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The Use of Sugarcane Bagasse in PP Matrix Composites: A Comparative Study of Bagasse

Treatment Using Calcium Hydroxide and Sodium Hydroxide **on** Composite **Strength** **1**

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e) huangsemin@gmail.com Abstract. Sugarcane fibers have been studied as reinforcement fibers to develop biocomposites of sugarcane fibers- polypropylene (PP). In this study, two different alkaline solutions were used to modify the sugarcane bagasse fibers, i.e. 10% v/v NaOH and 14% v/v Ca(OH)2 solution for 4 hours. Sugarcane fibers filled to polypropylene (PP) matrix was

3 cm, 5 cm and as their **original length.** 1

They were added in the various weight percentage ratios between sugarcane fibers to PP of 20/80, 25/75, and 30/70.

Fourier-Transform Infrared (FTIR) spectroscopy **was used to** study **the chemical structure of** 27

bagasse fibers after treatment. Composite strength was measured by tensile test which was performed in accordance with ASTM D638-03. The fracture surface of tensile tested composite specimens contained fibers from both treatment was evaluated using Scanning Electron Microscope (SEM). Results from the tensile test show that performing NaOH treatment for 4 hours could increase the tensile strength of the composites to 24.92 MPa when original length sugarcane fibers was used in the 25/75 weight ratio. Meanwhile the

highest strength of 11.30 MPa was obtained when 5 cm fibers 3

treated with Ca(OH)2 were added

in a weight % ratio of bagasse fibers/PP at 25/75. 3

The strength of composites

decreases when bagasse fibers were added to 30 wt.%. The 3

SEM evaluation shows the mixture between sugarcane fibers and PP was not quite homogeneous. In addition to that is

a greater number of fibers were found with **the** 1

transversal orientation or perpendicular to the direction of tensile stress. INTRODUCTION In the past decade, lightweight materials made from natural fibers composites with thermoplastics and thermosets have been embraced by automakers and suppliers to achieve weight reduction

in order to improve fuel economy of automobiles **and** reduction **of** 9

greenhouse gas emission. In the United States (US), there is a legislation and regulations in the form of

Corporate Average Fuel Economy (CAFE) standards 40

to improve the average fuel economy of cars and light trucks (trucks, vans, and sport utility vehicles) produced for sale in the 5

US. In 2025, CAFE standards is targeted to be 54.5 mpg (miles/gallon), an increase of 54% from current nominal standards (35.5 mpg) in 2016. [1, 2] Therefore

auto manufacturers selling in the United States must meet the 5

CAFE standards enforced by the National Highway Traffic Safety Administration (NHTSA). One of **the** 5

pathways for automotive industry to achieve CAFE standards for 2025 is to renew their focus on lightweight materials to achieve weight reduction. This can be achieved in structural and nonstructural components such as door panels, seatbacks, headliners, package trays, dash boards, front-end, and interior parts. The usage of lightweight, low cost

natural fibers such as kenaf, jute, sisal, **hemp,** and **flax are providing automobile** 4

makers benefits of reduction in

CO2, less dependence of **oil sources,** recyclability, **and** 39 they are renewable and sustainable resources. The

automotive industries around **the** globe **are continuously optimizing cost versus quality in order to remain competitive in the market. Moreover, increased** social awareness **of** 6

environmental problems is forcing automobile manufacturers to look for

renewable resources for raw materials and recyclability or biodegradability of the product at the end of its useful life. 6

This enhances the use of agro-based biofibers as biodegrable content in automotive applications. [3] Bio-fibers reinforced plastic composites are used in various applications in nearly

all the major car manufacturers in Germany (Mercedes, **Daimler Chrysler,** Audi Group, **Volkswagen, BMW, Ford and Opel).** Polypropylene and **natural** 15

fibers are used to produce for

interior trim components such as dashboards and door panels 30

for Daimler Chrysler.

Door trim panels made of polyurethane reinforced with mixed **flax/sisal** was used **in Audi A2 midrange car** 19

in 2000. [4] The

end of life vehicle (ELV) directive in Europe states that by 2015, vehicles must be constructed of 95% recyclable materials, with 85% recoverable through reuse or mechanical recycling and 10% through energy recovery or thermal recycling. 7

[5]

Though natural-fiber-reinforced plastic parts offer many 4

advantages

over synthetic or man-made fibers such as low cost, **low density, competitive specific mechanical properties,** CO2 subtraction, **sustainability, recyclability, and biodegradability;** 11

however several major technical considerations must be addressed before the engineering, scientific and commercial communities can widely accept **to** 4

have proven to meet the structural and durability demands of automobile interior and exterior parts. 9

Challenges include the homogenization of fiber's properties, adhesion between fibers **and** matrix, the **understanding of degree of polymerization and** 4

crystallinity, as well as moisture repellence, and flame retardant. In this research, sugarcane bagasse fibers were studied which combined with polypropylene matrix. Sugarcane

is an important crop for production of sugar 37

and the world's largest crop grown in

about 23.8 million hectares in more than 90 countries with a worldwide harvest of 14

about 1.69 billion tonnes in 2010. [6] Indonesia is rank 9th as major producers of sugar cane after Brazil, India, China (mainlands), Thailand, Pakistan, Mexico, Colombia, Philippines with a production of 28.7 million tonnes in 2012. [6] Bagasse is an agro-industrial by-product in Indonesia obtained from sugarcane stem after being crushed in a sugar mill

a lignocellulosic materials **consisting of 45-55% cellulose, 20-25%** hemicellulose, **and 18-24% lignin.** 10

[7] Polypropylene (PP) is utilized due to its

low density, high softening point, high rigidity, **hardness,** 38

and impact strength. [8] The

major disadvantage of natural fiber reinforced composites is 36

compounding difficulties between natural fibers and matrix due to

the inherent polar and hydrophilic nature of lignocellulosic fibers **and the non-polar characteristics of most thermoplastics.** This **result** 12

can

lead to non-uniform dispersion of fibers **within the matrix which impairs the composite** 21

mechanical properties. Therefore different chemical treatment may be employed on the fibers to modify the morphology

of the fibers surface **and** to improve **the** mechanical **properties of the fibers,** 10

thus resulting in the properties improvement of the composite. This work used alkali treatment

to modify the surface of bagasse **fibers** 8

or the whole fibers throughout. Two different alkaline solutions, NaOH and Ca(OH)2 were used. The results from Ca(OH)2 treatment has been reported elsewhere [9] and a comparative study on composite strength affected from those two different solutions on bagasse fibers was performed in this current study. EXPERIMENTAL Materials Details Sugarcane bagasse used in this study was obtained from a sugarcane juice seller after being crushed to extract the juice. They were neutralized by soaking in 70% ethanol for an hour with a volume ratio of ethanol (litre) to the weight of fibers (kg) was 2.5: 1. The aim of neutralization was to prevent bagasse to ferment furthermore. Neutralized sugarcane bagasse was then dried in an open air for 6 hours and continued with oven dried at 200°C for 30 seconds. After drying, the bagasse were soaked in two different alkaline solutions, i.e. 10 v/v% NaOH solution and 14 v/v% for 4 hours at 70°C. The quantity of alkaline solution used for soaking followed a ratio of 15 ml of alkaline solution to 1 g of bagasse fibers. Treated bagasse was rinsed with distilled water few times and they were ensured clean when the pH level of rinsed water reached 7 as indicated by pH meter. The drying in an open air for 6 hours followed by oven drying for 30 seconds was performed on rinsed bagasse. The dried bagasse was then prepared in different length of 3 cm, 5 cm, and some were kept as their original length. PP as the matrix was available in the form of long fibers. They were supplied by PT. Classic Prima Carpet Industries. To ease the mixing and in order to achieve homogeneous mixture between bagasse and PP, the

PP fibers were cut into 1 cm 1

length. Composite Samples Preparation and Characterization Bagasse and PP fibers were weighed to prepare composite samples with various wt. %

ratio of bagasse to **PP of 20/80, 25/75, and 30/70.** 1

Each composition was prepared in 60 gram total weight per mixture for a mixing process performed in a centrifugal blower mixing equipment. Each mixture was placed on the steel plate ready to be formed into composite sheet by hot pressing using a pressure of 9,8 kPa at heating temperature of 175°C for 3 minutes. Mechanical property of the final composites was evaluated using tensile test and the samples were prepared with dimensions in accordance to ASTM D638M-03 type I. The

test was performed using Instron 600 **DX.** 1

To understand the effect of alkali **treatment** on **the** 1

composite strength, microstructure

study using SEM or **Scanning Electron** Microscope **(FEI type Inspect S50) was** 1

implemented on surface of fibers in isolation and

on the fracture surface **of the tensile tested samples.** 3

Fourier-Transform Infrared (FTIR) spectroscopy (Thermo Scientific Nicolet 35

model) was used to investigate the chemical structure changes due to alkali treatment. Samples

thickness was also **measured using micrometer Links Brand with an accuracy of 0.01 mm** to study **the** effect **of** 1

hot pressing on the thickness obtained. RESULTS AND DISCUSSION Structural Evaluation of Bagasse Fibers

In order to understand the changes in the chemical structure of bagasse fibers **after** 2

alkali treatment using solutions of Ca(OH)2 and NaOH, infrared spectra (Fig. 1)

of the untreated and treated fibers 8

were acquired. Figure 1a shows the FT

-IR spectra of untreated sugarcane bagasse **and treated** 8

bagasse with Ca(OH)2 and NaOH.

The main features of these spectra are attributed to the existence **of lignin, hemicellulose, and cellulose;** they are **natural components of lignocellulose fibers.** 2

There are some similarities between infrared spectra of treated bagasse to the untreated ones but considerable changes occurred in the range from 2000-700 cm-1, which show that the alkali treatment promoted drastic modification in the chemical structure. The

absorptions of O-H stretching (H-bonded) usually **occur in 3100–3600 cm-1 range. The band observed at 3386 cm-1 seems to be characteristic of** O-H **groups present in lignin and carbohydrates.** 2

[10] The absorbance at 2894

cm-1 could be attributed to C-H aliphatic axial deformation of **CH2 and CH3 groups from cellulose, lignin and hemicellulose.**

2

It

is present in both untreated and treated fibers.

1

The band at 1730 **cm-1 is** referred **to** the the **C=O stretching of** the **acetyl groups**

22

present in hemicellulose. [11, 12] That band was absent in fibers after alkali treatment using both solutions which explains a hemicelullose fraction removed in each treatment. [9] The

relative absorbance of the primary and secondary peaks of **OH groups at 1051 cm-1 and 1165 cm-1 of bagasse**

2

fibers treated with NaOH is higher than the ones treated using Ca(OH)2 (Fig.1b). This can be understood from the fact that NaOH is a stronger alkali compared to Ca(OH)2

and consequently a higher severity which resulted in a more exposed structure.

2

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

1

which was only found in the fibers treated with Ca(OH)2 solution (Fig.1b). [9] This finding is further supported by Fig. 2c presenting scanning electron micrograph taken on the fiber surface after treatment.

The spectra 1250 cm-1 which is a lignin spectrum was no longer found in sugar cane fibers that have been given alkali treatment.

1

[9] Lignin is an amorphous phenolic macromolecule and has the

least water sorption of **the natural fiber** components. [13] **The** removal **of** 8

lignin during treatment was favorable

to improve interfacial bonding between fibers and matrix. 13

Alkali treatment has several effects on cellulose fiber modification. As reported in the literature that the treatment can produce fiber fibrillation [14-16] which leads to the reduction in fiber diameter, an increase in

aspect ratio and effective surface area accessible **for wetting by a matrix** 26

material. SEM was used to investigate

the morphology of the bagasse fibers **before and after alkali treatment** 33

using Ca(OH)2 and NaOH. The micrographs in Fig. 2 show the surface of bagasse fiber before and after 4 hours alkali

treatment in Ca(OH)2 (Fig.2b) **and in** 18

NaOH (Fig.2c). The differences of surface morphology between the untreated and treated fibers are very large. The unmodified fibers are found with a smooth surface with layers of pectin, lignin, and impurities. [16] The alkali treatment done in both solutions caused a definitive change on the morphological structure of the bagasse. Both treatments produced defibrillation of fibers as seen in Fig. 2b and 2c; therefore it caused

a decrease in **fiber length and** diameter. Besides **the** 25

defibrillation,

it was observed that bagasse fibers 25

treated with Ca(OH)2 were unclean with rough surface (Fig. 2b) compared to the surface of fibers treated with NaOH (Fig.2c). This result is in agreement with the FTIR spectra that the alkali treatment using NaOH was more effective compared with Ca(OH)2 in removing hemicellulose. It was also identified the presence of white tiny particles on the surface of Ca(OH)2 treated fibers. These particles have been recognized through FTIR analysis related to carbonates deposit. This deposit originated from the Ca(OH)2 solution used which contained CaCO3 which has also been identified during EDX analysis. [9] Absorbance (a.u) A) A) B) B)

Absorbance (a.u)

C) C) 4000 3500 3000 2500 2000 1500 wavenumber (cm-1) 1000 500 20

1350 1250 1150 1050 wavenumber (cm-1) 950 850 a) b) FIGURE 1. FTIR Spectra of a) in 500-4000 cm-1 region and b) in 850-1350 cm-1 region with A) untreated bagasse fibers, B) treated bagasse fibers using Ca (OH)2 solution and C) treated bagasse fibers using NaOH solution a) b) c) FIGURE 2.

SEM micrographs of a) untreated **bagasse fibers, b)** 17

Ca(OH)2

treated bagasse fibers, and c) NaOH **treated bagasse fibers.** 17

Thickness and Strength Characterization of Composites Figures 3 and 4 show the results from the thickness measurement on composite samples with the bagasse fibers treated in Ca(OH)2 and NaOH solution respectively. The measurement was done on tensile test samples using micrometer before test was performed. Thickness data were collected to understand the effect of pressure used during hot pressing on different conditions (treatment and length) of bagasse fibers mixed with PP. Figure 3

shows the relationship of composite thickness with fibers length and % wt. ratio of bagasse fibers **/PP** 1

(treatment using Ca(OH)2). The

increase in ratio or in number of bagasse 1

fibers as well as fiber length

added to PP increases the thickness of the composites. That phenomenon was understood that the bagasse fibers which were stiffer than PP formed like a preform in the mixture. 1

However, that phenomenon did not happen in all fiber conditions as shown in Fig. 4. The thickness range measured in composites contained 3 cm

length of bagasse fibers for **various wt.%** ratios **of bagasse/PP** 1 of 20/80, 25/75, and 30/70 are 2.17-2.95 mm, 2.27-3.0 mm, and 2.33-2.55 mm respectively. The thickness increases with the increase of fibers length but with lower increment compared to the effect from the increase of the amount of

bagasse fibers added to PP. Similar influence **was** 1

noticed in the composites contained fibers which were treated using NaOH (Fig. 4). The thickness increases

with the increase of fiber length and the wt.% **ratios of bagasse/PP** 3

excepting the thickness measured in the composites with a ratio of 20/80 using 3 cm fibers and the composites contained 30 wt.% of bagasse fibers with 5 cm length. This phenomenon might result from the inconsistent weight of bagasse/PP mixture during the hot pressing process compared to the other data obtained. Comparing the thickness of composites which contained fibers with different treatments, it was obvious that the

composites which used NaOH **treated fibers have** smaller **thickness** 1

range compared to the composites contained Ca(OH)2 treated fibers in all

fiber length and ratios of bagasse/PP. 1

For example in composites contained 30 wt.% of bagasse treated with NaOH, the thickness of composites was measured < 2.65 mm while the thickness measured in the composites contained the same quantity of bagasse but treated using Ca(OH)2, the thickness was recorded >2.78 mm. 3,8 3,6 Ca(OH)2 Treatment 3,4 3 cm 5 cm Original Length Thickness (mm) 3,2 3,0 2,8 2,6 Thickness (mm) 2,4 2,2 2,0 20/80 25/75 30/70 % weight ratio bagasse fiber/PP 3,5 3,3 3,1 2,9 2,7 2,5 2,3 2,1 1,9 1,7 1,5 NaOH Treatment

3 cm 5 cm Original Length 20/80 25/75 30/70 % weight ratio bagasse fiber/PP 1

FIGURE 3. Composite thickness against

bagasse fiber FIGURE 4. **Composite thickness against bagasse fiber length and wt. % ratio of bagasse fiber/PP** 1

(treated with

length and wt. % ratio of bagasse fiber/PP 1

(treated with Ca(OH)2) NaOH) Tensile

strength of the composites was also **affected by the alkali** solution used during **the** 1

treatment, i.e. Ca(OH)2 and NaOH solution. The strength data range obtained for composites using fibers which were treated with Ca(OH)2 for 4 hours were 4.84-11.3 MPa. The increase of baggase fibers

added to PP improves **the** strength **of the composites** as **was** 1

found in

composites which the fibers were treated 1

either with NaOH or Ca(OH)2 though this finding was not consistent for composites with 30 wt.% of Ca(OH) 2 treated fibers of

5 cm and original length fibers. 1

The strength dropped

when the bagasse fibers added up **to 30 wt.** % to PP. **The strength** 1

data obtained with the increment of fiber length in 20 wt.% Ca(OH)2 treated fibers used were 4.84 MPa (3 cm fibers), 8.51 MPa (5cm fibers), 10.10 MPa (original length fibers). Increasing the amount of bagasse fibers to 30 wt.%, the strength of composites was reduced into 7.36 MPa (5 cm fibers), 8.35 MPa (original length fibers) excepting for 3 cm fibers used with a strength of 6.78 MPa which needs to further investigated. The strength of composites using untreated bagasse fibers of original length was as also evaluated and their strength values obtained were 11.38 MPa, 7.38 MPa, and 7.22 MPa for a wt. % ratio of bagasse/PP 20/80, 25/75, and 30/70 respectively. Comparing the strength of the composites with untreated and Ca(OH)2 treated fibers, it was very obvious that the composites strength was not improved with Ca(OH)2 treatment. This low strength was contributed by the poor adhesion

of bagasse fibers/PP. Treatment **the bagasse fibers** 1 with NaOH increases the composite strength. The increase in strength was also affected by the increase in the

wt.% ratio of bagasse/PP as shown **in** Fig. 6. **The** 1

increase in fiber length used did not influence the strength consistently as the other factor such as the homogeneous mixture of bagasse fibers and PP was intervened in the result. The composites strength obtained with the increment of fiber length after NaOH treatment in 20 wt.% bagasse fibers used were 16.83 MPa (3 cm fibers), 8.31 MPa (5 cm fibers), 11.25 MPa (original length fibers). Increasing the amount of bagasse fibers to 25 wt.%, the strength of composites increases into 13.16 MPa (5 cm fibers), 12.94 MPa (original length fibers) excepting for 3 cm fibers used which gave a strength of 10 MPa. This finding was not clear yet in which other aspect such as the homogeneity of the bagasse/PP mixture had its influence and needs to be further studied. Adding bagasse fibers to 30 wt.% to PP increases the composites strength further into 20.59 MPa (3 cm fibers), 15.92 MPa (5cm fibers), 14.92 MPa (original length fibers).

tensile strength (MPa) 16 14 12 10 8 6 4 2 0 24

20/80 3 cm treated with

Ca(OH)2 5 cm **treated** with **Ca(OH)2** 18

original length treated with Ca(OH)2 25

/75 30/70 % weight ratio bagasse fiber/PP 1

30

25 20 tensile strength (MPa) 15 10 5 0 32

20/80 3 cm treated with NaOH 5 cm treated with NaOH original length treated with NaOH 25/75 %

weight ratio bagasse fiber/PP 30/70 **FIGURE** 1

5. Tensile strength of composite samples FIGURE 6.

Tensile strength of composite samples using different fiber **length** and % wt. ratio **of** 1 bagasse using

different fiber length and % wt. ratio of bagasse fiber/PP 1

(treated with Ca(OH)2) fiber/PP (treated with NaOH) Structural Characterization of Fracture Surface of Composites Figure 7a,b shows an evaluation of the fracture surfaces of 20/80 wt.% bagasse/PP composites reinforced with 5 cm bagasse fibers experienced an alkali treatment using Ca(OH)2. The micrograph in Fig. 7a shows that PP has wetted the fiber as it shows by the surface profile of the void's inner wall left by the fiber. The void observed was quite large in which possibly a bundle of fibers was pulled out from the matrix. Although wetting by the matrix occurred on

the fibers surface, **the adhesion between fiber and matrix was** 34

weak as it gives low strength (8.51 MPa) to the composite produced. This result has been well explained by the FTIR spectra and SEM evaluation on the fiber after alkali treatment using Ca(OH)2 that the treatment was ineffective in removing all substances which can hinder the adhesion, i.e. lignin and hemicellulose. The presence of CaCO3 deposit (Fig. 2b) on fiber surface gave another additional problem to develop good adhesion or interfacial strength. Clustering fiber bundles (Fig. 7b) was also observed in the structure. The SEM observation on fiber surface after NaOH treatment shows the removal of the lignin and hemicellulose by the NaOH solution (Fig. 2c). The structural study done with SEM (Fig. 7c and 7d) was confirmed by FTIR spectra on this fiber (Fig. 1). Figure 7c shows the cross section of fracture surface of tensile test composite sample. The cross section area shows very few sugarcane bagasse fibers which it seems less than their understood composition, i.e. 25/75 wt.% ratio bagasse/PP. The bagasse fibers orientations in that composite were shown in the tranversal orientation. Structural of this type contributes the tensile strength of composites to 10 MPa which is an increase of 18% compared to the strength of composites with bagasse fibber treated using Ca(OH)2. Sodium hydroxide treatment results in better mechanical bonding compared to the composite obtained with the treatment with Ca(OH)2. Composites with 4 hours NaOH treated sugar cane fibers increase tensile strength up to 20,59 MPa.

a) b) c) d) FIGURE 7. **SEM micrographs of fracture surface of** 28

tensile test samples of a), b) composites with 20/80 wt% bagasse fibers/PP with fiber length of 5 cm and treated with Ca(OH)2 and c), d) composites with 25/75 wt% bagasse fibers/PP with fiber length of 3 cm and treated with NaOH CONCLUSIONS Sodium hydroxide treatment on bagasse fibers was found more effective than alkali treatment using Ca(OH)2 solution. FTIR spectra and SEM study on fibers surface revealed a significant change on their surface structure.

Lignin and hemicellulose were removed from the fibers **surface** and fibrillation **of** 13

cellulose fibers was obvious with treatment in Ca(OH)2 solution. Tensile strength of the composites produced from Ca(OH)2 treated fibers reinforced PP was found lower in all fiber length compared to composites using NaOH treated fibers.

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