

Functional Properties of Polylactic Acid Starch-Based Loose-Fill Packaging Foams¹

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ABSTRACT

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In this study, attempts were made to improve the characteristics of starch-based plastic foams by blending starch with polylactic acid (PLA), a biodegradable polymer made from renewable sources. Formulations of the foams (types of starch, ratio of starch to polymer, and moisture content) were optimized. Physical and mechanical properties of the foams, including expansion, unit density, bulk density, water solubility index (WSI), spring index, and compressibility on both single piece and bulk samples were determined. The addition of the PLA polymer to regular (25% amylose) and waxy corn starches significantly improved the physical and mechanical properties of the extruded foams. Foams made from waxy starch had better radial expansions, higher WSI, higher compressibilities, and

lower spring indices than those of regular starch foams. Both regular and waxy starches produced foams with similar unit and bulk densities, bulk compressibilities, and bulk spring indices. Increasing PLA polymer contents increased the radial expansions and spring indices and significantly reduced the unit and bulk densities and bulk compressibilities. Changing the PLA content had no significant effect on WSI, compressibilities, and bulk spring indices. Increasing the moisture content adversely affected foam characteristics. The formulation containing waxy starch, 40% PLA, and 19% moisture produced a loose fill foam with the best physical characteristics and mechanical properties.

Loose-fill cushioning materials provide cost-effective protection for relatively lightweight products against shock or vibration during shipping and handling (Torok 1990). In the packaging industry, ≈19.6 billion pounds of plastic resins are consumed annually for rigid and flexible packaging in the United States alone. Only ≈1.61 billion pounds of postconsumer plastic packaging materials were recycled in the United States in 1996, which is <10% of the total amount of plastics used for packaging (Mastio 1998). Traditionally, loose-fill packaging materials have been prepared from petrochemical products such as polystyrene and polyethylene. Expanded polystyrene foam (EPS) is the most commonly used due to its low density, high resilience, and water resistance. During manufacturing of loose-fill materials, chlorofluorocarbons (CFC) are used as a blowing agent. CFC affect the ozone layer of the atmosphere (Molina and Rowland 1974). Also, the disposal of the used plastic packaging materials has become a public concern (Larson 1989). Also the manufacturing of petroleum-based loose-fill packaging materials increases oil imports and pollutes the environment.

Numerous research efforts have been made to find alternatives to replace the petrochemical-based loose-fill packaging materials. Wang et al (1995) studied the physical properties of two biological cushioning materials made from wheat and corn starches. Wheat and corn starch samples were mixed with 3% polyethylene glycol and 0.5% silicon dioxide. Samples were then extruded using a single-screw extruder. Compared with conventional EPS foams, the starch foams had significantly higher bulk densities, more unrecoverable plastic deformation, and higher water absorption. Lin et al (1995) extruded 70% amylose starch for loose-fill packaging materials. The starch was adjusted to 22% (wb) moisture content and then extruded using a corotating, intermeshing, self-wiping twin-screw extruder. Water sorption affected the mechanical properties of the extrudates. Both energy index and compression strength increased with increasing water activity. The spring index behaved similarly at higher water activity levels.

However, packaging materials made from pure starches usually do not have satisfactory physical and mechanical properties and

they are highly water-soluble. To improve the physical and mechanical properties and the resistance to water of the starch-based foams, Bhatnagar and Hanna (1995) extruded 25% amylose corn starch with polystyrene and polymethyl methacrylate in ratios of 70:30. Bicarbonate, urea, and siloxane were used as blowing and cross-linking agents. Water solubility indices (WSI) were reduced to 4–13% compared with 97% for the pure commercial starch loose-fill packaging material. However, the unit densities were too high at 30–59 kg/m³ compared with 8.9 kg/m³ for the commercial polystyrene foam. Also, the use of polystyrene lowered the biodegradability of the foam as well.

Lay et al (1992) holds a U.S. patent on polymer compositions containing destructurized starch. The polymer compositions have potential in manufacturing loose-fill packaging materials. Chinnaswamy and Hanna (1993) hold U.S. and Australian patents on manufacturing loose-fill foams made of 70% starch and 30% polystyrene. The combination of starch and polystyrene reduced the use of petroplastic and improved the biodegradability while providing water resistance.

Wang and Shogren (1997) extruded regular corn starch blended with biodegradable polymers to make loose-fill foams. Effects of nucleating agent (talc), blowing agents (water, ethanol, and propanol), and different polymers (polyvinyl alcohol, polylactic acid, polycaprolactone, cellulose acetate, polybutylene succinate-adipate, and polyester-amide) and extrusion conditions on foam density, resilience, and compressibility were examined. Results indicated that 0.5–1.0% talc addition was the optimum range. No significant differences existed among the blowing agents. Addition of 10–15% polylactic acid (PLA) gave foams the lowest density and the highest resilience.

Cha et al (1999a,b) conducted extrusions to study the rheological properties of blend melts and effects of temperature and moisture contents on mechanical properties of starch-based foams containing wheat and corn starches, polyethylene-covinyl alcohol, polystyrene, plasticizers, and nucleating and blowing agents. The blend melts transformed from dilatant to pseudoplastic fluid when temperature increased from 403 to 433 K. Bulk compressibility decreased as extrusion temperature and moisture content increased. Bulk resiliency increased as temperature increased and moisture content decreased. The mechanical properties of the starch foams were compared with those of commercial polystyrene foams.

The loose-fill packaging industry also has gone a long way in their efforts to commercialize starch-based foams. Ecofoam, a commercially available polystyrene-free foam (American Excelsior Co., Arlington, TX) is made of 95% hydroxypropylated high-

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amylose starch and 5% polyvinyl alcohol. This foam is biodegradable and has good mechanical properties, but it dissolves in water readily. Other commercially available starch-based foams include Biofoam (Biofoam Corporation, Atlanta, GA), starch packaging peanuts (Clean Green Packing Co., Minneapolis, MN), corn loose-fill packaging (Lewis Pulp Products, Jefferson, WI) and sorghum packaging peanuts (Bifpak, Tempe, AZ). All of the above-mentioned products are either water soluble or contain nondegradable plastics. Therefore, it is justified from points of view of improving the water resistance and the physical and mechanical properties of the loose-fill foams to incorporate biodegradable polymers into starch-based foams. The products would be expected to be completely biodegradable and possess excellent functional properties.

The overall objective of this study was to address the issues of improving physical and mechanical properties and water resistance of the starch-based loose-fill packaging materials by blending starch and PLA polymers.

In this study, the operating conditions of the extrusion were fixed, based on the results of preliminary studies. Only the formulations of the samples were varied. The independent variables included type of starch, ratio of starch to polymer, and moisture content. Physical properties, including radial expansion ratio, unit and bulk densities, WSI, and mechanical properties including spring index and compressibility on single piece and bulk samples were dependent variables.

MATERIALS AND METHODS

Materials

Semicrystalline polylactic acid (PLA) resin of MW_n 85,000 was purchased from Cargill, Inc. (Minneapolis, MN). It contained ≈93% L-lactide, 2% D-lactide and 5% mesolactide. The resin was dried at 40°C for 24 hr before use. The regular (25% amylose) and waxy corn starches were purchased from A.E. Staley Co. (Decatur, IL). The starches were agglomerated into spherical granules of 2f-4 mm in diameter to facilitate feeding into the extruder. Talc (magnesium silicate) was obtained from Barret Minerals, Inc. (Dillon, MT) and was used as a nucleating agent to ensure the uniformity of the extrudate cell void spaces (Bhatnagar and Hanna 1996). The talc had a median particle size of 1.2 μm, a bulk density of 120 kg/m³ and a specific gravity of 2.8. Distilled water was used to adjust the moisture content levels of the samples.

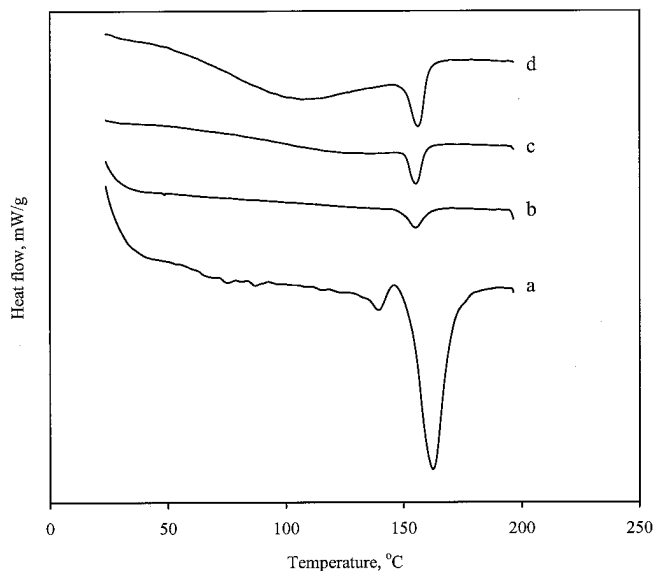


Fig. 1. Thermographs of polyactic acid (PLA) and PLA and starch foams: (a) PLA, (b) regular starch with 10% PLA, (c) waxy starch with 25% PLA, and (d) waxy starch with 0% PLA.

Equipment

A laboratory twin-screw extruder with corotating mixing screws (model CTSE-V, C. W. Brabender, Inc., S. Hackensack, NJ) was used to conduct the extrusions. The conical screws had diameters decreasing from 43 to 28 mm along a length of 365 mm from the feed end to the exit end. The rotating speeds for both screws were set at 150 rpm. The average feed flow rate was 125 g/min. The temperature at the feeding section of the barrel was set at room temperature while the other two sections and the die were maintained at 150°C. A 3-mm opening die nozzle was used to produce cylindrical extrudates, which were then cut into finite lengths for testing purposes using a rotating cutter mounted on the die face of the extruder. The extruder was controlled by a Plasti-Corder (Type FE 2000, C. W. Brabender, Inc., S. Hackensack, NJ). Data including screw rotating speeds, barrel temperature profiles, pressure profiles, and torque reading were recorded by a computer for subsequent analyses.

Experimental Design

A completely randomized factorial blocking experimental design was used for this study. Enclosed in the study were two types of corn starch (regular and waxy), three levels of polymer addition (10, 25, and 40%), and three levels of moisture content (19, 22, and 25% db). There were 18 treatment combinations. To eliminate the day-to-day variation, days were used as blocks (i.e., three replications were conducted on three different days). A total of 54 extrusions were conducted. All the data were analyzed using SAS statistical software (SAS Institute, Cary, NC).

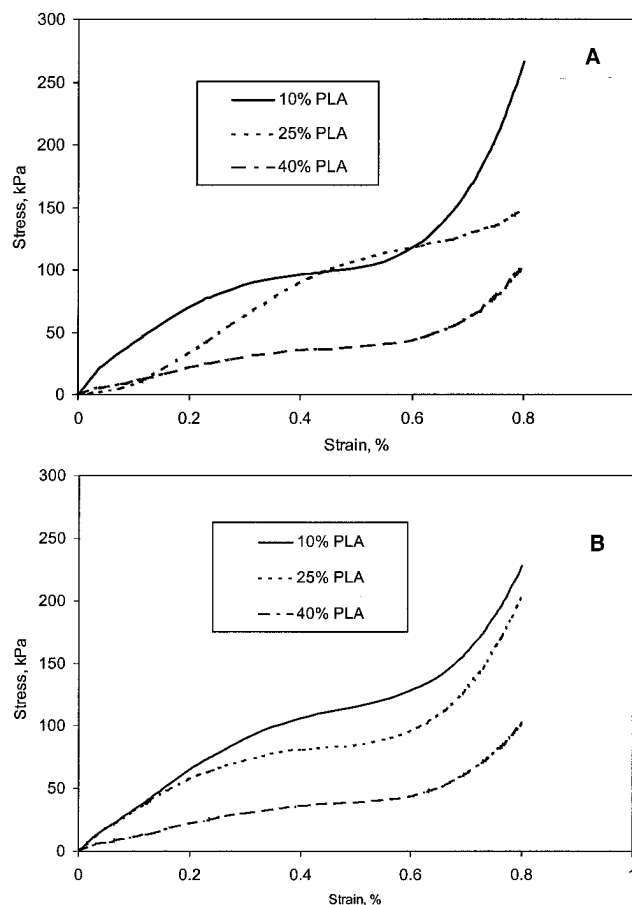


Fig. 2. Stress-strain curves of polyactic acid (PLA) and starch foams: **A**, 25% amylose starch; **B**, waxy starch.

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) was conducted on all the raw materials and selected extrudates to study the glass transition and melting temperatures. A DuPont differential calorimeter with a 910 cell base (TA Instruments, Inc., New Castle, DE) was used to run the DSC analyses. The instrument was first calibrated using indium and purged with nitrogen gas. Samples (≈ 10 mg) were sealed in an aluminum pan, allowed to equilibrate to 25°C and scanned from 25 to 180°C at a constant heating rate of 10°C/min. Three replicates were scanned for each sample.

Physical Properties

Physical properties of the extrudates including radial expansion ratio, unit density, bulk density, and WSI were studied. Radial expansion ratio values of the extrudates were calculated by dividing the mean cross-section area of the extrudates by the cross-section area of the die nozzle. Each mean value was the average of 10 measurements.

Unit density was determined using a glass bead displacement method (Bhatnagar and Hanna 1995). Glass beads of 0.1 mm in diameter were used as the displacement media to determine the volume of the extrudate. Unit density was obtained by dividing the mass by the volume of the extrudate being measured.

Bulk density was measured using a cylindrical Plexiglas container (ASTM D1895-96). The container had a diameter of 160 mm and a height of 160 mm. A funnel with an opening of 160 mm at the top and an opening of 64 mm at the bottom was mounted at a height of 160 mm above the container. Bulk density of an extrudate was calculated from the mass of the sample divided by the volume of the container.

WSI was measured using the method developed by Anderson et al (1969) with some modifications. To measure the WSI, extrudates were first ground into fine powders in a grinder and sieved through a 60-mesh U.S. standard sieve. Each sample (≈ 1 g) was dispersed in 100 mL of distilled water in a centrifuge tube. The dispersed samples were held for 30 min at 30°C with periodic stirring and then centrifuged at $5,000 \times g$ for 10 min. The supernatants were decanted and solute concentrations were determined by the phenol sulfuric acid method (Dubois et al 1956, Bhatnagar and Hanna 1994). WSI were determined at room temperature and expressed as the percentages on a dry matter basis.

Mechanical Properties

Before the mechanical property measurements were conducted, all specimens were conditioned under $43.2 \pm 0.4\%$ rh in a desiccator with saturated potassium carbonate (K_2CO_3) aqueous solution for 10 days at 25°C. The weight changes of the samples were closely monitored. After seven days, the sample weight was basically held constant, an indication of equilibrium.

Spring index refers to the ability of a material to recover its original shape after it has been deformed. It is a measure of the elastic characteristics or resilience. An Instron universal testing machine (model 5566, Instron Engineering Corp., Canton, MA) with a 6-mm diameter cylindrical probe was used to compress the sample to achieve a deformation of 80% of its original dimension at a loading rate of 30 mm/min (Altieri and Lacourse 1990). The cylindrical extrudate samples were cut into pieces 20 mm long using a razor blade. For each compression run, five pieces were used. The forces required to initially compress the samples and the forces required to recompress the same sample 1 min after releasing the initial load were recorded. Spring indices were calculated by dividing the values of the recompression forces by the values of the initial compression force.

Compressibility of the samples (i.e. the peak forces required to achieve a deformation of 80% of the original dimensions) was determined using the same Instron universal testing machine with the same testing conditions used in the spring index measurements. Compressibility values indicate how rigid the samples are. Because

of the differences in radial expansion of the test specimens, the peak forces were normalized by dividing them with the corresponding sample unit density.

Bulk compressibility and spring index of the samples were tested on bulk samples using the same Instron universal testing machine. A cylindrical aluminum container with a volume of 365 cm³ (6.93 cm in diameter and 9.68 cm in depth) was used to confine the bulk samples. The forces required to initially compress the samples to 80% of their original volumes and the forces required to recompress the same samples 1 min after releasing the initial load were recorded. Bulk spring indices were calculated by dividing the values of the recompression forces by the values of the initial compression forces. Bulk compressibility was expressed as the maximum force required to compress the sample to 80% of its initial volume.

RESULTS AND DISCUSSION

Typical DSC thermographs for the virgin PLA and PLA and starch foam samples are presented in Fig. 1. As shown in Fig. 1a, the virgin PLA resin had a melting temperature of 164°C. It also shows a clear peak, indicating that the PLA resin contained a crystalline region. The thermographs of foam samples containing regular starch blended with 10% PLA (Fig. 1b), waxy starch with 25% PLA (Fig. 1c), and waxy starch with 40% PLA (Fig. 1d) show a melting point at 157°C, which corresponds to that of PLA. The reduction in the PLA melting temperature from 164 to 157°C after extrusion was probably due to different thermal history, plasticization by water, or hydrolysis decomposition of PLA induced during extrusion. Also, the magnitude of the downward peaks corresponding to the PLA melting point increased as the PLA content increased from 10 to 40%. The DSC thermographs indicated clearly that at least part of the PLA formed a separate phase from that of the starch.

Radial expansion ratio, unit density, and bulk density are the most important physical characteristics of PLA and starch foams to be used as loose-fill packaging materials. They are all indirect measures of how well the extruded foams expanded. Table I shows the effect of starch type on the radial expansion ratio, unit density, and bulk density. Starch type had a significant effect on the expansion ratio. Waxy starch is $\approx 100\%$ amylopectin, while regular starch contains $\approx 25\text{--}30\%$ amylose. Waxy starch resulted in significantly higher radial expansion than did the regular starch. This was due mainly to the differences in molecular structures. Amylopectin is a very large and highly branched molecule, and amylose is essentially a linear molecule. Launay and Lisch (1983) showed that expansion ratio decreased as amylose content increased. Higher amylopectin content provided a light, elastic, and homogeneous texture with a smooth surface. In contrast, high-amylose starch resulted in a harder and less expanded extrudate. There was perhaps greater cross-linking due to amylose, thus restricting expansion. On the other hand, the effects of starch type on both unit density and bulk density were not significant.

Table II presents the effects of PLA polymer content on the radial expansion ratio, unit density, and bulk density. Polymer content had a significant influence on the radial expansion ratio and bulk density. As polymer content increased, the radial expansion ratio increased, which resulted in significant reductions in unit and bulk densities. Higher PLA polymer contents may have contributed in the increase of elasticity of the cell walls, helping trap more gas in the cells. This would increase the radial expansion and reduce the unit and bulk densities. The higher elasticity of the cell walls also would help prevent the cell structure from collapsing when the extrudates exited the die nozzle.

During starch foam extrusion, water is heated up and vaporizes when pressure is reduced as the product exits the extruder; water functions as a blowing agent. Also, the presence of water helps in the gelatinization of starch. The moisture effect on the physical properties of PLA and starch foam are shown in Table III. At mois-

ture content of 19% (db), the PLA and starch foam extrudates achieved the highest radial expansion and the lowest unit density and bulk density. As the moisture content increased, the radial expansion ratio decreased, and the unit density and bulk density increased. The foams became denser and heavier. During the extrusion runs, the cell structures of the extrudates at higher moisture contents collapsed after exiting the extruder die nozzle. Stearns (1974) pointed out a similar relationship between extrudate expansion and moisture content and attributed it to the inability of the thin-wall cells to maintain integrity as the cells were expanded by a large amount of steam flashing off during extrusion.

WSI measures how well the starch-based foam resists water. The good water solubility of pure starch-based foam makes it easier to degrade. This, however, also is a disadvantage in high-moisture environments. One of the objectives of this research was to use blends of degradable polymers and starch to achieve good water resistance while maintaining biodegradability. Table I shows the effect of starch type on WSI. Foams made from waxy starch had significantly higher WSI values than did those made from regular starch. Waxy starch foam dissolved more readily in water than the regular starch. Neither the PLA content nor the moisture content significantly affected WSI (Tables II and III). Overall, the WSI values (21–28%) were improved significantly compared with 97% for commercially available starch foams (Bhatnagar and Hanna 1995).

Compressibility measures the rigidity of the foam. A foam sample with a high value of compressibility indicates that it is rigid. Spring index measures the resilience of the foam. A perfect spring is an ideal elastic body that will deform on application of a load and regain its original shape after removal of the load (Bhatnagar and Hanna 1995). For a foam to be used as loose-fill packaging materials, higher spring index values are preferred. Figure 2 presents the strain-stress curves of PLA and starch foams made from the various formulations. Table IV summarizes the mechanical properties of both the single piece and the bulk samples, as affected by starch type. The regular starch foams had significantly lower compressi-

bility values and higher spring indices than the waxy starch foams. However, the effects of starch type on the mechanical properties of the bulk samples were not significant.

The PLA polymer content had varying influences on the mechanical properties of the foams (Table V). For the single piece samples, PLA content did not affect the compressibility significantly but did affect the spring indices significantly. As the PLA content increased, the values of compressibility changed slightly; the spring indices increased from 80.8 to 90.4% as the PLA content increased from 10 to 40%. Increasing the PLA content made the foam more elastic. For the bulk samples, increases in PLA content decreased the compressibility values and made the foam softer. The changes in PLA content, however, did not affect the bulk spring indices significantly. At all levels of PLA content, the bulk samples had impressive spring indices, ranging from 94.6 to 97.2%, comparable to 97% for the commercially available EPS foam (Bhatnagar and Hanna 1995).

Moisture content of the feed materials had a significant affect on foam compressibility (Table VI). As the moisture content increased, the extruded foam became more rigid. This was mainly due to the collapsing of the cell structures of the foams at higher moisture content. Moisture content did not affect the spring indices significantly. The spring indices only decreased from 86.6 to 83.9% as the moisture content was increased from 19 to 25%.

Among all the foams of various formulations, the foam made of waxy starch, 40% PLA, and 19% moisture content had the best physical and mechanical properties. However, addition of 40% PLA would significantly increase the raw material cost and make the foam more expensive than the commercially available EPS foam because PLA resin currently sells up to \$10.00/kg.

CONCLUSIONS

The addition of PLA polymer to regular and waxy corn starches significantly improved the physical and mechanical properties of extruded starch foams. Foam made from waxy starch had higher radial expansion ratio and WSI, higher compressibility, and lower spring index. Both regular and waxy starches produced foams with

TABLE I
Main Effect of Starch Type on Polylactic Acid (PLA)
and Starch Foam Physical Properties^a

Starch	Radial Expansion Ratio	Unit Density (kg/m ³)	Bulk Density (kg/m ³)	Water Solubility Index (%)
Regular	13.4a	41.3a	24.1a	20.3a
Waxy	19.9b	43.3a	24.2a	28.1b

^a Values followed by the same letter are not significantly different ($P < 0.05$).

TABLE II
Main Effect of Polylactic Acid (PLA) Content on PLA
and Starch Foam Physical Properties^a

PLA Content (%)	Radial Expansion Ratio	Unit Density (kg/m ³)	Bulk Density (kg/m ³)	Water Solubility Index (%)
10	15.2a	54.2a	30.8a	25.3a
25	15.9ab	46.0ab	23.9b	24.1a
40	19.2b	41.2b	17.7c	23.2a

^a Values followed by the same letter are not significantly different ($P < 0.05$).

TABLE III
Main Effect of Moisture Content on Polylactic Acid (PLA)
and Starch Foam Physical Properties^a

Moisture Content (% db)	Radial Expansion Ratio	Unit Density (kg/m ³)	Bulk Density (kg/m ³)	Water Solubility Index (%)
19	18.4a	42.0a	24.1a	27.9a
22	16.7ab	44.4a	27.8ab	23.8a
25	15.0b	59.9a	37.4b	20.9a

^a Values followed by the same letter are not significantly different ($P < 0.05$).

TABLE IV
Main Effect of Starch Type on Polylactic Acid (PLA)
and Starch Foam Mechanical Properties^a

Starch	Compressibility (N)	Spring Index (%)	Bulk Compressibility (N)	Bulk Spring Index (%)
Regular	211.5a	87.4a	736.4a	97.4a
Waxy	430.9b	82.7b	756.4a	95.0a

^a Values followed by the same letter are not significantly different ($P < 0.05$).

TABLE V
Main Effect of Polylactic Acid (PLA) Content on PLA
and Starch Foam Mechanical Properties^a

PLA Content (%)	Compressibility (N)	Spring Index (%)	Bulk Compressibility (N)	Bulk Spring Index (%)
10	340.6a	80.8a	1,125.2a	94.6a
25	279.2a	83.6a	690.2b	97.2a
40	365.3a	90.4b	423.9b	96.9a

^a Values followed by the same letter are not significantly different ($P < 0.05$).

TABLE VI
Main Effect of Moisture Content (MC) on Polylactic Acid (PLA)
and Starch Foam Mechanical Properties^a

MC (% db)	Compressibility (N)	Spring Index (%)
19	215.1a	86.6a
22	274.5a	85.1a
25	526.5b	83.9a

^a Values followed by the same letter are not significantly different ($P < 0.05$).

similar unit and bulk densities, bulk compressibilities, and bulk spring indices. Increasing the PLA polymer content improved the radial expansion ratios and spring indices and reduced the bulk densities and bulk compressibilities significantly. Changes in PLA content had no significant effects on unit densities, WSI, compressibilities, and bulk spring indices. Increasing moisture content adversely affected foam characteristics. The formulation containing waxy starch, 40% PLA, and 19% moisture content produced a loose-fill foam of the best physical characteristics and mechanical properties. The PLA and starch foam looks promising as a loose-fill packaging material to replace the petrochemical-based products.

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