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Effects of Different Plasticizers on Some Properties of Biodegradable Starch-based Foams

Jea Yoon Cha
Member

ABSTRACT

전분 완충재의 물리적, 기계적 특성을 개선하기 위한 노력들이 많이 이루어지고 있지만 기존의 플라스틱 완충재와 비교하면 더 많은 노력이 요구되어진다. 그래서 본 연구에서는 전분 완충재의 물리적 기계적 특성에 대한 유연제(plasticizers)와 첨가제(additives)의 영향을 규명하였다. 전분과 합성수지 그리고 여러 유연제 또는 첨가제를 혼합한 뒤 Brabender사의 일축 압출기를 이용하여 여러 축속도(60~120 rpm)에서 압출 가공한 후 물리적, 기계적 특성을 조사하였다. 전분 완충재의 밀도(bulk density)와 압축성(bulk compressibility)은 혼합물에서 물의 비율이 증가함에 따라 증가하였지만, 복원성(bulk resiliency)은 조금 감소하였다. 측정되어진 전분 완충재의 특성은 변형전분(hydroxypropylated starch)의 사용으로 향상되었다. 또한, glycerol, glycerol monostearate 그리고 alkylglucosides의 첨가도 전분 완충재의 밀도와 압축성을 증가시켰다. 염화철(II) 0.5%을 포함한 전분 완충재는 첨가제를 포함하지 않은 전분 완충재에 비하여 밀도 42%와 압축성 58%가 각각 감소한 반면, 복원성은 13% 포인트 증가하였다. 변형전분과 염화철(II)이 첨가된 전분 완충재는 기존의 플라스틱 완충재에 비하여 밀도와 압축성은 떨어지지만, 대체 사용되어질 수 있는 것으로 판단되었다.

Keywords : Biodegradable starch-based foam, Properties, Plasticizer, Surfactant.

1. Introduction

Huge quantities of such plastic products as refuse and retail bags, fast-food containers, egg cartons, packaging films, and loose fillers are used. For example, about 23,000 tons of expandable polystyrene loose fillers were consumed in 1988 (Frey and Star, 1988). Disposal of used plastic products from petroleum sources has become a public concern because of their nondegradability. Much effort has been put forth to produce environmentally friendly alternatives to plastic products because of the widespread concern over their long-term survival in landfills and the potential for toxic by-products from their incineration. Starches are probably the most abundant and lowest cost natural polymers commercially available. The use of starch in plastic production would greatly reduce the demand for petroleum as well as the negative impact on the environment caused by discarding nonbiodegradable materials. It is because starch easily degrades into small molecules that can be metabolized by microorganisms such as bacteria, yeast, and fungi.

Extrusion processing technology in conjunction with normal or modified starches has been used since the late 1980s to develop starch-based plastic foams as alternatives to expanded polystyrene loose fillers. Lacourse and Altieri (1989), Sachetto et al. (1991), Silbiger et al. (1991), Bastioli et al. (1991), and Neumann and Seib (1993) patented the technology to make biodegradable starch-based foams. A starch-based foam prepared from the mixture of 95%

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hydroxypropylated high amylose corn starch and 5% polyvinyl alcohol is used commercially as an alternative to polystyrene loose fill. The physical and mechanical properties of starch-based foams from various mixtures have been studied by Moore et al. (1990), Warburton et al. (1990 and 1992), Lourdin et al. (1995), Bhatnagar and Hanna (1995), Wang et al. (1995), Cha et al. (1999), and Cha et al. (2001). The power law correlation between mechanical properties and bulk density of starch-based foams was found by impact, compression, tension, and/or flexure tests (Hutchinson et al., 1987; Warburton et al., 1992; Cha et al., 1999). Cha et al. (2001) reported the power law correlation among bulk density, extrusion temperature, and moisture content. Water is an effective plasticizer that plays an important role in starch gelatinization. It is known that the expansion ratio of starch depends mainly on its degree of gelatinization (Chinnaswamy and Bhattacharya, 1984), which in turn is determined by the temperature and shear rate during extrusion and the moisture content of feed materials.

Much efforts has been put forth to improve the physical and mechanical properties of starch-based foams because an increase in starch levels decreases their density, compressibility, flexibility, and elasticity. Still, starch-based plastic foams have poor physical and mechanical properties compared to pure plastic foams. In this study, starch-ethylene vinyl alcohol copolymer (EVOH) formulations were used to produce starch-based foams. The specific objectives were to (1) study the effects of different types of plasticizers and additives on the physical and mechanical properties of a starch-EVOH composite foam, and (2) evaluate their properties by comparing those of polystyrene foams and commercial starch packaging foams.

2. Materials and Methods

A. Materials

Hydroxypropylated (HP, 4.9%) wheat starch (Midsol 40 (12% w.b.)) was obtained from Midwest Grain Products, Inc. (Atchison, KS). High amylose corn starch (Amylomaize VII (11% w.b.)) containing 70% amylose and HP (4.6%) corn starch (Amylomaize 2370 (11% w.b.)) containing 50% amylose were obtained from American Maize Products Co. (Hammond, IN). Both starches were in their non-gelatinized forms. Pelleted ($Ø$=2.0 mm, and l=5 mm) poly(ethylene-co-vinyl alcohol) (EVOH) containing 38 mol % ethylene was EVOH3803 from Eval Company of America (Lisle, IL). The EVOH was reported to be a random copolymer by the manufacturer. Safoam FPT, a chemical nucleating and blowing agent, is a proprietary mixture that consists of encapsulated sodium salts of carbonic and polycarboxylic acids from Reedy International Corporation (Keyport, NJ). Methanol was obtained from Fisher Scientific (Fair Lawn, NJ). Silicon dioxide (Sipernat 22) from Degussa Corporation (Teterboro, NJ) was used. Alkylglucosides (Glucopon 220 (C_8-C_{12}) and Glucopon 600 (C_{10}-C_{16})) were obtained from Henkel Corp. (Hoboken, NJ). Glycerol monostearate (GMS) was obtained from Grinsted Products, Inc. (Industrial Airport, KS), and other chemicals were reagent grade from Fisher Scientific (Fair Lawn, NJ). The commercial starch packaging foam (Eco-foam) was obtained from American Excelerior (Arlington, TX). The commercial polystyrene foam (Styrofoam) manufactured by Dow Chemical Company (Midland, MI) was obtained from National Bag Company (Hudson, OH).

B. Methods

(1) Mixing procedure

Methanol (5.5%) and water (5.5%) were mixed in a beaker with a glass stirring rod. The premixed liquid ingredients were slowly blended with wheat starch (35%), corn starch (35%), and EVOH 3803 (17%) in a dough mixer (model KSM5, Kitchen Aid Co., St. Joseph, MI) with a wire-whip. During mixing, each blend was scraped and stirred every 2 min. Then, each wetted starch-polymer blend was transferred to a tumbler (model 3M127A, Dayton Electric MFG. Co., Chicago, IL) containing Safoam FPT (1.5%), silicon dioxide (0.5%), and other optional dry ingredients (plasticizers or additives). The contents were tumbled

together for 10 min to yield a moistened blend. The amount of water added into the formula varied from 2.5 to 10%, while the levels of the starches were adjusted so that the sum of all ingredients remained at 100 parts. Different plasticizers and other additives were added into the above blends by several percentages. After mixing, each blend was packed into a sealed vinyl bag and kept for 3 h at 25℃ for moisture equilibration.

(2) Extrusion procedure

The moistened blends were extruded into foamy collets using a laboratory-scale extruder (Model 2503, C. W. Brabender, New Hackensack, NJ) equipped with a 2.4 mm (3/32 in.) cylindrical die and a 19 mm (3/4 in.) diameter single screw with a compression ratio of 5:1. The extruder consisted of a 19 mm inside diameter barrel with a 25:1 barrel length to inside diameter ratio. The temperatures of melted blends at three heating zones from the feed end to the melt end of the extruder barrel were set at 120, 170, and 130℃. Thermocouple probes (type SERP, OMEGA Engineering Inc., Stamford, CT) were flush-mounted on the extruder barrel. The temperature of melted blends in the extruder can be measured accurately by contacting the thermocouple probes with the melted blends. The temperature at each zone was set by an independent controller attached to and maintained automatically by the extruder heating and cooling system. Each blend was fed by hand at a rate sufficient to maintain a full- feed channel at the inlet. The flow rates of the blends were dependent on screw speed, which was varied from 60 to 120 rpm. Expanded collets (semi-rigid foams) were cooled and cut into packaging peanut shapes having a length of 30 mm. The starch-based foams were equilibrated at 60% relative humidity and 30℃ for 7 days. The moisture contents of the starch- based foams were measured after the foams had been dried at 130℃ for 24 h in an air oven following American Association of Cereal Chemists (AACC) method 44-15A (AACC, 1983). The moisture content of a commercial polystyrene foam (Styrofoam) was measured after the foams had been dried at 50℃ for 24 h in the air oven following American Society of Testing Materials (ASTM) method D570-81 (ASTM, 1992a).

(3) Bulk density

The bulk densities of the starch-based foams and polystyrene foam, equilibrated at 60% relative humidity and 30℃, were measured using a cylindrical plexiglass container of 126 mm diameter and 150 mm height inside dimensions, and a funnel having a 300 mm opening at the top and a 64 mm opening at the bottom following ASTM method D1895-65 (ASTM, 1992b). Each starch-based foam was poured into the funnel with the bottom end closed; then that end was opened, and the foam flowed into the container until it overflowed. The top of the container was struck off with a straightedge without shaking the container. The foam in the container was weighed, and the bulk density was calculated as the ratio of the mass to the internal volume of the container.

(4) Bulk compressibility

The bulk compressibility of a sample, defined as the maximum force required to compress the bulk sample to two-thirds of its original volume, was determined using an Instron Universal Testing Machine (model 4502) equipped with a personal computer. A random bulk sample of each foam was randomly loaded to fill the cylindrical container used to measure bulk density. The moving crosshead, on which the container was placed, was moved at 25 mm/min to compress the sample down to a height of 100 mm. The force required for compression was measured by the 1 kN load cell mounted on the fixed crosshead and continuously recorded at the data sampling rate of 25 values per minute into the computer. The bulk compressibility test for each bulk sample was duplicated five times at ambient condition.

(5) Bulk resiliency

Bulk resiliency refers to the degree that a sample is able to recover to its original volume after the determination of bulk compressibility. Following the initial bulk compressibility determination, the moving crosshead was returned to its original position and held there for 1 min before the next compression to the 100-mm height at the same crosshead speed. The volume recovery (resiliency) of the sample was calculated by subtracting the contact distance in the second compression cycle from the total displacement

distance (50 mm) of the first compression and dividing the difference by the total displacement distance. The result is expressed as a percentage. Five replicates for each bulk sample were conducted at ambient condition

(6) Experimental design

This study used a completely randomized design to evaluate the effects of moisture content, screw speed, emulsifiers, and divalent metals on selected properties of starch-based foams and to compare these properties of starch-based foams to those of commercial polystyrene foam. The response variables were bulk density, bulk compressibility, and bulk resiliency. A general linear model procedure was used for the analyses of variances and least significant difference (LSD). The Statistical Analysis System (SAS Institute, Cary, NC) was used for all statistical analysis. Means were compared by the LSD test at α=0.05 level.

3. Results and Discussion

A. Effect of moisture content

Starch-EVOH mixtures, which contained about 70% starch (Midsol 40 + Amylomaize VII), 17% EVOH, and 7.5% foam enhancers, were mixed with various amount of water at 2.5, 5.5, 7.5, and 10.5% of formula. The total moisture content of the feed material was then turned out to be from 10.5 to 17.5% considering the water originally contained in the starch. The mixtures were extruded at screw speed of 80 rpm. The mechanical properties of the foams are presented in Table 1. The bulk density of starch-based foams significantly increased from 27.8 to 46.8 kg/m^3 as the amount of water increased from 2.5 to 10.5%, and their bulk compressibility also significantly increased from 0.73 to 1.32 kN, whereas their bulk resiliency decreased from 41 to 38% ($P<0.05$). Increasing the total water above 13.5% decreased expansion of the starch-based foam because the extrudate partly collapsed after expansion, which was observed visually. Lower moisture content of blends might restrict the flow rate through the extruder barrel, increasing the effect of shear because of long residence time, which would increase the degree of starch gelatinization and expansion, whereas the higher viscosity of blend melts due to lower moisture content built up high pressure before die to result in greater expansion and lower bulk density. Also, the low water content in the foam, as it leaves the die, would decrease collapse due to high viscosity. The bulk densities of commercial polystyrene foam (Styrofoam) and starch packaging foam (Eco-foam) were 3.62 and 10.51 kg/m^3, respectively. The bulk compressibility and bulk resiliency of Styrofoam were 0.14 kN and 56.2%, respectively. It is evident that the starch-based foams were more dense and rigid than Eco-foam and Styrofoam.

B. Effect of Hydroxypropylation of Corn Starch

Hydroxypropylated corn starch (Amylomaize 2370) was used instead of unmodified corn starch (Amylomaize VII) in the formula containing 5.5% water to study the effect of hydroxypropylation on the properties of starch-based foams. The selected

Table 1 Properties of starch-based foams extruded from starch-EVOH blends with various water levels (screw speed : 80 rpm)

	Water Added (%)			
	2.5	5.5	7.5	10.5
Moisture content (%)	7.96 a	8.63 b	8.68 b	8.75 b
Bulk density (kg/m^3)	27.8 a	39.3 b	43.5 c	46.8 d
Bulk compressibility (kN)	0.73 a	0.85 b	1.07 c	1.32 d
Bulk resiliency (%)	41.0 a	37.0 b	37.4 b	38.0 b

* Values followed by the same letter in the same row are not significantly different ($P<0.05$).

properties of starch-based foam were significantly improved by using Amylomaize 2370 ($P<0.05$) (Table 2). The starch-based foam extruded from the combination of Midsol 40 and Amylomaize 2370 displayed significantly higher bulk resiliency (55%), lower bulk density (22.4 kg/m^3), and lower bulk compressibility (0.31 kN) ($P<0.05$). The improvement might be due to the improved compatibility between hydroxypropylated starch and EVOH. This result agreed with the findings in Cha et al. (1999), who reported the mechanical properties of starch-based foams by comparison between hydroxypropylated and native wheat starches. The hydroxypropylated starch has relatively low lipid content due to hydroxypropylation conducted under alkaline condition. The low lipid content in hydroxypropylated starch might be affected to perform better properties of starch-based foams compared to those with high lipid content in undefatted corn starch. In regards to the most desirable amylose levels in starches for foaming, Chinnaswamy and Hanna (1988a) reported that corn starch with 50% amylose content gave the greatest expansion ratio among the starches with various amylose levels.

C. Effect of screw speed

Starch (Midsol 40 + Amylomaize VII) - EVOH mixtures with 7.5% foam enhancers, were extruded at the screw speeds of 60 to 120 rpm, and the flow rates of 33 to 68 g/min (Table 3). The flow rate was mainly dependent on the screw speed, and minimally on the melting temperature. The bulk density and bulk compressibility of the starch-based foams significantly decreased as screw speed increased from 60 to 100 rpm, whereas those increased as screw speed increased from 100 to 120 rpm ($P<0.05$). The starch-based foams extruded with the flow rate of 60 g/min at screw speed of 100 rpm represented the best foam properties by its low bulk density (32.0 kg/m^3), low bulk compressibility (0.62 kN), and the highest bulk resiliency (44%). It is speculated that the higher bulk density, bulk compressibility and lower bulk resiliency on either side of the extrudates at 100 rpm, may be

Table 2 Properties of Eco-foam, Styrofoam, and starch-based foams extruded from starch-EVOH blends containing Amylomaize VII and Amylomaize 2370 (screw speed : 100 rpm)

	Eco-foam	Styrofoam	AM[a] VII	AM[a] 2370
Moisture content (%)	11.19 a	1.87 b	8.63 c	8.15 c
Bulk density (kg/m^3)	10.51 a	3.62 b	32.0 c	22.4 d
Bulk compressibility (kN)	0.07 a	0.14 b	0.62 c	0.31 d
Bulk resiliency (%)	60.5 a	56.2 b	44.0 c	55.0 b

*Values followed by the same letter in the same row are not significantly different ($P<0.05$).
[a] AM : Amylomaize.

Table 3 Properties of starch-based foams extruded from starch-EVOH blend with 5.5% water at different screw speeds

Screw speed (rpm)	Flow rate (g/min)	Bulk density (kg/m^3)	Bulk comp.[a] (kN)	Bulk res.[b] (%)
60	33.2	58.6 a	1.90 a	36.0 a
80	48.0	39.3 b	0.85 b	37.0 a
100	60.0	32.0 c	0.62 c	44.0 b
120	68.0	34.8 d	0.78 b	42.5 b

* *Values followed by the same letter in the same column are not significantly different* ($P<0.05$).
[a] Bulk comp.: Bulk compressibility. [b] Bulk res.: Bulk resiliency.

due to lower levels of gelatinization of starch at high screw speed and feed rate, or molecular degradation of starch at low screw speeds and feed rate since both would affect the residence time (Owusu-Ansah et al., 1983; Colonna et al., 1983).

D. Effect of plasticizer

Besides water levels in starch(Midsol 40 + Amylomaize VII)-EVOH blend, effects of several other plasticizers and surfactants on foam properties were examined. Overall, the bulk density, bulk compressibility, and bulk resiliency were negatively affected by the addition of glycerol, alkylglucosides (glucopon 220 and 600), and GMS in comparison with the starch-based foam that contained only water as a plasticizer (Table 4). The bulk density increased greatly with the higher level (10%) of glycerol although the bulk compressibility and resiliency were improved. However, the cell structure (size and uniformity) appeared to be improved because the plasticizer might reduce the strain on the cell wall and prevent bubbles from collapsing due to the increase of the plasticity of the cell wall. The addition of surfactants such as Glucopon 220 and 600 and GMS, significantly increased the bulk density and bulk compressibility of starch based foams ($P<0.05$). The surfactants are known to form helical complexes with amylose (Karkalas and Raphaelides, 1986; Czuchajowska, et al., 1995). The complex formation of amylose molecules and their alignment with EVOH molecules thus would be restricted.

E. Effects of other additives

The addition of urea gave large-sized cells evaluated visually and denser products. The bulk compressibility significantly decreased with the increase in urea level ($P<0.05$) (Table 5). The foam cells appeared to become coarser with the increase in urea level. Urea is considered a hydrogen bond breaking agent. Chinnaswamy and Hanna (1988b) reported that urea degraded the starch molecules. The molecular degradation of starch during extrusion was known to reduce the expansion ratio (Owusu-Ansah et al., 1983; Davidson et al., 1985).

Several other additives were also examined in an effort to improve the mechanical properties of the starch-EVOH composite foams. The iron salts were added to generate free radicals by way of a redox system, if any peroxide were present. Free radicals could create covalent bonds between starch and

Table 4. Properties of starch-based foams extruded from starch EVOH-blends with 5.5% water and various plasticizers (screw speed: 80 rpm)

Plasticizer	Content (%)	M.C.[a] (%)	Bulk density (kg/m^3)	Bulk comp.[b] (kN)	Bulk res.[c] (%)
Water	5.5	8.75 a	39.3 a	0.85 a	37.0 a
Glucopon 220	5	8.80 a	51.7 b	1.34 b	30.0 b
Glucopon 220	10	7.86 b	52.6 b	0.88 a	32.0 b
Glucopon 600	5	12.18 c	49.1 b	1.17 b	38.0 a
Glucopon 600	10	8.52 a	57.0 c	1.29 b	30.0 b
Glycerol	5	8.25 ab	49.2 b	0.83 a	40.0 a
Glycerol	10	8.64 a	78.0 d	0.30 c	52.0 c
GMS[d]	2	7.65 b	58.6 c	1.18 b	40.0 a
GMS[d]	4	8.50 a	43.5 e	0.96 a	37.0 a

* Values followed by the same letter in the same column are not significantly different ($P<0.05$).
[a] M.C.: Moisture content. [b] Bulk comp.: Bulk compressibility.
[c] Bulk res.: Bulk resiliency. [d] GMS: Glucose Monostearate.

Table 5. Properties of starch-based foams extruded from starch-EVOH blends with 5.5% water and various additives

Additives	Content (%)	Screw speed (rpm)	M.C.[a] (%)	Bulk density (kg/m^3)	Bulk comp.[b] (kN)	Bulk res.[c] (%)
Water	5.5	100	8.63 b	32.0 e	0.62 g	44.0 ce
$FeCl^2$	0.5	80	7.93 a	22.8 a	0.24 a	50.0 b
$FeCl^2$	1.0	80	8.52 b	27.0 b	0.27 a	46.0 cdf
ZnO	0.5	100	7.02 c	27.1 b	0.45 b	46.0 cdf
ZnO	1.0	100	7.84 a	36.5 c	0.87 c	47.5 d
MnO	0.5	100	8.08 a	30.7 d	0.53 d	46.5 df
MnO	1.0	100	8.42 b	37.0 c	0.95 e	43.0 e
Urea	1.0	80	8.26ab	32.8 e	0.76 f	45.0 cfg
Urea	5.0	80	8.05 a	39.5 c	0.59 g	37.0 a
Urea	10.0	80	7.27 c	64.0 f	0.40 h	55.0 h

* Values followed by the same letter in the same column are not significantly different ($P<0.05$).
[a] M.C.: Moisture content, [b] Bulk comp.: Bulk compressibility, [c] Bulk res.: Bulk resiliency.

EVOH. At the level of 0.5%, the ferrous chloride decreased the bulk density and compressibility to 22.8 kg/m^3 and 0.24 kN, respectively. However, zinc oxide and manganous oxide just caused slight improvement compared to the starch-based foam without additives. However, the cell size of foams with ferrous chloride appeared to become coarser and the cell wall thinner. Zinc oxide and manganous oxide are relatively insoluble in water, which might explain their ineffectiveness.

4. Conclusions

A starch-based foams for loose fill packaging can be produced from the blend containing only biodegradable materials. The bulk density and bulk compressibility significantly increased from 27.8 to 46.8 kg/m^3 and from 0.73 to 1.32 kN, respectively with the increase of water content from 2.5 to 10.5%. However, the bulk resiliency decreased from 41 to 38%. The starch-based foams extruded at 100 rpm represented the best foam properties by its low bulk density (32.0 kg/m^3), bulk compressibility (0.62 kN), and the highest bulk resiliency (44%). Hydro-xypropylation of corn starch contributed to improve the properties of starch-based foams; bulk density from 32.0 to 22.4 kg/m^3, bulk compressibility from 0.62 to 0.31 kN, and bulk resiliency from 44 to 55%. The addition of ferrous chloride significantly improved the mechanical properties of starch-based foams; bulk density from 32.0 to 22.8 kg/m^3, bulk compressibility from 0.62 to 0.24 kN, and bulk resiliency from 44 to 50%. The bulk compressibility of Eco-foam is needed to improve because it appears to be soft as a cushioning material. Starch-based foams were much denser than commercial polystyrene foam and Eco-foam; however, the starch-based foam from the blend with Amylomaize 2370 and ferrous chloride have similar bulk compressibility and bulk resiliency to those of commercial polystyrene foam. Therefore, they may be useful until such biode- gradable loose-fill materials having much improved mechanical properties, durability, and insensitivity to moisture and become available commercially.

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