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Modification in surface properties of sugarcane bagasse: a comparative study from the pretreatment using sodium and calcium hydroxide Juliana Anggono,1,*) Hariyati Purwaningsih, 2) Béla PukÃinszky,3) Suwandi Sugondo,1) Steven Henrico, 1) and Sanjaya Sewucipto1) 1)		

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email:	BPukanszky@mail.bme.hu ABSTRACT				
	Sugarcane is the world's largest crop grown	4			
	with a total harvest of about 1.69 billion tons in 2010.	4			
	Sugarcane bagasse is an agro-industrial by-product	8			
of sugar mill.					
	About 30-32% by weight of the cane produced as	4			
by-products. Like other natural					
	fibers, bagasse is a fibrous residue,	34			

have been relatively unexplored for fibrous

application.

To improve interfacial adhesion between fibers and matrix in



the composite, alkali pretreatment was performed on the bagasse fibers. This current research studied the effect of soaking time during alkali treatment

using sodium hydroxide (NaOH) and calcium hydroxide (Ca(OH)2)

so- lution on the surface properties modifications on bagasse fiber. Calcium hydroxide is chosen as low cost and less agressive alkaline solution to the environment compared to sodium hy- droxide. Pretreatment with NaOH was found more effective than with Ca(OH)2 with major removal of lignin and minor of hemicellulose as . The weight loss obtained on bagasse after pretreated with NaOH was found as 40.5-57.75%, while using Ca(OH)2, the weight loss was measured as 25-46%.

The chemical structure of the cellulose fibers after treatment was ana- lysed using Fourier Transform Infrared (FTIR). Scanning electron microscope (SEM)

was per- formed

to study the effect of treatment on the surface morphology of the

fibers. Those techniques identified that most lignin has been removed and cellulose fibers obtained from treatment using both solutions. Introduction Indonesia is a major producer of agricultural products which the

sugarcane is one of the main agricultural products

with a total production of 28,7 metric tons per year (rank 9th in the world).[1]

Agricultural production inevitably generates residues (by-products)

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that have limited use.

Sugarcane stems consist of three major parts: the pith (5%), fibers (73%), and the rind (22%).[

Sugarcane bagasse is the remaining substance (waste) after crushing the

canes for sugar. Bagasse is a fibrous residue, one among the three other main byproducts of the sugar industry, i.e. cane tops, filter muds, and molasses. Currently sugarcane bagasse is used as pri- mary energy source as fuel for the boilers in the sugar mills and the remaining is stored. However, the important part of the bagasse as an industrial waste is still underutilized. Added- value compounds that can be produced using bagasse are bioethanol, [3-6] protein-enriched ani- mal feed (†single cell proteinâ€[™]),[7] and enzymes. Therefore various research works have been done to develop methods for further processing of bagasse to produce

fuel and chemicals that offer economic, environmental, and strategic advantages.

However, there were relatively fewer studies reporting on obtaining fibers from sugarcane bagasse compared to other agricultural by-products.

Bagasse, as well as other lignocellulosic materials consist of cellulose, hemicelluloses, lignin, and small amounts of extractives and mineral salts.

Several research has studied bagasse as a potential resource as a reinforcement element in biocomposites. [8-15] There are a wide ray of applications in building, construction, and automobiles that exist for the fabrication of bagasse based composites. The use of lignocellulosic materials require pretreatment step. One of the main problems working with natural fibers used as reinforcement components is their high moisture sorption and hydrophobicity of the fibers which thereby influences the mechan- ical bonding with the matrix.

Therefore chemical pretreatments are considered in modifying the fiber surface properties.

Chemical pretreatments using acid have

been successfully applied to sugarcane bagasse

fiber. [10,13,16]

Acids hydrolyze hemicellulose and produce a liquid phase rich in xylose, with minor amounts of lignin derivatives and it







has been successfully applied to sugarcane bagasse.[17,18] The principle of

alkaline pretreatment method is the removal of lignin (major loss) whereas cellulose and a part of the hemicelluloses remain in the solid material (minor loss of hemicellulose). Pretreatment

using alkaline solution (mercerization) has been proven to be effective for removal of lignin in the fibers therefore enhanced fiber suface adhe- sion

which allowed an effective stress transfer from matrix to fiber.

Relatively fewer studies have investigated the surface modification on lignocellulosic fibers obtained from sugarcane baggase. This current research work focused on the study of surface and structural modification on bagasse fibers pretreated using two different alkaline solution, i.e.

sodium hydroxide (NaOH) and lime or calcium hydroxide (Ca(OH)2).

Calcium hydroxide was chosen due to its less hazardous characteristics and low cost. Surface properties on morphology and structure of modified bagassse fibers after pretreatment were studied to evaluate the delignification degree for bagasse fiber performed by both solution in different time length of pretreatment ranging from 2 to 6 hours at 60-70oC. Characterization on the untreated and treated fiber

was carried out using Fourier Transform Infrared (FTIR) and X-ray diffractometer (XRD)

tests to determine the modified structure and chemical composition on the fiber surface. Scanning electron micro- scope (SEM) was used to study the effectiveness of both pretreatments through the changes of surface morphology of bagasse fibers. Experimental The first step in the bagasse preparation was neutralization of the bagasse by soaking in a 70% ethanol for an hour to eliminate the stench generated due to bacterial fermentation reac- tion.[19] A ratio of volume of ethanol (litre) to the weight of bagasse (kg) used was 2.5:1. The neutralized fibers were drained and dried for 6 hours in air at room temperature.

The fibers were then dried in the oven (Memmert type UN450) at

a temperature 200oC for 30 seconds. Pretreat- ment was applied to the dried fibers by soaking the fibers in two different solutions of 10% vol. NaOH and 14 % vol. Ca(OH)2 at 60-70oC for various soaking time of 2, 4, and 6 hours. At the end of pretreatment, bagasse fibers were rinsed couple of times in distilled water

to ensure they were clean from the alkaline solution.







Before the rinsed fibers went to the oven for drying, they were dried in air for 6 hours at room temperature. Lastly, those fibers were oven dried at 200oC

for 30s. Dried fibers were then

examined with XRD (X'pert PRO tipe PANalytical)

and FTIR (Thermo Scientific Nicolet model) to analyse the

structural changes on the fiber surface after treatment. Mass loss after neutralization and pretreatment stages was measured to check the effectiveness of lignin removal. SEM (FEI type Inspect S50) was used to evaluate

the surface morphology of the fibers before and after the alkaline treatment



in different soaking time. Un- treated bagasse fibers were also evaluated to study the surface changes after both treatment. Results and discussions Modification of Sugarcane Bagasse †Weight Loss of Sugarcane Bagasse After Pretreatment The goals of alkaline pretreatment

include the removal of lignin and disruption of the crystalline structure of cellulose

(Fig. 1). Chemicals used in alkaline pretreatment were sodium and calcium hydroxide. Fig. 1 Schematic structure of natural fibers before and after pretreatment.[20] Weight loss measurement on bagasse samples due to pretreatment was carried out aiming to determine the delignification degree of both pretreatment. The weight loss values obtained can inform the effectiveness of lignin removal both in different alkaline solution and soaking time. Bagasse samples were weighed when they were received as wet fibrous residue. Two other weight measurements were performed after oven dried bagasse samples both after neutralization and alkali treatment processes. Neutralization of bagasse using 70% ethanol aimed to prevent bacterial fermentation as that solution is an effective desinfectant or antiseptic agent. The weight loss obtained after neutralization and oven drying was 42.5%. Apart from the three basic chemical compounds (lignin, hemicellulose, and cellulose)

that lignocellulose consists of, water is also present in the complex.

Typically, sugarcane bagasse has a moisture content of between 45 and 55% on a wet basis.[21]

Furthermore, minor amounts of proteins, minerals and other components can be found in the lignocellulose composition as well. Therefore the weight loss of



neutralized fibers was mainly due to the loss of water/moisture content after drying process. Following the neutralization step, the dried fibers were pretreatment using sodium and calcium hydroxide for 2, 4, and 6 hours. After pretreated and oven dried, the fibers were weighed to calculate a further weight loss due to this treatment. Fig. 2 shows the weight loss after both pretreatments. Significant loss was measured in NaOH pretreated fibers (40.5%-57.75%) than in the fibers pretreated using Ca(OH)2 (25%-46%). The weight loss due to NaOH pretreatment increased by 60% during 2 hours soaking time compared with the same soaking time during pretreatment in Ca(OH)2. When soaking time increased to 4 and 6 hours, the weight loss during NaOH pretreatment increased only by 30% compared with treatment using Ca(OH)2. However the weight loss increment with the additional two-hour soaking time was noted relatively higher in Ca(OH)2 solution (10%-11%) than in NaOH (4.5-12.75%). Major component removed during NaOH and Ca(OH)2 pretreatment was lignin. Hemicellulose removal was contributed as minor loss to the weight loss.[22] Fig. 1 Weight loss of bagasse fibers after pretreatment Higher value of weight loss was obtained in NaOH pretreatment than the same loss experienced in Ca(OH)2 meaning that the more efficient lignin removal experienced by bagasse in NaOH than in Ca(OH)2. The action of Ca(OH)2 during pretreatment is slower compared to sodium hydroxide.[22] FTIR and SEM results support the findings on the weight loss and will be explained in the next section. Characteristics of Modified Bagasse Bagasse is a lignocellulosic material consisting

of cellulose 43.8%, hemicellulose 28.6%, lignin 23.5%, ash 1.3%, and other components 2.8%.[

19] Studies performed

with different varieties of sugarcane bagasse reported that their main chemical composition does not differ significantly. [23] The

hemicellulose fraction is a heteropolymer of pentoses (C5) and hexoses (C6), predominated by xylans, and the cellulose fraction,

a major structural component of cell walls and it provides mechanical strength and chemical stability to plants, is 3

a homopolymer of glucose. Lignin is the most complex natural polymer;

polymer of aromatic compounds produced through a biosynthetic process and forms a protective layer for the plant walls.

It is an amorphous three-dimensional polymer with phenylpropane units as the

predominant building blocks.

The

major effect of alkaline pretreatment is the removal of lignin from the

bagasse.

FTIR spectroscopy was used to obtain information about the chemical structure of the bagasse before and

after pretreatment. The FTIR spectra for bagasse fibers after both pretreatments are shown in Figure 3a and 3c. The spectra for untreated fibers were also shown for a comparative study to identify considerable

changes in the chemical composition on the surface as a consequence of

modification due to alkali pretreatment. The similarity of the FTIR spectra betweeen untreated and pretreated bagasse fibers was found in the 4000-2700 cm-1 region where the

OH and CH stretching vibrations existed. The

strong broad band observed in the region of 3700-3000 cm-1 is assigned to different OH stretching modes and another band in the region of 3000-2800 cm-1 is attributed to the stretching of asymmetric and symmetric methyl and methylene cellulose groups.

Fig. 3b and 3d show a 1800-800 cm-1 region in the spectra that

revealed several bands. The band at 1642 cm-1 is associated with adsorbed water in cellulose and probably some hemicelluloses. The C-H stretch at

2894 cm-1 is present in both untreated and treated fibers. The

C-OH bending peaks at 670 cm-1 of cellulose are indicated in all

fibers. The obvious dif- ferences between the untreated bagasse fibers and treated



fibers were noted in the range from 2000-700 cm-1

(Fig. 3a and b). Lignin spectrum at 1512 and 1250 cm-1

was no longer found in sugar cane fibers that were given

pretreatment, either in NaOH or Ca(OH)2. The

peak 1730 cm-1 indicates the carbonyl peak, C=O stretching of the acetyl groups of hemicellulose [24,25] and can be seen in untreated fibers. The disappearance of that peak indicates the removal of hemicellulose from the fiber surfaces after treatment.

Pretreatment with

sodium hydroxide causes the disruption of H-bonding in cellulose and hemicellulose, breakage of ester linkages between lignin and xylan, and deprotonation of phenolic groups. As a result, swelling of cellulose and the partial solubilization of hemicellulose and lignin occurs.

[26]

There is an indication of carbonate peaks in the range of 1500-1400 and 872 cm-1 on the

fiber pretreated using Ca(OH)2 (Fig. 3d). XRD analysis and SEM study performed on the bagasse fiber pretreated using Ca(OH)2 confirmed the existence of carbonate compound. 2 hours treatment 4 hours treatment 6 hours treatment no treatment Absorbance (a.u) 1800 1600 1400 wavenumber (cm-1) 1200 1000 800 a) b) no treatment 2 hours



NaOH and c) Ca(OH)2 and in the



region between 1800-800 cm-1 in

b) NaOH and d) Ca(OH)2.

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Figure 4 shows X-ray diffractograms obtained from untreated and pretreated fibers both using NaOH and Ca(OH)2 at different soaking time. The diffractogram of untreated bagasse fiber shows the main diffraction peaks at 2? angles: 15.60 and 220 which confirmed the typical cellulose-I structure. The major diffraction peak between 220 and 230 referred to cellulose (002) crystallographic planes was found in all pretreated fibers but the weak peak at 2? angle 15.60 disappeared. (Fig. 4a and 4b). Cellulose is a crystalline phase with

four different polymorphs of cellulose are known, including cellulose I, II, III, and IV.

[27] Alkaline pretreatment

in natural cellulose fibers results in the structural transformation from cellulose I (native cellulose) to cellulose II.[

28, 29] This structural transformation was observed in the XRD patterns for both pretreatments which the significant change in diffraction pattern from double peaks as indication of cellulose I to a single peak indicating formation of cellulose II structure. Another peak at 2? angle 29.80 was noted at the bagasse sample pretreated with Ca(OH)2 in all soaking time. That peak was an indication of the presence of insoluble calcium carbonate that might be obtained from the mixture with the starting powder of calcium hydroxide used and from being recoverable from water by the reaction with carbon dioxide. [22] 6 hours pretreatment - NaOH 4 hours pretreatment - NaOH a.u. 2 hours pretreatment - NaOH untreated fibers 5 10 15 20 25 30 35 40 2? (degree) a) 6 hours pretreatment - Ca(OH)2 a.u. 4 hours pretreatment - Ca(OH)2 2 hours pretreatment - Ca(OH)2 Untreated fibers 5 10 15 20 25 30 2? (degree) 35 40 b) Fig.4

X-ray diffractograms of untreated and pretreated bagasse

fibers in a) NaOH and b) in Ca(OH)2 Morphological Property of Modified Bagasse

To evaluate the effect of alkaline treatment on the morphological surface of

bagasse fibers, the samples were studied using SEM. Morphological characteristics of fibres before and after pretreatment were microscopically observed. Figure 5 shows SEM micrographs of un-modified sugarcane fibers, providing the

elementary fibrils and bundles are cemented by lignin and pectin intercellular substances.

The fiber

obtained from sugarcane bagasse was reported to have a length of 2.5

to 20 cm.[2] Schematic structure of natural fibers before treatment in Fig.1 represents the morphology of unmodified bagasse fiber shown in Fig.5. It shows that cellulose fibers are in one piece and intact due to the presence of lignin.

Cellulose is a semi crystalline polysaccharide made up of D-glucopyranose units linked together by Î²-(1-4)-glucosidic bonds [30] and the large amount of hydroxyl group in cellulose gives natural fiber hydrophilic

proper- ties. Figure 5 SEM micrographs of longitudinal view of untreated sugar cane fibers

Hemicellulose is strongly bound to cellulose fibrils presumably by hydrogen bonds. Hemicellulosic polymers are branched, fully amorphous and have a significantly lower molec- ular weight than cellulose. Because of its open structure containing many hydroxyl and acetyl groups, hemicellulose is partly soluble in water and hygroscopic. [31] Lignins are amorphous, highly complex, mainly aromatic, polymers of phenyl propane units

form a protective layer for the plant walls [32]

but have the least water sorption of the natural fiber. components.[

31] Sugarcane fiber pretreated with NaOH for 2 hours shows that the treatment has been able to eliminate most of the lignin and minor hemicellulose. FTIR spectroscopy detected

the removal of lignin and hemicellulose (Fig. 3b). The surface

micrograph in Fig. 6a shows a cleaner surface of the fiber compared to untreated one in Fig. 5. An increase of NaOH soaking time to 4 hours increases the weight loss of the bagasse from 40.5% to 45%. That weight loss is evidenced by a definitive change in the morphogical structure occurred after 4 hours treatment which the cellulose fibers observed in the micrographs in Fig.6a are partially decom- posed. Increase soaking time in NaOH treatment to 6 hours increased the weight loss to 57.75%. This loss was supported by FTIR







spectra that reported the absence of lignin and hemicellulose spectra. Figure 6c shows deeper contour of cellulose fibers morphology compared to the fiber surface after soaking in NaOH for 2 hours (Fig.6a). a) b) c) Figure 6 SEM micrographs of bagasse fibers after pretreated using NaOH

for a) 2 hours, b) 4 hours, and c) 6 hours

SEM observations on bagasse fibers pretreated with Ca(OH)2 for 2 to 6 hours show the defibrillation of cellulose fibers. The defibrillation occurred

due to the removal of lignin and hemicellulose.

Fig. 7a-7c show the cellulose fibrils became more exposed. The weight loss data obtained from pretreatment using Ca(OH)2 are about 62%-80% of the weight loss experienced by the bagasse fiber pretreated with NaOH. There was an increase in weight loss about 10%-11% with the addition of two-hour soaking time. The presence of CaCO3 precipitate as it has been identified by FTIR and XRD results was evident in the SEM observation. Bright small particles of CaCO3 was found on the fibers surface in all soaking time. The cleanliness of the fiber surface pretreated with Ca(OH)2 was not as good as the fiber surface pretreated with NaOH. The removal of hemicellulose is not as effective as done with NaOH (Fig.7c) a) b) c) Figure 7 SEM micrographs of bagasse fibers after pretreated with Ca(OH)2

for a) 2 hours, b) 4 hours, and c) 6 hours

Conclusions Pretreatment bagasse fibers using 10 vol. % NaOH results in more significant removal of lignin and hemicellulose compared to pretreatment done with 14 vol.% Ca(OH)2 as indicated by FTIR and XRD spectra. Reaction of Ca(OH)2 with the bagasse is slower than with NaOH. That finding is supported by the weight loss data of bagasse after the NaOH and Ca(OH)2 pretreatments, i.e. 40.5-57.75% and 25-46% respectively. SEM evaluation on the morphological property on bagasse fiber surface shows significant fibrillation of cellulose bun- dle pretreated with Ca(OH)2

with bright small particles of CaCO3 deposited on the fiber surface.

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