Preparation and Characterization of Moulded Pulp Container Made by Hot Compression Moulding

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ABSTRACT

Single-use foam packages made of synthetic polymers have been replaced by biodegradable material from renewable resources, leading to ecological benefits. Molded pulp container is one possible alternative packaging for environmentally friendly products. Molded pulp containers in this research were made by the chemical pulp of sugarcane bagasse and binders. The chemical pulp of sugarcane bagasse was prepared by soda pulping process. Response surface methodology was employed to evaluate the optimum soda pulping condition which contributed the highest yield with good quality pulp. The optimum condition of the alkaline process was 20% sodium hydroxide cooking at a temperature of 154 ºC for 104 minutes. Polyvinyl alcohol (PVOH) and cassava starch paste with 15% solid content were used as the binder of the molded pulp containers. Molded pulp containers with a mixed adhesive of PVOH and starch (1:2) showed superior mechanical performance. The mixtures of alkaline pulp and binders which were conditioned to moisture level about 30% had been formed by hot compression molding at 165 ºC for 3 minutes. The water resistance of molded pulp containers was improved by the use of paraffin wax and PLA coating.

Keywords : *molded pulp, alkaline pulping, PLA coating, polyvinyl alcohol*

Introduction

Molded pulp package is a bio-based package material which is made from natural fibre. It is an environmental friendly product which is made from renewable resources and biodegradation. Molded pulp describes a three-dimensional package. It can be classified into many groups. Williams (1970) classified the molded pulp packages into 3 groups by their density: soft (0.2-0.5 grams/cc.), hard (0.5-1.2 grams/cc.), and fully molded (1.2-1.35 grams/cc.). Paine (1991) categorized the molded pulp packages through the production process into 2 groups: injection- and suction- molding. Brody and Marsh (2000) classified molded pulp packages in accord with the methods of fabrication into 2 groups: plain molding and precision molding. The unique characteristic of molded pulp package provides precise multiple compartment designs at a high strength-to-weight ratio. They can be produced in practically any shape and pattern, in either single or multi-compartmental styles. The molded pulp packages are readily biodegradable after use, and weigh considerably less than other package options.

These packages have long been used for low cost retail products, such as egg trays and boxes. However, more complex molded pulp products have been applied in the package of the industrial products such as electrical and electronic appliances such as printers and computers (Anonymous, 2007). The disadvantage of molded pulp package made from fiber is their hygroscopic nature that makes them very sensitive to environmental conditions containing moisture(Sørensen and Hoffmann, 2003., Sørensen and Hoffmann, 2004. Sørensen and Risbo, 2005). Moisture greatly affects the physical and mechanical properties of molded pulp, leading to a substantial degradation at elevated levels of moisture content.

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The molded pulp packages in this experiment were produced by using the soda pulp of sugarcane bagasse, used cassava starch paste and PVOH as the binder. The shape of the fiber containers was formed by hot compression molding.

Polyvinyl alcohol (PVOH) is manufactured by the polymerization of vinyl succinate monomer, followed by hydrolysis of the polyvinyl succinate (Jaffe and Rosenblum, 1990). It is the largest synthetic water-soluble polymer produced by volume in the world (Ramaraj, 2007). It is also a versatile polymer with many industrial applications because of its biodegradability, biocompatibility, chemical resistance, and excellent physical properties. However, the price of PVOH is quite high. In order to make PVOH more economical to use, PVOH can be blended with native starches to reduce the cost of materials. Cassava starch is available from large tuberous roots of a plant which is cheap. There have been a number of studies reporting the properties of cassava (Camargo *et al*., 1988; Sriroth *et al*., 1999; Charles *et al*., 2005; Taiwo, 2006). It is biocompatible and consumable by microorganisms as well as PVOH. Both PVOH and cassava paste can be used as the adhesives for food and non-food package.

Molded pulp packages are characterized by their cellular structure which imparts high compressive strength at a relatively low weight. However, they are prone to absorb water and moisture from the environment, especially when stored under high humidity conditions or in contact with high moisture materials. Absorption of water or moisture affects the physical and mechanical strength of molded pulp packages.

The water resistance of molded pulp packages may be obtained by changing the wettability of the surface with hydrophobic coating materials such as paraffin wax. However, the effect of paraffin wax on the package materials make it difficult to separate, recycle or compost them after use.

Poly(lactide) (PLA) is biodegradable, biocompatible and commercially available with good characteristics as a coating material. It is a polymer resulting from the polymerization of lactic acid by the ring opening of its cyclic dimmer, lactide (Conn *et al*., 1995). PLA has comparable mechanical performance, especially in terms of its high elasticity modulus and high strength (Garlotta, 2001; Drumright et al., 2000). In addition, PLA is "Generally Recognized AS Safe" (GRAS) when used in fabricated articles for food contact surfaces and its environmentally friendly nature is also recognized.

Fewer studies have been published dealing with molded pulp packages made by hot compression molding and PLA coating. The purpose of this study was to investigate the characteristics of molded pulp containers made by the soda pulp of sugarcane bagasse and the efficiency of using mixed adhesives between cassava paste and PVOH. In addition, the effects of various coating materials on repelling water were also determined.

Materials and Methods

Sugacane bagasses were collected from the vicinity of the Prince of Songkla University, Songkhla Thailand. Cassava starch was obtained from the Siam Modified Starch Co., Ltd., Pathumthani, Thailand. Fully hydrolysed PVOH grade, BF-17H, was obtained from Chang Chun Petrochemical Ltd., Taipei, Taiwan. Commercial grade Poly-L-lactide (PLA) was supplied by NatureWorks LLC, Minnesota, USA. Paraffin wax and dichloromethane were purchased from Sigma-Aldrich Pte. Ltd., Singapore.

Raw material preparation

Sugarcane bagasses were cleaned and the dust particles were removed by water. They were taken as air dry. The raw materials for pulping were chopped to 2-4 cm. lengths with a chopper then they were stored in plastic bags in cold storage. The fiber morphology and chemical composition of the sugarcane fibers were determined in accord with the respective standard methods: ash content (T211-om-93); Kappa number (T236-cm-85); pentosan (T223 om-84); cellulose content (Van Soest and Wine, 1967); and holocellulose (Cordeiro *et al*., 2004). The fiber lengths were determined by a Kajaani FS-200 instrument. The fiber width and cell wall thickness of the fibers were analyzed by a microscopic instrument.

Pulping process

The cooking process of sugarcane bagasse was undertaken by alkaline pulping. The conditions employed were varied as follows: sodium hydroxide (NaOH) concentration from 10 to 20 percentages; maximum cooking temperatures from 120 to 160 $^{\circ}$C; and cooking time from 60 to 120 minutes. The liquor-to-bagasse ratio of 10:1 was used. The cooked pulps were washed with tap water in several steps to achieve complete washing. The pulps were screened on a flat screen with 0.25 mm slits. The screened pulps were centrifuged and dried in a hot air oven. The oven-dry weight of the pulps was determined as the screened pulp yield. The kappa number of screened pulps was examined according to the TAPPI standard (T236-cm-85).

The Box and Behnken Design was used to outline the composition of the experimental process conditions made up around a central combination. Fifteen conditions of the soda pulping of sugarcane bagasse were examined to measure the optimum condition which gave the highest yield of screened pulps (Table 1). The results were subjected to multiple linear regressions as implemented in a polynomial model.

Table 1 Box and Behnken design of pulping conditions with three independent variables

Sample	Temperature(°C)	Time (min.)	Concentration(%)
1	160 (1)	120 (1)	15 (0)
2	160 (1)	60 (-1)	15 (0)
3	120 (-1)	120 (-1)	15 (0)
4	120 (-1)	60 (-1)	15 (0)
5	160 (1)	90 (0)	20 (1)
6	160 (1)	90 (0)	10 (-1)
7	120 (-1)	90 (0)	20 (1)
8	120 (-1)	90 (0)	10 (-1)
9	140 (0)	120 (1)	20 (1)
10	140 (0)	120 (1)	10 (-1)
11	140 (0)	60 (-1)	20 (1)
12	140 (0)	60 (-1)	10 (-1)
13	140 (0)	90 (0)	15 (0)
14	140 (0)	90 (0)	15 (0)
15	140 (0)	90 (0)	15 (0)

Molded pulp manufacture

The molded pulp containers were made from the soda pulps of sugarcane bagasse and binder. PVOH and cassava paste with 15% solid content were used as the binder of the molded pulp containers. The PVOH glue and cassava starch paste were mixed in the ratio of 1:0, 1:1, 2:1, 1:2 and 0:1, respectively. The binders were applied to the pulp in the ratio of 70:30 (wet weight) and the moisture content of the mixed pulp was adjusted to 30%. The mixed pulp was weighed to 60 g and pressed in the hot compression molding to an average density of 1.3 x 10^3 g/m^2 at 165 $^{\circ}$C for 3 minutes. The main mechanical properties of the molded pulp containers were evaluated as functions of the compression strength (ASTM D642), and puncture strength (ASTM D781).

Paraffin wax and PLA coating

The water resistance of molded pulp containers was improved by PLA and paraffin wax. The paraffin wax and PLA coating solutions were prepared by dissolving 1, 3 and 5 g of coating materials in 100 ml of dichlorometane and stirring using a magnetic stirrer. The coating solution was applied on the surface of the molded pulp containers by the dipping method. The coated containers were dried in a hot air oven with a temperature of 70 $^{\circ}$C. Coating weights were determined by subtracting the weight of the uncoated pulp containers from the weight of the coated pulp containers. The water resistance of coated pulp containers was measured using the modified method of Shogren *et al*.(2002). Then 100 ml of distilled water were poured over the fiber containers and the water was discarded after 120 seconds. The excess surface water on the pulp

container was removed by wiping with tissue paper. The weight of the pulp container after water absorption was compared to the initial weight of the pulp container. The compression strength and puncture resistance of coated pulp containers were examined according to ASTM D642 and ASTM D781, respectively. The residue of dichloromethane in the coated pulp containers was investigated by gas chromatography (GC). GC analyses were performed with a HP 6890 gas chromatograph. The coated pulp products were soaked in 99% methanol (HPLC grade) overnight and heated at 50°C. The air in the headspace was injected to the gas chromatography for dichloromethane analysis.

Results and Discussion

Characteristics of bagasse fiber

The fiber morphology and chemical composition of sugarcane bagasse, as a raw material of this research, are shown in Table 2. The characteristics of bagasse fiber are shown in terms of fiber length, fiber width and the ratio of fiber length and width. The mean length and width of the bagasse fibers were 1.9 and 0.02 mm, respectively. The fiber length of the sugarcane bagasse was practically similar to the dimensions of hardwood fibers. It was a short fiber when compared to the other non-wood fibers such as jute, and bamboo (Han, 1998). The fibrous products made from short fiber are often of low strength because the length of fiber has affected the interfiber bonding. However, short fiber contributes to the smooth surface of fibrous products (Smook, 1998).

Table - 1 Box and Behnken design of pulping conditions with three independent variables

Items	Unit	Value
Fiber length	mm.	1.90 ± 0.32
Fiber width	mm.	0.02 ± 0.003
L/D ratio		95 : 1
Cell wall thickness	mm.	0.0048 ± 0.0006
Ash	%	1.67 ± 0.45
Cellulose	%	38.95 ± 3.21
Holocellulose	%	69.73 ± 5.60
Lignin	%	16.11 ± 0.98
Pentosan	%	28.24 ± 4.34

L/D ratio and cell wall thickness are the indicators used to evaluate the strength of fibrous products. The L/D ratio of bagasse fiber was 95:1. This means that the sugarcane baggasse pulp can be used as a raw materials for fibrous packages. For paper making, the optimum L/D ratio was about 100:1 (Retulainen *et al.*, 2000). The cell wall thickness of bagasse fiber was 4.8 m. This influences the fiber flexibility and interfiber bonding. A thicker cell wall resists collapse and does not contribute to interfiber bonding.

Chemically, sugarcane bagasses are rich in holocellulose (69.73%) and cellulose (38.95%) as illustrated in Table 2. These are important parameter for pulping. The lignin content (16.11%) was lower than normally found in wood fiberi. such as eucalyptus with 22% (Alcaide *et al.*, 1990). The functional significance of lignin has long been associated with mechanical support for plant organs that enables increased growth in height (Boudet, 2000). Pentosan was chiefly in those which yield xylose and arabinose on hydrolysis. The pentosan content of the sugarcane bagasse (28.24%) was 50% higher than that of hardwoods (Casey, 1980).

Pulping process

Fifteen conditions of soda pulping were trialled to measure the optimum conditions for bagasse pulping. They varied in terms of the cooking time, cooking temperature and chemical charges. The pulp yield and pulp lignin content were used to evaluate the effects of the main cooking parameters in the soda pulping of sugarcane bagasse. Regression analysis was conducted for fitting the following second order polynomial equation:

$$Y = 388.764 + 5.442X_1 + 0.568X_2 \; 0.086X_3 \; 0.001X_1X_2 \; 0.003X_1X_3 \; 0.018X_1^2 \; 0.003X_2^2 + 0.02X_3^2$$

$$K = 85.369 \; 0.367X_1 \; 0.131X_2 \; 0.184X_3$$

Where Y and K are the pulp yield and kappa number of soda pulp, respectively. X_1, X_2 and X_3 are the cooking temperature, cooking time and soda concentration, respectively.

The correlation coefficient for the pulp yield (R^2 = 0.91) and pulp lignin content (R^2 = 0.85) are high for a response surface. The effects of the cooking conditions on the pulp yield and lignin content are clearly shown in Figure 1 and Figure 2, respectively. The optimum conditions to achieve the above responses are a cooking time of 104 min, a cooking temperature of 154 °C and a soda concentration of 20 %. The multiple regression equations were solved for the optimum pulp yield (33.25 %) and the optimum pulp lignin content (Kappa number = 11.44).

Results from the regression analysis show that the yield was affected mainly by the cooking temperature and cooking time, followed by the chemical charge concentration. Figure 1 illustrates the relationship between the variables in the quadratic model of pulp yield. The positive value of the regression coefficient of the cooking temperature indicates that the pulp yield increased with an increase in the cooking temperature. At a certain cooking temperature level, the pulp yield stopped increasing. There was an optimal value for this variable.

Cooking time made a similar effect as cooking temperature on the pulp yield. The pulp yield increased with increased cooking time

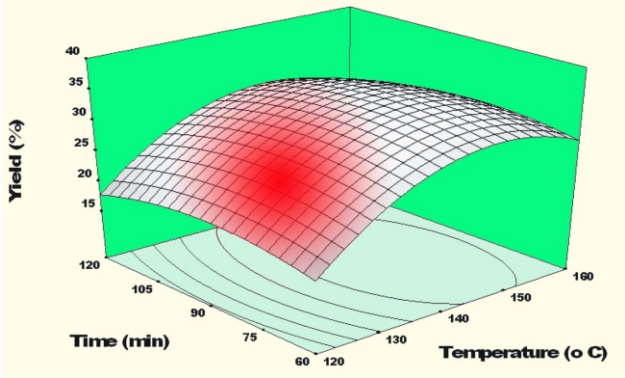

Figure 1 Effects of cooking time and cooking temperature on the pulp yield at 10% chemical charge concentration.

until it reached an optimum value at an intermediate level after which the pulp yield began to decrease.

The negative value of the regression coefficient suggested that the chemical charge concentration inversely affected the efficiency of the pulp production. An increase in soda concentration resulted in a decrease in the pulp yield.

On the other hand, the pulp lignin content or kappa number is given in the linear model. Figure 2 shows the 3D surface based on the 15 data point of the measured kappa number. The linear model has negative values for the regression coefficient. The cooking temperature, cooking time and soda concentration showed inverse effects on the kappa number. It can be concluded that the kappa number decreased with an increase in the pulping conditions.

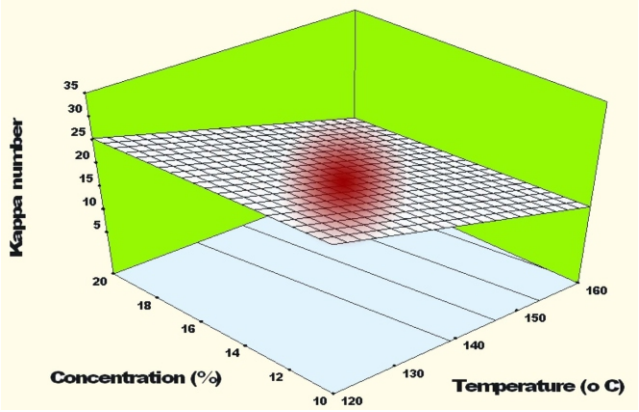

Figure 2 Effects of cooking temperature and chemical charge concentration on the Kappa number at a cooking time of 90 minutes.

An increase in the alkaline charge accelerated the delignification and decreased the pulp yield. Alen (2000) has established that during the alkaline pulping of lignocellulosics, alkali catalysed reactions (primary peeling) is mainly responsible for the loss of yield. This involves a stepwise elimination of monosaccharide moieties from the carbohydrates starting at their reducing ends and continuing along the polymeric chain until an alkali stable end group has been formed by a competing reaction.

Molded pulp manufacture

Molded pulp containers were formed by hot compression molding of the mixed pulp which was composed of the binder and chemical pulp of the sugarcane bagasse (Figure 3). Fully hydrolyzed polyvinyl alcohol (BF17) and cassava paste were used as the binder for molded pulp making. The color of the molded pulp containers made from alkaline pulp was pale brown. The molded pulp containers from the alkaline pulp contributed to the smooth surface and obviously fibrous texture.

Figure 3 Molded pulp container

The mechanical properties of the molded pulp packages suggested that the molded articles were durable to use. The compression strength and puncture resistance of the molded articles depended on the ratio of cassava starch paste and PVOH used as a binder. The role of adhesive is to develop the interaction between dissimilar bodies when they are in contact. The fibers of the molded pulp packages were sticky and the articles were strengthened. The effects of cassava starch paste and PVOH on the strength of molded articles are shown in Figure 4 and 5, respectively.

The compression strength of molded pulp containers made from alkaline pulp with different ratios of PVOH and cassava starch paste are shown in Figure 4. The type and composition of binders were the important parameters that affected the strength of molded articles.

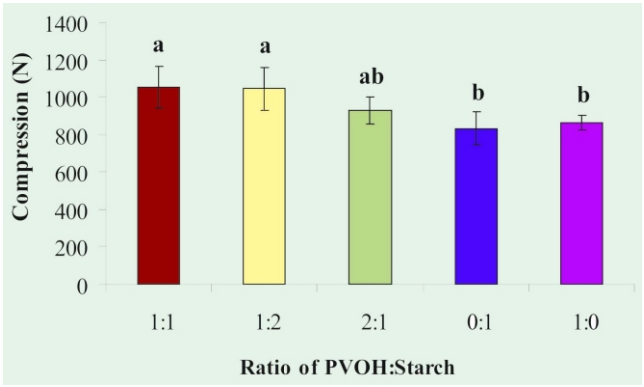

Figure 4 Compression strength of molded pulp containers made from various ratios of PVOH and cassava starch paste. Mean value with different letter are significantly different ($p < 0.05$)

The molded pulp package using a mixed binder of PVOH and cassava starch paste in the ratio of 1:1 gave the highest compression resistance before collapse. On the other hand, using only cassava starch paste as a binder gave the molded article the lowest compression strength. The presence of hydroxyl groups (-OH) tend to form strong hydrogen bonding among the molecules subsequently leading to synergistic stability and improved miscibility of the starch and PVOH (He *et al*., 2004; Rahmat *et al*., 2009; Saddaramaiah *et al*., 2003). Mixing starch and PVOH showed improvement in providing physicomechanical properties to the products. However, the compression strength of molded pulp packages made from the mixed adhesives of PVOH and cassava starch paste in the ratio of 1:1 and 1:2 were not significantly different ($p \geq 0.05$).

The puncture resistance of molded pulp packages using the various ratios of PVOH and cassava starch paste are shown in Figure 5. This test was used to determine the strength of the texture of the material. The type of adhesives also affected the puncture

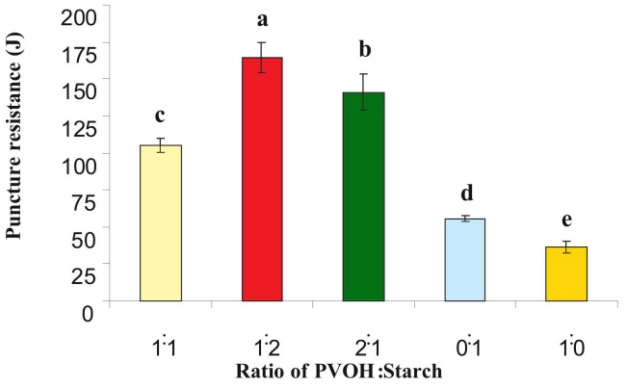

Figure 5 Puncture resistance of molded pulp containers made from the various ratios of PVOH and cassava starch paste
Mean value with different letter are significantly different ($p < 0.05$)

resistance. Figure 5 illustrates that the molded pulp package using PVOH and cassava starch paste in the ratio of 1:2 as a binder gave the highest puncture resistance. It can be concluded that the alkaline pulp of sugarcane bagasse mixed with PVOH and cassava starch paste in the ratio of 1:2 provided durable molded pulp packages.

Effect of coating on the mechanical properties of molded pulp container

A molded pulp package is sensitive to water. Water absorption deformed the molded pulp packages. Barrier coatings can to improve water absorption. PLA and Paraffin wax were used as the coating materials. They were dissolved in dichloromethane and coated on the surface of the molded fiber container by dipping. The results of water absorption on the molded pulp packages after coating are shown in Figure 6.

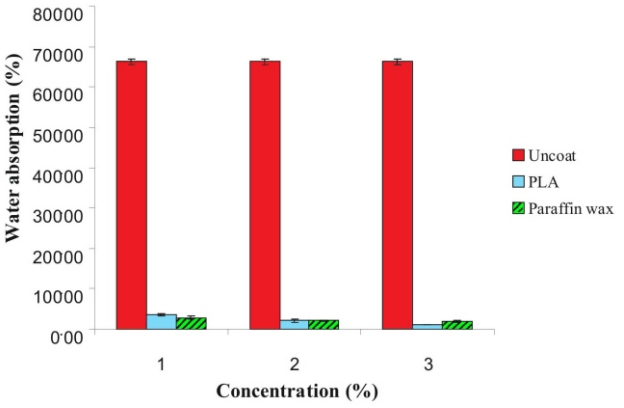

Figure 6 Water absorption of molded pulp containers after coating with PLA and paraffin wax at various concentrations

Molded pulp packages are hygroscopic, that is they are able to absorb moisture from the air. They contain hydroxyl radicals that are hydrophilic. When the molded pulp package was exposed to moist air or water, the OH groups attracted water molecules. Paraffin wax and PLA coatings are used to acts as a barrier to water absorption. After coating, the paraffin wax and PLA remain on the surface of molded pulp containers as a continuous film. The porous fibrous structure of the molded pulp container was covered and filled with coating materials (Rhim *et al*., 2007). The PLA coated pulp container contributed a smooth surface and the glossiness visually increased after coating. The molded pulp containers coated with both coating materials would have gained a little weight. The coating weights of paraffin wax on the pulp containers were 0.61%, 2.61% and 3.10% at coating concentrations of 1, 3 and 5%, respectively. In addition, the coating weights of PLA on the pulp containers were 1.26%, 2.62% and 3.48% at coating concentrations of 1, 3 and 5%, respectively.

Figure 6 shows that the water absorption of molded pulp packages was reduced by increasing the concentration of coating materials. However, a higher concentration of paraffin wax coating tended to have a lower efficiency of water protection compared to a PLA coating at the same concentration. Khwaldia et al. (2010) reported that the disadvantage of coating was brittleness, lack of homogeneity and presence of pinhole and cracks in the surface of coating. The paraffin wax at a high concentration might not completely cover the surface of the molded pulp as a continuous film after drying. The pinholes or cracks in the wax film were ways for the transmission of water and water vapour through the molded pulp. Thus the water absorption of PLA coating is lower than that of paraffin wax coating at higher concentrations of coating materials.

The compression strength and puncture resistance of uncoated and coated pulp containers are shown in Figure 7 and 8, respectively. The results showed that the compression strength of coated pulp containers increased with an increase in the concentration of coating solution. The trend for puncture resistance

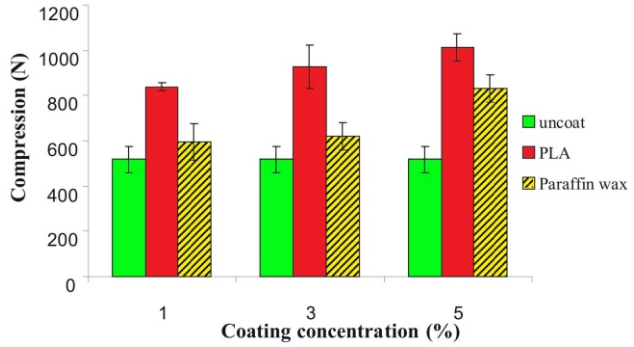

Figure 7 *Compression strength of molded pulp with coating and uncoating materials*

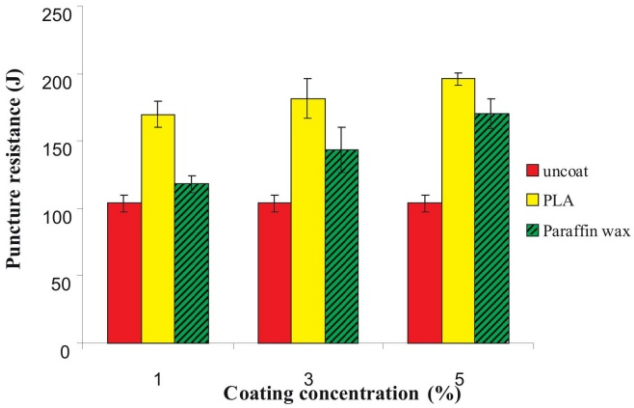

Figure 8 Puncture resistance of molded pulp with coating and uncoating materials

was similar with respect to compression strength. A higher solid content of coating gave a higher strength of molded pulp. It can be concluded that adding coating materials improves the mechanical properties of molded pulp containers.

The residue dichloromethane was also analyzed. Gas chromatography was used to investigate the residue of dichloromethane in the molded pulp products. A qualitative assessment of the residue of dichloromethane solvent in the molded pulp products was done by comparing peak areas with the pure dichloromethane at a concentration of 50.0 ppb. The results showed that the peak of dichloromethane disappeared completely in the PLA and paraffin wax coated products. It can be concluded that there were no residues of dichloromethane in the samples of paraffin wax and PLA coating. Dichloromethane has a boiling point of 40°C, thus the samples, after drying in the hot air oven at a temperature of 70°C, had no dichloromethane.

Conclusions

The development of molded pulp packages from sugarcane bagasses that are produced by hot compression is probably the greatest challenge. There are many factors affecting molded pulp making, including fiber characterization, types or composition of binder and types of coating materials.

The pulping process can be optimized by using RSM. A prediction model was established to identify the optimum pulping conditions for pulping yield and kappa index by using the best fitting second order regression model and linear regression model, respectively. These models, based on cooking temperature, pulping time and chemical charge concentration, related to the pulp yield and kappa index.

The compression strength and puncture resistance of molded pulp packages could be improved through using better binder composition. The optimum ratio of mixed adhesives between PVOH and cassava starch paste was 1:2. This contributed to higher mechanical properties of molded pulp packages.

The coated pulp packages using PLA and paraffin wax are promising because they provide greater strength and water resistance than uncoated pulp packages. In addition, PLA is biodegradable polymer which makes the coated pulp packages entirely biodegradable.

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