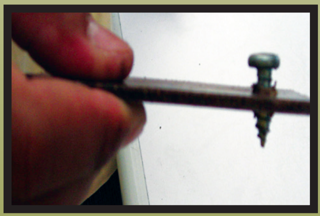
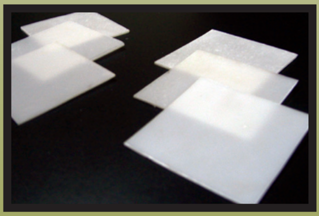
TECHNICAL BULLETIN

Vol. 2 No. 2
ISSN 2243-8483





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2012

ISSN: 2243-8483

Bibliographic Citation:
E.D. Flores, R.C. Martinez, M.C.B. Gragasin, R.P. Gregorio,
and D.S. Ognayon. 2012. Biomass Utilization as Biofiller for Biocomposite
Materials Development. PHilMech Technical Bulletin No. 4.
Philippine Center for Postharvest Development and Mechanization.
Science City of Muñoz, Nueva Ecija. 34p.

Cover photos by E.D. Flores

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CLSU Compound, Science City of Muñoz, Nueva Ecija, 2012

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ABSTRACT

The project aimed to utilize biomass as biofiller in the development of biocomposite materials. Potato and cassava starch and agriwastes such as corn cobs, bagasse and cocohusk were used in the study.

Biocomposites filled with starch and agriwastes were prepared by hot mixing and compression method. Biocomposites developed were characterized based on the International Organization for Standardization (ISO) standards. Density, water absorption and thickness swelling of starch and agriwaste-filled biocomposites increased with increasing biofiller content. Similarly, water absorption and thickness swelling of the biocomposites increased with increasing biofiller content.

Agriwastes-filled biocomposites which have smaller particle size of biofiller have lower water absorption and thickness swelling than with larger particles. Agriwastes-filled biocomposites with non-pretreated biofiller have lower water absorption and thickness swelling than with a pretreated one. Melting temperature of starch and agriwastes-filled biocomposites increased with the increasing starch and agriwaste content. Agriwastes-filled biocomposites with smaller particle sizes of biofiller have lower melting temperatures than with larger ones. Starch and agriwastes-filled biocomposites showed a decreasing tensile strength with increasing biofiller content. Biofiller particle size did not show any significant influence on the strength of agriwastes-filled biocomposites. The elongation at break of starch-filled biocomposites decreased with increasing starch content.

Biodegradation was observed for both starch and agriwastes-filled biocomposites as indicated by their reduction in weight under natural soil burial test. Increasing the amount of biofiller also increased the biodegradation of the biocomposites. The corn cobs and bagasse-filled biocomposites with 20 wt% biofiller met the specified range of properties of conventional plastic composites. A water resistant, termite and decay control additives can be incorporated depending on the composite utilization purposes. For exterior applications, an ultraviolet stabilizer could be incorporated as protection from UV light degradation.

The measured properties of starch-filled biocomposites were comparable to some properties of low density polyethylene (LDPE) and within the range properties of high density polyethylene (HDPE). The starch-filled biocomposites can be potentially used as thermoformed, injection molded and blown film plastic products with some modifications depending on the specific applications. Sustainability assessment should be done to determine the socio-economic and environmental impacts and benefits that can be derived from using biomass as filler in the development of biocomposite materials.

INTRODUCTION

The world supply of fossil resources is becoming limited and too expensive. It is estimated that known global resources of oil will run dry in 80 years, natural gas in 70 years and coal in 700 years, but the economic impact of diminishing resources could hit much sooner, as prices will likely soar as resources dwindle (Grengross and Slater, 2000).

Aside from the energy and transportation sectors, the world's plastic industry is heavily dependent on petroleum resources. About 245 million tons of plastic are produced annually from petroleum oil. Likewise, problems on the disposal of plastic wastes made from petroleum have become a public concern due to their non-biodegradability and their potentially hazardous degradation products from incineration (Nabar and Narayan, 2004).

In Asia, the Philippines ranked second to Hongkong in the importation of plastic materials in the form of resin, pre-formed sheets and other finished packaging products (Zadgaonkar, 2004). Another environmental problem is the isue of global deforestation. It is estimated that 11 million hectare of forestland is lost globally each year due to deforestation and production of wood for industrial purposes. Wood has been traditionally the most extensively used material in the production of pulp, furniture and boards of various types. as well as being a source for fuel and energy. Increasing demand for these raw materials, coupled with social-economic and environmental factors, make it necessary to find alternative sources of lignocellulosic material (Garay, et al., 2009) and to develop novel products that are environment friendly and use less fossil fuel.

Currently, total replacement of petroleum-based materials in terms of cost and performance perspective is hard to attain. However, it is not necessary to make 100 percent substitution for petroleum-based materials at once. One promising solution is to blend the distinct features and benefits of both petroleum and biomass resources to produce useful product containing the pre-requisite cost and performance properties for applications. Alternative materials especially “green composites” sometimes called biocomposites fit well into this paradigm shift (Mohanty et al., 2005).

Biocomposites are the combination of natural fillers with polymer matrices both from renewable and nonrenewable resources. Biocomposite products are categorized as low biobased content product (20 percent or less biobased content), medium biobased content product (21 to 50 percent biobased content) and high biobased content product (51 to 90 percent biobased content)(Mohanty et al., 2005).

One of the commercially available biodegradable polymer matrices is the *Polybutylene succinate (PBS)* (Kunioka et al., 2009; Liu et al., 2009) produced by Showa High Polymer Co. and Mitsubishi Co. (Mizukoshi, 2006; Kato et al., 2006). PBS had been developed and synthesized as fully biobased product from biomass resources to reduce greenhouse gas emissions and provide sustainable alternatives to the limited petroleum based resources (Masuda and Kunioka, 2009). However, other properties of PBS such as softness and flexibility are insufficient for various end-use applications. To modify its properties, some methods such as the incorporation of additive, inorganic filler, nanocomposites and other polymer were developed (Ray and Okamoto, 2003). Promotion of high biobased materials requires the use of additives from renewable resources. It is fortunate that Philippines is endowed with abundant biomass resources from agricultural crop wastes (Elauria, 2005). About 5.99 million tons of sugarcane bagasse, 1.3 million tons of corn cobs and 4.34 million tons of coconut husks are produced yearly (PCARRD, 2007; Baconguis, 2007). Starch, a naturally occurring renewable polymer is a potential raw material for the manufacture of biodegradable packaging material. Plastic products containing starch are considered biodegradable because starch is easily transformed by microorganisms into simple compounds (Ohkita and Lee, 2005).

Utilization of these biomass resources for the development of biocomposite materials will reduce dependence on petroleum resources, reduce agricultural wastes and generate more economic opportunities for the agricultural sector.

Recently, PHilMech successfully plasticized PBS with furfural (Flores et al., 2009), a biobased chemical compound produced from biomass resources such as corn cobs, rice hull, sugarcane bagasse and forestry wastes like wood chips and others (Martinez-Gomez et al., 2004). Biomass as renewable resources hold great promise as component of Clean Development Mechanism (CDM) strategies for the reduction of greenhouse gases (GHG) emissions. Biomass is considered a carbon dioxide neutral and does not contribute to global climate change (JBPA). Therefore, the utilization of biomass as a renewable resource is sustainable alternative to costly and limited fossil resources.

OBJECTIVES

General:

To utilize biomass as biofiller in the development of biocomposite materials.

Specific:

1. To explore and collect biomass potential for biofiller
2. To quantify the potential recovery of biofiller from biomass in powder form
3. To prepare biocomposite materials by hot mixing and compression method.
4. To characterize the properties of biocomposite materials based on ISO standards.

METHODOLOGY

Exploration and Collection of Biomass

Agricultural wastes such as corn cobs, bagasse and coconut husk were collected from the major agricultural producing areas that generate vast amount of biomass wastes in the Philippines. Breakdown of biomass materials (by volume or weight) from production per province were undertaken as basis for area of collection of biomass resources. The computation was based on the top 10 crop yield producing provinces obtained from the Bureau of Agricultural Statistics - BAS 2008 and the established crop to waste ratio of 23 percent for cob to corn kernel ratio, 30 percent for bagasse to sugarcane and 30 percent husk to coconut. The collection of biomass samples were done at nearest provinces of Nueva Ecija for immediate collection of samples and other practical reasons.

Aside from agriwastes, starch derived from energy crop biomass such as cassava and potato were also used as biofiller. Typical starch available in the market was purchased and used in this project.

Quantification of Recovery Potential of Collected Agriwastes as Biofiller

Collected agriwastes biomass such as corn cobs, bagasse and coconut husk were prepared as biofillers in powder form with particle sizes ≤ 180, 180-500 and 500-1000 μm. The potential recovery of biofiller from biomass stocks after drying, grinding and fractionation process was quantified.

The collected biomass stocks were manually pre-cleaned by removing dirt and foreign matters (Figure 1). After cleaning, the biomass samples were cut and shredded using a motorized shredding machine. The shredded samples were then washed with distilled water and dried by oven drying method. The dried samples were pulverized using a laboratory grinder, after which the milled samples were fractionated. Fiber surface treatment was also done for other shredded samples by soaking them in an aqueous solution for one hour with stirring to make the samples soft. After soaking, the samples were drained and washed thoroughly with distilled water. Immediately after washing, the samples were disintegrated using a laboratory blender (osterizer) and subsequently dried in an oven at 50^{o}C for 24 hours. The dried samples were further refined using the laboratory grinder and fractionated using a laboratory sieve with vibrator.

The fractionation of biofiller was done using sieve apertures of 180, 500 and 1000 μm (International Organization for Standardization, ISO) (Figure 2). The percent bio-filler recovery was computed by dividing the weight of fractionated samples to the weight of biomass multiplied by 100.

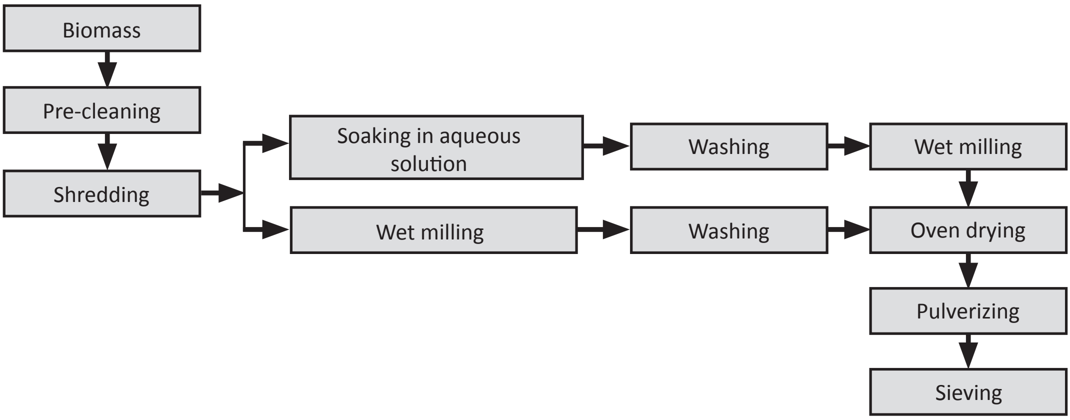

Figure 1. The preparation and quantification of biofiller from agriwastes biomass

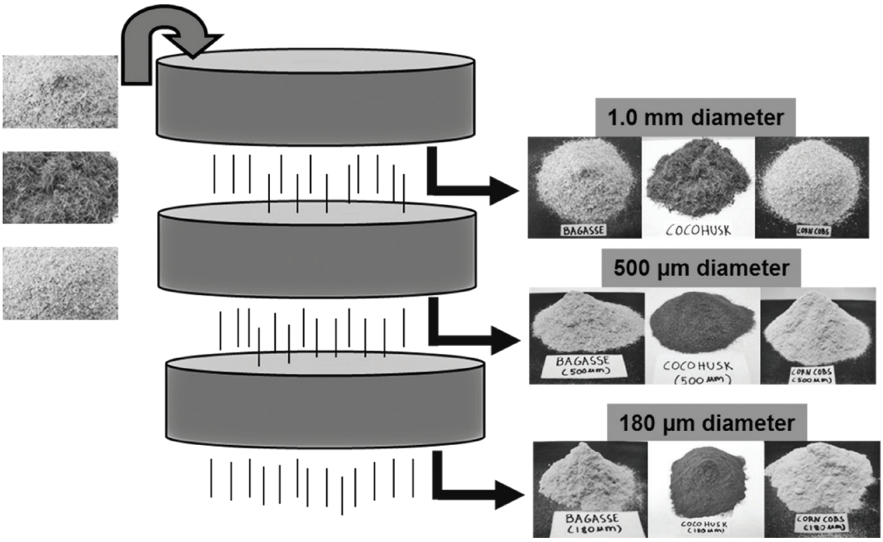

Figure 2. The fractionation of agriwastes biofiller using sieve apertures of 180, 500 and 1000 µm.

Preparation of Biocomposite Materials

The biocomposite materials were prepared by hot mixing and compression method. Biodegradable polymer such as *Polybutylene succinate* (PBS) was used as binder. Starch (potato and cassava) and agriwastes (corn cobs, bagasse and coconut husk) were used as biofillers. The starch and agriwastes were separately blended with the polymer matrix at varying ratio of 20, 40 and 60 wt% using the two-roll mill machine model (Figure 3). The particle sizes of agriwastes used were ≤ 180 µm (small) and ≥ 500 µm (large). After mixing, the samples were hot pressed using the compression molding machine (Figure 4). The biocomposites were conditioned for at least a week at ambient room temperature before laboratory analysis.

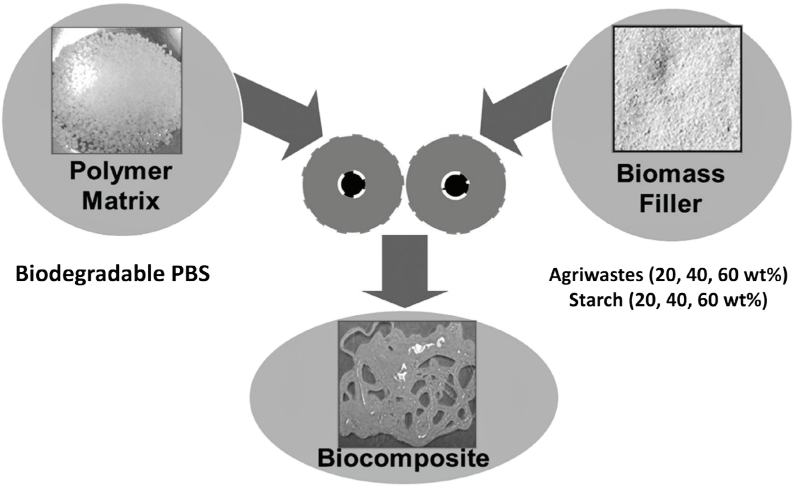

Figure 3. Mixing of composite samples

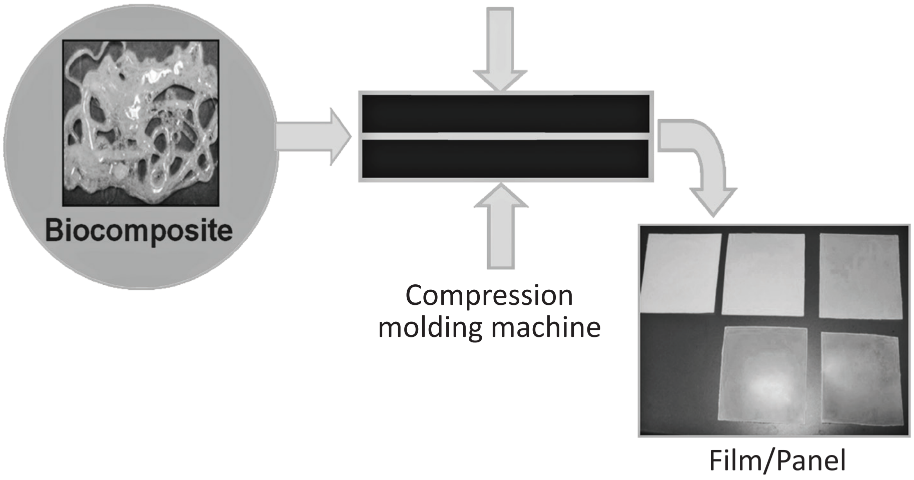

Figure 4. Hot pressing of composites samples

Characterization of Physico-Thermo-Mechanical Properties of Biocomposite Materials

The physico-thermo-mechanical properties of the developed biocomposite materials were determined based on ISO/ASTM standard testing procedures. Biodegradation was also undertaken using the natural soil burial test.

The biocomposite materials prepared were characterized by its properties to determine its potential application in the plastic industry. A data bank describing the properties and existing types of biofillers being used in the industry was prepared. The information was used as reference in matching the properties of the developed biocomposite materials. The properties of the biocomposites developed in the present study were compared with the properties of some traditional plastics commonly used in the plastic industry nationwide.

Physical Properties

Physical properties such as density, water absorption and thickness swelling of the biocomposite materials were measured. An average value was taken from measurements of various samples under the same conditions for each biocomposite film.

The **density** of the biocomposite materials was determined based on ISO 1183 (ASTM D792) method of test using the following formula:

$$\text{Density,} \quad \rho = \frac{W_a \times 0.9975}{W_a + W_w - W_b}$$

Where:

ρ = density of the composite materials, g/cm^3

W_a = weight of the specimen when hung in the air, g

W_w = weight of partly immersed wire holding the specimen, g

W_b = weight of the specimen when immersed fully in distilled water, along with the partly immersed wire holding the specimen, g

0.9975 is the density of distilled water in , g/cm^3 at 23^{0}C

The **water absorption** of the composite materials was determined based on ISO 62 (ASTM-D570) method of test by immersing the specimen to distilled water for 24 hours at room temperature.

The specimen was conditioned by ovendrying for 24 hours at 50°C, and then cooled down in a desiccator. The weight of the samples was recorded before and after soaking the samples in distilled water for 24 hours. The weight of the samples was measured using an electric weighing balance. The water absorption of the samples in percentage was calculated using the formula:

$$\text{Water absorption, } W_{ab} = \frac{W_f - W_i}{W_i} \times 100$$

Where:

W_{ab} = Amount of water absorbed, %
W_i = weight of the specimen before immersion, g
W_f = weight of the specimen after immersion, g

The **thickness swelling** of the biocomposite materials was determined during the water absorption test by measuring and recording the thickness of the specimen before and after soaking the samples for 24 hours at room temperature. The thickness swelling was calculated using the formula:

$$\text{Thickness swelling, } T_s = \frac{T_f - T_i}{T_i} \times 100$$

Where:

T_s = Thickness swelling, %
T_i = Thickness before soaking the specimen, µm
T_f = Thickness after soaking the specimen, µm

Thermal Properties

The melting temperatures (T_m) of the biocomposite materials were determined using a differential scanning calorimeter system (DSC model) following the procedure described in Standard Testing Division (STD), ITDI-DOST. The cut films (10 mg in an aluminum pan) were heated under a nitrogen gas flow at a rate of 10°C/min. The values of the T_m were calculated for the first heating scan.

Mechanical Properties

The melt-pressed composite films/panels were cut into standard specimens based on ISO 527 (ASTM D 638-91) standard test method for tensile properties of plastic. Mechanical properties such as strength and elongation of the biocomposite materials were measured using tensile tests. Tensile tests were carried out using a Shimadzu Autograph Mechanical Property Testing Machine (Universal Material Testing Machine) at room temperature. A grip distance of 25 to 50 mm at a tensile test speed rate of 50 mm min^{-1} was applied.

The tensile strength at break, *δb* (MPa), was determined as the ultimate strength required to break the materials on the stress-strain curve while the elongation at break, *εb* (%), was determined to be the maximum elongation on the stress-strain curve. An average value was taken from measurements of five samples under the same conditions for each composite film. The mechanical properties of the composite materials was done at the Standard Testing Division (STD)-ITDI, DOST using the ISO/ASTM standard testing procedure.

Biodegradation

The biodegradability or capability of the biocomposite materials to undergo decomposition in a specified period of time was investigated under natural soil burial test at PHilMech compound. The natural soil burial test was known to be a slow process however it was noteworthy that it reflected real-life condition better than any other tests (Bastioli, 2005).

The biodegradation rate of the materials was observed each week over a period of four months (120 days). The buried specimen was dug out, washed and dried in an air-drying oven for 24 hours at 50°C and placed in a desiccator before undergoing weight loss determination using an electronic weighing balance (model). The soil texture was composed of 50 percent clay, 21 percent silt and 29 percent sand and the pH was between 5.0 and 6.0. Micro-organisms such as Trichoderma and Fusarium present in the soil used were isolated and identified using direct plating method using Dichloran Rose Bengal Chloramphenicol (DRBC) .

The specimens were weighed and wrapped in synthetic net and placed separately in a 30 cm depth holes. Weight after every week was determined by digging the specimen, washing it with distilled water to remove dirt particles and ovendried for 24 hours. The weight of the specimens was recorded at regular time interval. The percentage weight loss of the specimen as a function of time was determined using the formula:

$$\text{Weight loss, } W_l = \frac{W_b - W_a}{W_b} \times 100$$

Where:

W_l = Weight loss, %

W_b = Weight of the specimen before burying, g

W_a = Weight of the specimen after digging and conditioned, g

Statistical Data Analysis

The data were analyzed using ANOVA and by DMRT for the mean difference observed. Each set of data was run using the STATGRAPHIC plus.

RESULTS

Exploration and Collection of Biomass

Tables 1 to 3 showed the top 10 crop producing provinces of corn, sugarcane and coconut in the Philippines (BAS, 2008). The amount of biomass was calculated based on the top 10 crop yield production and the established crop to waste ratio of 23 percent for corn to cob ratio, 30 percent bagasse to sugarcane ratio and 30 percent husk to coconut ratio. The biomass samples were collected from Pangasinan for corn cobs, Tarlac for bagasse and Quezon province for coconut husk. These were brought to PHilMech in Muñoz, Nueva Ecija.

Table 1. *Top 10 corn cobs producing provinces in the Philippines, BAS, 2008*

RANK	PROVINCES	CORN PRODUCTION (tons/year)	COBS PRODUCTION* (tons/year)
1	Isabela	1,009,420	232,167
2	Bukidnon	740,869	170,400
3	Maguindanao	553,288	127,256
4	South Cotabato	409,076	94,087
5	North Cotabato	368,162	84,677
6	Lanao del Sur	299,034	68,778
7	Cagayan Valley	297,984	68,536
8	Lanao del Norte	249,641	57,417
9	Pangasinan	211,229	48,583
10	Sultan Kudarat	204,419	47,016

* 23% corn cob ratio

Table 2. *Top 10 coconut husk producing provinces in the Philippines, BAS, 2008*

RANK	PROVINCES	COCONUT PRODUCTION (tons/year)	COCOHUSK PRODUCTION* (tons/year)
1	Davao Oriental	1,099,604	329,881
2	Quezon	1,090,941	327,282
3	Davao del Sur	838,497	251,549
4	Leyte	735,312	220,594
5	Zamboanga del Norte	684,651	205,395
6	Lanao del Norte	571,618	171,485
7	Zamboanga del Sur	565,130	169,539
8	Misamis Oriental	512,279	153,684
9	Maguindanao	502,305	150,691
10	Misamis Occidental	490,459	147,138

* 30% coconut husk ratio

Table 3. *Top 10 bagasse producing provinces in the Philippines, BAS, 2008*

RANK	PROVINCES	SUGAR CANE PRODUCTION (tons/year)	BAGGASE PRODUCTION* (tons/year)
1	Negros Occidental	12,156,699	3,647,010
2	Bukidnon	3,421,408	1,026,422
3	Batangas	2,429,635	728,891
4	Negros Oriental	2,404,790	721,437
5	Iloilo	1,007,752	302,326
6	Tarlac	807,951	242,385
7	North Cotabato	688,127	206,438
8	Davao del Sur	574,099	172,230
9	Leyte	553,057	165,917
10	Capiz	551,595	165,479

*30% sugarcane bagasse ratio

Quantification of Recovery Potential of Collected Biomass as Biofiller

The percent biofiller recovery of each biomass with pretreatment (P) and with no pretreatment (NP) was determined. The biofiller recovery values obtained for non-pretreated bagasse, corn cobs and cocohusk, were 91.8 percent, 90.8 percent and 89.6 percent, respectively. For treated samples, recovery values obtained were 70.2 percent, 80.7 percent and 80.2 percent recovery for bagasse, corn cobs and cocohusk, respectively.

The total percentage recovery of non-pretreated biofiller was significantly higher than the pre-treated samples due to material loss during washing (Figure 5). Bagasse is significantly different ($\rho \leq 0.05$) from corn cobs and cocohusks.

However, Figures 6, 7 and 8 show the effect of pretreatment on the particle sizes of biofillers from bagasse, coconut husk and corn cobs. Samples with pretreatment have yielded higher percentage recovery of particle sizes ($\leq 500\mu m$) than those samples without pretreatment.

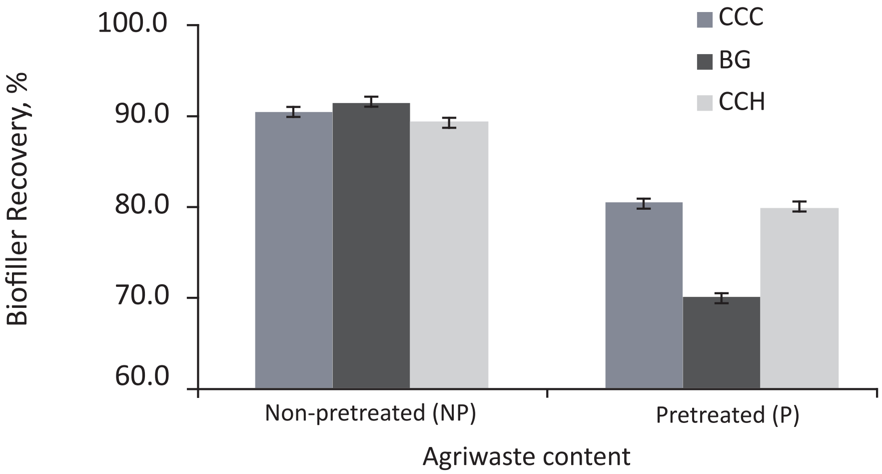

Figure 5. Total biofiller recovery of treated and non-treated biomass samples, %

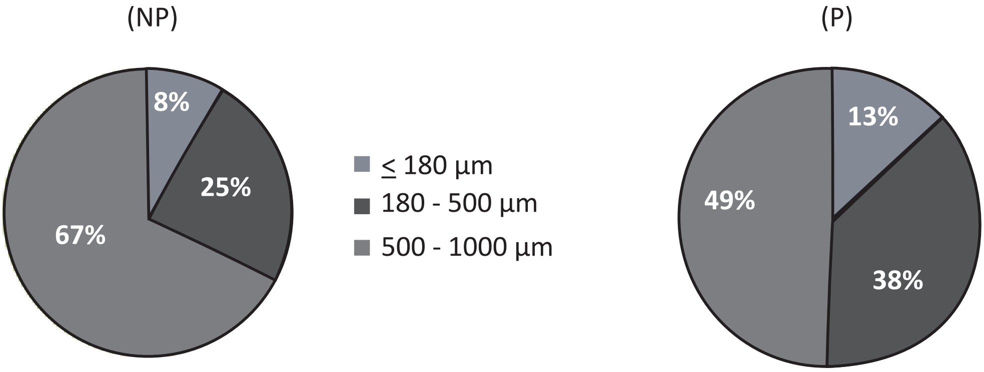

Figure 6. Particle size composition of non-pretreated (NP) and pretreated (P) bagasse, %

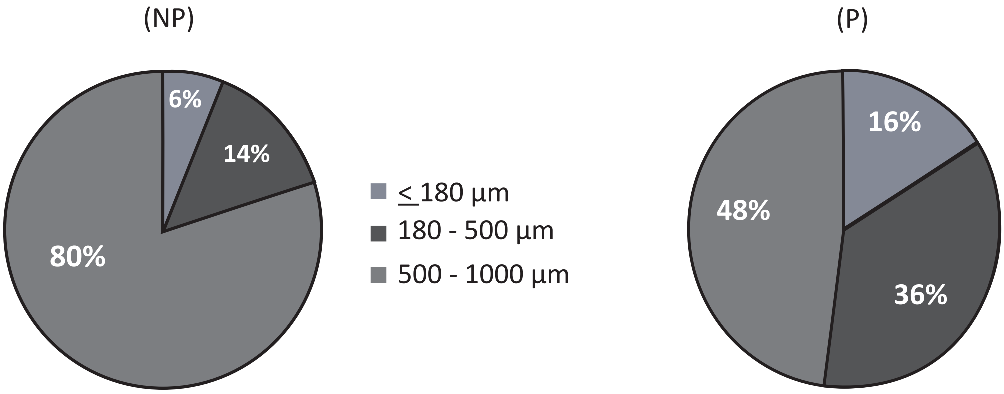

Figure 7. Particle size composition of non-pretreated (NP) and pretreated (P) coconut husk

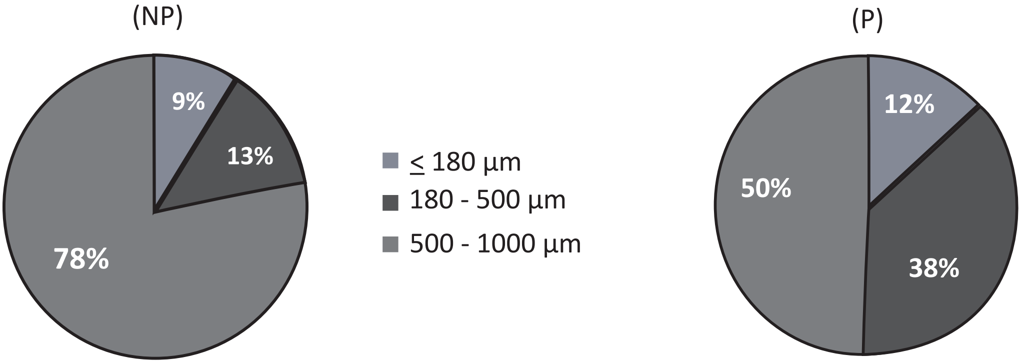

Figure 8. Particle size composition of non-pretreated (NP) and pretreated (P) corn cobs

Characterization of agriwastes filled biocomposites

Density

The density of agriwastes-filled biocomposites as affected by varying content, particle sizes and pretreatment of agriwaste is shown in Figure 9. Results revealed that increasing the amount of agriwaste increased the densities of the biocomposites. Particle sizes of agriwaste biofiller whether small (S) or large (L) and pretreatment (with and without) of agriwastes showed no significant effect on the density of the biocomposites ($\rho \leq 0.05$) .

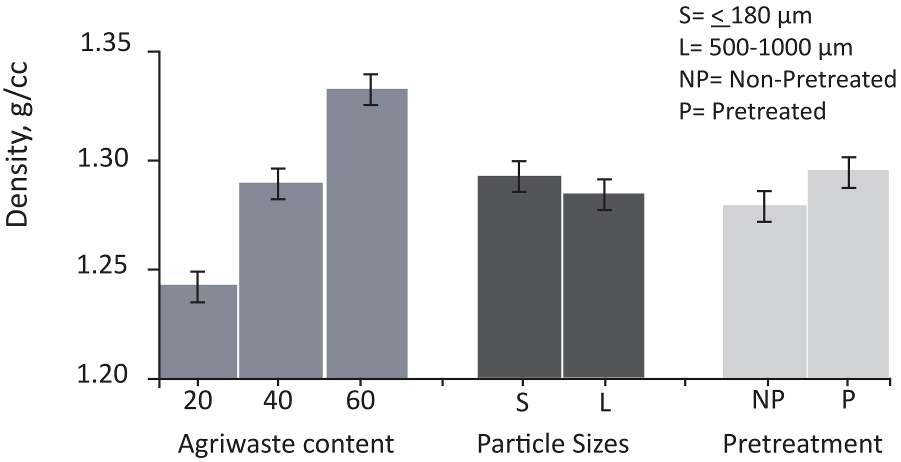

Figure 9. Density of agriwastes-filled biocomposites as affected by varying content of agriwaste, particle sizes and pretreatment of biofiller

The effect of varying content of agriwaste on the density of corn cobs, bagasse, and cocohusk-filled biocomposites was shown in Figure 10. Results showed no significant difference between the densities of corn cobs, bagasse and cocohusk-filled biocomposites at 20 wt% content. The same trend was observed at 40 and 60 wt% content, respectively.

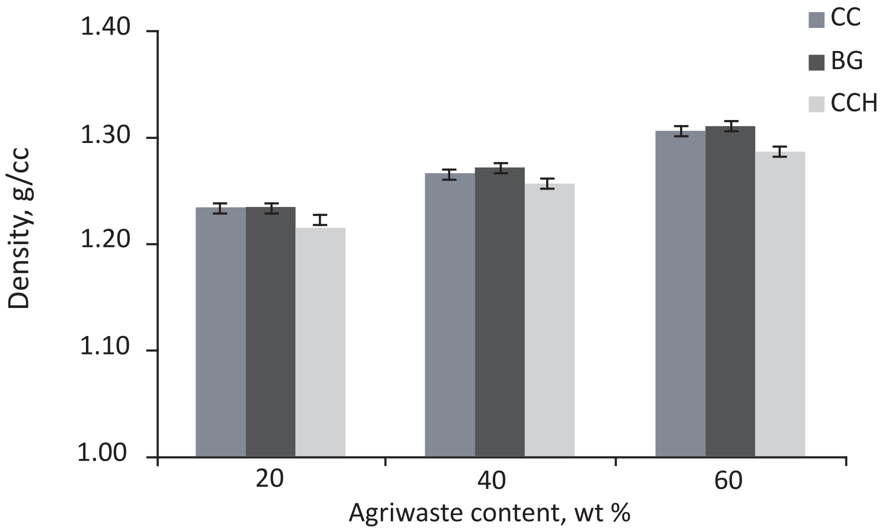

Figure 10. Effect of varying agriwaste content on the density of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites.

Water absorption

The water absorption of agriwastes-filled biocomposites as affected by the varying content, particle sizes and pretreatment of agriwaste is shown in Figure 11. Results showed that increasing the amount of agriwastes increased the water absorption of the biocomposites. Particle sizes and pretreatment of agriwaste showed a significant effect on the water absorption of the biocomposites ($\rho \leq 0.05$). Biocomposites filled with small (S) particle sizes of agriwaste have lower water absorption than biocomposites filled with large (L) particle sizes. Likewise, biocomposites filled with non-pretreated agriwaste (NP) have lower water absorption than biocomposites filled with pretreated (P).

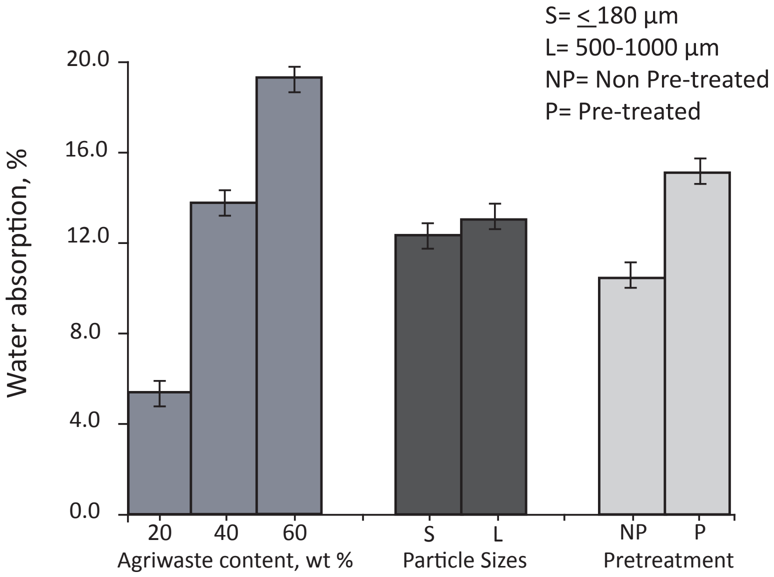

Figure 11. Water absorption agriwastes-filled biocomposites as affected by the varying content, particle sizes and pretreatment of agriwaste biofiller

The effect of varying content of agriwaste on the water absorption of corncobs (CC), bagasse (BG) and cocohusk (CCH) filled biocomposites is shown in Figure 12. It was found out that the water absorptions of corn cobs, bagasse and cocohusk-filled biocomposites at 20 wt% content of agriwaste had no significant difference ($\rho \leq 0.05$) with each other. At 40 wt%, the water absorptions of corn cobs and cocohusk-filled biocomposites which had no significant difference ($\rho \leq 0.05$) were lower than bagasse-filled biocomposites. The same result was observed at 60 wt% content of agriwaste.

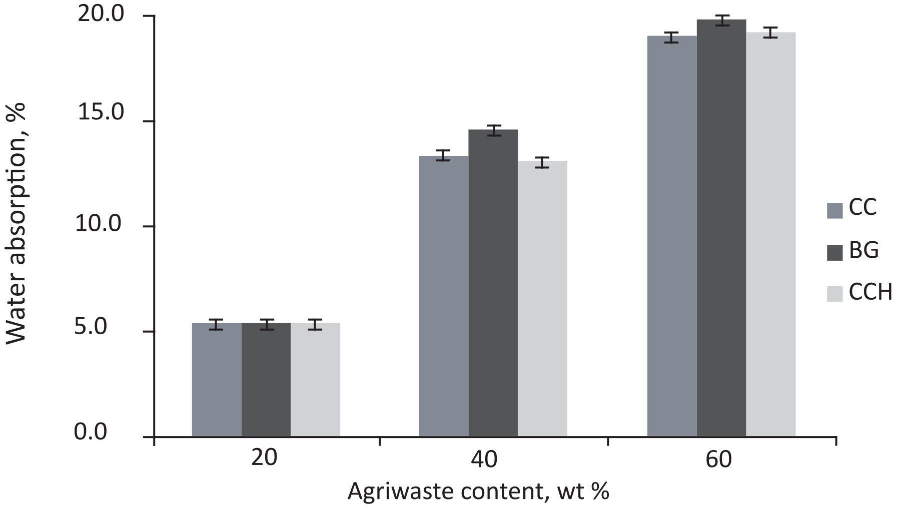

Figure 12. Effect of varying agriwaste content on the water absorption of corn cobs (CC),bagasse (BG) and cocohusk (CCH) filled biocomposites.

The effect of particle sizes of agriwaste on the water absorption of corn cobs, bagasse and cocohusk-filled biocomposites is presented in Figure 13. For small particle sizes of agriwaste biofiller, results showed that the water absorption of corn cobs and cocohusk-filled biocomposites which had no significant difference with each other were lower than bagasse-filled biocomposites. The same trend was observed for large particle sizes of biofiller. It was also indicated that lower water absorption was observed for all biocomposites filled with small particle sizes than with large particle sizes.

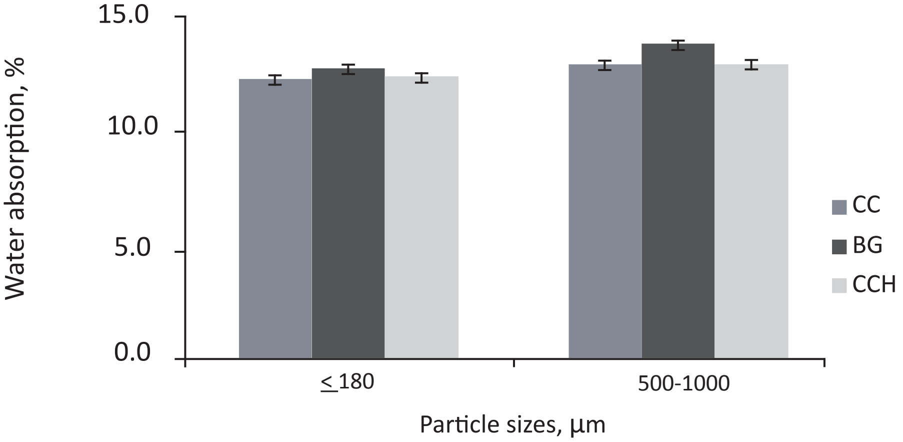

Figure 13. Effect of particle sizes of agriwaste on the water absorptions of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites.

Figure 14 shows the effect of pretreatment of agriwaste on the water absorption of corn cobs, bagasse and cocohusk-filled biocomposites. All biocomposites filled with non-pretreated (NP) agriwastes absorbed lesser water than biocomposites filled with pretreated agriwastes (P). For non-pretreated agriwastes, the water absorptions of corn cobs and cocohusk-filled biocomposites which differed insignificantly were lower than the water absorption of bagasse-filled biocomposites. The same trend was observed for the water absorption of biocomposites filled with pretreated agriwastes.

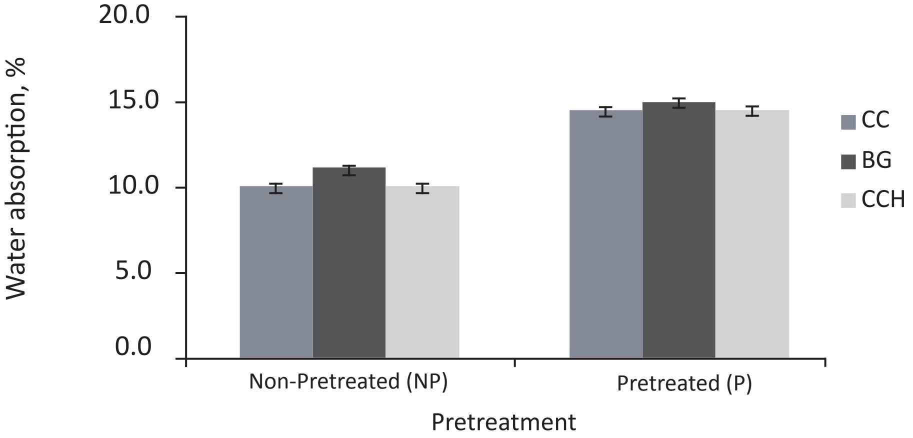

Figure 14. Effect of pretreatment of agriwaste on the water absorptions of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites.

Thickness swelling

The thickness swelling of the agriwastes-filled biocomposites as affected by the varying content, particle sizes and pretreatment of agriwaste is shown in Figure 15. As in the case of water absorption, results showed that increasing the amount of agriwastes increased the thickness swelling of the biocomposites. Particle sizes and pretreatment of agriwaste showed a significant effect on the thickness swelling of the biocomposites ($\rho \leq 0.05$). Biocomposites filled with smaller particle sizes of agriwaste have lower thickness swelling compared to biocomposites filled with larger particle sizes. Likewise, biocomposites filled with non-pretreated agriwaste (NP) have lower thickness swelling than biocomposites filled with pretreated (P) agriwaste.

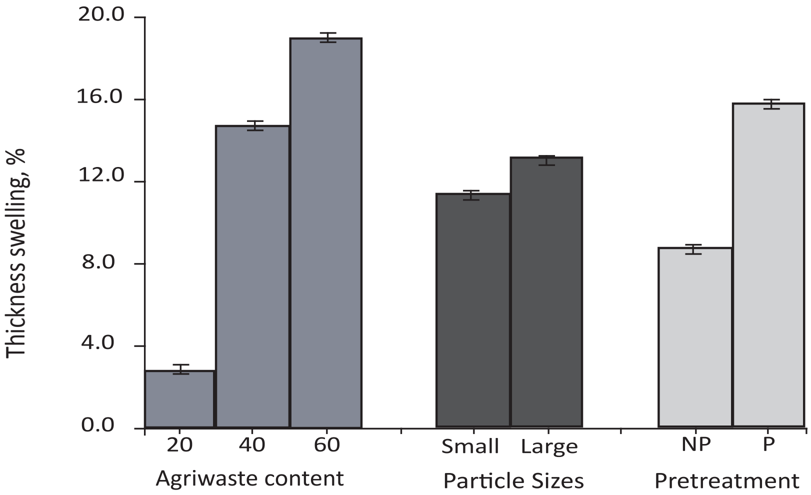

Figure 15. Thickness swelling of agriwastes-filled biocomposites as affected by the varying content (wt %), particle size and pretreatment of biofiller.

Figure 16 showed the effect of varying content of agriwaste on the thickness swelling of corn cobs, bagasse and cocohusk-filled biocomposites. At 20 wt% content, the thickness swelling of corn cobs filled biocomposites was observed to be lower compared to cocohusk and bagasse-filled biocomposites which have no significant difference with each other ($\rho \leq 0.05$). At 40 wt%, the thickness swelling of corn cobs and cocohusk-filled biocomposites which showed no significant difference was lower than bagasse-filled biocomposites. The same trend was observed at 60 wt% content of agriwaste. Results also indicated that increasing the amount of biofiller increased the thickness swelling of all the agriwastes-filled biocomposites.

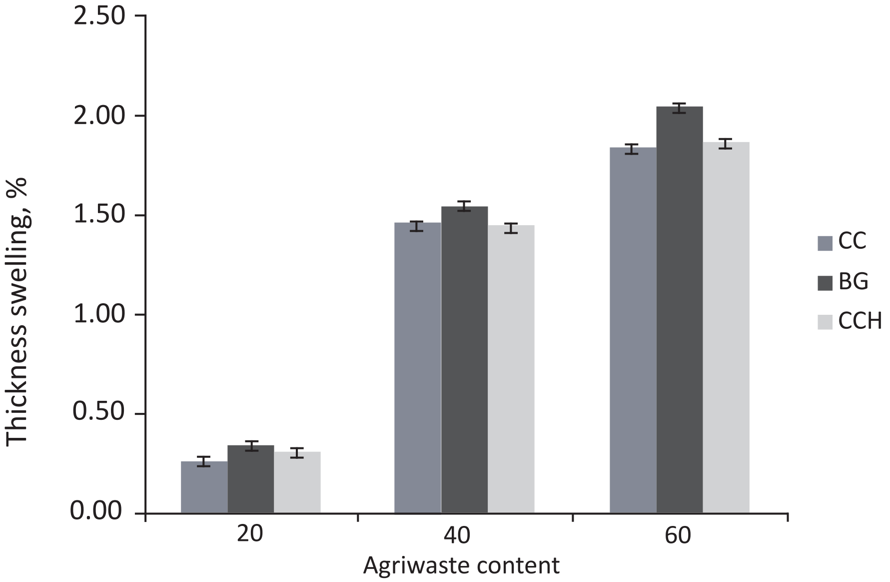

Figure 16. Effect of varying content of agriwaste to the thickness swelling of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites.

Figure 17 shows the effect of particle sizes of agriwaste on the thickness swelling of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites. Results showed that all biocomposites filled with small particle sizes of biofiller have lower thickness swelling than biocomposites filled with larger ones. For biocomposites filled with small particle sizes of biofiller, corn cobs and cocohusk-filled biocomposites did not differ significantly and have lower thickness swelling than bagasse-filled biocomposites. The same was observed for biocomposites filled with large particle sizes of biofiller.

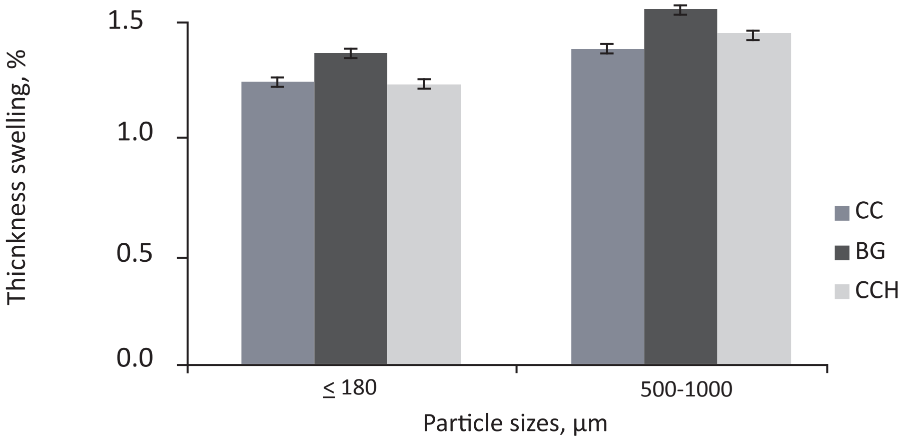

Figure 17. Effect of particle sizes of agriwaste on the thickness swelling of corn cobs (CC), bagasse (BG), and cocohusk (CCH)-filled biocomposites.

Following the trend of water absorption, Figure 18 shows that all biocomposites filled with non-pretreated (NP) agriwastes have lower thickness swelling than the biocomposites filled with pretreated (P). For biocomposites filled with non-pretreated agriwastes, corn cobs and cocohusk-filled biocomposites have lower thickness swelling than bagasse-filled biocomposites. The same trend was observed for biocomposites filled with pretreated ones. It was indicated that non-pretreated agriwaste gave the biocomposites with lower thickness swelling than the pretreated.

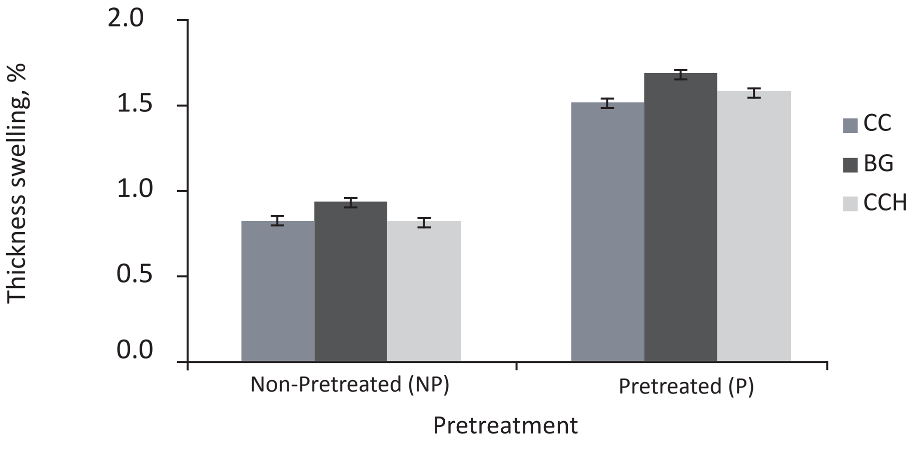

Figure 18. Effect of pretreatment of agriwaste on the thickness swelling of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites.

Melting temperature

The melting temperature of the agriwastes-filled biocomposites as affected by the varying content, particle sizes and pretreatment of agriwaste is shown in Figure 20. Results showed that increasing the amount of agriwaste increased the melting temperature of the biocomposites. Biocomposites filled with smaller (S) particle sizes of agriwastes have lower melting temperatures than biocomposites filled with larger (L) sizes. Pretreatment of agriwastes showed no significant effect on the melting temperatures of the biocomposites at 5 percent level of significance.

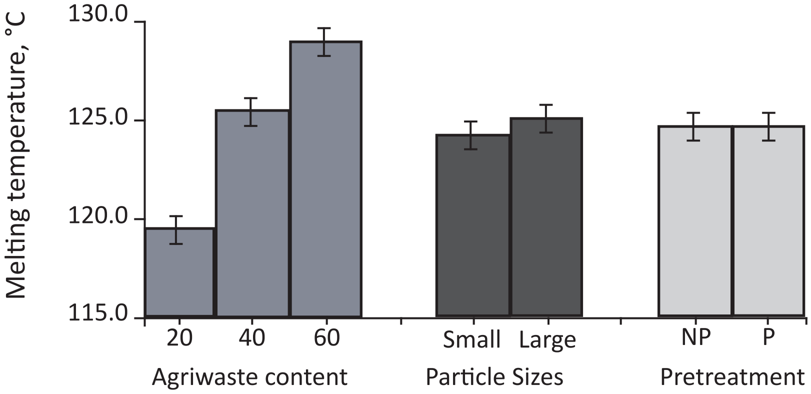

Figure 19. The melting temperature of each agriwaste filled composites as affected by varying content (wt %) of agriwaste, particle size and pretreatment of biofiller.

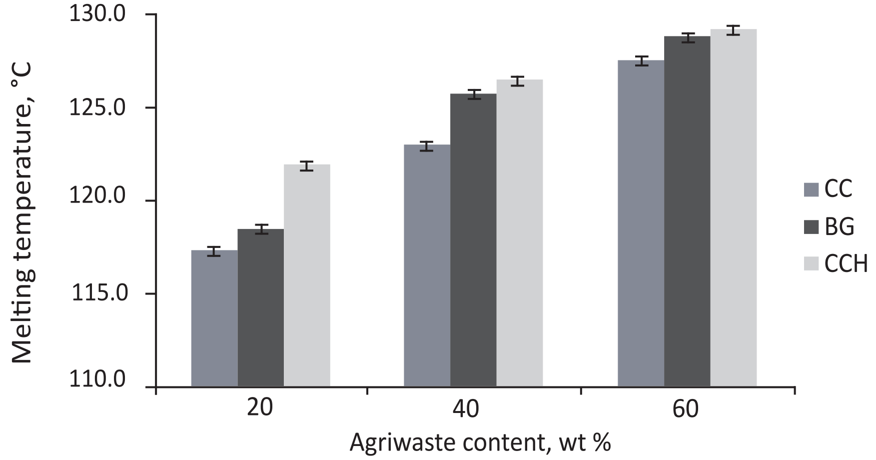

Figure 20. Effect of varying amount of agriwaste to the melting temperature of corncobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites.

Figure 20 showed the effect of varying content of agriwaste on the melting temperature of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites, respectively. It was observed that increasing the amount of agriwaste increased the melting temperature of all the biocomposites. At 20 wt% content, lowest melting temperature was observed for corn cobs-filled biocomposites, followed by bagasse-filled biocomposites and lowest for cocohusk-filled biocomposites. Similar trend was observed at 40 wt% content. At 60 wt%, lowest melting temperature was still observed for corn cobs-filled biocomposites followed by bagasse and cocohusk-filled biocomposites which showed no significant difference with each other.

Figure 21 presents the effect of agriwaste particle sizes on the melting temperatures of corn cobs, bagasse and cocohusk-filled biocomposites. For biocomposites filled with small particle sizes of biofiller, lowest melting temperature was observed for corn cobs-filled biocomposites, followed by bagasse-filled biocomposites and highest for cocohusk-filled biocomposites. For biocomposites filled with large particle sizes, corn cobs and bagasse-filled biocomposites had no significant difference ($\rho \leq 0.05$) with each other and observed lower melting temperatures than cocohusk-filled biocomposites.

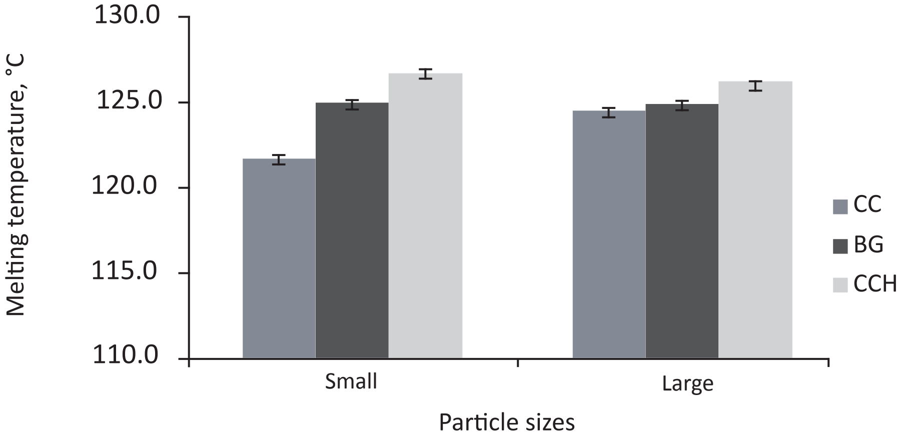

Figure 21. The effect of agriwaste particle size on the melting temperature of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled composites.

Tensile strength

Results shown in Figure 22 indicated that increasing the amount of agriwaste decreased the tensile strength of the biocomposites. The particle sizes of agriwaste whether small or large has no significant effect to the tensile strength of the biocomposites. Non-pretreated biofillers were observed to give higher tensile strength for the biocomposites than the pretreated. The effect of pretreatment reduced the tensile properties of the biocomposites.

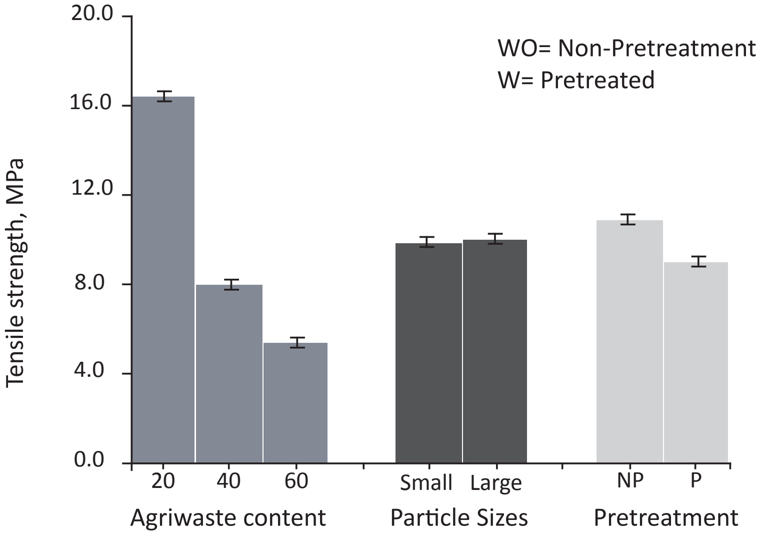

Figure 22. The tensile strength of each agriwaste filled composites as affected by varying content of agriwaste, particle size and pretreatment of biofiller.

Figure 23 shows the effect of varying content of agriwaste on the tensile strength of corn cobs, bagasse and cocohusk-filled biocomposites. At 20 wt% content of biofiller, highest tensile strength was observed for corn cobs-filled biocomposites, followed by bagasse-filled biocomposites and lowest for cocohusk-filled biocomposites. At 40 wt %, highest tensile strength was still observed for corn cobs-filled biocomposites followed by bagasse-filled biocomposites which have no significant difference with cocohusk-filled biocomposites. At 60 wt%, bagasse-filled biocomposites was highest, followed by corn cobs-filled biocomposites which have no significant difference with cocohusk-filled biocomposites.

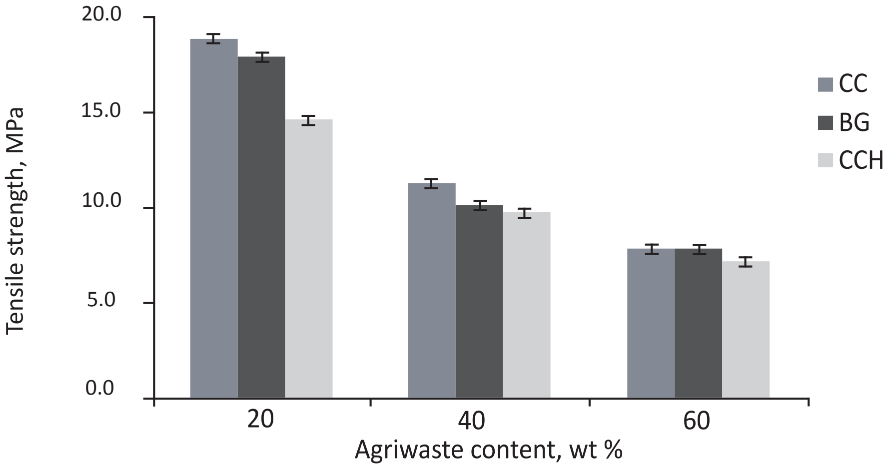

Figure 23. The effect of varying amount of agriwaste to the tensile strength of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites.

Figure 24 shows the effect of pretreatment of biofiller on the tensile strength of corn cobs, bagasse and cocohusk-filled biocomposites. For biocomposites filled with non-pretreated biofiller, corn cobs-filled biocomposites was observed to have the highest strength, followed by bagasse-filled biocomposites while cocohusk-filled biocomposites obtained the least. For biocomposites filled with pretreated biofiller, bagasse-filled biocomposites was highest followed by corn cobs-filled biocomposites and lowest for cocohusk-filled biocomposites.

Highest strength was observed for corn cobs-filled biocomposites with non-pretreated biofiller followed by bagasse-filled biocomposites with non-pretreated and pretreated biofiller which showed no significant difference with each other. Cocohusk-filled biocomposites with non-pretreated biofiller and corn cobs-filled biocomposites with pretreated biofiller ranked third while cocohusk-filled biocomposites with pretreated biofiller obtained the lowest.

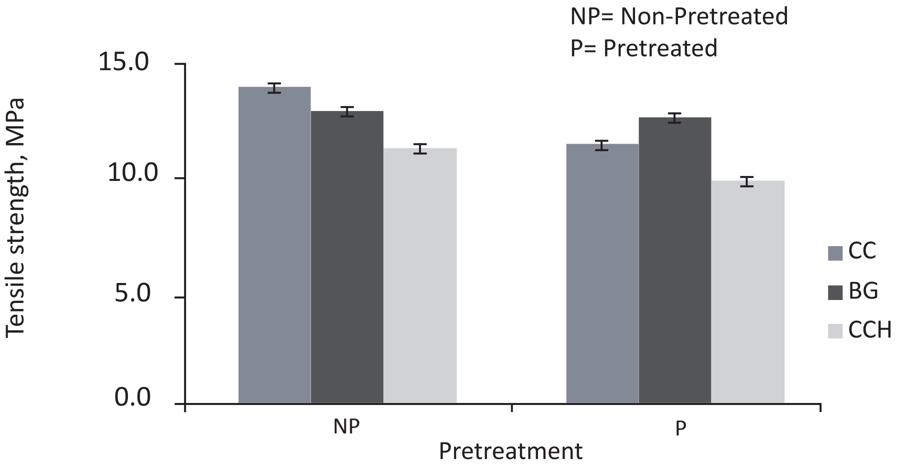

Figure 24. The effect of pretreatment of agriwaste to the tensile strength of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled composites.

Biodegradation

The biodegradability based on percentage weight loss of the agriwastes-filled biocomposites as affected by the varying content and particle sizes of agriwaste was shown in Figure 25. Results indicated that increasing the amount of agriwaste, increased the percentage weight loss of the biocomposites by natural soil burial test. Likewise, particle sizes of agriwaste showed a significant effect on the percentage weight loss of the biocomposites at 0.05 level of significance.

Figure 26 shows the effect of varying content of agriwaste on the percentage weight loss of corn cobs, bagasse and cocohusk-filled biocomposites. At 20 wt % agriwaste content, corn cobs-filled biocomposites was highest, followed by bagasse-filled biocomposites. Meanwhile lowest result was observed for cocohusk-filled biocomposites. Likewise, at 40 wt%, highest biodegradation was observed for corn cobs-filled biocomposites, followed by bagasse and cocohusk-filled biocomposites,both of which showed no significant difference with each other. At 60 wt %, corn cobs-filled biocomposites was still the highest, followed by bagasse-filled biocomposites and lowest for cocohusk-filled biocomposites.

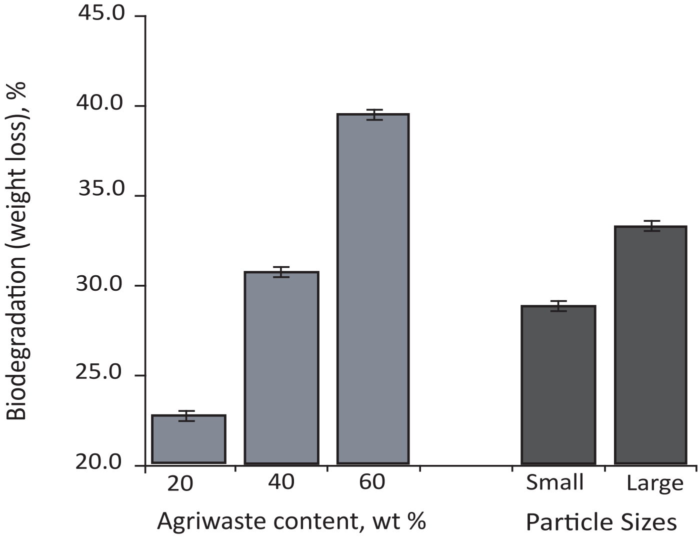

Figure 25. Biodegradability based on percentage weight loss of agriwastes-filled biocomposites as affected by varying content of agriwaste and particle sizes of biofiller.

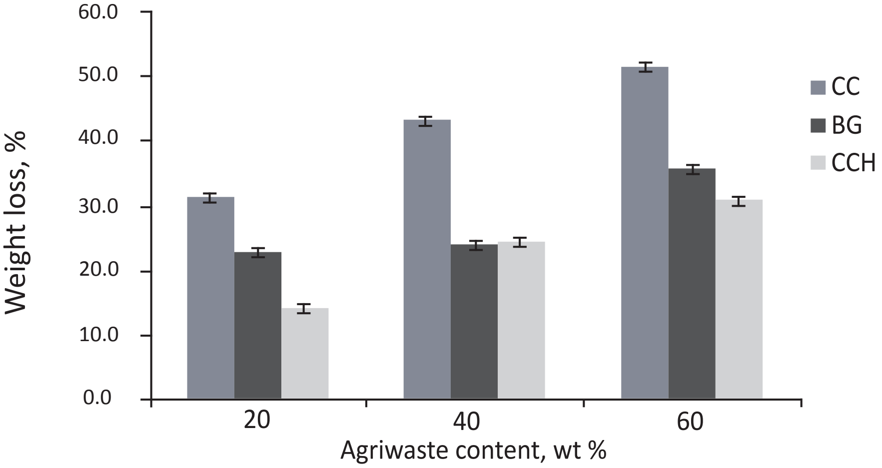

Figure 26. The effect of varying amount of agriwaste to the biodegradation of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites.

Figure 27 shows the effect of agriwaste particle size on the percentage weight loss of corn cobs, bagasse and cocohusk-filled biocomposites. Biocomposites filled with larger particle size of agriwaste have higher percent biodegradation than biocomposites filled with smaller particle size, except for biocomposites filled with cocohusk which showed no significant difference with each other. For biocomposites filled small particle size of agriwaste, highest percentage weight loss was observed for corn cobs-filled biocomposites followed by bagasse-filled biocomposites and lowest for cocohusk-filled biocomposites. The same trend was observed for biocomposites filled with larger particle size. Corn cobs-filled biocomposites was still the highest, followed by bagasse-filled biocomposites while cocohusk-filled biocomposites was the lowest.

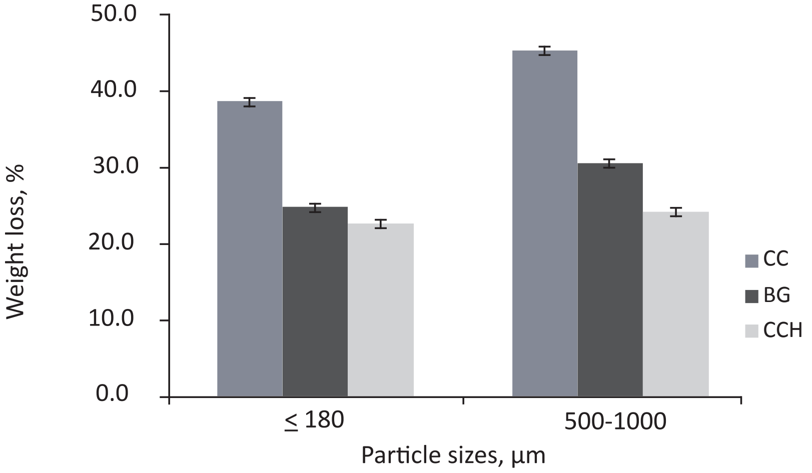

Figure 27. The effect of agriwaste particle size to the percent biodegradation of corn cobs (CC), bagasse (BG) and cocohusk (CCH)-filled biocomposites.

Characterization of starch-filled biocomposites

Density

The density of the starch-filled biocomposites, showing the effect of varying starch content is presented in Figure 28. Results showed that there was no significant difference on the measured densities of cassava (CS) and potato (PS) starch-filled biocomposites ($\rho \leq 0.05$). Results also indicated that both potato and cassava starch-filled biocomposites showed an increasing density as the starch loading increased.

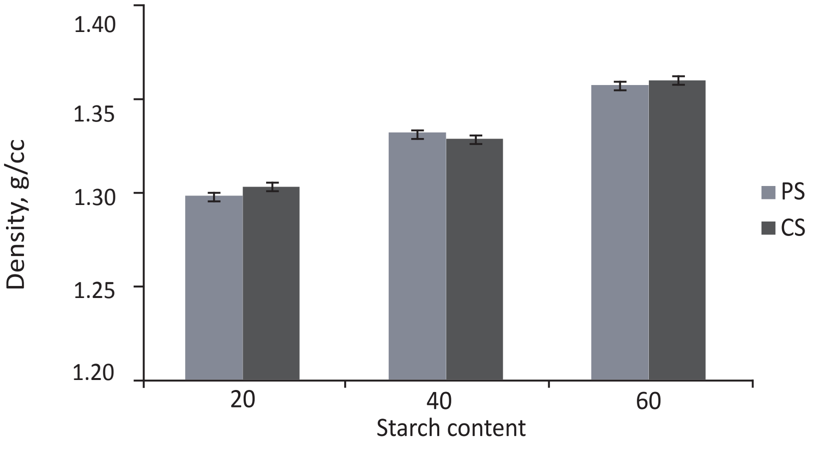

Figure 28. Density of potato (PS) and cassava starch (CS)-filled biocomposites as affected by varying starch content

Water absorpt*ion*

The water absorption of starch-filled biocomposites, showing the effect of varying starch content is shown in Figure 29. Results showed that biocomposites filled with cassava

(CS) and potato (PS) starch showed an increasing water absorption as the starch loading increases. At 20 wt% ratio, the water absorption of the potato starch-filled biocomposites have no significant difference with the water absorption of cassava starch-filled biocomposites. However at 40 and 60 wt%, the water absorption of the potato starch-filled biocomposites was lower than the water absorption of cassava starch-filled biocomposites.

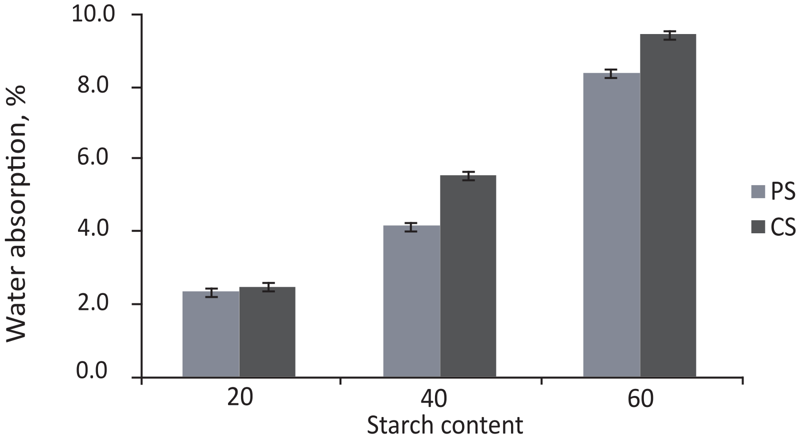

Figure 29. Water absorption of potato (PS) and cassava starch (CS) filled biocomposites as affected by varying starch content

Thickness swelling

The thickness swelling of the starch-filled biocomposites, showing the effect of varying starch content is presented in Figure 30. As in the case of water absorption, results showed that both potato and cassava starch-filled biocomposites showed an increase in thickness swelling as the biofiller loading increased. At 20 and 40 wt% starch content, the thickness swelling of potato starch-filled biocomposites was not significantly different with the thickness swelling of cassava starch-filled biocomposites. However at 60 wt%, the thickness swelling of the potato starch-filled composites was lower than the thickness swelling of the cassava starch-filled biocomposites.

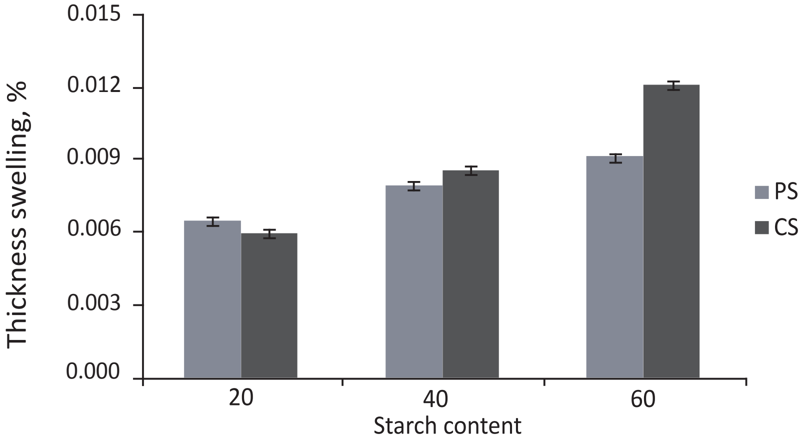

Figure 30. Thickness swelling of potato (PS) and cassava starch (CS)-filled biocomposites as affected by varying starch content

Melting temperature

The melting temperature of starch-filled biocomposites, showing the effect of varying starch content is shown in Figure 31. Results showed no significant difference on the melting temperature of cassava and potato starch-filled biocomposites at 5 percent level of significance. Statistical analysis also indicated that both potato and cassava starch-filled biocomposites showed an increase in melting temperatures as the biofiller loading increased.

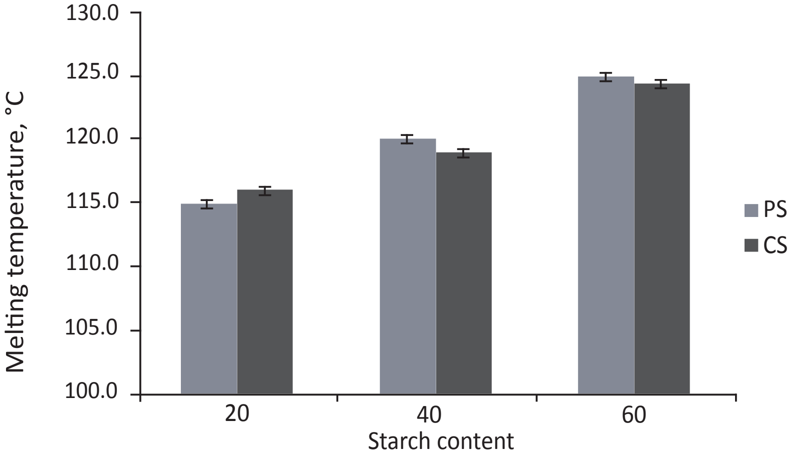

Figure 31. Melting temperatures of cassava (CS) and potato starch (PS)-filled biocomposites as affected by varying starch content

Tensile strength

The tensile strength of the biocomposites filled with potato and cassava starch, showing the effect of varying starch content is shown in Figure 32. Results showed that there was no significant difference between the tensile strength of cassava and potato starch-filled biocomposites in all starch contents at 5 percent level of significance. Results also showed a decreasing tensile strength for both potato and cassava starch-filled biocomposites as the starch loading increases.

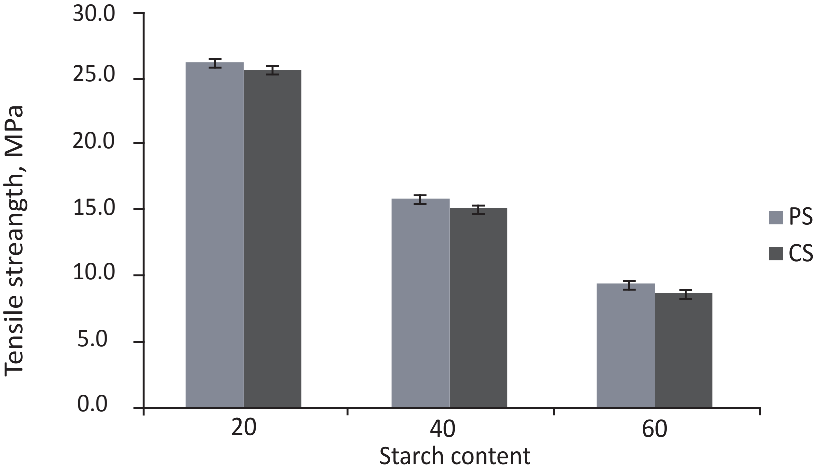

Figure 32. Tensile strength of potato (PS) and cassava starch (CS)-filled biocomposites as affected by varying starch content

Elongation at break

The elongation at break of the biocomposites filled with potato (PS) and cassava starch (CS), showing the effect of varying starch content is presented in Figure 33. Results showed that there was no significant difference between the elongation at break of cassava and potato starch-filled biocomposites in all starch contents. Results also showed a slightly decreasing elongation at break and increasing starch content.

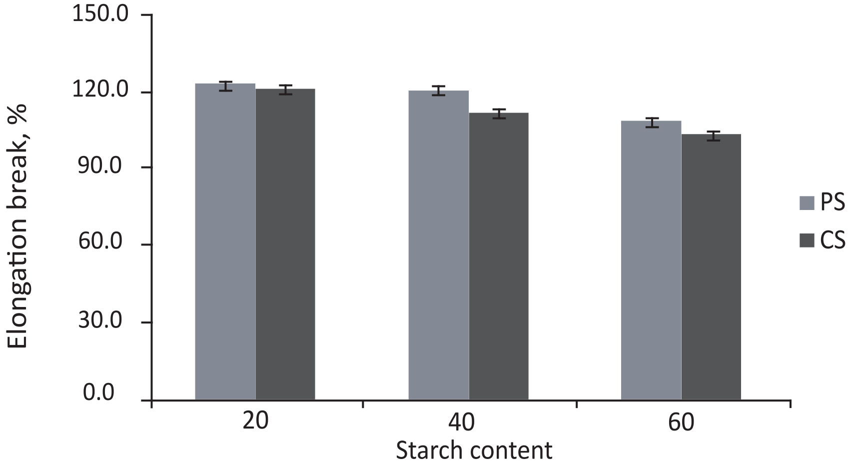

Figure 33. Elongations at break of potato and cassava starch-filled biocomposites as affected by varying starch content.

Biodegradation

The biodegradability based on percentage weight loss of potato and cassava-filled biocomposites as affected by the varying starch content are presented in Figure 34. Results showed that there was no significant difference between the percentage weight loss of cassava and potato starch-filled composites at 20 and 40 wt% starch content. At 60 wt%, cassava starch-filled biocomposites had higher percentage weight loss than potato starch-filled biocomposites. Results also indicated an increase in biodegradability for both potato and cassava starch-filled biocomposites as the starch loading increased.

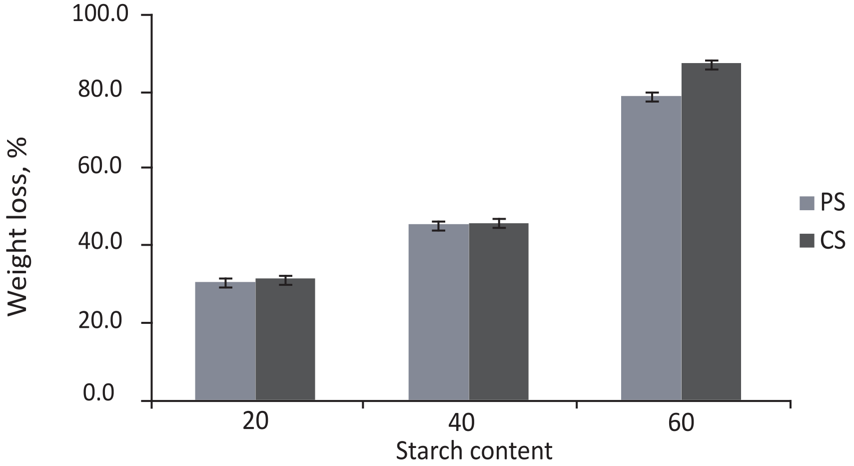

Figure 34. Biodegradation (weigh loss) of starch-filled biocomposites as affected by varying starch content.

Potential uses of developed biocomposite materials

Agriwastes-filled biocomposites

Figure 35 shows the developed agriwastes-filled biocomposites. These biocomposites can be nailed and screwed similar to single pressed board (medium density fiberboard) applications (Figure 36). Tables 4 and 5 present the measured properties of agriwastes-filled biocomposites compared with the traditional plastic composites for general purpose use.

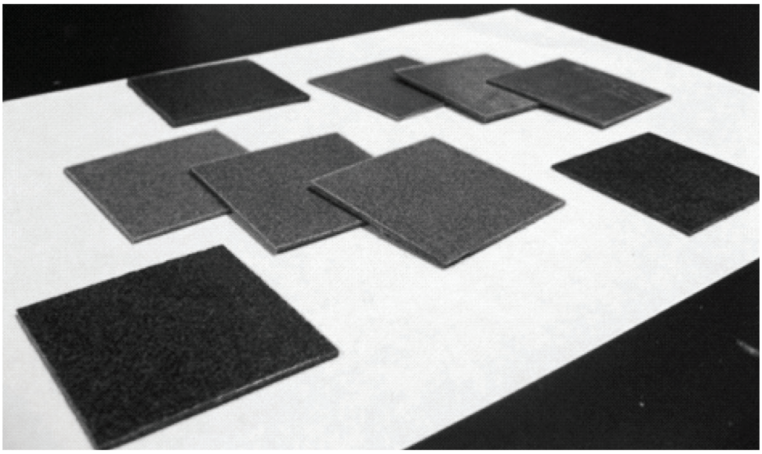

Figure 35. Agriwastes-filled biocomposites

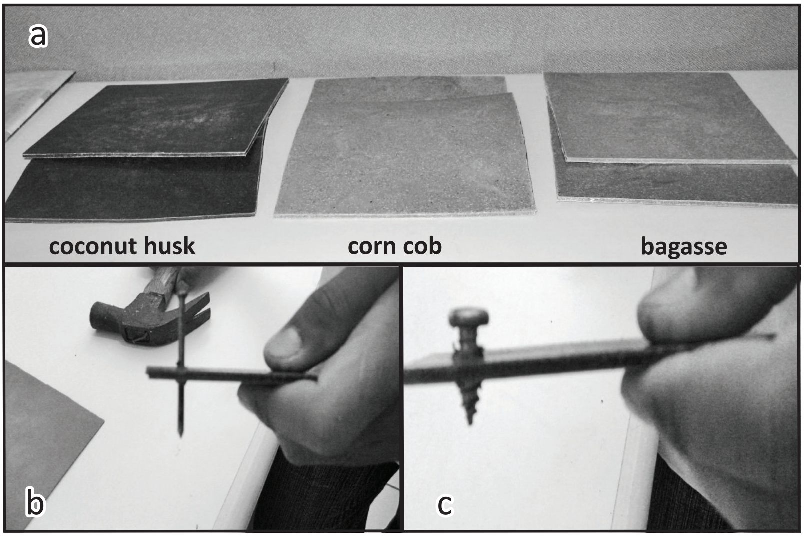

Figure 36. Agriwastes-filled composites (a) can be nailed (b) and screwed (c)

The corn cobs-filled biocomposites and bagasse-filled biocomposites, especially at 20 wt% agriwaste filler loading, met the specified range of properties of the traditional plastic composites for general purpose use. However at 40 and 60 wt% agriwaste filler content, the biocomposites were observed to be less superior in terms of water absorption, swelling and tensile strength to the traditional plastic composites. At these biofiller loadings, a coupling agent which serves as compatibilizer and which provides bridging effect between the filler and the matrix maybe be incorporated to improve their properties. A water resistant, termite and decay control additives could be incorporated depending on its application. For exterior applications, an ultraviolet stabilizer could be incorporated at the time of manufacture as protection from UV light degradation.

Table 4. Properties of biocomposites filled with non-pretreated agriwastes compared with the properties of traditional plastic composites for general purpose use.

Properties	Corn cobs-filled biocomposites (20-60wt %)		Bagasse-filled biocomposites (20-60wt %)		Cocohusk-filled biocomposites (20-60wt %)		*Plastic composites
	180 µm	500 µm	180 µm	500 µm	180 µm	500 µm	
Density, g/cc	1.24-1.30	1.23-1.31	1.25-1.31	1.24-1.32	1.22-1.29	1.22-1.27	1.00-1.40
Water absorption, %	4.53-10.63	4.87-15.33	4.43-15.33	4.84-16.77	4.64-14.31	4.97-15.51	1.0-6.0
Thickness swelling, %	0.15-1.0	0.25-1.36	0.21-1.32	0.45-1.46	0.15-1.26	0.26-1.35	1.0-6.0
Tensile strength MPa	9.5-22.5	9.6-17.2	9.9-18.0	10.8-18.4	8.73-14.5	8.34-14.9	17.2**

Table 5. Properties of biocomposites filled with pretreated agriwastes compared with the properties of traditional plastic composites for general purpose use

Properties	Corn cobs-filled biocomposites (20-60wt %)		Bagasse-filled biocomposites (20-60wt %)		Cocohusk-filled biocomposites (20-60wt %)		*Plastic composites
	180 µm	500 µm	180 µm	500 µm	180 µm	500 µm	
Density, g/cc	1.25-1.32	1.25-1.33	1.25-1.33	1.24-1.33	1.23-1.32	1.21-1.31	1.00-1.40
Water absorption, %	5.95-23.05	6.02-23.52	5.77-23.25	6.54-23.73	5.76-23.19	6.18-23.67	1.0-6.0
Thickness swelling, %	0.27-2.20	0.35-2.51	0.24-2.55	0.47-2.80	0.24-2.23	0.58-2.60	1.0-6.0
Tensile strength MPa	6.21-19.2	6.46-16.7	8.53-17.4	11.07-18.3	6.04-14.5	5.98-14.87	17.2**

*Panels meet the requirements in standard specification for medium density fiber board for general purpose use (22) ** unsaturated polyester (USP) composites.

Starch-filled biocomposites

Table 6 presents the properties of starch-filled biocomposites compared with some traditional, non-biodegradable plastics like high density polyethylene (HDPE) and low density polyethylene (LDPE). These traditional plastics are used for a wide variety of applications such as packaging, plastic film sheets, containers and other products. The developed starch-filled biocomposites shown in Figures 37 and 38 mimic the strength properties of LDPE and within the range of HDPE. Likewise, their measured elongations are within the range of elongation/strain properties of LDPE and HDPE. The starch-filled biocomposites however have higher densities and absorbed water more readily than the traditional non-biodegradable polymers.

As presented in Table 7, the mechanical properties of the developed starch-filled biocomposites were within the range of mechanical properties for processing grades of plastic products. The starch-filled biocomposites can be potentially used and processed as thermoformed, injection molded and blown film products.

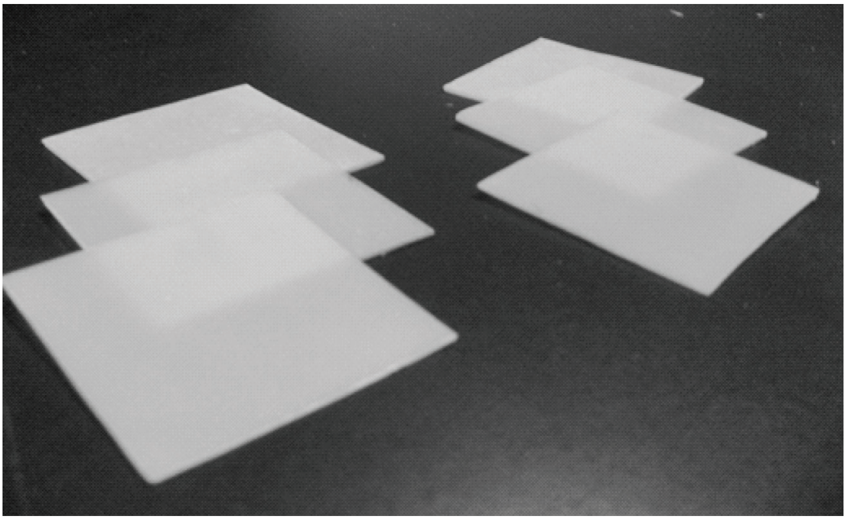

Figure 37. Starch-filled biocomposites

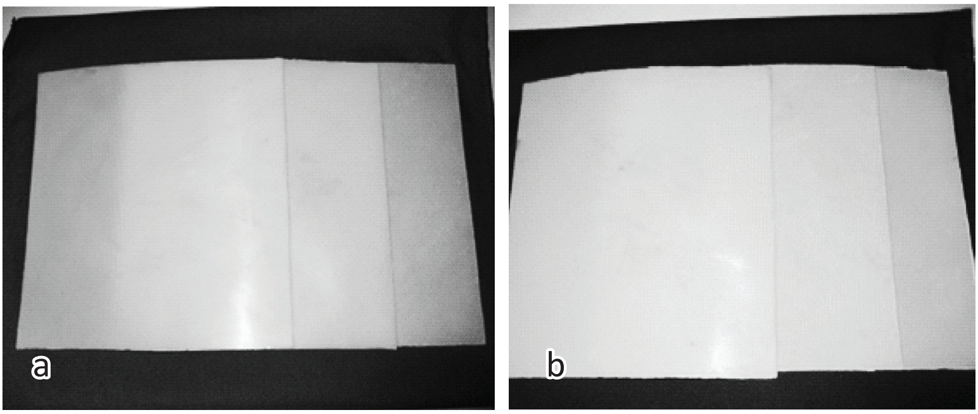

Figure 38. Starch-filled biocomposites (a) potato starch (b) cassava starch

Table 6. Properties of biocomposites filled with potato and cassava starch compared with the properties of some traditional non-biodegradable plastics

Properties	Biocomposites filled with starch (20-60wt %)		Traditional, non-biodegradable plastics	
	Potato starch	Cassava starch	LDPE	HDPE
Density, g/cc	1.298-1.357	1.304-1.360	0.910-0.940	0.941-0.965
Water absorption, %	2.34-8.39	2.46-9.45	1.0	1.0
Thickness swelling, %	0.01	0.01	-	-
Melting temperature, °C	115-125	116-124	98-115	130-137
Tensile strength, MPa	9.39-26.50	8.78-25.70	4.13-15.86	21.37-37.92
Elongation, %	108-123	103-121	100-650	10-130
Biodegradation (weight loss), %	30.60-78.90	31.90-87.24	-	-

Table 7. Mechanical properties of developed starch-filled biocomposites compared with the processing grade requirement of plastics

Properties	Starch-filledbiocomposites (20-60 wt %)		Processing grades *		
	Potato starch	Cassava starch	Thermoforming	Injection molding	Blown filming
Tensile strength, MPa	9.39-26.50	8.78-25.70	15.0-25.0	8.0-25.0	6.9-44.8
Elongation, %	108-123	103-121	20-60	10-500	10-600

*Metabolix Natural Plastics. International Degradable Plastic Symposium, 2006

SUMMARY, CONCLUSION AND RECOMMENDATIONS

Aside from energy crop biomass such as potato and cassava starch, agriwastes biomass such corn cobs, bagasse and cocohusk were used as biofiller in the development of biocomposite materials. Corn cobs, bagasse and cocohusk were prepared as biofiller in powder form. Non-treated agriwastes have higher percent biofiller recovery than treated agriwastes. However, treated agriwastes yielded higher percentage of smaller particle sizes than non-treated.

Biocomposites filled with starch and agriwastes can be prepared by hot pressing method. The properties of biocomposites were determined based on ISO standards. Biodegradation was also undertaken by natural soil burial test.

For agriwaste-filled biocomposites, the density of corn cobs, bagasse and cocohusk-filled biocomposites have no significant difference with each other. Particle sizes and pretreatment of biofiller have no significant effect on the density of biocomposites. Increasing the amount of biofiller increased the density of biocomposites.

The water absorption of corn cobs and cocohusk-filled biocomposites were lower than the bagasse-filled biocomposites. Biocomposites filled with smaller particle size of biofiller absorbed lesser water than with larger sizes. Likewise, biocomposites filled with non-pretreated biofiller have lower water absorption than with pretreated. Increasing the amount of biofiller increased the water absorption of the biocomposites.

As in the case of water absorption, the thickness swelling of the corn cobs and cocohusk-filled biocomposites were lower than bagasse-filled biocomposites. Biocomposites filled with smaller particle sizes of biofiller have lower thickness swelling than with larger particle sizes. Likewise, biocomposites filled with non-pretreated biofiller have lower thickness swelling than with pretreated. Increasing the amount of biofiller also increased the thickness swelling of the biocomposites.

The melting temperature of biocomposites increased with increasing biofiller content. Biocomposites filled with smaller particle sizes of biofiller have lower melting temperatures than with larger particle sizes. Pretreatment of biofiller have no significant effect on the melting temperatures of biocomposites.

The tensile strength of biocomposites decreased with increasing biofiller content. The particle sizes of biofiller have no significant effect on the tensile strength of each biocomposites. Biocomposites filled with non-pretreated biofiller have higher tensile strength than with the pretreated.

Biodegradability of the biocomposites increased with increasing biofiller content. Biocomposites filled with larger particle sizes of biofiller have higher percent weight loss than with smaller particle sizes.

For starch-filled biocomposites, the density of potato and cassava starch-filled biocomposites have no significant difference with each other. The density of starch-filled biocomposites increased with increasing starch content.

The water absorption and thickness swelling of the starch-filled biocomposites increased with increasing starch content.

The melting temperatures of the potato and cassava starch-filled biocomposites have no significant difference with each other. Increasing the starch content increased the melting temperature of the starch-filled biocomposites.

The tensile strength of starch-filled biocomposites decreased with increasing starch content. The strength of potato and cassava starch-filled biocomposites has no significant difference with each other. The same trend was observed for the measured elongations of the biocomposites.

Biodegradation was observed for starch and agriwastes-filled biocomposites due to their reduction in weight under natural soil burial test. For agriwastes-filled biocomposites, corn cobs-filled biocomposites have highest percent weight loss, followed by bagasse and lowest for cocohusk-filled biocomposites. For starch-filled biocomposites, the percent weight loss of potato and cassava-filled biocomposites were not significantly different. Increasing the starch and agriwastes content also increased the biodegradation of the biocomposites.

The corn cobs and bagasse-filled biocomposites, especially at 20 wt% biofiller loading, met the specified range of properties of the traditional plastic composites. At 40 and 60 wt% biofiller loadings, a coupling agent which serves as compatibilizer and provides bridging effect between the filler and the matrix maybe incorporated to improve their properties. Water resistant, termite and decay control additives can be incorporated depending on its application. For exterior applications, an ultraviolet stabilizer could be incorporated at the time of manufacture as protection from UV light degradation. The agriwastes-filled biocomposites can be potentially suitable alternative, particularly in the face of universal concern for ecological degradation caused by the continuing dependence of depleting forest/wood resources.

The measured properties of starch-filled biocomposites were comparable to some properties of LDPE and within the range properties of HDPE. The starch-filled biocomposites however have higher densities and water absorption than LDPE and HDPE. At the time of manufacture, water resistant additives can be added to increase its resistance to water absorption. The starch-filled biocomposites can be potentially processed as thermoformed, injection molded and blown film plastic products with some modifications depending on the specific applications.

Sustainability assessment should be done to determine the socio-economic and environmental impacts and benefits derived from using biomass as biofiller in the development of biocomposite materials.

ACKNOWLEDGMENTS

The authors would like to thank the Department of Science and Technology (DOST) for allowing the project team to prepare biocomposite materials using their laboratory equipment and conduct laboratory testing and analysis. Thank you also to Dr. Jessie C. Elauria and Prof. Omar F. Zubia of the CEAT-UPLB for their technical comments and suggestions during the TEG.

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For more information, please contact:

The Executive Director
Philippine Center for Postharvest Development and Mechanization
CLSU Cmpd., Science City of Muñoz, Nueva Ecija
Tel. Nos.: (044) 456-0213; 0290; 0282; 0287
Fax No.: (044) 456-0110
Website: www.philmech.gov.ph

PHilMech Liaison Office
3rd Floor, ATI Building
Elliptical Road, Diliman, Quezon City
Tel. Nos.: (02) 927-4019; 4029
Fax No.: (02) 926-8159