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PAPER SHEETS MADE FROM SUGARCANE BAGASSE AND LEMONGRASS BY-PRODUCTS: SYNTHESIS AND PROPERTIES

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ABSTRACT

Plastic single-use packaging is one of the largest contributors to plastic pollution in Vietnam as well as in many other countries. Alternative materials, especially materials derived from natural and renewable sources, should be developed to solve this global issue. In this study, we aimed to investigate the synthesis and properties of packaging paper sheets from sugarcane bagasse and lemongrass by-products. The delignification of the biomass was implemented at different NaOH/biomass ratios and hydrolysis times while the paper making process was studied at various sugarcane bagasse/lemongrass ratios and different amounts of glycerol and starch additives. The obtained paper sheets were then tested for their mechanic properties and water absorption through ASTM (American Society for Testing and Materials) procedures, and the biodegradability by scanning electron microscopy (SEM). The results showed that the paper sheets at the optimized conditions had low thickness (0.3mm), density (0.4 g cm^{-3}), and water absorption but high tensile strength (19 N mm^{-2}) and flexural modulus (17 N). These properties and their biodegradability suggest that the paper sheets could potentially be used in packaging.

Keywords: Biodegradability, lemongrass, optimization, paper sheet making, sugarcane bagasse, tensile strength.

1. INTRODUCTION

White pollution or plastic pollution has become a global problem that has many severely negative impacts on humans and the ecosystem. One of the biggest sources of plastic pollution is about 400 million tons of single-use packaging that accounts for 36% of the total plastic production annually (UNEP, 2018). It is urgent to find out alternative sustainable materials to replace plastic in the packaging industry. Renewable agricultural by-product fibers have attracted much attention from researchers worldwide to serve as substitutes for petroleum-based polymers which can be highly contaminated by hazardous substances such as PAH (Rochman *et al.*, 2013), PCB (Pascall *et al.*, 2005) or heavy metals (Alam *et al.*, 2018a; Alam *et al.*, 2018b).

There is a lot of agricultural waste containing fiber in Vietnam such as sugarcane bagasse, lemongrass bagasse (by-product of lemongrass after essential oil distillation), rice straw, banana pseudostems and leaves, and Zingiberaceae leaves. Sugarcane bagasse compositions are mainly cellulose (about 60%), hemicellulose (20%), glucose (10%), and some other ingredients. Although sugarcane bagasse is considered the ideal raw material for producing many new products due to its abundant supply and stable price, it needs to be modified to obtain the desired mechanical properties. For example, cellulosic fibers from bagasse were combined with powders to form composite materials; combined with gelatin, starch, and agar for making tableware; or mixed with wood pulp resins and other natural fibers (Loh *et al.*, 2013). The cellulose content in lemongrass by-products is about 40% (Kaur & Dutt, 2013). When combined with sugarcane bagasse, the difference in cellulose content and polymeric chain lengths of the different fibers may enhance some mechanical properties of the obtained composites (Agustina *et al.*, 2019).

It was also published that fibers from agricultural by-products could be used to make paper sheets. In Oman, Khalsa Al-Sulaimani and his research group (Al-Sulaimani *et al.*, 2017) produced handmade paper from bagasse and banana stalk fiber. The research team investigated three composition formulas: formula 1 had only raw materials from bagasse and banana, formula 2 and 3 were fibers mixed with $CaCO_3$ fillers (2% and 5%), respectively, and starch (2%-5%) for adhesion. The research results showed that adding additives increased adhesion and the whiteness of the paper while reducing the paper thickness. Bagasse is more rigid than banana fiber so it is suitable for wrapping paper, while banana fiber is very suitable for making soft paper such as tissue paper.

Although these fibers are important resources, there are few environmental-friendly applications of these materials in use now in Vietnam. The major quantity of sugarcane bagasse in Vietnam is used for low value thermal purposes resulting in carbon release. Sugarcane and lemon bagasse have been sometimes used as microbiological fertilizer (Pham, 2017) or animal feed (Hoang, 2017). However, huge amounts of these by-products are discharged into the environment or burned, resulting in a waste of resources and increased greenhouse gas emissions.

In our previous initial study, we found that sugarcane and lemongrass bagasse could be combined to generate bio composites (Ngo *et al.*, 2018). In this study, we aimed to systematically investigate the synthesis and properties of paper sheets made by different combinations of sugarcane bagasse and lemongrass by-products.

2. MATERIALS AND METHODS

2.1. Raw materials preparation

The bagasse and lemongrass by-products were collected in Hanoi. The collected materials were dried in an oven (42°C) for 3 days, cut into small pieces with average lengths of ~3cm, again dried at 42°C overnight, and then kept in plastic seal pack bags until further utilization.

Delignification (Isolation of lignin from biomass)

To produce paper sheets, the biomass was first delignified to separate the fibers from the lignin, a group of substances that attach the fibers with each other. To determine the optimized parameters of delignification, the samples were treated with NaOH at different ratios (from 4 to 20% NaOH/biomass) and different NaOH concentrations (1M to 5M) and the samples were denoted as TN1.1 to TN1.4. The samples were treated for various periods of time (from 0.5h to 2h) corresponding to samples TN2.1 to TN2.4. After the delignification, the solid fractions were separated by muslin cloth, rinsed with water until neutral, and dried in the oven (42°C) for 3 days. The solid fractions were weighed before being mixed with 1L of water and grinding to form a pulp mixture. The paper making yield (%) was determined by Formula (1):

Making yield (%) = Paper sheet weight/ Delignified biomass weight × 100 (1)

2.2. Paper Making

The above-mentioned pulp was used to make sheets of paper. In this step, additives (glycerol and starch) were added to the pulp and the ratios of the biomass were varied to enhance the properties of the paper. Glycerol was added at the amounts of 0, 2, 4, 6, and 8ml for 50g of initial biomass (TN3.1 to TN3.5). The mass ratios of the sugarcane bagasse and lemongrass by-products were 100-0, 90-10, 80-20, and 70-30 (TN4.1 to TN4.4). Starch was added at 2, 4, and 6% for 2 ratios of the biomass (TN5.1 to TN5.6)

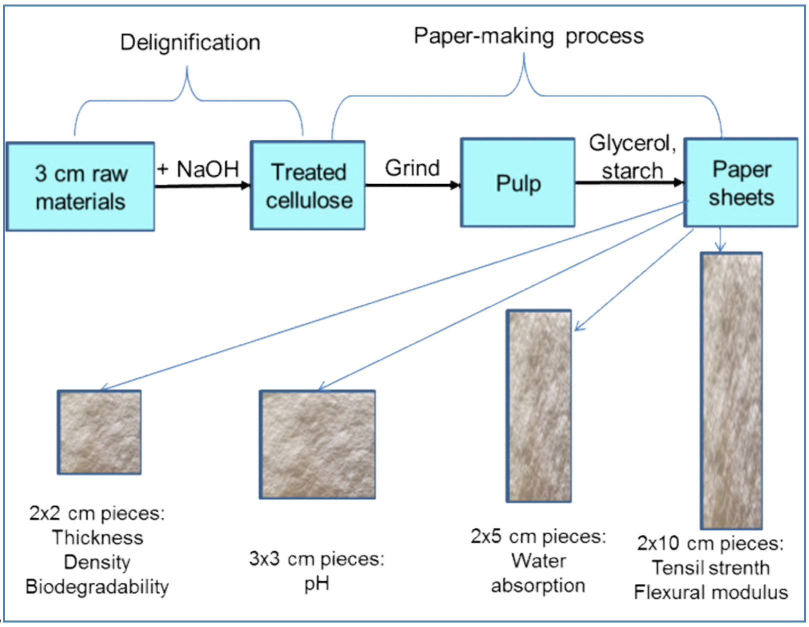


Figure 1. Synthesis and characterization diagram of the paper sheets

2.3. Paper testing

The pH, thickness, density, tensile strength, flexural modulus, and water absorption were determined from the obtained paper sheets according to respective ASTM. In addition, the biodegradability test was carried out according to the published procedure of Marichelvam *et al.* (2019).

2.3.1. pH test

To determine the pH of the paper sheets, 3 × 3cm pieces were immersed in 50ml of distilled water for 5h. The pH values of the solutions were then measured with a pH meter (HANNA HI98107).

2.3.2. Thickness and density determination

The thicknesses of paper pieces sized 2 × 2cm were determined with an electronic caliper (Mitutoyo) (Figure 1). The pieces were also weighed in an analytical balance. The density of each sample was calculated by Formula (2):

$$d = \frac{m}{2 \times 2 \times h} \quad (2)$$

in which d, m, and h are the density (g cm^{-3}), the mass (g), and the thickness (cm) of the sample, respectively.

2.3.3. Tensile strength and flexural modulus

Based on ASTM D638 and ASTM D790, the tensile strength and flexural modulus were measured in a universal testing machine. Three paper bands of 2×10 cm for each sample were tested to determine the average values.

2.3.4. Water absorption

Water absorption of the paper sheets was determined at room conditions (ASTM D570-98). The paper pieces of 5 × 2cm were weighed before (w_i) and after (w_f) the water absorption process. Each sample was tested 3 times to get an average value. The percent of absorbed water was calculated by Formula (3):

$$\% \, w_{water} = [(w_f - w_i)/w_i] \times 100 \quad (3)$$

where w_{water} is the weight percentage of the water absorbed by the paper, w_i is the weight of the dry paper sample, and w_f is the weight of the saturated paper sample.

2.3.5. Biodegradability Test

The biodegradability of the best sample (paper with high tensile strength, low density, and low water absorption) was determined. This sample was cut into pieces of 2 × 2cm. Soil (500g) from a soybean field with a slight moisture content was collected and stored in a container. Three pieces of the paper sample were buried inside the soil at a depth of 5cm and 3 others were buried at a depth of 10cm for 15 days at room conditions. The weights of the pieces were measured before and after the testing. The biodegradability of each sample was calculated by Formula (4):

Weight Loss (%) = $[(w_o - w)/w_o] \times 100$, (4)

where w_o and w are the average masses of the pieces before and after the test, respectively.

The microstructures of the samples before and after the biodegradability test were recorded by scanning electron microscopy (SEM) on an FEI Nova NanoSEM system.

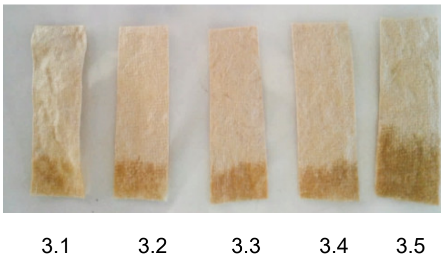
2.4. Data analysis

Data were processed in Excel 2010, Mathlab 2016, and Origin 8.0 software. One-way Anova analysis was performed to compare samples with significant differences at $p < 0.05$.

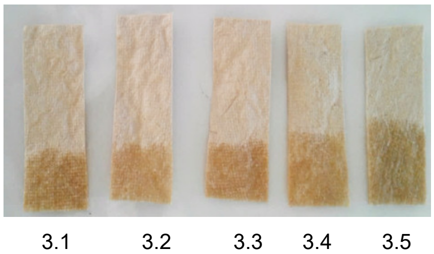
3. RESULTS AND DISCUSSION

3.1. Delignification

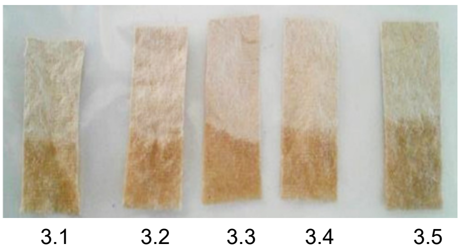
The main component of paper is cellulose fibers. In sugarcane bagasse and lemongrass by-products, in addition to the main component of cellulose, there are also other components of hemicellulose and lignin. Therefore, it was necessary to use alkali to separate the hemicellulose and lignin from the biomass. Sodium hydroxide solution can separate lignin, hemicellulose, and other solutes (sugar, ash, wax, and protein, among others) from biomass. NaOH is capable of breaking down cell walls because the alkali dissolves the lignin and hemicellulose and also breaks down the α-ether links between the lignin and hemicellulose. The remaining lignin in the pulp makes it become dark yellow or brown (affecting the sensory value of the paper) and hard (Kido, 2016). Therefore, the ratio of NaOH/biomass is an important factor affecting the hydrolysis of the biomass. For a certain amount of biomass, if too little NaOH is used, it will not be enough to thoroughly remove the lignin. Conversely, if the amount of NaOH used is too high, when the hydrolysis process has ended, the remaining NaOH will lead to waste of chemicals and water to neutralize the biomass. Therefore, the NaOH/biomass ratio was first investigated. The results are shown in Table 1.



(a)



(b)



(c)

Figure 2. Water absorption of the TN3 series at (a) 15s, (b) 30s, and (c) 1 min

Table 1. Effects of the NaOH/biomass ratio on delignification

Sample	NaOH/ biomass ratio (%)	Delignified biomass weight (g)	Paper sheet weight (g)	Papermaking yield (%)	Thickness (mm)	Obtained fibers	Obtained paper sheet
TN1.1	4	42.75^{a} ± 0.26	34.06^{a} ± 0.36	80.9 ± 1.0	1.61^{a} ± 0.38	Difficult to grind, hard fibers with the size range of 2-2.5 cm	
TN1.2	10	32.90^{b} ± 0.15	30.38^{b} ± 0.23	92.7 ± 0.8	1.41^{a} ± 0.24	Less difficult to grind, hard fibers with the size range of 1.5-2 cm	
TN1.3	14	28.80^{c} ± 0.18	26.14^{c} ± 0.27	91.2 ± 1.1	1.11^{b} ± 0.08	Easy to grind, soft and short fibers	
TN1.4	20	28.49^{c} ± 0.07	25.26^{c} ± 0.32	89.1 ± 1.1	1.05^{b} ± 0.16	Easy to grind, soft and short fibers	

Note: Values with the same letter within each column are not significantly different at the 5% level.

The results of Table 1 show that, when the ratio of NaOH/biomass increased from 4 to 20%, the remaining biomass weight after hydrolysis decreased. When this weight increased to 20%, the remaining amount of biomass decreased insignificantly compared to that at the ratio of 14%. This reveals that the 14% ratio is enough to hydrolyze the biomass.

In addition, the paper-making yield was only different when the NaOH/biomass ratio used was 4%. The reason for this is that the low paper yield was due to the fact that the cellulosic fibers had not been separated due to an incomplete lignin reaction, thus their ability to bond when paper-making was low. In the remaining three cases, the paper making yields were not significantly different, and about 90% of the weight of the delignified biomass could be converted into a paper sheet. However, in the samples TN1.2 and TN1.1, the cellulose fiber bundles were difficult to split, and the raw fibers were clearly visible on the finished paper. Meanwhile, samples TN1.3 and TN1.4 had much better fineness. Thus, combining the above parameters, the NaOH/biomass ratio of 14% (TN1.3) was selected to carry out the next studies.

The effects of hydrolysis time on the lignification (Table 2) show that when the hydrolysis time increased, the weight of the delignified biomass weight decreased, in which there was no observed significant difference between TN2.3 and TN2.4. The paper sheet yield did not vary much among the samples, however, the resulting paper had a good smoothness in the TN2.3 and TN2.4 formulas. Thus, we chose these two samples to perform hydrolysis for 2h in subsequent tests. In a published study involving the sugarcane bagasse/starch/PVA composite, the NaOH treatment changed the surface of the fibers which in turn changed the wettability of the samples (Zhu *et al.*, 2019).

3.2. Papermaking process

3.2.1. Effects of the glycerol amount on the paper properties

Adding glycerol (2, 4, 6, or 8ml) to the pulp made it easier to form paper sheets with a much smaller thickness compared to the sample without glycerol (Table 3). Table 3 also shows that the added amount of glycerol had effects on the tensile strength and flexural modulus. However, 2ml of glycerol (TN3.2) was not enough to significantly change the tensile strength and flexural modulus for the obtained paper compared to the sample without glycerol. When the added glycerol amount increased to 4 ml and higher, the tensile strength and flexural modulus of these samples increased significantly compared to the TN3.1 and TN3.2 samples. Among samples TN3.3, TN3.4, and TN3.5, there were some significant changes in the tensile strength and flexural modulus. Increasing the glycerol amount appeared to raise the tensile strength and flexural modulus of the samples. It has been reported that glycerol could lubricate the cellulose fibers to make the links between cellulose fibers tighter, resulting in pulp that was then easier to make into paper with a smaller thickness, and greater density, flexural strength, and tensile strength (Cruz *et al.,* 2017). Although, glycerol is water-soluble, in order to select the appropriate glycerol ratio, it was necessary to further consider the water absorption properties of the paper. Water absorption is greatly affected by the structural integrity of the paper. The cause of this phenomenon is that the natural capillaries found in bagasse make the paper quickly reach hydration equilibrium. The results of the water absorption test of this sample series are presented in Figure 2 and Figure 3. When not using glycerol, after only 15s in contact with water, the paper bands absorbed water more than 50% of their weight. After 30 s, they absorbed 100% of the water. At the contact time of 15s, TN3.2 (2ml glycerol added) was the least water absorbent sample. However, after 60s, the water absorbed amounts in TN3.2, TN3.3, and TN3.4 became close to that of each other. These differences were not statistically significant. TN3.1 and TN3.5 showed the highest water absorption. This can be explained by the fact that without glycerol (TN3.1), water was more easily able to go into the cellulose fibers in the biomass, while with too much glycerol (TN3.5), water could be absorbed into the paper sheets through the glycerol dissolution process. These results suggested that 4 ml of glycerol was enough to absorb and connect the fibers in the biomass. However, based on the three properties of tensile strength, flexural modulus (Table 3), and absorption, the TN3.4 condition (6ml glycerol) was chosen to proceed with other experiments.

3.2.2. Effects of the sugarcane bagasse/ lemongrass ratio on the paper properties

Table 4 shows the effect of the lemongrass composition on the paper properties. It was obvious that when adding lemongrass, the paper sheets became thinner with a higher density, higher tensile strength, and higher flexural modulus. The differences between the strength of the cellulose fibers (due to the length of the cellulose chains) of the sugarcane bagasse and lemongrass could be the reason for the significant changes in these parameters between TN4.2, TN4.3, or TN4.4 and TN4.1. Figure 4 also demonstrates that the higher the ratio of lemongrass, the less water was absorbed into the paper sheets. These results also suggested that the combination could be an effective approach to controlling the paper properties. In our previous study, the composite samples with lower contents of lemongrass also showed lower tensile strength and flexural modulus (Ngo *et al.*, 2018). For further experiments, the sugarcane bagasse/lemongrass ratios of 80/20 and 70/30 were chosen. Another study showed a similar dependence of the composite mechanical properties on their composition (Agustina *et al.*, 2019). Different ratio mixtures of sugarcane bagasse and pineapple leaves were also investigated to prepare paper pulp resulting in papers with varied tensile and tearing strengths (Evelyn *et al.*, 2019).

3.2.3. Effects of the added starch amount on the paper properties

All the researched samples were neutral. The thickness and density of the samples in this experiment series did not show much variation. The results in Table 5 reveal that adding starch at the ratios of 4% and 6% for both ratios of biomass (80/20 and 70/30) made the tensile strength and flexural modulus of the samples significantly ($P < 0.5$) greater than that at the ratio of 2%. This showed that when starch was used, the links between the cellulose fibers increased leading to stronger and more flexural paper. The tensile strength and flexural modulus of the samples at the ratios of 4% and 6% starch were not significantly different at the 5% significance level. There ws a similar trend in the water absorption of the samples (Figure 5).

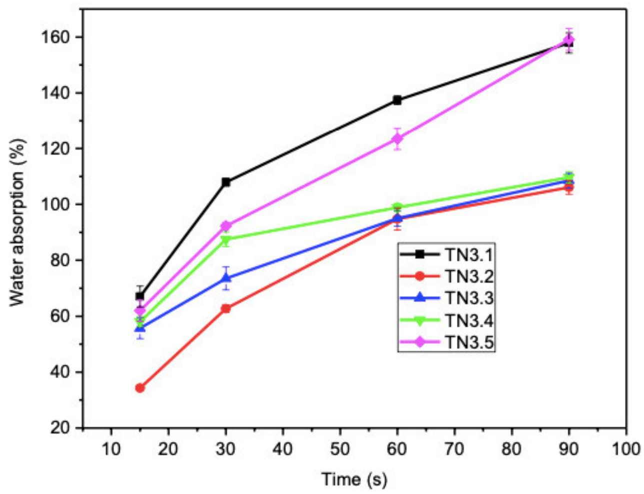


Figure 3. Effect of the added glycerol amount on the water adsorption capacity

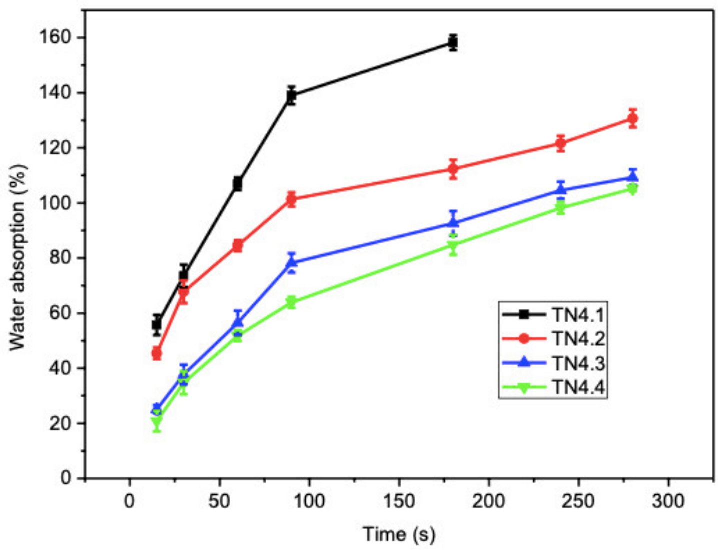


Figure 4. Effects of the sugarcane bagasse/lemongrass on the water absorption of the paper sheets

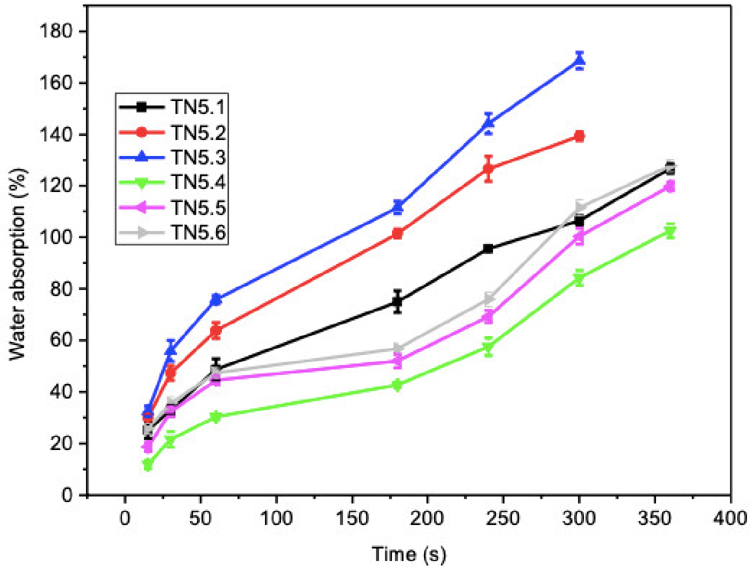


Figure 5. Effects of the starch amount on the water absorption of the paper sheets

Table 2. Effects of reaction time on the delignification of the biomass

Sample	Time (h)	Delignified biomass weight (g)	Paper sheet weight (g)	Papermaking yield (%)	Thickness (cm)	Obtained fibers	Obtained paper sheet
TN2.1	0.5	29.43 ± 0.32	28.96 ± 0.12	92.1 ± 1.1	1.13[a] ± 0.20	Difficult to grind, hard fibers with the size range of 2-2.5cm	
TN2.2	1	27.18 ± 0.21	27.00 ± 0.25	99.3 ± 1.2	0.94[a] ± 0.26	Less difficult to grind, hard fibers with the size range of 1.5-2cm	
TN2.3	2	24.52 ± 0.17	24.33 ± 0.13	99.2 ± 0.9	0.77[b] ± 0.11	Easy to grind, soft and short fibers	
TN2.4	3	24.03 ± 0.18	23.71 ± 0.10	98.7 ± 0.8	0.73[b] ± 0.10	Easy to grind, soft and short fibers	

Note: Values with the same letter within each coulmn are not significantly different at the 5% level.

Therefore, 4% starch was an appropriate amount for the papermaking process. The data showed that if the links between cellulose fibers were too tight, then the tensile and flexural strength would increase very little when more starch was used. The ratio of 4% was the ratio of the flexural strength and the tensile strength of the hearing.

3.3. Biodegradability properties

The biodegradability test results are shown in Figure 6. The optimized sample pieces of TN5.5 were buried in soil at different depths of 5 cm and 10 cm. The initial average weight of the TN5.5 pieces was 0.0562 ± 0.0017. After 15 days, the weights of the TN5.5 pieces at 5 cm and 10 cm-depths were 0.0367 ± 0.0015 and 0.0282 ± 0.0018g, respectively. These 34.7% and 49.8% weight losses indicated the biodegradation of the samples. The SEM images (Figure 6) of the samples also showed a decrease in the thickness of the samples. There was a difference in the biodegradability rate between the 2 depths. This result differs from those published by Mirachelvam *et al.* (2019). This could be due to the differences in the composition of the samples in the current study and referent research.

Table 3. Effects of added glycerol amount on the paper properties

Formula	Glycerol amount (ml)	pH	Thickness (mm)	density (g cm^{-3})	Tensile strength (N mm^{-2})	Flexural modulus (N)
TN3.1	0	6.4	0.75^{a} ± 0.05	0.28 ± 0.03	6.30^{a} ± 0.10	0.61^{a} ± 0.10
TN3.2	2	6.3	0.48^{b} ± 0.05	0.35 ± 0.06	8.23^{b} ± 0.05	0.70^{a} ± 0.10
TN3.3	4	6.5	0.47^{b} ± 0.04	0.32 ± 0.06	14.33^{c} ± 0.11	0.93^{b} ± 0.05
TN3.4	6	6.5	0.46^{b} ± 0.04	0.34 ± 0.04	17.70^{d} ± 0.70	1.10^{bc} ± 0.10
TN3.5	8	6.4	0.46^{b} ± 0.08	0.34 ± 0.09	24.00^{e} ± 0.81	1.23^{c} ± 0.05

Note: Values with the same letter within each column are not significantly different at the 5% level.

Table 4. Effects of the sugarcane bagasse and lemongrass byproduct ratio on the paper properties

Formula	Sugarcane bagasse/lemongrass by-product ratio	pH	Thickness (mm)	density (g cm^{-3})	Tensile strength (N mm^{-2})	Flexural modulus (N)
TN4.1	100/0	6.3	0.46 ± 0.09^{a}	0.31 ± 0.09	16.27^{a} ± 2.04	1.13^{a} ± 0.06
TN4.2	90/10	6.3	0.29 ± 0.03^{b}	0.48 ± 0.07	18.07^{ab} ± 1.72	1.31^{b} ± 0.10
TN4.3	80/20	6.2	0.30 ± 0.03^{b}	0.42 ± 0.05	20.43^{bc} ± 0.81	1.33^{b} ± 0.06
TN4.4	70/30	6.3	0.33 ± 0.03^{b}	0.43 ± 0.07	21.46^{c} ± 0.33	1.43^{b} ± 0.05

Note: Values with the same letter within each column are not significantly different at the 5% level.

Table 5. Effects of the added starch amount on the paper properties

Formula	Sugarcane bagasse/lemongrass by-product ratio	Starch amount (%)	pH	Thickness (mm)	density (g cm^{-3})	Tensile strength (N mm^{-2})	Flexural modulus (N)
TN5.1	80/20	2	6.3	0.32^{a} ± 0.01	0.43 ± 0.05	11.87^{a} ± 0.31	0.93^{a} ± 0.15
TN5.2	80/20	4	6.4	0.30^{a} ± 0.01	0.50 ± 0.04	18.33^{b} ± 0.15	1.17^{ab} ± 0.15
TN5.3	80/20	6	6.4	0.30^{a} ± 0.02	0.44 ± 0.07	18.80^{b} ± 0.41	1.31^{b} ± 0.10
TN5.4	70/30	2	6.3	0.31 ^{a}± 0.01	0.48 ± 0.03	16.30^{c} ± 0.10	1.32^{b} ± 0.11
TN5.5	70/30	4	6.4	0.32^{a} ± 0.01	0.44 ± 0.04	18.90^{b} ± 0.36	1.63^{c} ± 0.11
TN5.6	70/30	6	6.3	0.33^{a} ± 0.02	0.39 ± 0.02	19.60^{b} ± 0.56	1.77^{c} ± 0.06

Note: Values with the same letter within each column are not significantly different at the 5% level.

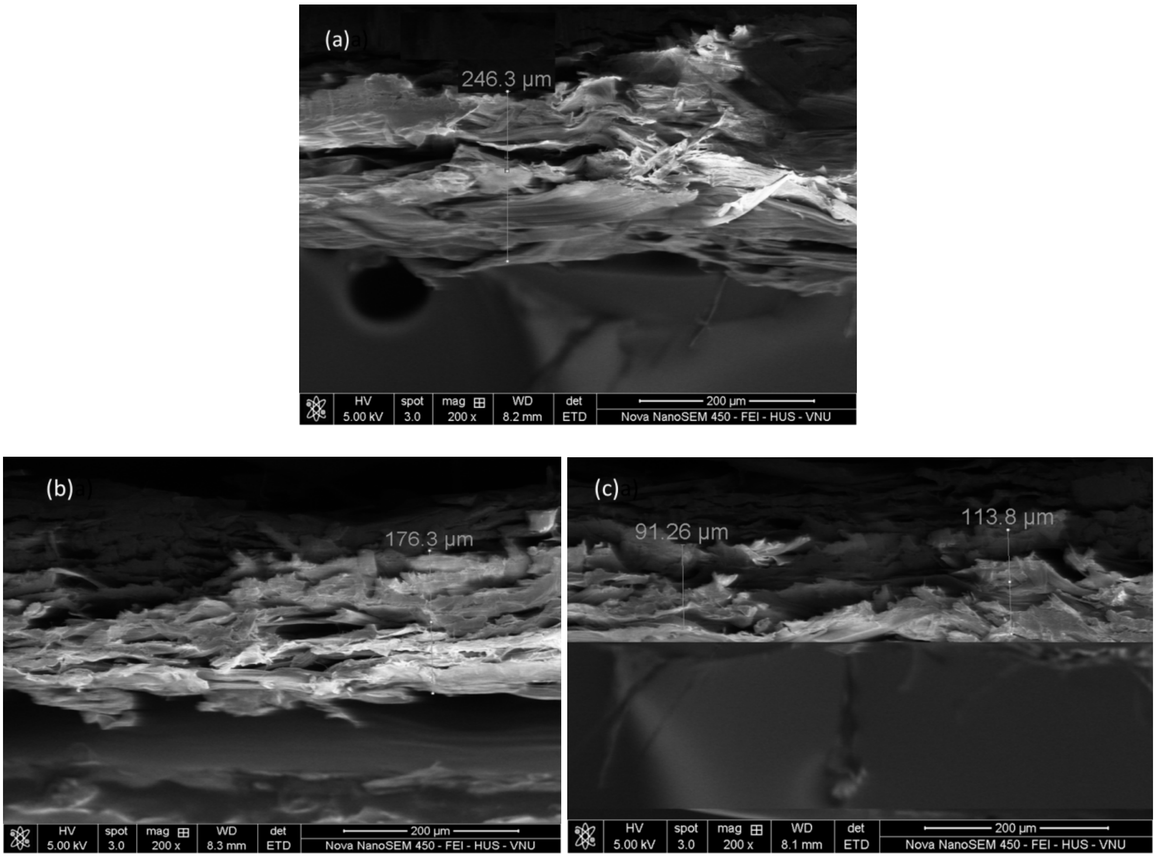


Figure 6. SEM images of the TN5.5 pieces before (a) and after the biodegradability test at 5-cm (b) and 10-cm depth (c)

4. CONCLUSIONS

In conclusion, the optimized conditions of the synthesis of paper sheets from sugarcane bagasse and lemongrass by-products have been investigated. To obtain paper sheets with high tensile strength and flexural modulus, and with low thickness and water absorption, the biomass should be treated with 14% NaOH (w/w to biomass) for 2h. The sugarcane bagasse/lemongrass ratio could be 80/20 or 70/30, and should then be mixed with 6ml glycerol and4% starch before making starch. The obtained paper sheets had good physical properties with the thickness of ~0.3 mm, small density of ~0.4 g cm^{-3}, tensile strength of ~19 N mm^{-2}, and flexural modulus of ~1.7 N. The paper sheets also exhibited low water absorption and the ability to biodegrade up to 49.8% in the soil layer at a 10 cm depth after 15 days. Based on the obtained results, it would be possible to prepare bio bags or bio containers from the optimized paper sheets.

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