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MIGRATION OF SOY METHYL-ESTER BASED PRODUCTS THROUGH BOTTLES

Ву

Paweena Limjaroen

Submitted to
Michigan State University
In partial fulfillment of the requirements
For the degree of

MASTER OF SCIENCE

School of Packaging

ABSTRACT

MIGRATION OF SOY METHYL-ESTER BASED PRODUCTS THROUGH BOTTLES

By

Paweena Limjaroen

Soy methyl-ester based products are a new category of products made from soybean oil. High-density polyethylene was used as a container for distributing these products, but migration of the product and package distortion (paneling) occurred. Then the container was changed to high-density polyethylene with high levels of fluorination. However, the cost of the container was high and it was not available in all the desired shapes. Also, migration and paneling sometimes still occurred.

In order to find a solution to these problems, six polymers, polypropylene (PP), polyvinyl chloride (PVC), polyethylene terephthalate (PET), glycol-modified PET (PET-G), polyacylonitrile (PAN), high-density polyethylene/nylon blend (SELAR) and polypropylene/ high density

polyethylene (PPAL), were tested for usefulness as containers without the migration and package distortion. High-density polyethylene and low-density polyethylene were included to be the baseline for the test. For some plastics, more than one type of the same polymer was included in the test. Three test methods were used: the migration test, the immersion test and the bubbling test. The migration test played an important role in selection of suitable containers, while the others were not helpful. Bean-e-doo, a product with a high concentration of methyl ester, and pure soy methyl ester from Franmar Chemical Inc. were used as the test samples. PET, PVC and PAN showed absolutely no migration of the products. However, some bottles of PVC and PAN showed paneling problems.

To my mother, Panadda Amornjarusiri.

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INTRODUCTION

Soy methyl ester-based products are a new category of products made from soybean oil. Biodiesel, solvent cleaner and printing ink are among the examples of them. For distribution of soy methyl ester-based products, the package is an important factor that should be considered. The appropriate packaging will properly deliver the product to the market so it reaches the consumers. containers now are the most interesting containers for containing a variety of products. Plastic containers are low cost for many reasons. For example, there is a lot of competition among the plastic manufacturers and many new methods for producing plastic continue to be developed by many plastic manufacturers. They can also be clear and transparent, which encourages the sales appeal of the packages. Also, plastic containers are lightweight when compared to glass or metal containers, and they resist breakage. As a result, plastic containers were considered to distribute soy methyl ester-based products. Generally, plastic packaging, polyethylene first for will be considered for use with the product because it usually does not react or cause any damage to the product. Thus, highdensity polyethylene was first chosen to deliver the newly created soy methyl ester-based products. However, problems between the polyethylene containers and soy methyl esterbased products were reported. Migration of the product, or sweating, and package distortion (paneling) Bubbling on the surface of the bottles was also reported. Soy methyl ester itself was determined to cause failures of the packages. As a result, the concentration of soy methyl ester was limited in soy methyl ester-based products. Fluorinated high-density polyethylene containers, which are known as solvent-resistant materials, were selected to replace ordinary high-density polyethylene containers. A high level of fluorination of high-density polyethylene was found to solve or reduce the problems. However, the cost of this container was high, and it was not available in all the desired shapes. Also, it didn't always work well. Therefore, this effort to select suitable packaging for distribution of soy-methyl esterbased products, which contain high concentrations of soy methyl ester, without the incompatibility problems and at reasonable low cost, began.

For this research, the goal was to find suitable packaging that can be used to distribute soy methyl esterbased products without packaging distortion (paneling and

bubbling) or product migration, and at an inexpensive cost as well.

Three tests for selecting suitable plastic containers for distribution of high concentrations of soy methyl ester-based products were designed. These three tests were the migration test, immersion test and bubbling test. First, in the migration test, a soy methyl ester-based product called Bean-e-doo, which is a general cleaner containing a high concentration of soy methyl produced by Franmar Chemical Inc., was used as the sample product for testing with a variety of different types of plastic bottles. To investigate the belief that the soy methyl ester itself caused the failure of the package, the same test using pure soy methyl ester instead of Bean-e-doo was designed to compare the results. Bean-e-doo had been reported to exhibit problems between the product package, package distortion (paneling), and migration of the product from high-density polyethylene containers. Bubbling on the surface of the bottles was also reported. The second and third tests, the immersion test and the bubbling test, are two more tests that were designed to support and complete the test results.

CHAPTER 1

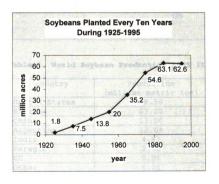
LITERATURE REVIEW

<u>History of soybeans</u>, the raw material for soy methyl ester, in the United States

Soybeans (scientific name: Glycine max (L) Merrill) are the world's foremost provider of protein and oil. originated in the north and central regions of China as early as 5000 years ago (1), and were introduced to the United States in 1804 by the sailors of a Yankee clipper ship (2). When the ship left China, the sailors loaded the ship with soybeans as inexpensive ballast. Then they dumped the soybeans to make room for cargo when they arrived in the United States. After that, soybeans could be found in the U.S. In 1829, farmers in the U.S. first grew soybeans (1). In the late 1800s, farmers started to grow soybeans as forage for cattle (1). In 1904, a study of soybeans used as a protein and oil source was begun (1). In the late 1920s, William Morse, the first president of the American Soybean Association, brought thousands of new soybean varieties from China to the United States for U.S.

researchers to study and create new varieties of soy-based products for industrial use from soy protein and soy oil, besides food products (2). As a result, the first U.S. soybean processing plant was opened in 1922. Then the large-scale production of soybeans had begun. The methyl ester of soybean oil, called soy methyl ester or soyate ester, is a newly developed soy product as a raw material for a variety of industrial uses such as solvent cleaner, biodiesel, lubricant and printing ink. The following graph shows U.S. soybean acres planted every ten years during 1925-1995.

Figure 1: Sovbean Acres Planted 1925-95 (1)



Today, farmers in over 29 states in the U.S. grow soybeans and export to the world market (2). As a result, now the U.S. is the world leader for producing and exporting soybeans (see Figure 2 and Table 1), according to the American Soybean Association.

World Soybean Exports 1995

China
18
Other
Paraguay
58
Brazil
108

States 72%

Figure 2: World Soybean Exports 1995 (1)

Table 1. World Soybean Production 1995 (1)

Country	Production		
	(million metric ton)		
United States	58.56		
Brazil	23.20		
China	13.50		
Argentina	12.64		
India	4.47		
Paraguay	2.30		
EU	0.94		
Other	8.04		
Total	123.65		

Soy Methyl Ester

Soy methyl ester is an important ingredient or component for a variety of products such as solvent cleaner, biodiesel, lubricant and printing ink. Soy methyl ester is a fatty acid methyl ester product from the transesterification process (see Figure 3).

Figure 3: Soy Methyl Ester from Transesterification

Triglyceride Methanol Glycerol Fatty acid methyl ester

Generally, transesterification is a reaction of animal or vegetable fats and oils with alcohols, in the presence of an acid or alkaline catalyst (3). The alcohol components of an ester, triglyceride, are chemically replaced by the other alcohol components from methanol or ethanol, so that transesterification can be called alcoholysis as well. If methanol is used in the reaction, the fatty acid is called methyl ester. On the other hand, if ethanol is used instead of methanol, the fatty acid ester is called ethyl ester. Normally, methanol is used in the process because

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half as much methanol is required as ethanol to form fatty acid ester in the transesterification reaction (4). other reason is the methanol price is only one-third of the ethanol (4). For soy methyl of cost ester transesterification, soybean oil (see composition soybean oil in Table 2) is heated to 80 °C, and 1.6 molar excess of methanol and methanolic solution of 5% potassium hydroxide (KOH) are added. Then the mixture is agitated for 30 minutes, and left to settle. Four hours later, there will be two separate layers of the product, which are the glycine layer and the ester layer. The ester layer is taken and acidified with 0.1% phosphoric acid. ester is washed with hot water (about 90 °C) a couple of times until the wash water is neutral. Finally, the ester layer is dried under a vacuum to remove the last of the There are other transesterification processes moisture. available to produce soy methyl ester, as well.

The suitable purity of soy methyl ester is dependent on the needs of the users. For instance, fuel suppliers need 97% purity for their typical uses. The purity of soy methyl ester can be improved by using distillation (3).

The main soy methyl esters are methyl oleate and methyl linoleate, which account for 75% of soy methyl ester (see Figure 4). In addition, the following soy methyl

esters are present in significant amounts: methyl palmitate, methyl stearate and methyl linolenate (3).

Table 2. Fatty Acid Composition of Soybean Oil (5)

Fatty Acid	Quantity (% wt.)		
Saturated			
Lauric	0.1		
Myristic	Less than 0.5		
Palmitic	7-12		
Stearic	2-5.5		
Arachidic	Less than 1.0		
Behenic	Less than 0.5		
Unsaturated			
Plamitoleic	Less than 0.5		
Oleic	20-50		
Linoleic	35-60		
Linolenic	2-13		
Eicosenoic	Less than 1.0		

Figure 4: Methyl Oleate and Methyl Linoleate

Methyl Oleate

 $CH_3 (CH_2)_7 CH = CH (CH_2)_7 COOCH_3$

Methyl Linoleate

 $CH_3 (CH_2)_4 CH = CHCH_2 CH = CH (CH_2)_7 COOCH_3$

Research about the kinetics of transesterification of soybean oil shows that the following three consecutive reactions are involved in the transesterification process of soy methyl ester (6):

Triglyceride (TG) Methanol Diglyceride (DG)

Diglyceride (DG) Monoglyceride (MG)

Monoglyceride (MG) Glycrol (GL)

<u>Overall</u>

Triglyceride + 3 CH₃OH ⇔ Glycerol + 3 ROOCH₃

Each reaction is reversible, and thus the overall reaction is also reversible. Three soy methyl ester molecules will be produced from one soybean fatty acid molecule in the reaction.

Soy methyl ester-based products

Diesel Fuel

Vegetable oil has been of interest for use as fuel for engines since the late 1800s (4). Nonetheless, vegetable oils did not gain much attention as fuel because there was plenty of petroleum available. However, in the late 1970s the price of petroleum became high due to the Organization Petroleum Exporting Countries (OPEC) limiting supply. Also, Western countries were insecure about Middle East politics (4). As a result, vegetable oil reconsidered for use as diesel fuel. Now researchers pay more attention to diesel fuel from soybean oil because the methyl ester of soybean oil is an environmentally friendly Biodiesel is non-toxic and biodegradable. It also fuel. has a higher cetane number than diesel fuel, and offers the potential for reduction of emissions of carbon monoxide, unburned hydrocarbon and particulate matter.

As described above, triglycerides of soybean oil are converted to methyl esters or ethyl esters by a transesterification process, also called alcoholysis (4). Methyl ester is preferred because half as much methanol is required as ethanol to form the ester, and methanol's price is about one-third the cost of ethanol (4). Methyl ester from soybean oil is referred to as "methyl soyate." The properties of Soy Diesel (methyl soyate) are compared with No.2 diesel fuel in Table 3.

SoyDiesel is biodegradable, nontoxic and free of sulfur and aromatics. However, the price of SoyDiesel is higher than the current prices of diesel fuel. Currently, SoyDiesel is of interest for fueling urban buses in cities to reduce air pollution problems.

SoyDiesel is developing and improving under the National SoyDiesel Development Board (NSDB), located in Jefferson City, Missouri, which was established in 1992.

Printing Ink

Newspaper printing primarily uses lithography or offset printing and letterpress printing. Ink in both cases is composed of pigment (such as carbon black for black ink or various organic compounds for colored inks), a vehicle, and other minor proprietary ingredients. The

vehicle is used as the carrier for the pigment. Normally, the vehicle is composed of a resin and a petroleum-derived hydrocarbon solvent such as mineral oil. Recently, soy methyl ester has been used as a solvent for printing ink.

Table 3: Comparative Properties of Diesel and Soy Diesel
Fuels (4)

ASTM fuel property	Soy diesel	Diesel
	(methyl	(No. 2)
	soyate)	
Total acid number	0.45	0
(mg KOH/g)		
Water and sediments	<0.005	0.01
Max. (vol %)		
Color	0.5	3.5
Ash, max. (wt.)	0.001	0.01
Relative density	0.883	0.840
(@15°C)		
Distillation temp.		
(°C)	313	100.9
Initial point	335	270.2
50%	345	351.1
90%	354	370.2
95%	357	380.5
End point	96	96.5
Recovery	1	1.5
Residue		
Carbon residue max.	0.19	0.08
(wt%)		
Cetane number	51.8	51.0
Phase changes (°C)		
Flash point	118	64
Cloud point	-3	-21
Viscosity at 37.8 °C	4.70	3.25
(min)		
Sulfur (wt%)	<0.01	<0.21

In 1985, the American Newspaper Publishers' Association (ANPA) produced the "first-generation" soybean oil-based new ink (3). It was composed of alkali-refined soybean oil, a hydrocarbon resin, a pigment, and antioxidant. Soybean oil was used as a solvent for a vehicle instead of mineral oil. This ink is available for both black and color printing. The performance of ANPA ink, soybean-based ink manufactured by ANPA, is as good or better than conventional mineral oil-based ink (4). The main advantages of this ink are superior print qualities, brighter color, cleaner run and environmental benefits.

A "second generation" of soybean-based ink was developed by USDA's NCAUR (National Center for Agricultural Utilization Research) (4). The vehicle of this new ink is totally derived from soybean oil, eliminating the need for a petroleum-based component.

The USDA's NCAUR ink ingredient has no petroleum-based component; the solvent and resin have been replaced with soybean-based materials, so the cost of this ink is cheaper. Also, the light color of the NCAUR vehicle allows less pigment to be used, so the cost of colored ink is reduced as well. Because of the above reasons, soybean-based ink is cost-competitive with petroleum-based ink.

The quality of this ink meets the standard requirements and is above the industry standard for printing quality, ease of cleanup, rub-off resistance, tack, and biodegradablity.

According to the NCAUR group's research, soybean-based ink is biodegradable. Soybean-based ink containing a 100% soy-based vehicle can degrade 82-92%, compared with 58-68% biodegradability for the ANPA vehicle, and 20% biodegradability for a petroleum-based vehicle (4).

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CHAPTER 2

TESTING MATERIALS AND METHODS

Test selection and test design

According to previous investigation, high concentration of soy methyl ester was believed to cause failure of the high-density polyethylene containers. the effort to seek a new suitable type of plastic packaging While a high level of fluorination of highoccurred. density polyethylene containers was being used to contain the product, the high cost and the limited variety of shapes were problems. Also, they didn't always work well. Therefore, there was a desire to seek a new type of plastic packaging. The goals were to select a suitable plastic container, which removes the packaging distortion migration, bubbling (paneling), sweating or and the problems of existing containers. Inexpensive packaging and an unlimited variety of shapes and sizes were also included in the requirements for the new selected packaging.

The first test for selecting a suitable plastic container was migration testing of the plastic bottles.

The migration of the product from the bottles was observed. Also, any physical change of the bottles such as paneling and bubbling was observed. Bean-e-doo, a general cleaner with a high concentration of soy methyl ester, from Franmar Chemical Inc., which actually showed the packaging problems described above, was selected to use as a sample product. Nine types of plastic materials were selected for testing. They were low-density polyethylene (LDPE), High-density polyethylene (HDPE), polypropylene (PP), high-density polyethylene/nylon blend (SELAR), polypropylene/highdensity polyethylene blend (PPAL), polyvinyl chloride (PVC), polyethylene teraphthalate (PET), glycol-modified polyethylene teraphthalate (PET-G), and polyacrylonitrile (PAN) bottles. Polyethylene was known to be problematic with soy methyl ester. However, high-density polyethylene and low-density polyethylene were included in the test as a baseline for comparison. Low-density polyethylene was expected to be a poorer container than high-density High-density polyethylene/ nylon blend polyethylene. (SELAR) is known as good barrier packaging. The barrier properties, or permeation prevention, for many hydrocarbons and organic solvents are improved. Its barrier is equally effective as high-density polyethylene (HDPE) with surface chemical modification. Also, its stiffness at elevated

temperatures is increased, and it is better to use at higher temperature than surface treated HDPE. Thus, SELAR was selected to test because it might be suitable with soy methyl esters. Polypropylene (PP), which is resistant to strong acids, bases and most solvents, was an alternative choice for soy methyl-ester based products. Polypropylene/ high-density polyethylene blend (PPAL), which has both good polypropylene properties and high-density polyethylene properties, was also selected to seek for compatibility with soy methyl esters because it was reported to be a better barrier than either alone. Polyvinyl chloride (PVC) is an inexpensive material. Also, it is an excellent barrier for oil, alcohol and petroleum solvents. Because of its barrier properties, PVC was chosen. Polyethylene terephthalate (PET) is successful for packaging carbonated soft drinks and is also used to package a variety of other products. It is used for products ranging from edible oil to industrial products. Since soy methyl ester is a fatty acid methyl ester from soybean oil, it might be compatible with PET in the same way as is edible oil. Glycol-modified polyethylene terephthalate (PET-G) was also included in the test to complete the PET category. Polyacrylonitrile (PAN) was the last material chosen to test to qualify as a soy methyl ester container because of its well-known

outstanding chemical resistance and barrier. package household generally used to and industrial chemicals. For some plastics, more than one type of plastic bottle of the same polymer was tested, meaning containers from different resins or suppliers. There were types of high-density polyethylene two (HDPE) different suppliers. There were two types of polyvinyl chloride bottles from different PVC resins. Four types of polyethylene terephthalate (PET) containers were tested with three made from different resins and the other from a different supplier. Two types of polyacrylonitrile (PAN) from different resins were used in the test. The test was carried out at three different temperatures, low, medium and high. The low temperature was at ambient conditions, about 23 °C and 50% relative humidity (RH), to represent room temperature in the store or on the shelf. It is an actual condition for distribution of the products. acceleration of the results and testing at severe conditions, higher temperatures were used. temperatures of 30 °C, 60% RH and 40 °C, 75% RH were Then to prove that soy methyl ester causes the selected. failure of the packages, a test with pure soy methyl ester was designed. The test was the same as the test with Beane-doo, only pure soy methyl ester was used instead of Beane-doo. For some types of plastic containers, the experiments were duplicated with both Bean-e-doo and pure soy methyl ester tests.

The second test was an immersion test. It was a test designed to obtain more information about the dimensional changes, length, width and thickness, and weight changes when the plastic strips were immersed in the soy methyl ester based product, Bean-e-doo. Plastic strips were first measured to determine the dimensions and weight, and then immersed in Bean-e-doo for a specific time, 7 or 14 days, at a specific temperature, ambient temperature or 40 °C. After that, they were taken out and the dimensions and There were four sets of test weight re-measured. conditions. The first set was immersion for 7 days at ambient temperature. The second was immersion for 7 days at 40 °C. For the third set, the plastic strip was immersed for 14 days at ambient temperature. The last set was at 40 °C for 14 days. Only some plastic types were selected: polyethylene (HDPE), polyethylene high-density terephthalate (PET), polypropylene (PP) and high-density polyethylene / nylon blend (SELAR). Ionomer, named by Du Pont because of ionic bonds in the molecule in addition to the normal covalent bonds that are present in polyethylene,

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was included in this test. It is tough, durable transparent thermoplastic mostly used for consumer industrial products. The third test was a bubbling test. Information from Franmar Chemical Inc. showed that containers of soy methyl ester based product were bubbling all over the surface of the bottles. Therefore, the test was designed to determine whether bubbles would occur on pieces of plastic material immersed in a high concentration of soy methyl ester, Bean-e-doo, or on bottles containing the product as they were observed in the first test, the migration test. The types of polymer used in this test were the same ones used in the immersion test. The test was set at 40 °C to accelerate the results, doing the test at severe conditions, where the bubbling would be expected to occur more rapidly than at ambient temperature.

Testing materials and testing methods

Migration Test of Plastic Bottles

Materials

1. Plastic bottles; the description of each plastic bottle is in Appendix B. Also, some pictures of plastic bottles are shown in Chapter 3. High-density polyethylene (HDPE)

- 2 different HDPE

Low-density polyethylene (LDPE)

Polypropylene (PP)

High-density polyethylene/nylon blend

(SELAR)

Polypropylene/high-density polyethylene (PPAL)

Polyvinyl chloride (PVC)

-2 different PVC

Polyethylene terephthalate (PET)

-4 different PET

Glycol-modified PET (PET-G)

Polyacrylonitrile (PAN)

-2 different PAN

- 2. Bean-e-doo
- 3. pure soy methyl ester
- 4. plastic caps
- 5. labeled tag
- 6. marker
- 7. corrugated board card

Methods

1. Label each plastic bottle with labeled tag.

- 2. Fill the bottles with Bean-e-doo or pure soy methyl ester.
- 3. Cap the plastic bottles tightly.
- 4. Clean all the completed bottles with a towel to remove any liquid on the bottle surface, which can disturb the migration results.
- 5. Put the finished bottles on the corrugated board card at the specified temperature, ambient, 30 °C, or 40 °C. The purpose of the corrugated board is to provide a stainable surface for evidence of migration, as well as a smooth base for the bottles.
- 6. Observe the migration of the product and record the time of product migration from the bottle.

 Observe for paneling of the bottle, and any damage of the bottle every day. (The first sign of migration is usually smearing of the ink on the label. Then the bottles will become oily on the surface.)

Immersion Test of Plastic Strips

Materials

1. Plastic strips

Ionomer

High-density polyethylene (HDPE)

Plastic strips of HDPE and Ionomer were made in a dumbbell shape as described in ASTM D638-91 (Standard Test Method for Tensile properties of Plastics). The dimensions were 63.5×9.63×3.3 mm (type V)

High-density polyethylene / nylon blend
(SELAR)

Polyethylene terephthalate (PET)

Plastic strips of SELAR and PET were cut from SELAR bottles and PET bottles, respectively, in a rectangular shape.

Polypropylene (PP)

Plastic strips of PP were made by compression molding (see Appendix A) and cut in rectangles with dimensions of 2 \times 0.265 inch \times 0.13 inch

- 2. Bean-e-doo
- 3. Labeled tag
- 4. Marker

- 5.400-ml Beakers
- 7. Micrometer (precision; \pm 0.005 mil)
- 8. Caliper (precision; \pm 0.005 millimeter)
- 9. LDPE film
- 10. Rubber bands
- 11. Chamber (40 °C, 75% RH)

Methods

- 1. Label each plastic strip. Each plastic type was done in triplicate to get average data for the dimension and weight changes in Bean-e-doo.
- 2. Measure the width and length of the plastic strip using a caliper, and then measure the thickness by using a micrometer.
- Measure the weight of the plastic strip using the analytical balance.
- 4. Fill each 400-ml beaker with 200 ml of Bean-e-doo.
- 5. Put one plastic strip in each beaker. The control test used water instead of Bean-e-

- doo. A control was run with each plastic type.
- 6. Close each beaker with LDPE film and rubber band.
- 7. Store the beakers for the specified time and temperature.

Set 1; at ambient for 7 days

Set 2; at ambient for 14 days

Set 3; at 40 ° C for 7 days

Set 4; at 40 °C for 14 days

- 8. After storing for the specified time, take the strip out. Dry the strip with a towel, then re-measure the dimensions and weight.
- 9. Calculate the percent changes.

Bubbling Test

Materials

1. Pieces of plastic sample

Ionomer

High-density polyethylene (HDPE)

Polypropylene (PP)

The pieces of HDPE, Ionomer and PP were made by a compression molding

process (see Appendix A). Then they were cut into a square shape, 3×3 inch $\times 0.13$ inch

High-density polyethylene / nylon blend
(SELAR)

Polyethylene terephthalate (PET)

The pieces of PET and SELAR were cut from SELAR bottles and PET bottles, respectively, in the square shape of 3×3 inch \times 0.013 inch.

- 2. Bean-e-doo
- 3. Labeled tag
- 4. Marker
- 5.400-ml beaker
- 6. LDPE film
- 7. Rubber bands
- 8. chamber (40 °C, 75% RH)

Methods

- 1. Label each plastic piece.
 - The experiment was duplicated for each type of plastic.

- 2. Fill each 400-ml beaker with 400 ml of Bean-e-doo.
- 3. Put one piece of plastic in each beaker.

 Close each beaker with LDPE film and rubber band.
- 4. Store at 40 $^{\circ}\text{C}$. The total time of the test is 131 days.
- 5. Observe for bubbling on the piece of plastic.

CHAPTER 3

RESULTS AND DISCUSSION

Migration Test

Table 3: Results of Migration Test of plastic Bottles with Bean-e-doo and pure soy methyl ester (complete results in Appendix C)

Plastic/	Average	Time for	Migration*
Test liquid	Ambient	30 °C	40 °C
	(days)	(days)	(days)
LDPE/	21	8	3
Bean-e-doo			
LDPE/Pure soy	12	5	4
HDPE-1/	36.5	24.5	7.5
Bean-e-doo			
HDPE-1/	50	23	8.5
Pure soy			
HDPE-2/	80	34	17
Bean-e-doo			
HDPE-2/	55	45	15
Pure soy			
PET-1/	>613	>195	>174
Bean-e-doo			
PET-1/	>550	>172	>172
Pure soy			
PET-2/	>564	>186	>172
Bean-e-doo			
PET-2/	>550	>172	>172
Pure soy			
PET-3/	>564	>186	>174
Bean-e-doo			L

PET-3/	>550	>172	>172
Pure soy PET-4/	> 5 C A	> 1 0 C	. 174
Bean-e-doo	>564	>186	>174
PET-4/	>550	>172	>172
Pure soy	2550	7112	7172
PET-G/	>564	>186	>174
Bean-e-doo	2004	7100	71,4
PET-G/	leaking	>172	>172
Pure soy			, , ,
PAN-1/	>613	>195	>174
Bean-e-doo			
PAN-1/	>550	>172	>172
Pure soy			
PAN-2/	>564	>186	>174 ·
Bean-e-doo			
PAN-2/	>550	>172	>172
Pure soy			
PVC-1/	>564	>186	>174
Bean-e-doo			
PVC-1/	>550	>172	>172
Pure soy			
PVC-2/	>564	>186	>174
Bean-e-doo		170	170
PVC-2/	>550	>172	>172
Pure soy PP/Bean-e-doo	69	17	6
PP/Beall-e-doo		1 /	0
PP/Pure soy	49	16	5
[]	'		
PPAL/	78	53	27
Bean-e-doo			
PPAL/Pure soy	74	62	2.5
SELAR/	69	41	20
Bean-e-doo			

^{*}The number of days from initiation of the test to the first appearance of migration.

> Means the bottle showed no migration on the last day of the test.

As expected, test results showed that low-density polyethylene (LDPE) was the worst container for soy methyl ester-based products. The bottles took the shortest time to show migration of the product at any temperature in both the Bean-e-doo and the pure soy methyl ester tests. the three different temperatures, low, medium and high, the bottles tested at low temperature took the longest time to migration of the product, while at the temperature the bottles took the shortest time to show first sign of migration was usually migration. The smearing of the ink on the label. Then the bottle would become oily on the surface. The bottles showed serious paneling at the highest temperature, 40 °C, in both the Bean-e-doo and the pure soy methyl ester tests. Also, they showed bubbling on the surface of the bottles at room temperature in both Bean-e-doo and pure soy methyl ester tests at the end of the experiment (see Figures 5 and 6). The bottles took a long time, almost 613 days for Bean-edoo and almost 550 days for pure soy methyl ester, before the bottle surface ambient showing bubbling on at temperature. Bubbling of the bottles would probably have occurred at the higher temperatures, both 30 °C and 40 °C, if the experiments had continued longer, and would likely have taken a shorter time than the bottles at room temperature. Even though it was already known that high-density polyethylene containers were not suitable for containing soy methyl ester-based products, low-density polyethylene, which was expected to be the poorest container, was included in the test to be a baseline to compare the results with other plastic containers.

For high-density polyethylene, two types of highdensity polyethylene containers were tested, HDPE-1 and HDPE-1 and HDPE-2 were from different suppliers. The results showed that HDPE-2 took more time than HDPE-1 before showing migration of the product out of the bottles The reason might be the greater at all temperatures. thickness of the container wall of HDPE-2 than HDPE-1. The thickness of HDPE-2 averaged 2 mil, but unfortunately the exact thickness of HDPE-1 was not measured. Both HDPE-1 and HDPE-2 showed bubbling at room temperature at the end of the test in both the Bean-e-doo and pure soy methyl ester tests (see Figures 8, 9 and 10). There is difference in the nature of the bubbling of these two types of HDPE bottles. Individual bubbles on the surface of the HDPE-1 bottle were bigger than on the HDPE-2 bottle (see The small bubbles on the surface of HDPE-2 Figure 7). might be an earlier stage in bubble development. showed, as expected, high-density polyethylene is not suitable to contain high concentrations of soy methyl ester-based products. Also, increasing the thickness of the high-density polyethylene containers cannot remove the packaging distortion and migration problems.

For polyethylene terephthalate (PET), four types of containers were evaluated, PET-1, PET-2, PET-3 and PET-4, made from different resins. They showed no migration of the product, no paneling or bubbling at any temperatures in both the Bean-e-doo and pure soy methyl ester tests. As a result, PET is qualified to be a good candidate for distribution of high concentrations of soy methyl esterbased products.

Glycol-modified polyethylene terephthalate (PET-G) bottles showed no migration, paneling and bubbling in the Bean-e-doo test, except for paneling in one of two bottles at 40 °C. In the pure soy methyl ester test at ambient temperature, the product leaked from the bottom of the bottle. Investigation showed that the bottom of the bottle was broken, which might be the result of a production defect rather than failure caused by the soy methyl ester. Unfortunately, the test was not duplicated, so further testing with PET-G would be required to determine whether or not this failure was caused by a defect in the bottle. The bottle with pure soy methyl ester at 30 °C showed no

migration and no paneling, while at 40 °C the bottle paneled with no migration. Therefore, PET-G is a questionable candidate for containing pure soy methyl ester-based products.

For polyacrylonitrile (PAN), generally available under the Barex trade name, two types of bottles were evaluated, PAN-1 and PAN-2, tested with both Bean-e-doo and pure soy methyl ester. In the Bean-e-doo test, one of two PAN-2 bottles at 40 °C paneled without migration of Bean-e-doo. The other bottles in the Bean-e-doo test exhibited no paneling or migration. In the pure soy methyl ester test, there were absolutely no migration or paneling problems. As a result, polyacrylonitrile (PAN) is still a candidate for high concentration of soy methyl ester-based products.

There were two types of polyvinyl chloride (PVC) bottles in both the Bean-e-doo and pure soy methyl ester tests. These two types are made from different resins, PVC-1 and PVC-2. For the Bean-e-doo test, all of the PVC-1 bottles at all temperatures showed paneling without migration, except one at 30 °C. A picture of the paneling is shown in Figure 11. On the other hand, all PVC-2 bottles showed absolutely no paneling or migration at the test end. For the pure soy methyl ester test, all PVC-1

bottles paneled with no migration at any temperature (see Figures 12 and 13), while all PVC-2 showed no paneling or migration. Because of the PVC-2 results, PVC is still considered as a candidate to distribute high concentration of soy methyl ester based products. However, further research about specific types of PVC resin should be carried out.

All polypropylene (PP) bottles showed migration of the product at all temperatures in both the Bean-e-doo and pure soy methyl ester tests. Even though all bottles showed migration, they did not exhibit paneling or bubbling at any temperatures. Because of the migration results, polypropylene (PP) is not suitable to distribute high concentration of soy methyl ester-based products.

PPAL is a polymer blend containing polypropylene and high-density polyethylene. It showed better performance than either high-density polyethylene homopolymer or polypropylene homopolymer. Even though PPAL had better performance, it still had a migration problem at all temperatures in both the Bean-e-doo and pure soy methyl ester tests. Also, the bottles at ambient temperature showed a bubbling on their surface in both the Bean-e-doo and pure soy methyl ester tests (see Figures 14 and 15). Therefore, this polypropylene/ high-density polyethylene

blend is not suitable as a container for soy methyl esterbased products.

A high-density polyethylene/nylon blend (SELAR) was the last polymer in the migration test. SELAR was tested only with Bean-e-doo, not with pure soy methyl ester. This polymer showed better performance than high-density polyethylene homopolymer. However, all the plastic bottles showed migration problems at all temperatures. Also, the bottles paneled at 30 °C, and seriously paneled at 40 °C. As a result, SELAR cannot be qualified to be a candidate for soy methyl ester-based products.

In evaluating whether soy methyl ester caused the failure of the packages, generally the results showed that the bottles with pure soy methyl ester exhibited migration or paneling problems faster than the bottles with Bean-edoo. This supports the conclusion that soy methyl ester causes the failure of the packages.

According to these results, then, there are three possible candidates that are suitable to contain or distribute high concentration of soy methyl ester-based products. These candidates are polyethylene terephthalate (PET), polyacrylonitrile (PAN), and polyvinyl chloride (PVC).

Immersion Test

According to the results (the complete results are shown in Appendix C), the data did not show a clear relationship between the sample bottles in the migration test and the sample strips in the immersion test of the same type of plastic. For example, PP and HDPE weight changes of plastic strips in the immersion test for 7 days at 40 °C showed weight gains for PP and HDPE were 7.3% and 1.38% respectively. The weight gains (in percentage) represent the liquid sorption of the plastic strip, so the sorption of PP was higher than HDPE. Sorption could cause changes in mechanical properties. Change in mechanical properties could cause unexpected package system failure due to a variety of factors. For example, sorption could affect the structure of the polymer such as swelling or dissolution, causing a decrease of tensile strength. As a result, if the tensile strength of the packaging material decreased significantly, the strength of the package may be severely reduced. From the PP and HDPE results, PP had an average 7.3% weight gain, higher than HDPE with an average 1.38% weight gain. From this result, PP might be expected to fail quicker than HDPE, but in the migration test HDPE failed, showing migration, quicker than PP. PET is an example as well. PET weight gain at 40 °C averaged -13.6%.

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This could indicate dissolution of PET, so PET might have a package system failure. The results for PET were also not consistent. For instance, the weight results of PET for the 7 day test showed 0.06% at ambient and -13.6% at 40 °C, while for the 14 day test the results showed 0.37% at ambient and 0.24% at 40 °C. Therefore, no general conclusions can be drawn from this test. The significant test or principal test for package selection was the migration test.

Bubbling Test

No bubbling on any pieces of plastic was found in the bubbling test (see Table 14 in Appendix C), even though some bottles did show bubbling during the migration test. example, high-density polyethylene (HDPE) bottles filled with Bean-e-doo at ambient temperature had bubbling, but the polyethylene (HDPE) strip showed no bubbling. the time of may be due to short the experiment. Polyethylene bottles in the migration test took about 613 days in Bean-e-doo and 550 days in pure soy methyl ester to show bubbling on their surface, but the experiment for bubbling was run only with Bean-e-doo and the experimental test time was 131 days. Even though the experiment was run

a*

at 40 °C, a longer time might be required for bubbling to occur.



Figure 5: Bubbling of LDPE in the Bean-e-doo Test



Figure 6: Bubbling of LDPE in the Pure Soy Methyl

Ester Test



Figure 7: The Difference in Bubbles between HDPE-1 (left) & HDPE-2 (right)



Figure 8: Bubbling of HDFE-1 in the Bean-e-doo $$\operatorname{\textbf{Test}}$$



Figure 9: Bubbling of HDPE-1 in the Pure Soy

Methyl Ester Test



Figure 10: Bubbling of HDPE-2 in the Pure Soy

Methyl Ester Test



Figure 11: Paneling of PVC in the Bean-e-doo Test



Figure 12: Paneling of PVC in the Pure Soy Methyl

Ester Test



Figure 13: The Unique Paneling of PVC



Figure 14: Bubbling of PPAL in the Bean-e-doo
Test



Figure 15: Bubbling of PPAL in the Pure Soy

Methyl Ester Test

CHAPTER 4

CONCLUSIONS

The migration test or stability test plays an important role in screening various types of plastics that will be successful for packaging soy methyl-ester based products with a high concentration of soy methyl ester. The immersion test and bubbling test did not provide useful information for making this selection.

The results show three plastic candidates for packaging soy methyl-ester based products: polyethylene terephthalate (PET), polyacrylonitrile (PAN), and polyvinyl chloride (PVC). These three candidates can be used to replace the current container, highly-fluorinated high-density polyethylene.

Fluorinated high-density polyethylene is high-density polyethylene whose inner surface is chemically altered by fluorine gas. Fluorine will replace hydrogen atoms on the polymer chain. As a result, it provides a barrier to solvent permeation. Only high levels of fluorination make HDPE suitable for products with high concentrations of soy methyl ester. Generally, fluorinated high-density

polyethylene is used to package automotive related products, household chemicals, industrial chemicals and agricultural products.

The polarity of soy methyl ester and of the packaging container might be a significant factor. Soy methyl ester is hydrophobic (non-polar), so it can be more readily absorbed by a plastic container which is also hydrophobic such as polyethylene and polypropylene, following a theory of the chemical similarity (like dissolves like) causing the migration and paneling problems. Soy methyl ester firstly wets the inner surface of the container, dissolves into the wall, diffuses through the container and then surface evaporates off the outer container called migration. When it is sorbed by the container, it might cause structural changes resulting in container paneling. On the other hand, soy methyl ester would not be absorbed as readily by a hydrophilic (polar) plastic container such as PET, PVC and PAN because of the chemical dissimilarity.

Among PET, PAN, and PVC, polyacrylonitrile (PAN), generally sold under the trade name Barex, is the most expensive, while polyvinyl chloride (PVC) is the cheapest. However, the prices of these three candidates are competitive to the current container, since a high level of fluorination increases cost.

While high-density polyethylene with high levels of fluorination, the current container, is not available in all desired styles such as tubs, polyethylene terephthalate (PET), polyacrylonitrile (PAN) and polyvinyl chloride (PVC) are available in all varieties of shapes and sizes.

Therefore, these three candidates, polyethylene terephthalate (PET), polyacrylonitrile (PAN), and polyvinyl chloride (PVC) are the candidate alternative polymers for distribution of soy methyl-ester based products with high concentration of soy methyl ester.

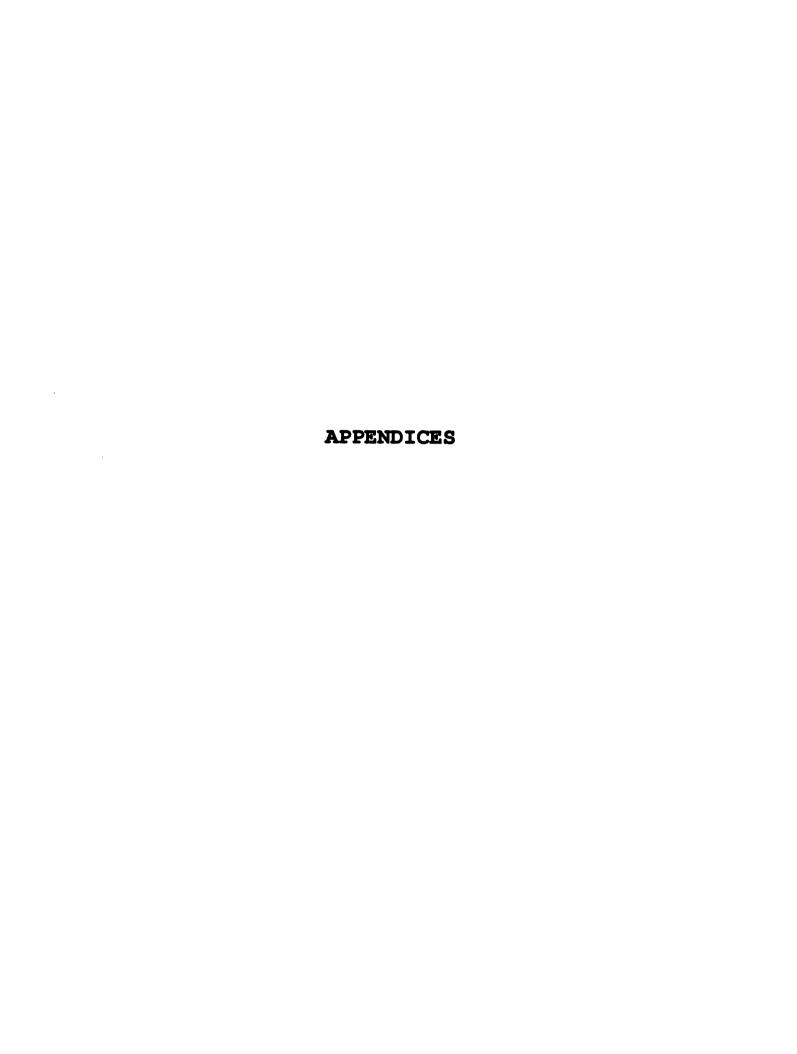
Franmar Chemical Inc. stated that the company has had problems with polyvinyl chloride (PVC) components in their equipment. However, the details were not disclosed. The company has now adopted polyacrylonitrile (PAN) containers for packaging their soy methyl-ester based products with high concentrations of methyl ester.

RECOMMENDATIONS

Since one out of the two types of PVC bottles tested showed paneling of the bottles at each temperature in both the Bean-e-doo test and the pure soy methyl ester test, further testing of specific types of PVC resin should be carried out to investigate whether or not the resin type influences performance.

The polymer materials in this research may be too limited, so further tests with other materials could be undertaken. Also, the materials in the tests only focus on single-layer materials. Therefore, multi-layer materials should be added. Duplication of the test results is also desirable. The duplication should be switched to five replicates because the results will be more accurate.

Environmental stress cracking (ESC) is also a big concern. Many polymeric materials are susceptible to failure due to cracking when exposed to mechanical stress in combination with certain kinds of liquids. Therefore, the candidates from the migration test should be further tested for environmental stress cracking in order to avoid this problem.



APPENDIX A

Diagram of compression molding process

Set the desired temperature of both platens of molding machine (HDPE = 150 °C, PP = 185 °C, IONOMER = 176 °F)

1

Wait for 12-15 minutes to heat up the system

1

Assemble three pieces of the extrudate in the mirror-like structure of Mylar sheet (PET sheet) and the metal platens, with the appropriate amount of plastic resin. (amount of resin: HDPE 58 g, PP 58 g, ionomer 50 g)

↓

Close the hydraulic chamber and put 30,000-psi load for 5 minutes

1

Adjust temperature to 50 °C and turn on cooling water

1

Wait for 5 minutes in order to let the system cool down to 50 $^{\circ}\text{C}$

1

Release the pressure and take out the frame

1

 $\hbox{Cut plastic into desired dimensions}\\$

APPENDIX B

Description of plastic bottles (Thickness of all plastic bottles was not measured.) HDPE-1 Color: white (transparent) Shape: cylinder round Height: 2.7760 inches HDPE-2 Color: white (opaque) Shape: cylinder round Height: 2.7155 inches LDPE Color: white (transparent) Shape: cylinder round Height: 2.5470 inches PPColor: white (transparent) Shape: bullet Height: 2.6245 inches SELAR Color: white (transparent) Shape: cylinder round

Height: 9.4375 inches

PPAL

Color: white (opaque)

Shape: flat oval

Height: 2.9545 inches

PVC-1

Color: clear

Shape: cylinder round

Height: 6.2030 inches

PVC-2

Color: clear

Shape: cylinder round

Height: 4.9435 inches

PET-1

Color: clear

Shape: cylinder round

Height: 2.9120 inches

PET-2

Color: clear

Shape: cylinder round

Height: 6.0625 inches

PET-3

Color: clear

Shape: bullet

Height: 4.2275 inches

PET-4

Color: clear

Shape: flat oval

Height: 5.6295 inches

PET-G

Color: clear

Shape: cylinder round

Height: 4.5410 inches

PAN-1

Color: clear

Shape: cylinder round

Height: 2.0245 inches

PAN-2

Color: clear

Shape: cylinder round

Height: 5.4335 inches

APPENDIX C

The complete results of migration test, immersion test and bubbling test

Results of Migration Test

Table 4: Results of Migration Test of Plastic Bottles with Bean-e-doo

Remarks		Both showed	bubbling at 578 days					Both bottles showed	serious paneling at	20 days.	Both showed bubbling	at 596 days.						
Time of	migration* (day)	21	21	21	8	8	8	3	3	3	32	17	3.98	97	23	24.5	9	6
Total days	of test (day)	613	613		195	195		174	174		613	613		195	195		74	174
Testing temp	(°C)	Ambient temp	Ambient temp	Average	೨₀0೯	ე₀0€	Average	J. 07	J. 07	Average	Ambient temp	Ambient temp	Average	3 ₀ 0£	3000	Average	℃ 7	℃ 0 \$
Plastic	bottle	LDPE1	LDPE2		LDPE1	LDPE2		LDPE1	LDPE2		$HDPE_1-1$	HDPE ₂ -1		$HDPE_1-1$	HDPE ₂ -1		$HDPE_1-1$	HDPE ₂ -1

00.00	Average	620	7.5	T C I
HDPE ₁ -2	Ambient temp	620	08	Both showed
HDPE2-2	Ambient temp	020	80	davs.
HDPE ₁ -2	30 °C	132	34	
HDPE ₂ -2	30℃	132	34	
	Average		34	
HDPE ₁ -2	40 °C	174	17	
HDPE ₂ -2	40 °C	174	17	
	Average		17	
PET1-1	Ambient temp	613		No migration
PET2-1	Ambient temp	613	1	No paneling
	Average			No bubbling
PET1-1	30 °C	195	1	No migration
PET2-1	30°C	195	1	No paneling
	Average	1.4		No bubbling
PET1-1	40 °C	174	,	No migration
PET ₂ -1	40 °C	174	1	No paneling
	Average	2.6		No bubbling
PET ₁ -2	Ambient temp	564		No migration
PET2-2	Ambient temp	564	1	No paneling
	Average	1.87		No bubbling
PET1-2	30 °C	186	ı	No migration
PET ₂ -2	30°C	186	1	No paneling
	Average	274		No bubbling
PET ₁ -2	40 °C	174	1	No migration
PET2-2	40 °C	174	-	No paneling
	Average			No bubbling

П																											
No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	It showed no	migration or	paneling at 125	The bottle was
1	1		1	1		1	1		1	1		ı	1		1	1		1	-		ı			1			1
564	564		186	186		174	174		564	564		186	186		174	174		564	564		186	186		174	564		174
Ambient temp	Ambient temp	Average	30 °C	30℃	Average	40 °C	40 °C	Average	Ambient temp	Ambient temp	Average	30 %	30,℃	Average	40 °C	40 °C	Average	Ambient temp	Ambient temp	Average	30 %	30℃	Average	40 °C	western temp		40 °C
PET_1-3	PET_2-3		PET_1-3	PET_2-3		PET ₁ -3	PET2-3		PET1-4	PET2-4		PET1-4	PET2-4		PET1-4	PET2-4		PET1-G	PET2-G		PET_1-G	PET2-G		PET1-G			PET2-G

																				\Box						_
days, but no migration		No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	No migration	No paneling	No bubbling	The bottle was	paneling at 114	days. However, no	No paneling or	migration	Section Const	The bottle was	paneling at 125	days. However, no	migration
	1	1	1		1	1		ı	1		-	1			1					1			1			
		613	613		195	195		174	174		564	564		186	186		174			174			564		194	
	Average	Ambient temp	Ambient temp	Average	30 °C	30,℃	Average	40 °C	40 °C	Average	Ambient temp	Ambient temp	Average	30 %	30°C	Average	40 °C			40 °C		Average	Ambient temp			
		PAN ₁ -1	PAN ₂ -1		PAN ₁ -1	PAN ₂ -1		PAN ₁ -1	PAN ₂ -1		PAN ₁ -2	PAN ₂ -2		PAN ₁ -2	PAN ₂ -2		PAN ₁ -2			PAN2-2	1		PVC ₁ -1			

PVC ₂ -1	Ambient temp	564	1	The bottle was
				days. However, no
	Average			man and a second
	30 °C	186		The bottle was
				paneling at 126
				days. However, no
				migration
PVC ₂ -1	30°C	186	1	No paneling or
				migration
	Average		1:	
PVC ₁ -1	40 °C	174	-	The bottle was
				paneling at 113
				days. However, no
				migration
PVC ₂ -1	40 °C	174	F	The bottle was
				paneling at 112
				days. However, no
				migration
	Average			
PVC ₁ -2	Ambient temp	564	-	No migration
PVC ₂ -2	Ambient temp	564	7	No paneling
	Average			No bubbling
PVC ₁ -2	30 %	186	25	No migration
PVC ₂ -2	30℃	186	1	No paneling
	Average			No bubbling
PVC ₁ -2	40 °C	174	of the sa	No migration
PVC ₂ -2	40 °C	174	1	No paneling
	Average			No bubbling

									Both bottles were	bubbling at the	Shoulder of the	bottles at 596 days								A little paneling at 101 days	Serious paneling at 90 days
69	69	69	17	17	17	9	9	9	89		88	78	53	53	53	27	27	27	69	41	20
613	613		195	195		174	174		613		613		195	195		174	174		132	132	132
Ambient temp	Ambient temp	Average	30 °C	30℃	Average	J. 07	2° 04	Average	Ambient temp		Ambient temp	Average	30 %	30℃	Average	2° 04	℃ 0 7	Average	Ambient temp	30 08	40 °C
PP ₁	PP2		PP1	PP ₂		PP1	PP ₂		PPAL1		PPAL ₂		PPAL ₁	PPAL ₂		PPAL ₁	PPAL ₂		SELAR	SELAR	SELAR

* The number of days from initiation of the test to the first appearance of

migration

Table 5: Results of Migration Test of Plastic Bottles with Pure Soy Methyl

Ester.

Plastic	Temperature	Total time	Time of	Remark
bottle	Testing (°C)	of test	migration	
		(days)	(၁့)	
$LDPE_1$	Ambient temp	250	12	Both bottles
				showed serious
LDPE2	Ambient temp	055	12	bubbling at 515
	Average		12	days.
LDPE1	၁့ 0 ေ	172	2	
LDPE2	೨₀0೯	172	2	
	Average		5	
LDPE1	J° 04	172	4	Both bottles
LDPE2	J. 07	172	4	Showed serious
	Average		7	paneling 7 days.
$HDPE_1-1$	Ambient temp	055	20	Both bottles
				showed serious
HDPE ₂ -1	Ambient temp	055	20	bubbling at 515
	Average		50	days.

						The bottle was	bubbling at 515 days.														The barries a branch	panelino ar or	davs. No migratio	The bottle showed	paneling at 50	GAYE. NO MIGRACIO
22	24	23	7	10	8.5	55		45	15	1			1	ı					ı	-	1	1	1	1	1	
172	172		172	172		550		172	172	550	550		172	172		172	172	100	550	172	172	550	172	172	550	172
30 %	30°C	Average	40 °C	40 °C	Average	Ambient temp		30 °C	40 °C	Ambient temp	Ambient temp	Average	30 %	300€	Average	40 °C	40 °C	Average	Ambient temp	30 %	40 °C	Ambient temp	30 °C	40 °C	Ambient temp	30 %
$HDPE_1-1$	HDPE ₂ -1		$HDPE_1-1$	HDPE ₂ -1		HDPE-2		HDPE-2	HDPE-2	PET_1-1	PET ₂ -1		PET ₁ -1	PET2-1		PET ₁ -1	PET2-1		PET-2	PET-2	PET-2	PET-3	PET-3	PET-3	PET-4	PET-4

°C 172 -	550	pottom of the	bottle failed,	causing leaking of	6117	the product	% No migration	77.	the parieting	°C 172 -	No market	1t temp 550 -	1t temp 550 -	rage	17.0	30 °C 1./2 -	30°C 172 -	rage	40 °C 172 -	40 °C 172 -	rage	ıt temp 550 -	30 °C 172 -	°C 172 -	it temp 550 - The bottle showed	days. No migration	°C 172 -	
40	Ambient temp							30		40		Ambient temp	Ambient temp	Average	0	30	30°	Average	40	40	Average	Ambient temp	30	40	Ambient temp		30	
PET-4	PET-G						PET-G	5 177		PET-G		PAN ₁ -1	PAN ₂ -1		Dant 1	PAN1-1	PAN ₂ -1		PAN ₁ -1	PAN ₂ -1		PAN-2	PAN-2	PAN-2	PVC-1		PVC-1	

PVC-1	40 °C	172	1	The bottle showed
				paneling at 55
				days. No migration
PVC-2	Ambient temp	550	ş	
PVC-2	3 ₀ 0E	172	-	
PVC-2	40 °C	172	-	
PP1	Ambient temp	550	49	
PP2	Ambient temp	550	49	
	Average		49	
PP1	30 08	172	12	
PP ₂	3008	172	20	-77
	Average		16	
PP1	40 °C	172	5	
PP ₂	40 °C	172	2	
	Average		2	
PPAL1	Ambient temp	550	7 L	Both bottles were
PPAL ₂	Ambient temp	550	7.4	bubbling at 515
	Average		74	days.
PPAL ₁	30 08	172	62	
PPAL ₂	3008	172	62	
	Average		62	
$PPAL_1$	40 °C	172	1	Both bottles were
				dubling aroung time
PPAL ₂	40 °C	172	4 '	sides (left and right) at 135
	Average		2.5	days.

Results of Immersion Test

Table 6: Weight Changes of Plastic Strip for Immersion Testing in Bean-e-doo for 7 days at Ambient Temperature and 40 °C

Sample	Temperature	Weight 0	Weight,	Weight	Weight
1	(၁)	day	day	Gain	Gain
		(g)	(g)	(g)	(&)
HDPEc	Ambient	1.1931	1.1939	8000.0	0.078
HDPE1		1.2000	1.2100	0.0100	0.83%
HDPE2		1.1763	1.1861	8600.0	0.83%
HDPE3		1.1987	1.2086	6600.0	0.83%
Average		1.1917	1.2016	6600.0	0.83%
HDPEc	J。07	1.2051	1.1926	- 0.0125	- 1.048
HDPE1		1.1943	1.2000	0.0057	0.48%
HDPE2		1.1942	1.2156	0.0214	1.79%
HDPE3		1.1708	1.1926	0.0218	1.86%
Average		1.1864	1.2027	0.0163	1.38%
IONOMER	Ambient	1.5359	1.5411	0.0052	0.348
IONOMER ₁		1.3752	1.3982	0.0230	1.67%
IONOMER ₂		1.3724	1.3953	0.0229	1.67%
IONOMER3		1.3729	1.3938	0.0209	1.52%
Average		1.3735	1.3958	0.0223	1.62%
IONOMERC	J。0 7	1.4096	1.3753	- 0.0343	- 2.43\$
IONOMER		1.3806	1.4429	0.0623	4.51

19.59	1.03%	8.38%	0.13%	0.23%	0.22%	0.228	0.228	- 12.98	3.078	- 9.438	11.55%	7.31%	0.32%	0.06%	0.078	0.05%	0.06%	- 14.28	- 11.18	- 17.68	- 12.18	- 13.6%	0.19%	2.61%	2.148	3.59%	2.78%	0.03%	4.25%
0.2709	0.0149	0.1160	6000.0	0.0019	0.0016	0.0017	0.0017	- 0.1585	0.0351	- 0.1160	0.1293	0.0822	0.0013	0.0002	0.0002	0.0002	0.0002	- 0.0683	- 0.0533	- 0.0797	- 0.0619	- 0.0650	0.0010	0.0152	0.0122	0.0158	0.0144	0.0001	0.0170
1.6537	1.4557	1.5174	0.6874	0.8459	0.7471	0.7863	0.7931	1.0697	1.1771	1.1147	1.2486	1.1801	0.4122	0.3120	0.3058	0.3815	0.3331	0.4131	0.4285	0.3723	0.4476	0.4161	0.5187	0.5975	0.5819	0.4561	0.5452	0.3437	0.4167
1.3828	1.4408	1.4014	0.6865	0.8440	0.7455	0.7846	0.7914	1.2282	1.1420	1.2307	1.1193	1.1640	0.4109	0.3118	0.3056	0.3813	0.3329	0.4814	0.4818	0.4520	0.5095	0.4811	0.5177	0.5823	0.5697	0.4403	0.5308	0.3436	0.3997
			Ambient					40 °C					Ambient					40 °C					Ambient					40 °C	
IONOMER ₂	IONOMER3	Average	PP_{C}	PP_1	PP ₂	PP ₃	Average	PP_{C}	PP1	PP ₂	PP_3	Average	PETc	PET1	PET ₂	PET3	Average	PET_{C}	PET ₁	PET ₂	PET3	Average	SELARC	SELAR	SELAR	SELAR	Average	SELARC	SELAR

SELAR2	0.5088	0.5308	0.0220	4.32%
SELAR3	0.5590	0.5821	0.0231	4.138
Average	0.4892	6605.0	0.0207	4.238

Table 7: Weight Changes of Plastic Strip for Immersion Testing in Bean-e-doo

for 14 days at Ambient Temperature and 40 °C

Sample	Temperature	Weight 0	Weight 14	Weight	Weight
	(၁)	day	day	Gain	Gain
		(g)	(g)	(g)	(&)
HDPEc	Ambient	1.8730	1.1882	6000.0	0.05%
HDPE1		1.2001	1.2128	0.0127	1.06%
HDPE2		1.1906	1.2048	0.0142	1.19%
HDPE3		1.1842	1.1986	0.0144	1.22%
Average		1.1916	1.2054	0.0138	1.16%
HDPEc	J. 07	1.2412	1.2416	0.0004	\$60.0
HDPE1		1.1850	1.2151	0.0301	2.548
HDPE2		1.1933	1.2220	0.0287	2.418
HDPE3		1.1926	1.2210	0.0284	2.38%
Average		1.1903	1.2194	0.0291	2.448
IONOMER	Ambient	1.3730	1.3783	0.0053	868.0
IONOMER		1.4289	1.4630	0.0341	2.39%
IONOMER ₂		1.3600	1.3917	0.0317	2.33%
I ONOMER3		1.3526	1.3851	0.0325	2.40%
Average		1.3805	1.4133	0.1560	2.37%
IONOMERC	J. 07	1.3792	1.4023	0.0231	1.67%
IONOMER1		1.3705	1.4847	0.1142	8.33%
IONOMER ₂		1.3558	1.4710	0.1152	8.50%
I ONOMER3		1.3247	1.4396	0.1149	8.67%
Average		1.3503	1.4651	0.1148	8.50%

PP ₁ PP ₂ PP ₃ Average		1			
PP ₂ PP ₃ Average PP _c		0.8151	0.8196	0.0045	855.0
PP ₃ Average PP _c		0.8399	0.8444	0.0045	0.548
Average PP _C		0.8720	0.8775	0.0055	0.63%
PP _C		0.8423	0.8472	0.0048	872.0
	40 °C	0.0614	1.0615	0.0001	%600 ° 0
PP_1		1.1168	1.1372	0.0204	1.83%
PP ₂		6:66:0	1.0145	0.0206	2.078
PP3		1.1443	1.1645	0.0202	1.778
Average		1.0850	1.1054	0.0204	1.89%
PET _C /	Ambient	0.4389	0.4389	0	% 0
PET1		0.4174	0.4187	0.0013	0.318
PET2		0.4475	0.4489	0.0014	0.31%
PET3		0.4431	0.4455	0.0024	0.54%
Average		0.4360	0.4377	0.0017	878.0
PET_{C}	40 °C	0.3293	0.3304	0.0011	% EE'0
PET1		0.3817	0.3826	0.0009	0.248
PET2		0.3167	0.3174	0.0007	0.22%
PET3		0.3538	0.3547	0.0009	0.25%
Average		0.3507	0.3516	0.0008	0.248
SELARc	Ambient	0.3963	0.3963	0	80
SELAR1		0.4457	0.4646	0.0189	4.248
SELAR ₂		0.4100	0.4276	0.0176	4.29%
SELAR3		0.5699	0.5931	0.0232	4.07%
Average		0.4752	0.4951	0.0199	4.20%
SELARc	40 °C	0.4638	0.4640	0.0002	0.04%
SELAR		0.4071	0.4251	0.0180	4.428
SELAR ₂		0.4785	0.4994	0.0209	4.378
SELAR3		0.5015	0.5234	0.0219	4.378
Average		0.4624	0.4826	0.0203	4.398

Table 8: Thickness Changes of Plastic Strip for Immersion Testing in Bean-e-doo

for 7 days at Ambient Temperature and 40 °C

_																							
& Gain			% 0	80	80	886.0	0.138	- 0.118	386.0 -	80	886.0	0.25%	80	398 .0	\$ 96.0	1.798	878.0	1.068	1.438	2.13%	1.418	1.66%	% 0
Thickness	Gain	(mil)	0	0	0	0.5	0.17	- 1.5	- 0.5	0	0.5	0.25	0	0.5	0.5	2.5	1.2	1.5	2.0	3.0	2.0	2.3	0
Thickness	7 day	(mil)	131.5	133	130	132	131.7	130	133	130.5	133	132.2	142	140.5	141.5	142.5	141.5	140	141.5	144	144	143.2	127
Thickness	0 day	(mil)	131.5	133	130	131.5	131.5	131.5	132.5	130.5	132.5	131.8	142	140	171	140	140.3	141.5	139.5	171	142	140.8	127
Temperature	(0,)		Ambient					J. 07					Ambient					J。07					Ambient
Sample	,		HDPEc	HDPE1	HDPE2	HDPE3	Average	HDPEc	HDPE1	HDPE2	HDPE ₃	Average	IONOMER _C	IONOMER	IONOMER ₂	IONOMER3	Average	IONOMER	IONOMER	IONOMER ₂	I ONOMER3	Average	PP_{C}

PP ₁		137	137		0
PP ₂		127	127.5		0.5
PP ₃		132	132.5		0.5
Average		132	132.3		0.3
PPc	40 °C	132.5	118.4	- 1	14.05
PP1		126.2	126.2		0
PP ₂		136.3	120.9	- 15	15.45
PP ₃		126.8	120.6	9 -	6.25
Average		129.8	122.5	- 7	7.23
PETc	Ambient	16.10	16.3	0.20	20
PET1		16.25	16.3	0.05	35
PET2		12.70	12.8	0.10	0.
PET3		14.65	14.7	0.05	2
Average		14.50	14.6	0.07	7
PETc	40 °C	14.95	15.06	0.11	1
PET1		13.86	15.00	1.14	4
PET2		13.70	13.17	- 0.53	53
PET3		13.78	16.22	2.44	4
Average		13.78	14.80	1.79	6
SELARC	Ambient	28.25	28.25	0	
SELAR		29.75	29.95	0.20	0
SELAR2		29.10	29.10	0	
SELAR3		23.75	23.90	0.15	2
Average		27.53	27.65	0.12	2
SELARC	40 °C	20.15	20.25	0.10	0
SELAR		22.10	22.75	0.65	2
SELAR ₂		28.00	28.65	0.65	2
SELAR3		30.00	30.60	09.0	0
Average		26.70	27.33	0.63	3

Table 9: Thickness Changes of Plastic Strip for Immersion Testing in Bean-e-doo

for 14 days at Ambient Temperature and 40 °C

& Gain			3.948	3.948	4.728	4.768	4.478	- 3.82%	- 2.33%	- 2.67%	- 3.79%	- 2.93%	8.08%	8.46%	6.02%	5.19%	6.56%	- 3.52%	- 1.448	- 1.448	- 1.43%	- 1.448	4.86%
Thickness	Gain	(mil)	5	5	9	9	5.6	- 5	- 3	- 3.5	- 5	- 3.83	10.5	11	8	7	8.6	- 5	- 2	- 2	- 2	- 2	6
Thickness	14 day	(mil)	132	132	133	132	132.3	126	126	127.5	127	126.8	140.5	141	141	142	141.3	137	137	137	138	137.3	129.5
Thickness	0 day	(mil)	127	127	127	126	126.7	131	129	131	132	130.7	130	130	133	135	132.7	142	139	139	140	139.3	123.5
Temperature	(၁,)		Ambient					J. 07					Ambient					40 °C					Ambient
Sample			HDPEc	HDPE1	HDPE2	HDPE3	Average	HDPEc	HDPE1	HDPE2	HDPE3	Average	IONOMER _C	IONOMER1	IONOMER ₂	IONOMER3	Average	IONOMER	IONOMER	IONOMER ₂	IONOMER3	Average	PP_{C}

PP ₁		121	126.5	5.5	4.55%
Γ		124	129.5	5.5	4.448
<u> </u>		124	134	10	8.06%
Average		123	130	<i>L</i>	5.68%
_	40 °C	122	117	S -	- 4.10%
Γ-		131	127	7 -	- 3.05%
Γ		115	111	7 -	- 3.48%
<u> </u>		132	128	7 -	- 3.038
Average		126	122	7 -	- 3.19%
-	Ambient	13.3	14.3	1	7.52%
		12.75	13.3	0.55	4.318
<u> </u>		12.5	13	05.0	48
I		12	12.55	0.55	4.58%
Average		12.42	12.95	0.53	4.30%
_	40 °C	11.80	11.93	0.13	1.10%
Γ		14.33	14.33	0	80
		11.40	11.53	0.13	1.148
Γ		13.23	13.23	0	% 0
Average		12.99	13.03	0.04	0.38%
SELAR	Ambient	26.00	26.70	0.70	2.69%
		24.00	24.60	09.0	2.50%
Γ		23.30	24.00	0.70	38
		29.90	30.60	0.70	2.348
Average		25.73	26.40	0.67	2.61%
SELARc	40 °C	27.00	27.60	09.0	2.22\$
Ι_		23.75	24.50	0.75	3.16%
		26.10	26.80	0.70	2.68%
SELAR		26.75	27.40	0.65	2.43%
Average		25.53	26.23	0.70	2.76%

Table 10: Width Changes of Plastic Strip for Immersion Testing in Bean-e-doo

for 7 days at Ambient Temperature and 40 °C

Width	Gain	(&)	80	80	80	80	80	0.33%	80	- 0.55%	- 5.01%	- 1.85%	80	80	80	80	80	- 4.89%	0.32%	1.28%	0.10%	0.57%	80	80
Width	Gain	(mm)	0	0	0	0	0	60.0	0	50°0 =	77. 0 -	91.0 -	0	0	0	0	0	- 0.47	0.03	0.12	0.01	50.0	0	0
Width ,		(ww)	9.03	9.12	9.14	9.14	9.13	9.11	6.07	80.6	8.35	8.83	10.23	9.43	9.42	9.43	9.43	9.13	9.47	10.52	9.55	9.85	6.15	6.53
Width o day	(ww.)		9.03	9.12	9.14	9.14	9.13	80.6	6.07	9.13	8.79	00.6	10.23	9.43	9.42	6.43	9.43	9.62	9.44	07.6	₹9.6	97.6	6.15	6.53
Temperature Width o day	(၁့)		Ambient					℃ 70 °C					Ambient					40 °C					Ambient	
Sample			$HDPE_C$	HDPE1	HDPE2	HDPE3	Average	2	HDPE1	HDPE2	HDPE3	Average	IONOMER	IONOMER	IONOMER ₂	IONOMER ₃	Average	IONOMER	IONOMER	IONOMER ₂	I ONOMER ₃	Average	PPc	PP_1

- 1

Table 11: Width Changes of Plastic Strip for Immersion Testing in Bean-e-doo

for 14 days at Ambient Temperature and 40 °C

Sample	Temperature Width o day	Width o day	Width 14	Width	Width
	(၁)	(mm)	day	Gain	Gain
			(mm)	(mm)	(&)
HDPEc	Ambient	9.03	9.03	0	% 0
HDPE1		9.03	9.06	0.03	958.0
HDPE2		9.07	9.10	0.03	0.33%
HDPE3		9.10	9.13	0.03	0.33%
Average		9.07	9.10	0.03	0.33%
HDPEc	J. 07	9.43	9.45	0.02	0.21%
HDPE1		9.21	9.30	60.0	886.0
HDPE2		9.10	9.18	0.08	878
HDPE3		9.02	9.09	0.07	0.78%
Average		9.11	9.19	80.0	888.0
IONOMER	Ambient	9.48	9.50	0.02	0.21%
IONOMER1		10.14	10.19	0.05	0.49%
IONOMER ₂		9.52	9.58	90.0	0.63%
IONOMER3		9.24	9.30	90.0	0.65%
Average		9.63	9.69	90.0	0.59%
IONOMERC	J. 07	9.44	9.45	0.01	0.11%
IONOMER		9.51	9.71	0.20	2.10%
I ONOMER ₂		9.43	9.63	0.20	2.12%
I ONOMER ₃		9.15	9.34	0.19	2.08%
Average		9:36	9.56	0.20	2.10%
$\mathtt{PP_c}$	Ambient	6.55	6.55	0	% 0

PP ₁	PP_2	PP3	Average	PPc	PP1	PP ₂	PP ₃	Average	PETc	PET1	PET2	PET3	Average	PETc	PET1	PET2	PET3	Average	SELARC	SELAR1	SELAR ₂	SELAR3	Average	SELARC	SELAR	SELAR2	SELAR
				40 °C					Ambient					40 °C					Ambient					40 °C			
6.64	99.9	95.9	6.62	6.71	6.80	6.73	6.65	6.73	23.93	24.48	24.09	24.68	24.42	25.16	25.06	25.02	24.93	25.00	25.40	25.43	25.38	25.41	25.41	25.37	24.70	25.41	25.39
99.9	6.70	6.59	6.65	6.68	6.88	6.84	6.74	6.82	23.93	24.48	24.09	24.68	24.42	25.00	24.90	24.99	24.93	24.94	25.45	25.75	25.70	25.68	25.71	25.43	24.97	25.68	25.65
0.02	0.04	0.03	0.03	- 0.03	0.08	0.11	0.09	60.0	0	0	0	0	0	- 0.16	- 0.16	- 0.03	0	90.0 -	0.05	0.32	0.32	0.27	0.30	90.0	0.27	0.27	0.26
0.30%	0.60%	0.46%	0.45%	- 0.45%	1.18%	1.63%	1.35%	1.39%	80	80	80	80	80	- 0.64%	- 0.64%	- 0.12%	80	- 0.25%	0.20%	1.26%	1.26%	1.06%	1.19%	0.248	1.09%	1.06%	1.02%

Table 12: Length Changes of Plastic Strip for Immersion Testing in Bean-e-doo for 7 days at Ambient Temperature and 40 °C

0.26% 0.248 Length 0.26% 0.23% Gain 0.08% 0.48% 1.428 1.40% 1.40% 1.37% æ 0 80 **&** (%) 80 80 80 80 80 80 **%**0 0.16 0.15 Length Gain 0.88 0.85 0.05 0.29 0.14 0.16 0.87 0.87 (mm) 0 0 0 0 0 0 0 0 0 0 0 Length , 60.95 61.09 62.95 61.04 61.05 62.07 62.93 61.05 61.19 61.28 61.17 62.33 62.28 61.77 61.88 61.1261.03 62.04 62.93 62.92 day (mm) Length o 62.28 60.95 61.09 61.12 61.00 60.90 62.33 61.04 61.05 61.19 61.14 61.01 62.07 62.04 62.03 62.06 62.04 62.10 62.07 44.73 61.77 day (mm) Temperature Ambient Ambient Ambient ပွ ပ (၁ 40 40 IONOMER_c IONOMER₁ IONOMERC I ONOMER2 I ONOMER3 IONOMER2 I ONOMER, Average IONOMER Average Average Average Sample $HDPE_{c}$ HDPE1 HDPE2 HDPE3 HDPE2 HDPE3 HDPE1 HDPE PP_{C}

PP ₁		45.40	45.40	0	
PP ₂		45.35	45.35	0	
PP ₃		45.66	45.66	0	
Average		45.47	45.47	0	
PPc	40 °C	67.74	67.23	- 0.51	
PP1		65.92	68.36	2.44	
PP ₂		66.20	68.13	1.93	
PP ₃		66.43	67.19	94.0	
Average	The second second second	66.18	67.89	1.71	
PETc	Ambient	31.26	31.26	0	
PET ₁		32.64	32.64	0	
PET2		32.71	32.71	0	
PET3		33.11	33.11	0	
Average		32.82	32.82	0	
PETc	40 °C	42.43	33.09	- 9.34	
PET1		42.01	33.36	- 8.65	
PET2		39.69	33.28	- 6.41	
PET3		42.41	33.68	- 8.73	
Average		41.37	33.44	7.93	
SELARC	Ambient	31.81	31.81	0	
SELAR		33.58	33.83	0.25	
SELAR ₂		34.19	34.38	0.19	
SELAR3		32.94	33.20	0.26	
Average		33.57	33.80	0.23	
SELARc	40 °C	30.90	30.96	90.0	
SELAR		31.93	32.26	0.33	
SELAR2		32.24	32.61	0.37	
SELAR3		32.62	32.98	0.36	
Average		32.26	32.62	0.35	

Table 13: Length Gain of Plastic Strip for Immersion Testing in Bean-e-doo for

14 days at Ambient Temperature and 40 °C

Sample	Temperature	Length o	Length 14	Length	Length
	(၁)	day	ç	Gain	Gain
		(mm)	(mm)	(mm)	(&)
$HDPE_{c}$	Ambient	61.93	61.96	0.03	0.05%
HDPE1		61.98	62.13	0.15	0.248
HDPE ₂		61.98	62.13	0.15	0.248
HDPE3		62.11	62.27	0.16	0.26%
Average		62.02	62.17	0.15	0.25%
$HDPE_{C}$	℃ 0 7	60.88	68.09	0.01	0.02%
HDPE1		61.29	61.73	0.44	0.72%
HDPE ₂		61.06	61.45	0.39	0.64%
HDPE3		61.00	61.41	0.41	0.67%
Average		61.12	61.53	0.41	0.68%
IONOMER	Ambient	62.88	62.95	0.07	0.11%
IONOMER1		62.89	63.30	0.41	0.65%
IONOMER ₂		62.84	63.24	0.40	0.648
IONOMER ₃		62.85	63.25	0.40	0.64%
Average		62.86	63.26	0.40	0.64%
IONOMER _C	℃ 40 °C	62.04	62.09	0.05	0.08%
IONOMER		62.02	63.38	1.36	2.19%
IONOMER ₂		62.00	63.46	1.46	2.36%
IONOMER3		62.03	63.50	1.47	2.378
Average		62.02	63.45	1.43	2.318

48.7548.7948.9249.0149.9550.0348.6848.7749.1849.2763.2463.2962.7763.11
+
39.86 39.95 39.14 39.14
40.59 40.59 40.18 40.18
H
32.08 32.02 31.90 31.84
31.83 31.77
\dashv
+
-
32.28 32.63
29.63 29.78
30.71 31.04
33.25 33.62
31.85 32.20

Results of Bubbling Test

Table 14: Bubbling Test of Plastic Pieces

Plastic sample	Time of experiment (days)	Results
HDPE	131	No bubbling
PET	131	No bubbling
IONOMER	131	No bubbling
PP	131	No bubbling
SELAR	131	No bubbling

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