Mechanical and Physical Properties of Protein-Starch Based Plastics Produced by Extrusion and Injection Molding

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ABSTRACT: Processing conditions, ingredient ratio, and moisture content were optimized for making soy protein/starch based plastics using a twin-screw extruder and an injection molding machine. A metering pump and a high speed mixer were used for ingredient mixing. The optimal processing temperature for injection molding was 130°C, and the moisture content of extruded pellets was 10–14%. Processing effects were investigated by measuring the tensile properties and water resistance of specimens. Reduction of water and glycerol in mixtures increased the barrel pressure of the extruder. Mold release was improved by incorporating 0.25 parts tallow per 100 parts of solid material (soy protein and starch). The water absorption of the specimens was reduced by adding acids to adjust the pH to the isoelectric point of soy proteins (pH 4.5). Effects of storage at different relative humidities were studied. The processibility of pellets was stable after a 4-wk-storage period, despite some moisture loss. Injection-molded specimens, after being stored for up to 6 mon at dry conditions [50 and 11% relative humidity (RH)] at room temperature and for 4 wk in a 50°C oven, showed no surface crack. However, humid (93% RH) storage at room temperature promoted fungal growth after storage for 3 mon, indicating that preservatives such as potassium sorbate and propionic acid were needed.

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KEY WORDS: Extrusion, glycerol, injection molding, mold releasing, proteins, starches, tallow, tensile strength, water absorption.

Soy protein has been widely used in foods to provide flavor, texture, and other functional properties (1). Soy protein also has been used in many industrial products, such as adhesives for wood and paper, emulsifiers in colloidal rubber products, and binders in coatings and paints (2–6). Corn starch is one of the most abundant, inexpensive, and widely used food and industrial ingredients. Normal, waxy, and high-amylose corn starches are often chemically modified to improve the viscosity, texture, and stability of pastes for coating, binding, and sizing applications (7).

New and increasingly tightened environmental regulations from the United States Environmental Protection Agency and concerns about environmental pollution have increased demand for biodegradable plastics made from renewable agricultural products. Previous research conducted in our laboratory demonstrated feasibility of compression (8–10) and injection (11,12) molding of plastics made from soy protein, corn starch, zein, and other biomaterials, and their biodegradability in marine and soil environments (13). Effects of crosslinking chemicals and cellulose fibers (9), preparation and processing conditions (8,11), and polyhydric alcohols as plasticizers (14) on mechanical properties and water resistance of soy protein-based plastics have been reported.

Extrusion technology is effective and efficient and has been widely used to process polymers and biopolymers, like proteins and starches, for various applications (15–17). Injection molding is the most popular processing method used by the plastics industry. Objectives of this study were (i) to investigate extrusion and injection-molding processes of soy protein/starch mixtures, and (ii) to study the relationships between tensile properties, water resistance, and storage stability of injection-molded specimens with biomaterial.

EXPERIMENTAL PROCEDURES

Materials. Soy protein isolate (Supro 760) was purchased from Protein Technologies International (St. Louis, MO). Acetylated high-amylose corn starch (50% high-amylose) was purchased from Cerestar, USA, Inc. (Hammond, IN). Tallow (bleachable fancy tallow, ET-8715) was a gift from Chemol Company (Greensboro, NC). Titanium dioxide was purchased from Johnson Matthey (Ward Hill, MA), glycerol from Fisher Scientific Co. (Pittsburgh , PA), and potassium sorbate from Sigma Chemical Co. (St. Louis, MO). All chemicals not otherwise noted were American Chemical Society grade.

Moisture determination. Moisture contents of ingredient mixtures were determined by using a Sartorius MA 30 moisture analyzer (Bohemia, NY) at 110°C for 6 min. Moisture contents of extruded pellets were determined at 120°C for 25 min.

Ingredient mixing. Protein and starch were mixed in a high-speed Thyssen Henschel mixer (model FM10; Houston, TX) at a mixing speed of 860 rpm. While mixing, a glycerol/water solution was added by using a Masterflex pump (Cole Parmer Instruments, Chicago, IL) at speed #7. Melted

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tallow was added manually. In a typical experiment, a solution of 0.9 kg of glycerol (30 parts per 100 parts of protein/starch mixture) and a calculated amount of water was added to a mixture of 0.51 kg each of soy protein and acetylated high-amylose corn starch to give a final moisture content of 11.5%. The mixtures were allowed to equilibrate overnight before extrusion.

Extrusion. The equilibrated mixtures were extruded using an extruder equipped with a set of co-rotating twin screws (Leistritz Micro 18, Somerville, NJ). Temperatures in the barrel were set at 75, 85, 95, 100, and 105°C for zones from input to output, and the die was set at 95°C. The screw speed was set at 100 rpm. An accurate feeder (Leistritz) was used to transfer mixtures to the extruder at speeds ranging from 11 to 20 rpm. A lab pelletizer (C.W. Brabender, South Hackensack, NJ) was used to pelletize extrudates.

Injection molding. The pellets were injection-molded into Type I ASTM tensile specimens by using a Boy-22S Dipronic injection-molding machine (Boy Machines Inc., Exton, PA). To study temperature effects on the properties of injection-molded specimens, seven temperatures, from 80 to 140°C in 10°C increments, were tested in the metering zone of the barrel. The temperature of the transition zone was set at 10°C below that of the metering zone. For other studies, the metering zone temperature of the molding machine was set at 130°C and the transition zone temperature at 120°C.

Tensile properties and storage effects. Tensile strength, percentage elongation, and Young's modulus of injection-molded tensile bar specimens were analyzed according to the ASTM D 638-86 method (8,18). For each treatment, five or more specimens were tested at 5 mm/min crosshead speed in an Instron machine (Instron model 4502; Canton, MA). The prepared specimens were stored in a closed chamber equilibrated with selected saturated salt solutions under dry (11% RH, lithium chloride), humid (93% RH, potassium nitrate), or medium humidity (50% RH, calcium nitrate) conditions for 7 d to 6 mon. Injection-molded tensile specimens also were stored at 50°C in an oven for up to 4 wk. Weight changes were recorded after storage.

Water absorption. A modified ASTM standard test method D570-81 using tensile bar specimens was adopted for water absorption tests (8,19). Three specimens for each treatment were used for the tests. The specimens, having the same surface area/volume ratio, were dried in an oven (50°C for 24 h), cooled in a desiccator, and immediately weighed. The conditioned specimens were submerged in distilled water at 25°C. After 24 h, they were removed, the surface moisture was wiped with a paper towel, and specimens were then immediately weighed. Water absorption was calculated as a percentage of initial weight. The specimens were checked for soluble matter loss during submersion by weighing them after drying in an oven for 24 h at 50°C. If any loss of water-soluble matter occurred, it was added to the water absorption calculation.

RESULTS AND DISCUSSION

Amylose, the primarily linear molecule of starch, is known to produce films of high tensile strength. High-amylose corn starch, which consists of 50% or more amylose, however, is difficult to process because of its very high gelatinization temperature (71–113°C). Acetylation is known to reduce starch gelatinization temperature and thus facilitate the processing of high-amylose corn starch (20,21). In experiments not reported here, comparison of specimens made by extrusion and injection-molding of soy protein blended with native normal corn starch and with acetylated high-amylose corn starch (50:50 parts, with 30 parts of glycerol) showed tensile strength at break of 3.0 and 3.7 MPa, respectively. The specimens made from soy protein/normal corn starch displayed a larger water absorption (132%) than those made from soy protein/acetylated high-amylose corn starch (88%). Because of the superior properties, we selected acetylated high-amylose corn starch for this study.

Effects of injection-molding temperatures. Soy protein/acetylated high-amylose corn starch (50:50 parts) in the presence of 30 parts glycerol and a 11.5% moisture content was injection-molded at seven different temperatures. Starting at 80°C, the temperature was increased in 10° increments to 140°C. The tensile strength of molded specimens increased as the injection-molding temperature increased, up to 130°C (Table 1). The specimens exhibited the greatest tensile strength (3.9 MPa) at a molding temperature of 130°C. This

TABLE 1
Effects of Injection-Molding Temperatures on the Properties of Protein/Starch Specimens[a]

Temperature[b] (°C)	Tensile strength at break (MPa)	Elongation at break (%)	Young's modulus (MPa)	Water absorption (%)
80	2.9 ± 0.1	89.5 ± 3.7	29 ± 3	102 ± 2
90	2.9 ± 0.1	94.7 ± 6.7	28 ± 2	104 ± 2
100	2.9 ± 0.1	94.5 ± 9.1	29 ± 1	100 ± 2
110	3.1 ± 0.1	69.4 ± 12.9	36 ± 2	91 ± 1
120	3.2 ± 0.1	74.1 ± 14.1	34 ± 1	89 ± 2
130	3.9 ± 0.1	85.1 ± 4.2	46 ± 5	86 ± 1
140	3.7 ± 0.1	74.5 ± 19.0	46 ± 4	88 ± 1

[a]The specimens were made from soy protein/acetylated high-amylose corn starch (50:50 in parts) with 30 parts of glycerol at pellet moisture of 11.5% under various injection-molding temperatures.
[b]Metering zone temperatures are shown; transition zone temperatures were 10°C lower.

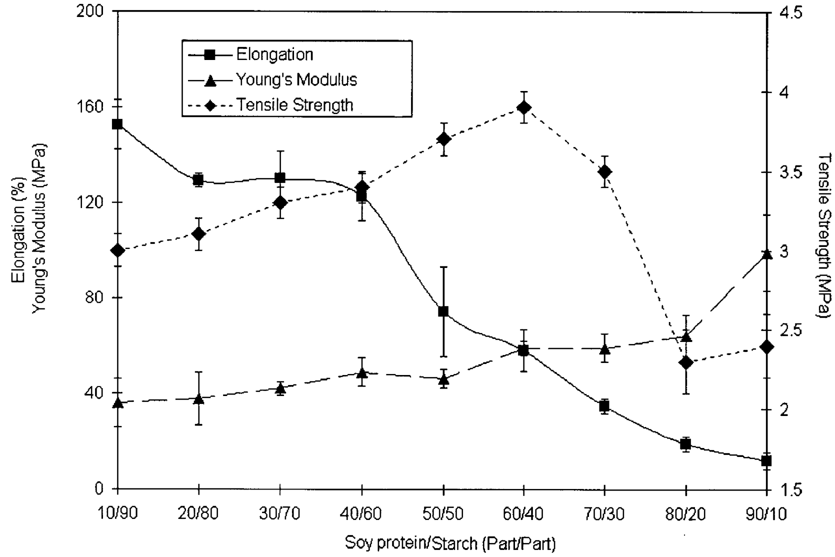

FIG. 1. Mechanical properties of specimens made from various ratios of soy protein/acetylated high-amylose corn starch by weight with 30 parts of glycerol per 100 parts of protein/starch mixture. Bars are standard deviations; *n* ≥ *5*.

could be attributed to the fact that soy protein was fully denatured and polymeric interaction between molecules was enhanced. Specimens were dark and displayed a decreased tensile strength (3.7 Mpa) at a molding temperature of 140°C, indicating the beginning of thermal degradation of soy protein and starch. Temperatures above 150°C led to thermal degradation of the specimens during injection molding and failed to produce adequate specimens.

Effects of different ratios of soy protein and acetylated high-amylose corn starch. Specimens were made with different formulations using various contents of soy protein and acetylated high-amylose corn starch at part ratios of 10:90, 20:80, 30:70, 40:60, 50:50, 60:40, 70:30, 80:20, and 90:10 by weight, with 30 parts of glycerol per 100 parts of protein/starch mixture. Properties of the specimens as analyzed are shown in Figure 1. The graph shows typical behaviors of blending two polymers, one flexible (starch) and another stiff and brittle (soy protein). The percentage elongation at break decreased, yet the modulus increased when the part ratio of soy protein increased. The tensile strength increased with the increase of soy protein content up to 60 parts but decreased when the soy protein content exceeded 60 parts. The result suggested that when gelatinized starch was the major phase (e.g., 10:90 = soy protein/starch), the plastic was flexible, as indicated by a large percentage elongation at break (153%). When the soy protein increased and became the major phase (e.g., 90:10 = soy protein/starch) the plastic became stiff and displayed a very low percentage elongation at break (11.9%). The blends displayed the largest tensile strength at 60:40 = soy protein/starch. To obtain specimens with relatively good tensile strength and percentage elongation, we selected the 50:50 ratio of protein and starch as the basic formulation for this study.

Effects of pellet moisture and glycerol on tensile properties. Effects of pellet moisture and glycerol content are shown in Figures 2 and 3, respectively. Reduction of moisture and glycerol in the mixtures increased the barrel pressure during extrusion. Specimen tensile strength and modulus also increased when moisture and glycerol contents decreased. The total water absorption decreased from 106 ± 2 to 88 ± 1% when the glycerol content increased from 10 to 30 parts per 100 parts of protein/starch mixture (Table 2). The total water absorption was determined by summing two parts, the water intake during 24 h immersion and the soluble loss from the specimens. Table 2 shows that the specimens of higher glycerol content had higher soluble loss, 25.6 ± 0.1% of specimens with 30 parts glycerol vs. 17.1 ± 1.4% of specimens with 10 parts of glycerol. However, water intake decreased to a larger extent as glycerol content increased so that the total water absorption decreased as glycerol content increased.

Effects of tallow on mold release and tensile properties. Mold release of injection-molded specimens was significantly improved by adding beef tallow. When tallow content was greater than 1 part per 100 parts of protein/starch mixture, however, the material could not be injected because of over lubrication. Results showed that tallow (0.25 part) in soy protein/starch (100 parts) mixtures improved mold release. Mold release was also improved by applying external lubricants such as silicone, lecithin, and tallow on the die surfaces. Tallow was a better external lubricant than silicone and lecithin for mold release; however, tallow decreased tensile strength and elongation (Fig. 4).

Effects of storage on the processing ability of pellets. Pellets composed of 20 parts glycerol and 0.25 parts tallow were tested over 4 wk of storage, and results showed good stability with no effects on the processibility, except for a slight moisture loss, from 8.5% at the beginning to 7.6% at the end of the fourth week (Table 3). The moisture loss resulted in increase of tensile strength and water absorption.

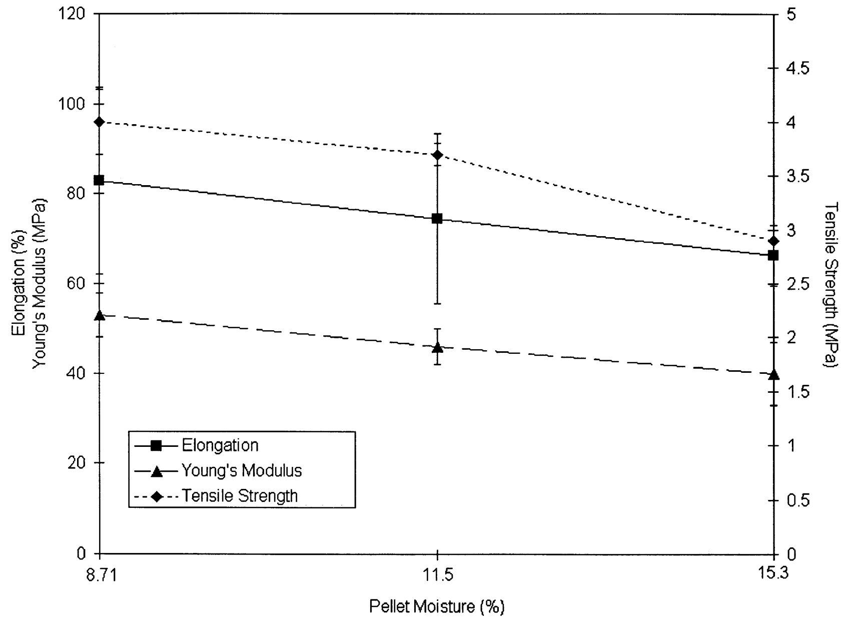

FIG. 2. Effects of pellet moisture content on the tensile properties of specimens made from soy protein and acetylated high-amylose corn starch (50:50) with 30 parts of glycerol per 100 parts of protein/starch mixture. Bars are standard deviations; $n \geq 5$.

Shelf life storage test. Commercial products normally require a 6-mon shelf life. The storage tests of the injection-molded specimens were conducted in two ways: (i) stored at 11, 50, and 93% RH for 6 mon, (ii) stored in an oven at 50°C for 4 wk. Specimen weights decreased after storage at 11 and 50% RH and increased at 93% RH as a result of moisture equilibration. The tensile strength of specimens significantly increased from 11 to 38 MPa and from 5 to 16 MPa after being stored at 11 and 50% RH for 6 mon, respectively. The specimens stored at 93% RH showed fungal growth after 3 mon (Table 4).

Chemicals such as titanium dioxide and potassium sorbate are commonly added to improve the shelf life of commercial products against physical and biological degradation. Titanium dioxide is a white pigment and also is an antioxidant that prevents tallow oxidation during thermal processing (22). Potassium sorbate extends shelf life in humid conditions by inhibiting mold and yeast growth (22). In an accelerated shelf life storage test at 50°C for 4 wk, all specimens showed no apparent cracking at the end of the storage period (Tables 5 and 6). Specimen weights significantly decreased over the first week and decreased continuously during 50°C storage.

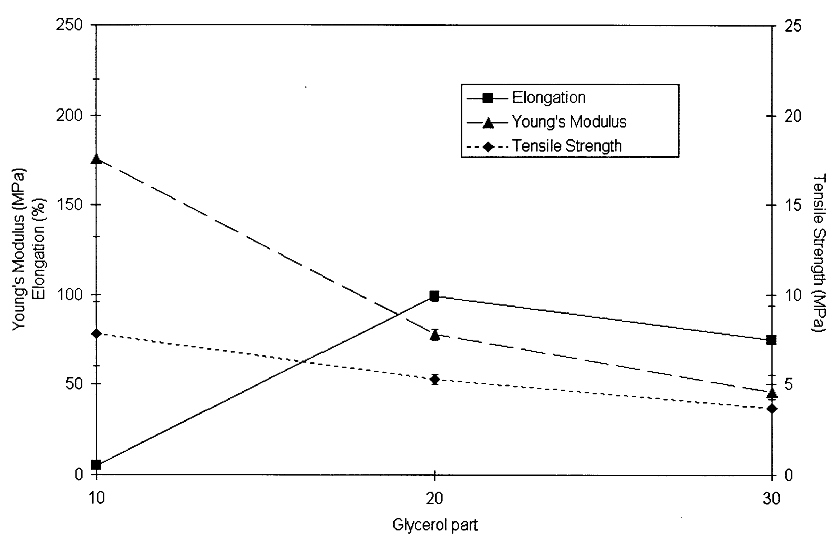

FIG. 3. Effects of glycerol content on the tensile properties of soy protein/acetylated high amylose corn starch mixtures (50:50 in parts) having pellet moisture content of 11% with 50% relative humidity storage. Bars are standard deviations; $n \geq 5$.

TABLE 2
Effects of Glycerol Content on Water Absorption Properties[a]

Part of glycerol[b]	Water Absorption (%)	Soluble material loss (%)	Total water absorption (%)
10	89.1 ± 0.9	17.1 ± 1.4	106 ± 2
20	73.4 ± 0.7	19.3 ± 0.5	93 ± 1
30	61.9 ± 0.8	25.6 ± 0.1	88 ± 1

[a]The specimens were made from soy protein/acetylated high-amylose corn starch (50:50 in parts) with various parts of glycerol at pellet moisture of 11.5% at 130°C injection molding temperatures.
[b]Per 100 parts of protein/starch mixtures.

The increase in tensile strength and decrease in elongation resulted from moisture loss during storage.

Effects of sodium acidic salt buffers and acids on water absorption. Sodium acidic salt buffer solutions (0.1 M, pH 4.5), such as acetate, phosphate, citrate, and propionate, were used to replace 25 parts water to adjust the pH to 4.5, i.e., the isoelectric point (pI) of soy protein. At its pI, soy protein has the minimum net charge and is the most water resistant. However, Table 7 showed that 0.1 M (pH 4.5) sodium acidic salt buffer solutions did not decrease water absorption, which could be the result of the high concentration of sodium salts. Acids also were added to soy protein/starch mixture to reduce the water absorption by adjusting the pH to 4.5. The water absorption of the molded specimens with 2 parts of the acid was *ca.* 83%, compared with 99% of the control samples (Table 8). The limited reduction in water absorption could be attributed to the presence of starch; water absorption of starch was not affected by the pH change. The water absorption of specimens with 1 part acid hovered between *ca.* 86 and 94%. The tensile strengths of the acid-treated specimens slightly increased at 50% RH conditions.

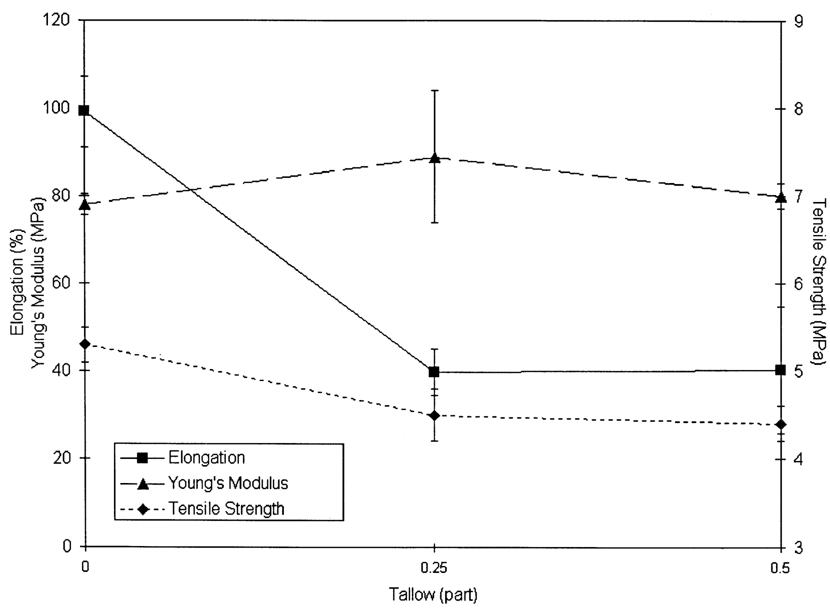

FIG. 4. Effects of tallow contents on the tensile properties of soy protein/acetylated high-amylose corn starch mixtures (50:50 in parts) with 20 parts of glycerol. Bars are standard deviations; $n \geq 5$.

TABLE 3
Effects of Pellet Storage on Tensile Properties of Specimens[a]

Storage period (wk)[b]	Pellet moisture (%)	Tensile strength at break (MPa)	Elongation at break (%)	Young's modulus (MPa)	Water absorption (%)
0	8.5	5.0 ± 0.1	35.5 ± 7.2	101 ± 3	96 ± 1
1	7.8	5.8 ± 0.5	32.2 ± 13.9	112 ± 8	103 ± 1
2	7.7	6.0 ± 0.3	42.3 ± 16.0	116 ± 4	105 ± 2
3	8.0	7.7 ± 0.5	32.1 ± 7.5	109 ± 7	104 ± 1
4	7.6	7.0 ± 0.6	54.9 ± 8.5	113 ± 13	103 ± 2

[a]The specimens composed of soy protein/acetylated high-amylose cornstarch (50:50 in parts) with 20 parts of glycerol and 0.25 part tallow. Injection molding temperature was 130°C.
[b]Stored at room temperature.

TABLE 4
Effects of Storage at Room Temperature on Properties of Specimens[a]

Storage period	Storage (RH%)	Tensile strength at break (MPa)	Elongation at break (%)	Young's modulus (MPa)	Water absorption (%)
7 d	11	11.3 ± 1.1	5.1 ± 0.7	216 ± 15	99 ± 2
7	50	5.4 ± 0.5	48.6 ± 6.7	101 ± 9	99 ± 2
7	93	2.9 ± 0.3	56.5 ± 15.2	50 ± 10	99 ± 2
1 mon	11	24.1 ± 1.5	5.0 ± 0.4	454 ± 25	99 ± 3
1	50	11.5 ± 0.6	17.4 ± 3.7	172 ± 16	99 ± 3
1	93	2.4 ± 0.2	30.2 ± 8.7	59 ± 19	99 ± 3
2 mon	11	35.2 ± 1.4	6.8 ± 0.2	543 ± 28	99 ± 1
2	50	14.1 ± 0.2	9.3 ± 0.6	241 ± 10	99 ± 1
2	93	2.4 ± 0.1	43.4 ± 2.7	36 ± 6	99 ± 1
3 mon	11	32.3 ± 3.6	5.0 ± 0.5	659 ± 49	98 ± 2
3	50	15.2 ± 0.4	7.6 ± 0.7	258 ± 11	98 ± 2
3	93	2.0 ± 0.1	35.2 ± 5.3	26 ± 67	98 ± 2
4 mon	11	32.1 ± 1.9	4.6 ± 0.7	697 ± 23	98 ± 1
4	50	14.6 ± 0.5	7.7 ± 1.1	256 ± 15	98 ± 1
4	93	1.7 ± 0.2	26.4 ± 7.4	46 ± 12	98 ± 1
5 mon	11	35.5 ± 8.1	5.1 ± 1.4	730 ± 99	99 ± 1
5	50	16.0 ± 0.3	7.9 ± 0.5	274 ± 6	99 ± 1
5	93	NA			
6 mon	11	37.6 ± 5.1	5.3 ± 0.6	723 ± 48	98 ± 1
6	50	16.3 ± 0.3	7.5 ± 0.5	294 ± 13	98 ± 1
6	93	NA			

[a]The specimens were made from soy protein/acetylated high-amylose cornstarch (50:50 in parts) materials with 20 parts of glycerol, 0.25 part tallow. The injection molding temperature was 130°C and the pellet moisture was 9.2%. RH, relative humidity; NA, not applicable.

TABLE 5
Effects of Storage at 50°C on the Properties of Specimens with Additives[a]

Storage period week	Tensile strength at break (MPa)	Elongation at break (%)	Young's modulus (MPa)	Water absorption (%)
0[b]	3.9 ± 0.2	87.6 ± 9.3	72 ± 11	100 ± 2
1	15.7 ± 3.6	3.3 ± 1.1	483 ± 68	100 ± 1
2	22.6 ± 7.3	4.0 ± 1.3	542 ± 15	99 ± 2
3	26.1 ± 3.5	3.6 ± 0.4	687 ± 23	100 ± 1
4	34.7 ± 4.6	5.7 ± 0.7	608 ± 26	99 ± 2

[a]Specimens were made from soy protein/acetylated high-amylose corn starch (50:50 in parts) with 20 parts of glycerol, 0.25 part tallow, 1 part titanium dioxide, and 0.25 part potassium sorbate. The injection molding temperature was 130°C, and the moisture content of the pellets was 8.8%.
[b]The injection-molded control samples were equilibrated in a chamber of RH 50% for 48 h before testing in tension. For abbreviation see Table 4.

TABLE 6
Effects of Storage at 50°C on the Properties of Specimens without Additives[a]

Storage period week	Tensile strength at break (MPa)	Elongation at break (%)	Young's modulus (MPa)	Water absorption (%)
0[b]	5.4 ± 0.5	48.6 ± 6.7	101 ± 9	99 ± 2
1	21.9 ± 3.0	4.8 ± 0.6	485 ± 30	99 ± 2
2	23.9 ± 3.7	3.8 ± 0.8	633 ± 97	99 ± 1
3	25.3 ± 4.4	3.5 ± 0.4	723 ± 65	98 ± 1
4	23.7 ± 2.6	3.2 ± 0.4	747 ± 26	100 ± 1

[a]Specimens were made from soy protein/acetylated high-amylose corn starch (50:50 in parts) with 20 parts of glycerol, and 0.25 part tallow. The injection molding temperature was 130°C, and the moisture content of the pellets was 9.2%.
[b]The injection-molded control samples were equilibrated in a chamber of RH 50% for 48 h before testing in tension. For abbreviation see Table 4.

TABLE 7
Effects of Sodium Buffer Solutions on Specimen Properties [a]

25-part solution	Pellet moisture (%)	Storage (RH%)	Tensile strength at break (MPa)	Elongation at break (%)	Young's modulus (MPa)	Water absorption (%)
Water	9.2	50	5.4 ± 0.5	48.6 ± 6.7	101.3 ± 9.0	99 ± 2
Acetate	10.0	50	5.5 ± 0.2	42.8 ± 7.2	109.0 ± 2.9	104 ± 1
Phosphate	11.2	50	5.5 ± 0.2	55.4 ± 16.2	108.9 ± 7.7	105 ± 1
Citrate	10.4	50	5.5 ± 0.5	59.0 ± 19.6	105.1 ± 7.4	107 ± 1
Propionate	11.0	50	5.7 ± 0.2	69.5 ± 5.1	102.6 ± 10.5	105 ± 3

[a]Specimens were made from soy protein/acetylated high-amylose corn starch (50:50 in parts) with 20 parts of glycerol and 0.25 part tallow, using 25 parts of various sodium buffer solutions at 0.1 M and pH 4.5 to replace water. Injection temperature was 130°C. For abbreviation see Table 4.

TABLE 8
Effects of Replacing Water by Various Acids on Properties of Specimens[a]

Solution	Pellet moisture (%)	Storage (RH%)	Tensile strength at breaka (MPa)	Elongation at break (%)	Young's modulus (MPa)	Water absorption (%)
Water	9.2	50	5.4 ± 0.5	48.6 ± 6.7	101 ± 9	99 ± 2
Added 1 part of acid						
Acetic	11.9	50	5.7 ± 0.1	69.5 ± 10.6	96 ± 15	86 ± 2
Phosphoric	11.1	50	5.7 ± 0.4	58.6 ± 19.8	101 ± 10	90 ± 3
Citric	10.2	50	5.9 ± 0.2	40.2 ± 3.6	111 ± 6	94 ± 0
Propionic	10.3	50	5.4 ± 0.2	49.2 ± 11.0	104 ± 6	92 ± 2
Added 2 parts of acid						
Acetic	11.6	50	6.0 ± 0.1	50.9 ± 8.0	112 ± 11	83 ± 1
Phosphoric	10.5	50	6.4 ± 0.3	59.6 ± 10.9	119 ± 5	84 ± 2
Citric	9.2	50	6.8 ± 0.3	39.6 ± 13.5	130 ± 4	84 ± 3
Propionic	9.8	50	6.0 ± 0.4	46.8 ± 13.0	124 ± 6	83 ± 0

[a]Specimens were made from soy protein/acetylated high-amylose corn starch (50:50 in parts) with 20 parts of glycerol and 0.25 part tallow, and 1 and 2 parts of various acids. Injection-molding temperature was 130°C.

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