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Effect of Coating Methods on the Properties of Poly(lactide)-coated Paperboard: Solution Coating vs. Thermo-compression Coating

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Abstract Poly(lactide) (PLA)-coated paperboards were prepared by solution coating and thermo-compression coating methods and their effect of coating on the packaging properties such as tensile, water resistance, water vapor barrier, and heat sealing properties was tested. Compared with uncoated control paperboard, tensile strength (TS) of PLA-coated paperboard increased profoundly (2.2-2.6 folds) with slight increase in elongation at break (E). Water absorptiveness (WA) of the paperboard decreased 74-170 folds and water vapor permeability (WVP) decreased 6.3-22.1 folds by coating with PLA, which indicates an increase in the hydrophobicity of the surface of paperboard. Compared with polyethylene (PE)-coated paperboard, both PLA-coated paperboard exhibited 2.3 time higher heat sealing strength. In addition, the PLA-coated paperboards showed equal or higher wet TS than PE-coated paperboard. All the test results showed that the paperboard coated by the thermo-compression coating method was similar to or better than those of coated by the solution coating method.

Keywords: paperboard, one-way paper cup, water barrier, poly(lactide), solution coating, thermo-compression coating

Introduction

Paperboard is one of the most widely used packaging materials for both food and non-food products. In addition to the environmentally friendly nature of paperboard, it offers a number of benefits such as structure, stiffness, a substrate for barrier and other functional components, and a surface for printing with good packaging performance at a relatively low cost. Its application covers a wide range of uses from simple cartons, trays, tubs, and cups to complex containers used even for liquid (1). Since paperboards, basically composed of cellulose fibers, are hydrophilic in nature, they are usually coated with hydrophobic materials such as paraffin wax or polyethylene for the use as packaging or containers for the direct contact with high moisture foods (2,3). However, the content of the polyethylene and paraffin wax of packaging materials makes it difficult to separate, recycle, or compost them after use (4).

One of the main uses of paperboard is a disposable one-way paper cup, which is commonly used for potable water, coffee, and other beverages. Since it was introduced a century ago, enormous amount of one-way paper cups are being used in daily life. Fifteen billion disposable coffee cups are used in a year in the USA, with projected numbers reaching 23 billion by 2010 (5). And more than 60 million paper cups are used daily in Korea (5). Though the used paper cups have a high potential for recycling since they are manufactured with virgin pulp, the synthetic plastic coating materials causes a difficulty in recycling of the paper cups. Only 13.7% of the used paper cups are recycled to make toilet paper and the remainders are land-filled or incinerated resulting in a serious waste of natural resources and environmental pollution (5).

In an effort to produce more environmentally-friendly materials, renewable and biodegradable biopolymers like proteins such as corn-zein (6,7), whey protein (8-11), wheat gluten (9), and soy protein (12,13), and carbohydrates such as alginate (13) and carrageenan (14), have been investigated as paper coating materials. However, most of the research work on biopolymer coated papers have been focused on improving lipid barrier properties of paper.

In order to improve water barrier properties of paper packaging materials, a new type of coating materials with hydrophobic and good film-forming properties is needed. Poly(lactide) (PLA), an aliphatic thermoplastic biopolyester synthesized from lactic acid derived from renewable resources such as corn or sugar beets, has been considered as a potential candidate for food packaging applications since it is biodegradable, biocompatible with good performance characteristics as a packaging material with fairy good water barrier properties (5,15-18). In addition to its environmentally-friendly nature, PLA can also be used for food contact surfaces and generally recognized as safe (GRAS) (19). The water vapor permeability (WVP) values of PLA film are known to be two orders of magnitude lower than those of natural biopolymer-based films such as soy protein isolate films (20,21). With these characteristics and recent lowering the production cost by new technology and large-scale production, the application of PLA has been extended to commodity areas such as packaging, textiles, and composite materials (17).

Basically, there are two types of coating processes for the application of a uniform layer or coating across a paperboard, i.e., solvent-borne and solvent-free process. The one is based on dissolving a polymer in a suitable solvent then applying to the paperboard and evaporating the solvent, while the other is based on a hot melt coating in which a molten thermoplastic sheet (usually low or medium density polyethylene) is extruded from a slotted die and cast onto the paperboard. Recently, Rhim *et al*. (5,

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15) reported on the preparation of PLA-coated paperboards using a solution coating method with chloroform as a solvent. They showed packaging properties of the PLA-coated paperboard such as water resistance, water vapor barrier, mechanical, and heat sealing properties have increased significantly. Though the solvent coating method is easy to apply, it needs extra step to remove the solvent after casting and there still exists a problem on the remaining solvent in the PLA film layer, which has been reported to be 13.7% of the film layer (20). Therefore, a solvent-free coating method should be used for the preparation of PLA-coated paperboard, especially, in the case of direct contact with food materials. As one of solvent-free film forming method, Rhim *et al*. (20) used a thermo-compression method for the preparation of free-standing PLA films. Such thermo-compression method can be easily adopted for the preparation of PLA-coated paperboards, which does not need such expensive equipment like extruder.

The main objective of the present study was to test the feasibility of a solvent-free PLA-coating method for paperboard used for the preparation of paper cups by testing the effect of PLA-coating methods, i.e., solution coating and thermo-compression coating.

Materials and Methods

Materials A hard-sized solid-bleached-sulfate (SBS) paperboard (basis weight: 165 g/m^2) and a polyethylene (PE)-coated paperboard (coating thickness: 30 μm), commonly used for manufacturing paper cups, were obtained from Daehan Pulp (Seoul, Korea). Poly(L-lactide) (Biomer L9000; weight-average molecular weight, Mw=200,000 g/mol) was purchased from Biomer, Inc. (Krailling, Germany). PLA resins were dried in a vacuum oven at 60°C for 24 hr before use. Analytical grade chloroform was obtained from J.T. Baker (Mallinkrodt Baker, Inc., Phillipsbury, NJ, USA).

Poly(lactide) (PLA) coating PLA-coated paperboards were prepared using both solution coating and thermo-compression coating methods. The solution coated paperboards were prepared using the method of Rhim *et al*. (5,15). The thermo-compression coated paperboards were prepared through 2 steps, i.e., preparation of PLA films and thermo-pressing the film over the base paperboard. First, thermo-compressed PLA film was prepared following the method of Rhim *et al*. (20), in which 3 g of PLA was placed between 2 stainless steel plates lined with Teflon cloth and inserted them on a hydraulic press (Carver laboratory press, model 3925; Carver, Inc., Wabash, IN, USA) heated to 190°C and applied a pressure about 10,000 psi (68.9 MPa) for 2 min. The PLA film obtained was placed on the base paperboard and thermo-pressed again using the same method as the preparation of the PLA film. Both PLA-coated paperboards were used as test samples, and non-coated and PE-coated paperboards were also tested for comparison.

Conditioning All the paperboard samples were cut for property testing and conditioned in a constant temperature humidity chamber (model FX 1077; Jeiotech Co., Ltd., Ansan, Korea) set at 25°C with 50% relative humidity (RH) for at least 48 hr before test (5).

Weight and thickness measurements Weight of paperboard samples was determined using a digital balance (MC1 Analytic 210S; Satorius AG, Göttingen, Germany). Coating weights were determined by subtracting the weight of the uncoated paperboard from the weight of the coated paperboard. Thickness of paperboard samples was measured using a hand-held micrometer (Dial thickness gauge No. 7301; Mitutoyo Co., Ltd., Kanagawa, Japan) with 0.005-mm accuracy (5).

Tensile strength (TS) and percentage elongation at break (E) Tensile testing was carried out on an Instron Universal Testing Machine (Model 4465; Instron Engineering Corporation, Canton, MA, USA) according to the ASTM (D882-97) method as described previously (5).

Ring crush test Ring crush strength of paperboard was determined according to the method described by Lee and Rhim (22). Paperboard sample was cut into strips (12×153 mm) using a ring crush cutter. Test sample was placed in the crush tester and the ring crush strength was measured using the same Instron Universal Testing Machine as used in tensile test with the compression mode. A 500 N load cell was used with the crosshead speed of 50 mm/min. At least 10 specimens for each paperboard sample were tested with triplicate replication, and their mean values were reported.

Heat sealing strength Heat sealing of paperboard strips was performed using a Y-peel test described previously (5).

Water vapor permeability (WVP) Water vapor transmission rate (WVTR; g/m^2·sec) was measured using a gravimetric method described by Gennadios *et al*. (23) in accordance with the ASTM (E96-00) method. Paper samples cut into 70×70 mm were mounted on the WVP cups with the coated face to the inside of the WVP cups filled with 18 mL of distilled water. The cups were placed in an environmental chamber (Model FX 1077; Jeiotech Co.) set at 25°C and 50% RH. The actual RH value (RH_I, %) beneath the paperboard sample of the WVP cup was calculated after accounting for the resistance of the stagnant air layer between the paperboard and the water surface in the cup using the method of Gennadios *et al*. (23). Then the WVP (g·m/m^2·sec·Pa) was determined as follow:

$$WVP = WVTR \times L/\Delta p \qquad (1)$$

where, L is the thickness (m) of paperboard and Δp is the actual water vapor partial pressure difference (Δp, Pa) between both sides of the paperboard sample.

Water absorptiveness Water absorptiveness of the paperboard samples was determined using the Cobb test according to the ASTM (D 3285-93) method as described previously (5).

Wet tensile strength Wet TS of paperboards was measured according to Tappi standard method (T494. om-88) using the Instron Universal Testing Machine with the same test conditions as tensile test. The effect of water contact at one face of the paperboard, i.e., a coated surface, was tested using both water (ca. 20ºC) and hot coffee (ca. 80ºC). First, paperboard sample sheets (21×27 cm) were fabricated into tray type by folding each side for about 1 cm high in order to hold water or hot coffee, and added about 300 mL of water or hot coffee to cover the surface of the paperboard. After 30 min, the water or coffee was discarded and measured TS after removing surface water.

Statistical analysis All the test property measurements of each sample were triplicated with individually prepared coated or non-coated paperboard as the replicated experimental units. Analysis of variance (ANOVA) was performed to determine statistical differences in the properties of paperboard samples. If significant differences were indicated ($p<0.05$), the Duncan's multiple range test was performed to separate the mean property values.

Results and Discussion

Coating thickness and coating weight PLA was coated homogeneously on the paperboard by both coating methods, i.e., solution coating and thermo-compression coating. Especially, the thermo-compression method was more convenient to prepare the PLA-coated paperboards compared with the solution coating method without further processing of removal of solvent. The PLA layer was adhered firmly to the paperboard, which indicates a high affinity of PLA with the cellulose fibers to make well coated paperboards (5,15). Table 1 shows the coating thickness and coating weight of PE or PLA-coated paperboards. The thickness of paperboards increased 26.9-35.6 μm after coating with PE or PLA; consequently the coating weight also increased 34.7-57.6 g/m^2 depending on the coating material and coating method. Note that the standard deviation values of coating thickness of both PLA-coated paperboards were larger than that of PE-coated paperboards, which indicates that the PE-coated paperboards were coated more evenly with less variation in the coating thickness compared to the PLA-coated paperboards. As suggested previously (5), it is mainly attributed to the difference in the processing method, i.e., the PE-coated paperboards were produced industrially using extrusion coating and calendaring process.

Tensile strength (TS) and elongation at break (E) The results of tensile testing of paperboards are shown in Table 2. Mechanical strength of the paperboard increased significantly ($p<0.05$) as a result of PE- or PLA-coating, i.e., the strength of the paperboard determined by the TS values increased 1.9, 2.2, and 2.6 times for PE-coated paperboard, PLA-coated paperboard with solution coating method, and PLA-coated paperboard with thermo-compression coating method, respectively, compared with the uncoated counterpart. The E of the paperboard, a measure of flexibility or ductility of a packaging material, also increased after coating with PE- or PLA. Usually, the E of packaging materials decreased with increase in the TS (24), however, the E of PE- or PLA-coated paperboards increased with increase in the TS. This is mainly due to the higher resiliency of the coating films (20). Contrary to the present result, the decrease in the TS has been observed with paper or paperboard coated with hydrophilic biopolymer coating materials such as alginate (12), soy protein isolate (SPI) (12), and whey protein isolate (WPI) (10). The decrease in TS of such biopolymer-coated paperboard is attributed to the decrease in strength between cellulose fibers composed of paperboard structure, which may be partially due to the fact that coating materials are impregnated into the cellulose structure of paper and interfere with fiber-to-fiber interaction. Han and Krochta (10) suggested that such interference by the coating materials causes a reduction of interaction force between the fibers of coated papers, consequently resulting in decreased TS of coated paper. However, PLA is less hydrophilic than those hydrophilic biopolymers and was

Table 1. Coating thickness and coating weight of paperboards

Paperboard	Thickness of PB[1)] (μm)	Coating thickness (μm)	Coating weight (g/m^2)
PB/uncoated	205.0±0.9[a2)]	-	-
PE-coated PB	235.8±0.5[b]	30.8±0.5[b]	34.7±0.1[a]
PLA-coated PB (SC)	231.9±1.7[b]	26.9±1.7[a]	50.8±0.1[b]
PLA-coated PB (TC)	240.6±1.8[c]	35.6±1.8[c]	57.6±0.1[b]

[1)]PB: paperboard.
[2)]Means of 3 replicates±SD. Any 2 means in the same column followed by the same letter are not significantly different ($p>0.05$) by Duncan's multiple range test.

Table 2. Tensile properties, ring crush strength, and heat sealing strength of paperboards (PBs)[1)]

Paperboard	TS (MPa)	E (%)	Ring crush strength (N)	Heat sealing strength (kPa)
PB/uncoated	35.8±1.4[a2)]	4.6±0.8[a]	437.0±4.1[a]	-
PE-coated PB	69.3±0.1[b]	7.3±0.3[c]	463.3±2.4[b]	118.1±32.8[a]
PLA-coated PB (SC)	77.6±6.2[c]	6.3±1.9[b]	460.7±2.8[b]	271.2±36.2[b]
PLA-coated PB (TC)	93.3±4.8[d]	6.4±1.6[b]	465.8±4.6[b]	276.4±38.8[b]

[1)]TS, tensile strength; E, elongation at break.
[2)]Means of 3 replicates±SD. Any 2 means in the same column followed by the same letter are not significantly different ($p>0.05$) by Duncan's multiple range test.

not impregnated into the cellulose structure to interfere with fiber-to-fiber interaction. On the contrary, Rhim *et al.* (15) reported that the TS of PLA-coated Kraft paper by solution coating method decreased significantly compared with the uncoated control paper, which does not agree with the present result. This is mainly attributed to the difference in the source of fiber materials and processing methods, such as hard sizing for SBS paperboard and PLA coating (thermo-compression method), causing it more hydrophobic and resulting in less penetration of PLA film layer into the paper matrix. The increase in E of PLA-coated paperboards indicates that the flexibility or ductility of paperboard increased by coating with PLA. The flexibility is an important property of packaging materials like paperboard used for making cartons or paper cups, which need endurance from frequent bending or pressing.

Ring crush strength Usually ring crush test is used as the standard test for the paperboard of fiberboard boxes or cartons to predict stacking strength of the final form of those boxes. The test is expected to serve as a good predictor of performance of paper cups stacked during storage and transportation. Table 2 also shows the ring crush test result of the paperboards. Ring crush strength of the paperboard increased significantly after coating with both PE and PLA ($p<0.05$). This is mainly due to the increased strength of the coated paperboards. There was no significant difference in the ring crush strength between coating materials and PLA coating methods. The increase in the ring crush strength in the present study (5-7%) is much lower than the previously reported for PLA-coated Kraft paper (1.5-1.9 fold increase) (22). This may be attributed to the higher ring crush strength of the base paperboard (437±4.1 N) than that of the Kraft paper (ca. 230 N).

Heat sealing strength The heat sealability of a packaging material is a crucial property to maintain the integrity of a package. To obtain a proper heat seal is an important requirement in packages like cartons and one-way paper cups, since seal failure cause not only damage to the package itself but also product deterioration. The heat sealing strength test result of PE- and PLA-coated paperboards are shown in Table 2. The heat sealing strength of PLA-coated paperboards were 2.3 times higher than that of PE-coated paperboard. However, there was no significant difference in the heat sealing strength of PLA-coated paperboards between PLA coating methods. Recently, Rhim and Kim (5) reported that the heat sealing strength of PLA-coated paperboards coated by solution coating method increased linearly with increase in PLA concentration in the range of 1 to 4%(w/v). They found the heat sealing strength of about 270 kPa for the PLA-coated paperboard coated with 3%(w/v) PLA coating solution, which is in good agreement with the present study. Considering the heat sealing strength of the PE-coated paperboard (118.1±32.8 kPa), the PLA-coated paperboards prepared by both solution coating and thermo-compression coating methods can be used for the manufacture of a paper cup or a carton.

Water absorptiveness (WA) The WA provides basic information on water resistance of the paperboard because it indicates how much the paperboard can absorb water when directly contacts with water. The water resistance of the paperboards was measured through direct contact of water with the surface of the boards and the results are shown in Table 3. The WA of the paperboards decreased tremendously by coating with PE or PLA. The WA of PE-coated paperboard was negligible and that of PLA-coated paperboard decreased by 73.7 and 170.2 times depending on the coating method. Thermo-compression coating method was more effective in reducing the WA, which may be explained by the fact that a more compact structure was formed through thermo-compression with less hydrophilic surface compared with the solution coated paperboard. Rhim *et al.* (20) demonstrated PLA films prepared with solution casting method retained 13.7% of solvent (chloroform) in the film matrix, which served as a plasticizer and affected film properties. Contrary to the present result, the WA of paper or paperboard coated with hydrophilic biopolymers such as WPI (8), alginate (12), and SPI (12) was reported to be increased after biopolymer coating, which may be caused by the accelerated water absorption at the biopolymer film layer and consequently elevated total water absorption capacity. This indicates that coating with PLA is more effective than hydrophilic biopolymers for the increase in water resistance of paperboard.

Water vapor permeability (WVP) WVP is a measure of resistance of packaging materials against water vapor transmission. A proper WVP property of packaging materials is necessary for preventing the water vapor from being transported through the wall of packaging materials to affect the product quality packaged with them and to reduce the mechanical strength of the packaging materials. Table 3 also shows the WVP values of paperboards and the calculated relative humidity values underneath the paperboard samples in the WVP cups (RH_I). The WVP of the paperboard decreased greatly after coating with PE and PLA. Depending on the coating material and PLA coating methods, they decreased from 6.3 to 99 times compare

Table 3. Water absorptiveness (WA) and water vapor permeability (WVP) of paperboards (PBs)

Paperboard	WA (g/m^2)	WVP ($\times10^{-10}$ g·m/m^2·sec·Pa)	RH_I[1)] (%)
PB/uncoated	22.12±0.01[b2)]	40.60±0.81[d]	72.5±0.2[a]
PE-coated PB	-	0.41±0.01[a]	99.6±0.0[c]
PLA-coated PB (SC)	0.30±0.01[a]	6.44±0.74[c]	97.2±0.5[b]
PLA-coated PB (TC)	0.13±0.10[a]	1.84±0.40[ab]	98.2±0.1[bc]

[1)]Real RH underneath the paperboards of WVP cup.
[2)]Means of 3 replicates±SD. Any 2 means in the same column followed by the same letter are not significantly different ($p>0.05$) by Duncan's multiple range test.

Table 4. Wet tensile strength (TS) of paperboards (PBs) determined by tensile test

Paperboard	Wet TS (MPa)	
	Contact with water	Contact with hot coffee
PB/uncoated	7.7±1.9[a1)]	4.3±0.2[a]
PE-coated PB	37.1±1.1[b]	33.5±1.7[b]
PLA-coated PB (SC)	39.2±5.4[b]	36.4±3.8[c]
PLA-coated PB (TC)	40.6±6.6[b]	36.7±0.9[c]

[1)]Means of 3 replicates±SD. Any 2 means in the same column followed by the same letter are not significantly different (p>0.05) by Duncan's multiple range test.

with the uncoated paperboard. As expected, the PE-coating was the most effective and followed by the PLA-coating with thermo-compression and solution coating methods. The WVP of PLA-coated paperboard coated with the thermo-compression method was significantly (p<0.05) lower than that coated with the solution coating method. This result is consistent with the WA test result. The RH_I values also support the result of WVP measurement. Rhim *et al.* (15) reported that the WVP of PLA-coated Kraft paper prepared with solution coating method (3 w/v% coating solution) decreased about 24 folds from (4.08 ±0.11)×10^{-9} g·m/m^2·sec·Pa for the uncoated Kraft paper to (0.17±0.04)×10^{-9} g·m/m^2·sec·Pa. Their results are in good agreement with the present study. On the other hand, Han and Krochta (8) found that WPI coating on pulp paper reduced WVP by 1.42 to 1.96 folds depending on the concentration of coating solution, which is not so profound increase compared with the PLA-coating. It is mainly due to the lower WVP of PLA film layer. The WVP values of PLA film (thickness: ca. 90 μm) prepared by solvent casting method and thermo-compression method were reported as (4.66±0.25)×10^{-11} and (2.61±0.07)×10^{-11} g·m/m^2·sec·Pa, respectively (20). Namely, the WVP values of PLA films are 2 orders of magnitude lower than that of paperboard.

Wet tensile strength (TS) The wet TS, a measure of ability of a packaging material to resist against disruption under direct contacting with water, is the one of the most important properties of a package used for water container like paper cup. In the present study, the wet TS of paperboards were tested after contacting water or hot coffee with only one side (coated face) of paperboards to simulate the real situation of using paper cups and the results are shown in Table 4. Generally, mechanical strength of all the paperboards was reduced more when they contacted with hot coffee. This may be caused by the higher diffusion rate of moisture, i.e., more adsorption of water, into the cellulose fibers to reduce interaction force between them. Another possibility for decrease in the wet TS of PLA-coated paperboard is the glass transition of the coated PLA layer, i.e., transition from the amorphous glassy state to the rubbery state at its glass transition temperature around 62.5°C (20), experiencing during contact with hot coffee. However, such effect on glass transition on the wet TS is expected to be relieved since the tensile test was performed at room temperature after the temperature of the coated surface decreased below the glass transition temperature of PLA. As expected, uncoated paperboard decreased in TS tremendously from 35.8±1.4 MPa (Table 2) at dry state to 7.7±1.9-4.3±0.2 MPa when they were wet. Though the coated paperboards also decreased in wet TS, they still retained appreciable amount of strength. In both cases of contacting with water and hot coffee, the wet TS of PLA-coated paperboards were higher than that of the PE-coated paperboard, though the differences are not statistically significant. The high wet TS of PE- or PLA-coated paperboards may be attributed to the improved hydrophobicity of the coated surface as indicated in the WA and WVP measurements. In addition, the higher wet strength of PLA-coated paperboard than that of PE-coated paperboard implies that the PLA-coated paperboard can be used for replacing PE-coated paperboard for packaging of high moisture food or beverage in the form of paper cup or carton.

Conclusively, mechanical and water barrier properties of PLA-coated paperboards for the use of one-way paper cup determined by TS, E, heat sealing strength, WA, WVP, and wet TS indicated that they were as well as or better than PE-coated paperboard. Between the two test methods for PLA coating, the thermo-compression coating method resulted in better mechanical and water barrier properties than the solution coating method. As a solvent-free hot melt coating method, the thermo-compression method shows a high potential for manufacturing environmentally-friendly PLA-coated paperboards utilizing the industrial extrusion coating process.

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