beakers containing 1.0, 3.0 and 5 % aqueous NaOH solutions for extraction of oily or greasy materials, lignin, hemicellulose and other impure surface materials for 2.5 hours at room temperature. The alkalized mukwa was then filtered, drained and thoroughly rinsed with distilled water to remove any excess NaOH prior to oven drying at 100 C for 24 hours [4].

$$Fibre-OH + NaOH \rightarrow Fibre-O^{-}Na^{+} + H_2O + [surface impurities] \quad (1)$$

Determination of sample weight loss percentage was calculated as follows:

$$Weightloss(\%) = [(W_0 - W_1)/W_0]x100 \quad (2)$$

Where W_0 was the initial weight of mukwa wood flour before mercerization and W_1 the residual weight of mukwa wood flour after NaOH treatments [12].

2.3. Flour characterization

The surface morphology of graphite coated untreated and treated mukwa flours were examined using scanning electron microscopy, model JSM-7100F. The mukwa flours crystallinity were assessed by XRD using Bruker D8 advance X-ray diffractometer, with generator operated at 40kV and 30mA. The samples were scanned in the 2θ

range of 5-50 . The crystallinity index (CrI) of the flours was determined using Segal empirical method [13], as follows:

$$CrI(\%) = [(I_{002} - I_{am})/I_{002}]x100 \quad (3)$$

Where I_{002} is the crystallographic counter reading and I_{am} is the amorphous phase counter reading at 2θ in the samples. The thermal stability of untreated and treated mukwa wood flour was examined and measured at a heating rate of 10 C/min, under nitrogen atmosphere in the temperature range of 25-700 C.

3. Results and discussion

3.1. SEM analysis of mukwa wood flours

The morphological changes due to thermalisation and NaOH treatment are shown in Fig.1. In the SEM image of untreated mukwa flour (Fig. 1a), considerable waxes, fats and surface particles can be identified. For the sample treated at 115C (Fig. 1b), fewer waxes, surface particles and impurities are observed when compared to untreated sample. Thermalisation affects surface properties and fibre structure without altering the chemical composition [7].

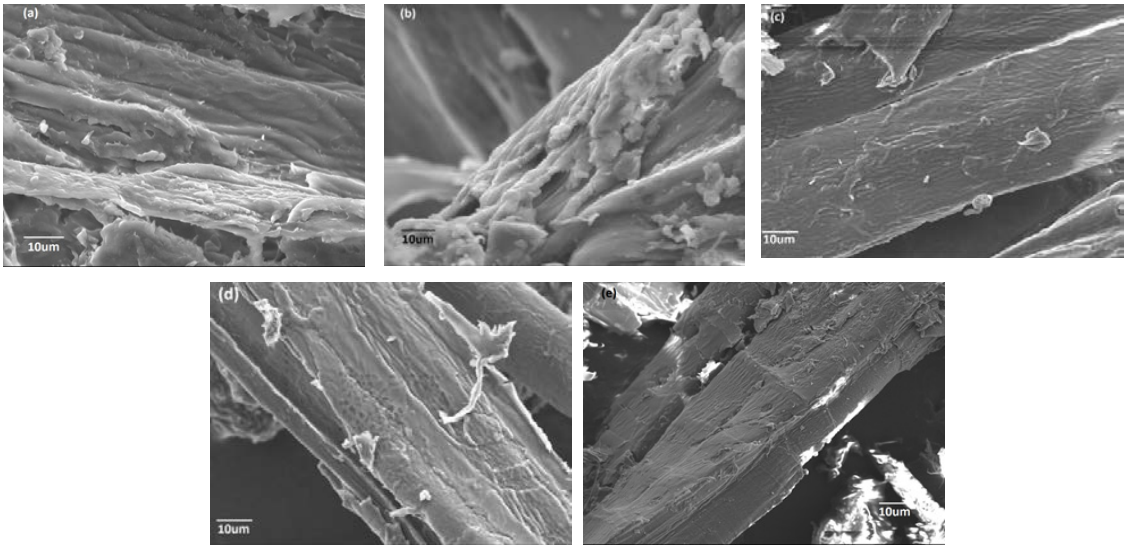

Fig. 1. SEM micrographs of (a) Untreated; (b) 115C treated; (c) 1%NaOH treated; (d) 3%NaOH treated and 5%NaOH treated mukwa flours

Partial dissolution of hemicellulose, lignin and surface impurities due to NaOH concentrations of 1 and 3% are of insignificant differences as observed in Fig.1 (c and d). It is thought that mercerisation affects cellulosic, non-cellulosic and fibre surface impurities. In addition, a pronounced fibre structure, with less waxes when compared to untreated and thermalized flours were found. This can be attributed to insufficient NaOH concentration, as major fibre surface cleaning was not achieved. A tremendous improvement was observed on the 5% NaOH (Fig. 1e), as the image shows a cleaner rough surface with fewer surface particles as cellulose cell walls are exposed (i.e. best). This is because 5% NaOH extracted more non-cellulosic materials leading to possibilities of fibrillation. Thus in agreement with the work reported by Zhong et al. [14]. Other researchers [15,16] have reported cleaning of fibre surfaces following alkalization, which successively made the fibre surfaces more structured. Investigations through SEM have shown the prospect of more fibre surface structuring that coalesces with improved crystallites order through XRD analysis and weight loss assessment.

3.2. Weight loss assessment

It is no doubt that mukwa fibre consists of cellulose, lignin and hemicellulose making majority of the total mass. The weight loss due thermalisation at 115 C is 6.78% (Table 1), aiding cellulose hydrolysis of fibre [5]. After measuring the dry weight of alkalized flours, the mercerisation decreased lignin and hemicellulose contents as expected [17].

Table 1, The weight loss of mukwa wood flours

Parameter	Mukwa wood flour			
(%)	Thermalised	NaOH concentrations (%)		
Weight loss		1	3	5
	6.78	14.20	16.05	16.89

Analysis indicated that weight loss of mukwa flours due to NaOH concentration treatments (1, 3 and 5%) are 14.20%, 16.05% and 16.89% respectively. Weight loss of alkalization is 2.09-2.49 times higher than that of thermally treated flour. Consequently, weight loss is defined as extractives being hemicellulose and lignin thereby affecting the overall mass of the flours. It is in agreement with the XRD and SEM analysis.

3.3. XRD analysis of mukwa wood flours

The X-ray diffractograms of untreated and treated mukwa flours are shown in Fig.2. A similar amorphous spectrum was observed on both untreated and treated mukwa fibre. Two major peaks for both samples can be observed at 2θ 22.2 and 34.5 , with a broader amorphous shoulder at around 16.1 2θ corresponding to (1-10) plane while crystallographic peak 22.2 2θ corresponds to (200) crystallographic plane. Crystallinity index (CrI) was calculated according to the method described in section 2.3, with results presented in Table 2. From the results, increasing NaOH concentration resulted in increased CrI, with 5%NaOH recording the highest. This is due to alkalization inducing changes in the molecular structure.

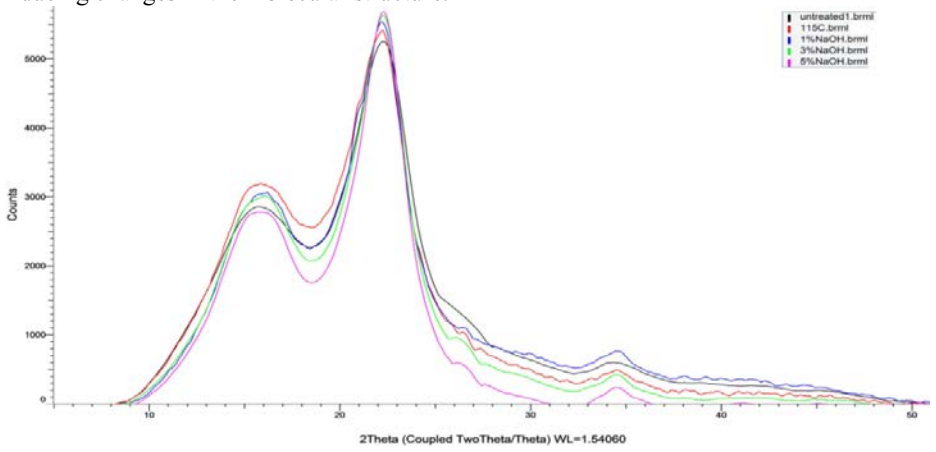

Fig. 2. X-ray diffractograms of untreated, 115C treated and NaOH treated mukwa wood flours

Table 2, Crystallinity index of untreated, thermalised and alkalised mukwa wood flours

%			NaOH concentrations		
Parameter	Untreated	115C	1	3	5
Crystallinity Index	35.0	38.1	39.9	41.1	43.7

Alkalization effect on lignocellulosic materials and their crystallinity has been observed by numerous researchers, [18,19] who found that realignment of cellulose molecules due to loss of amorphous lignin and hemicellulose results in an increase of crystallinity. Mwaikambo et al, [15] found that alkalization improves the order of crystallites as the cell wall thickens thereby increasing overall crystallinity index. Overall, there was a strong link between the microstructure, XRD and weight loss.

3.4. Thermogravimetric analysis of mukwa wood flours

The thermograms of untreated thermally treated and NaOH treated mukwa wood flours are shown in Fig.3. All thermographs show a weight loss at around 100 C corresponding to less than 10% weight loss of absorbed moisture in the flours. Above 100 C, thermal stability decreases as both flours display a comparable decomposition trend that is three stages of weight loss.

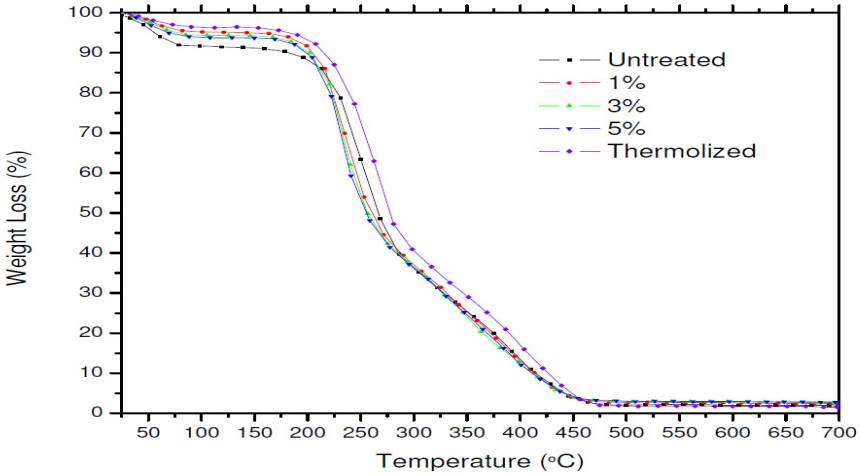

Fig 3: Thermograms of untreated, thermalised and (1, 3 and 5%) NaOH treated mukwa wood flours

The first stage occurs in the range of 150-225 C, 175-238 C and 163-240 C for untreated; NaOH treated and thermalized wood flours respectively. The weight loss in these temperature ranges was about 20%. It is attributed to the thermal depolymerisation of a portion of lignin, hemicellulose and destruction of crystalline regions [20]. The second stage has a minor weight loss in the temperature ranges of 238-300 C, 225-325 C and 240-325 C for mercerised, untreated and thermalized wood flours respectively, with an average weight loss of about 63% for NaOH modified, 65% and 64% for untreated and thermalized respectively. The final decomposition stage occurred in the range of 300-470 C, 325-463 C and 325-475 C for alkalized, untreated and thermalized flours, and a weight loss of about 95% for all the flours. It is thought that lignin is the last component to decompose as it requires higher temperatures of up to 900 C [20]. The results indicated that thermalized flour had a better thermal stability as structure reinforcing constituents such as lignin; hemicellulose and other impurities were not extracted when compared to alkalized flours.

4. Conclusion

The effects of thermalization and alkalization on mukwa wood flour have been investigated and the results showed that thermal treatment influences weight loss, surface morphology, thermal stability and crystallinity index while alkalization modification extracted non-cellulosic (hemicellulose, lignin and oils) material from fibre, thereby

improving the order of crystallites and eventually increasing crystallinity index. 5%NaOH treated mukwa wood flour showed better surface roughness, improved weight loss and the highest crystallinity index when compared to others. The thermal stability of thermalized wood flour was slightly superior when compared to others. Thus, the following conclusion could be drawn; higher NaOH concentration yields superior modification and exposed non-cellulosic material to reaction.

Acknowledgements

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