EASTMAN

Recycle Studies with Eastar® EB062

May 1

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This white paper presents the study conducted to assess the recyclability of Eastar® EB062 and its compatibility with the current PET PCR stream.

Specialty Plastics Business Organization

Introduction

What is Eastar® EB062?

Eastar® EB062 is a copolyester manufactured by Eastman Chemical Company for use in the extrusion blow molding process. EB062 enables brand owners to develop packaging that combines innovative designs, shelf appeal, and an ergonomic use experience that will support more premium positioning, maintenance of brand identity, greater awareness and consideration on the shelf, and consumer satisfaction.

Eastar® EB062 is not designed for use in injection stretch blow molding (ISBM) applications. Rather, it is a modified PET product with similar aesthetics but with inherently higher costs. The vast majority of EB062 is used in clear handleware applications in the food, beverage, and consumer packaging market. It is not cost competitive with ISBM PET products in non-handleware applications.

EB062 is not PETG and is much more tolerable than PETG in the PET recycle stream. As such, it can be used as a replacement to PETG or a replacement to extrusion blow molded PVC.

Based on Eastman's forecasts, Eastar® EB062 will be present in the North American market at less than 100M lbs. by 2018. In that year, it is estimated that there will be 12.6B lbs of ISBM PET in the North American market, whereby EB062 would represent no more than 1% of the total PET volume.

Purpose of this Study

This study will help one understand the potential impact of EB062 on the PET post-consumer recycle (PCR) stream. The amount of EB062 sold into the marketplace and how much gets recycled are key factors in this assessment. Additionally, a high-value demand exists for EB062 PCR and post-industrial recycle (PIR) in both non-food grade packaging as well as extruded PETG sheet. While Eastman will work with converters and brokers to ensure the EB062 post-industrial recycle (PIR) is directed to the high value markets that exist for it (and not directed into the PET PIR stream) certain amounts of EB062 will still enter the PET recycle stream. Beyond the options of hand sorting along with PVC handleware or auto-sorting using near-infrared technology, this study attempts to assess the impact of the EB062 that remains in the PET PCR stream.

Recycling Facts

A key part of this study involved understanding the amount of EB062 that would enter the PET PCR stream AND understanding how big that PET PCR stream may be. NAPCOR's (National Association for PET Container Resources) 2006 Report on Post Consumer PET Container Recycling Activity[1] provided several important numbers in this assessment. First, the report notes that in 2006 there were 5.424B lbs of PET bottles on U.S. shelves. From 2002 to 2006, the compounded annual growth rate of PET bottles on U.S. shelves is 7.9%. Therefore, we can estimate that in 2010 there will be approximately 6.870B lbs of PET bottles on U.S. shelves (12.6B lbs in 2018). The recycling rate, which had been in the range of 19.9%-23.1% since 2002, was 23.5% in 2006. Furthermore, the recycling rate over the last 11 years has been an average of 23.5%. We applied this recycling rate to the 2010 volume of PET bottles on U.S. shelves and determined that approximately 1.6B lbs of PET would make up the PET post-consumer recycle stream in 2010.

To compare Eastar® EB062 to this number, Eastman forecasted the amount of EB062 that will be in the marketplace in 2010 and applied our own safety factors that rounded this number up to 100M lbs. Because recyclers typically attempt to cull all clear handleware bottles from the recycle stream (to reduce the chance that clear PVC handleware bottles will contaminate the stream), the ultimate recycling rate for clear handleware or custom multi-serve containers is about 10%. This 10% applied to the 100M lbs. of EB062 taken to be sold in the U.S. in 2010 leaves 10M lbs. of EB062 to integrate into the 1.6B lbs PET PCR stream. This is a 0.6% loading level. Applying a 5X spiking factor takes this loading level to only 3.1%.

These loading levels, volumes, and recycling rates are important factors in the rest of this study.

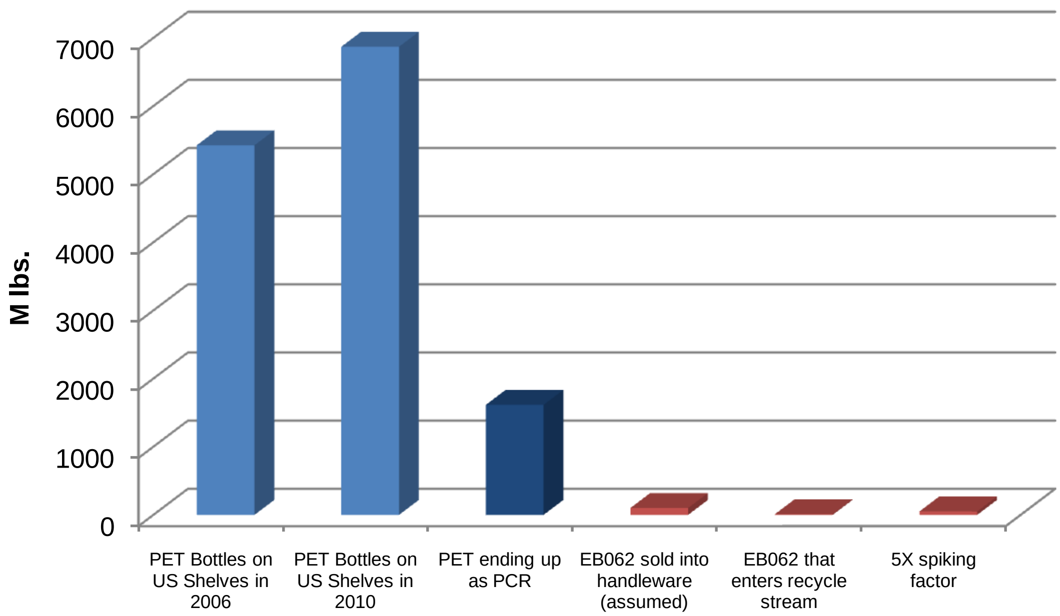

Figure 1 - PET PCR Stream vs. EB062

[1] www.napcor.com/plastic/bottles/reports.html

APR PET Critical Guidance Protocol

Typical PET Post-Consumer Recycle Procedures

Recycling is not a charitable event. Recyclers are ultimately attempting to convert waste into profitable products. Although a few end use products (such as plastic lumber) can utilize commingled plastic materials, the vast majority of end use products require well separated incoming materials to make their operations profitable. Generally, recyclers will not hesitate to send poorly sorted or contaminated incoming materials into the landfill…while acknowledging the potentially lost profit. Thus, to maximize the profitability of their operations, recyclers demand high quality incoming materials.

Most of the plastic bottles recycled today originate from curbside collection programs. Although some programs require that homeowners or collection crews separate materials based on SPI resin identification code, many curbside collections are "single source" (i.e. commingled glass, aluminum, steel, paper, cardboard, and plastic). Single source collection relies on the material recovery facility (MRF) to perform basic separation and has the greatest potential for cross-contamination. Higher purity source materials are found where municipalities or states have mandated bottle deposit laws or use drop-off centers, but some cross-contamination can occur even in these situations.

Most plastics are sorted at the bottle (not flake) level and this operation generally takes place at the materials recovery facility (MRF), or at an intermediate processing center (IPC). Sorting of commingled recyclable materials is an especially labor-intensive (i.e. expensive) effort since the plastic must first be separated from the glass, aluminum, steel, paper, and cardboard. Sorting of whole bottles can occur manually (by visual inspection) or automatically (via optical detection systems).

In manual sorting, flattened and crushed bottles are passed on a conveyer in front of a team of human observers. The operators attempt to separate acceptable bottles via shape, color, and/or product recognition. However, human error naturally limits the accuracy of this method. The containers, crushed to reduce the cost of transportation, now are virtually unrecognizable to the human eye. In addition, some bottles of the same design may be fabricated from different plastic polymers.

In automatic sorting, machines utilizing an X-ray, visual light and/or near-infrared analysis, can identify and separate plastic bottles based on color, resin type or both. Automatic sorting can greatly improve the quality and efficiency of the separation process, but the technology may be more expensive than a MRF or IRF can afford and is still not 100% efficient.

Most plastic bottles contain an SPI (or similar) resin identification code which supposedly facilitates recycling efforts, but in reality these stamped codes are of limited value to manual sorting personnel, as the rate at which the bottles pass on the conveyer precludes looking at the bottom of every bottle passing by. Slowing the conveyer rate would render the system unprofitable. These codes are also meaningless to automatic sorting equipment. Thus, these codes merely reflect the general recycle stream in which a material is best suited.

After sorting, the bottles are ground into flake. The flake is then air elutriated to separate light material, such as labels, from the heavier flake. The flake is then washed to remove any remaining dirt, ink and glue. The cleaned flake is then run through a water bath to further separate any residual material from caps, basecups, attachments, etc that might have been on the bottle. Materials with a density greater than one are separated from those below one in this process. This process separates the PP caps from PET bottles, for example. Note that it is also possible to "auto-sort" flake at this step in the process.

Sorted, ground, and washed PET PCR flake is then subjected to some or all of the following procedures, depending on the final end use. Typical end used for PET PCR flake include fiber (49%), bottles (22%), strapping (15%), and film (9%). (Percentages are based on the 2006 NAPCOR Report on Post Consumer PET Container Recycling Activity.) It is in these steps where EB062 could cause issues.

- Drying
    - PET PCR flake is typically dried at 160°C for 4 hours and the new material must not cause flakes to stick to the walls of the dryer or cause clumping.
- Pelletizing
    - The new material must not change the IV after pellitization or compromise the ability to filter contaminants during pelletization.
- Solid Stating
    - The new material must not change the solid state rates needed to obtain the specified final IV of the PCR-based product.
- End Use
    - The color, haze, and amount of black specs of the final PCR product must not significantly increase due to introduction of the new material. The melt point and crystallizability of the final PCR-based product must also not be affected.
    - The manufacturability and properties of the primary end uses (fiber, strapping, film, bottle) must not be adversely affected.

Protocol Guidance

The Association of Post-Consumer Recyclers (APR) has developed a PET Critical Guidance Document protocol that helps an innovator evaluate many of these concerns, including solid-state rate, melt point, color, haze, black specs, etc. This protocol is similar to that proposed by PETCORE in Europe. The testing called for in the protocol involves:

- Selection of an appropriate control material
- Caustic washing of the control and test resin
- Separate extrusions of the control resin and test resin at conditions that would simulate their respective primary use processing conditions

- A second extrusion of the control resin and mixtures of the test resin at 25% and 50% in the control resin
- Solid-stating of the extruded control resin and extruded mixtures
- Molding of the solid stated control and mixtures into plaques
- Assessment of properties – IV, melt point, color, haze

The protocol suggests that if the new material is intended for use in ISBM applications, it should be tested after blending at 25% and 50% levels into a control material. For this study, Eastman's CB12 PET was used as a control, as listed in the APR PET Critical Guidance Protocol. At these loading levels, the haze and color of the EB062 mixtures were unacceptable, with haze values greater than 20%. Nevertheless, since EB062 is intended for extrusion blow molded (EBM) handleware applications and not injection-stretch blow molding applications, EB062 is expected to be present in the marketplace at less than 1% compared to ISBM PET (less than 100M lbs of EB062 alongside 12.6B lbs of PET in 2018). As noted in the APR Critical guidance document,

> *"The testing called for in this document is **intentionally rigorous** with regard to test concentrations of the innovation 25 and 50%. As stated in the preamble, "Inability of an innovative bottle to meet specific critical values **does not imply recycling failure**, but should be a clear message that a **significant issue might exist** under certain circumstances and **mitigation of the issue may be needed** to avoid degrading the value of the stream of recyclable bottles". APR's Criteria to consider when evaluating the recyclability of a PET variant in the PET bottle stream" suggests the **variant be evaluated at a multiple of the expected market penetration**. The multiples suggested are between 2 and 10. A test at 5 times the expected developed market penetration is frequently used to reflect actual recycling impact."*

Therefore, this study was rerun at lower levels, at various multiples of the anticipated market penetration rate of EB062 (1%).

Evaluation at Market Multiples

To assess the effect in the general post-consumer stream, EB062 was blended into PET at various multiples of expected market penetration, according to the sample preparation procedures defined in the Protocol. These blends were tested according to the test methods defined in the Protocol and the test results compared against critical guidance values.

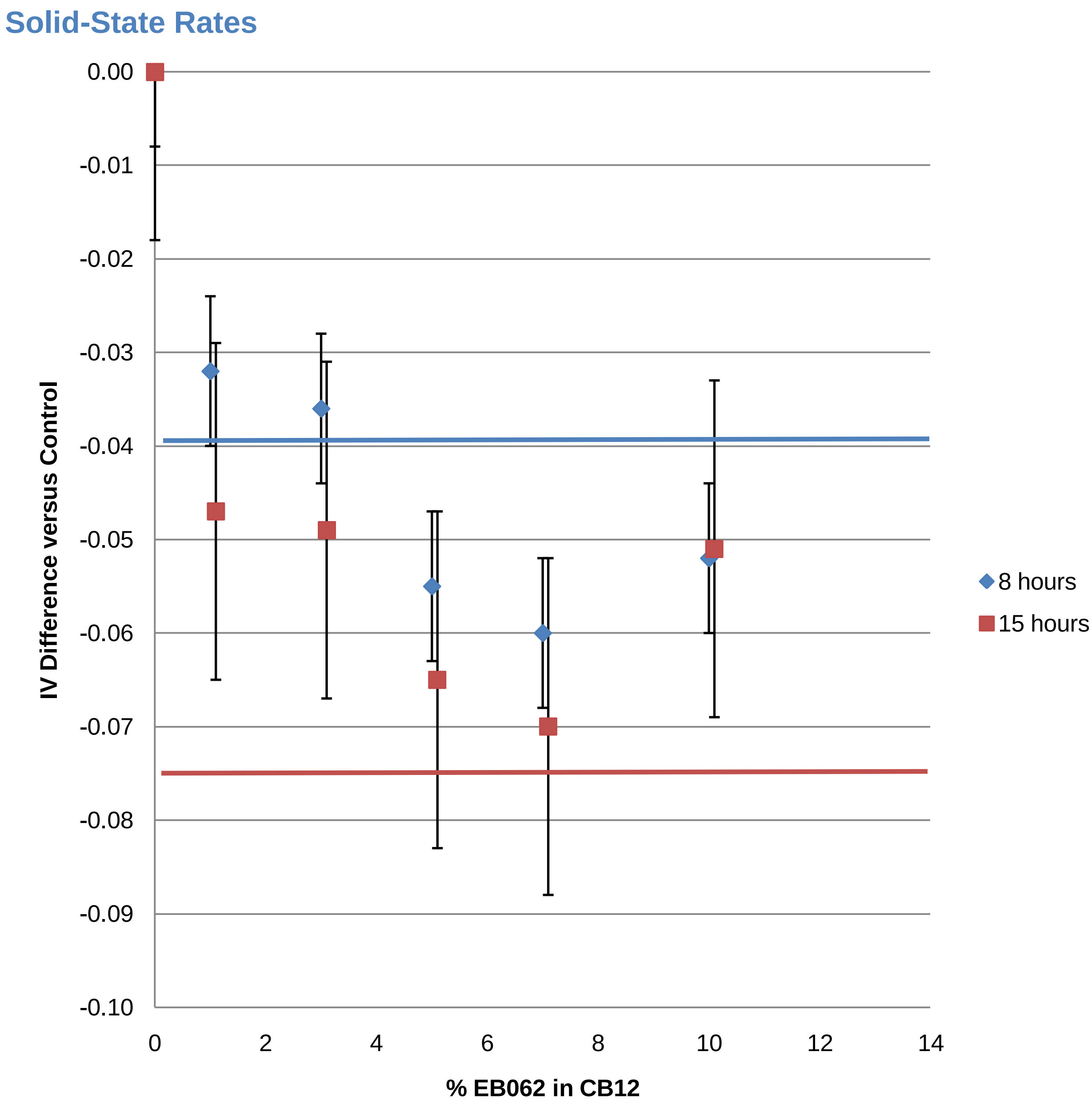

Figure 2 - Solid-State Rates

The Protocol states that at 8 hours, the IV difference versus the control should not be greater than 0.040 and at 15 hours, not greater than 0.075. In figure 1, it can be seen that EB062 has a difference of less than 0.040 up until about 4%. At 15 hours, EB062 is less than a difference of 0.075 all the way to 10% loading.

2^{nd} Scan T_m of Solid-Stated Pellets

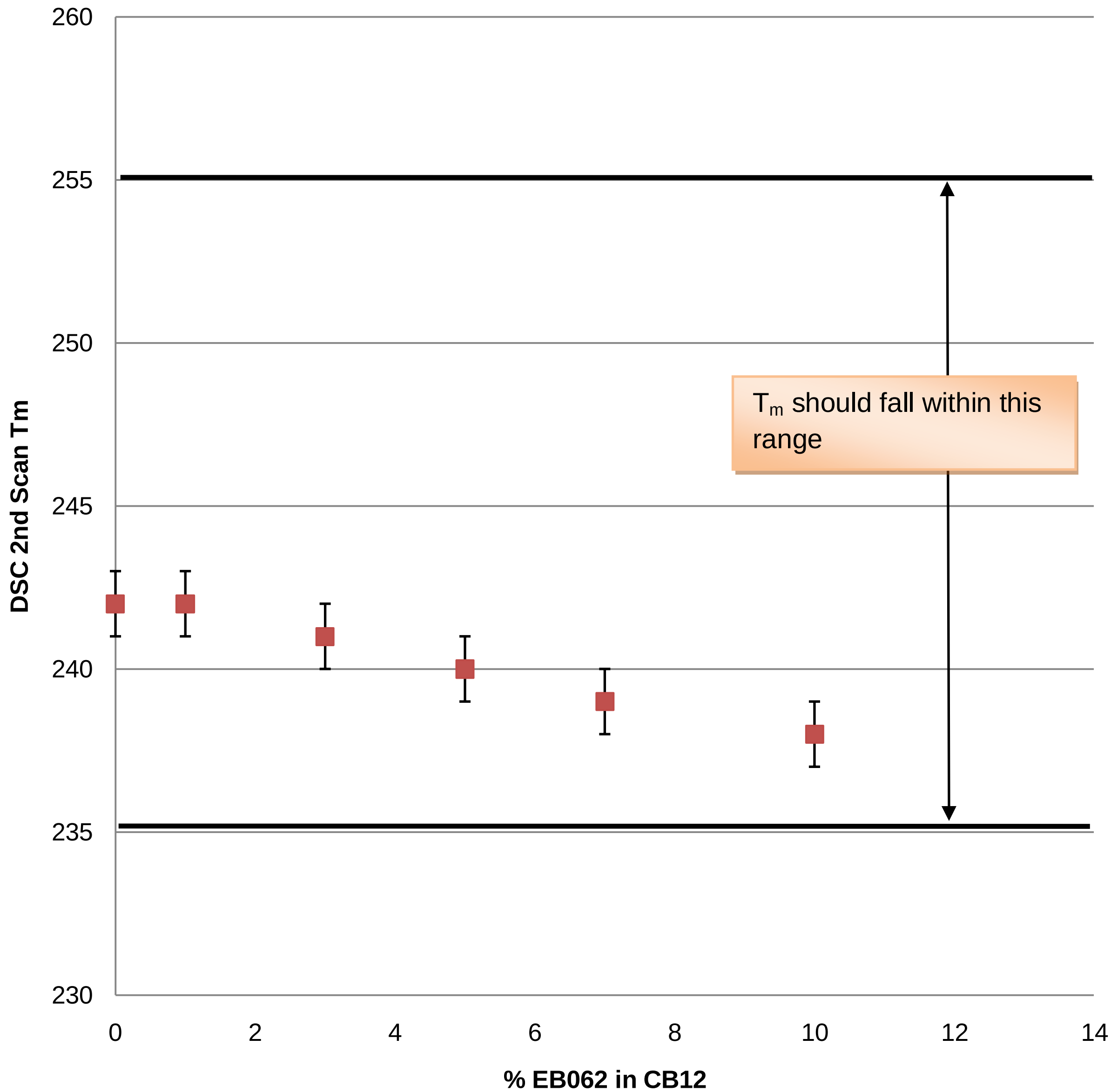

Figure 3 - 2nd Scan T_m of SS Pellets

For the differential scanning calorimetry (DSC) 2^{nd} scan melt point, all of the loading levels tested for EB062 (0 to 10%) fell within the acceptable range of 235°C to 255°C.

% Haze in 3.2mm Plaques

Figure 4 below shows the results for various loading levels of EB062 in CB12 for haze measured in 3.2mm plaques. The APR Protocol identifies three regions of results: “should not be a problem”, “needs study”, and “likely noticeable”. None of the plaques tested with EB062 fell within the “likely noticeable” region.

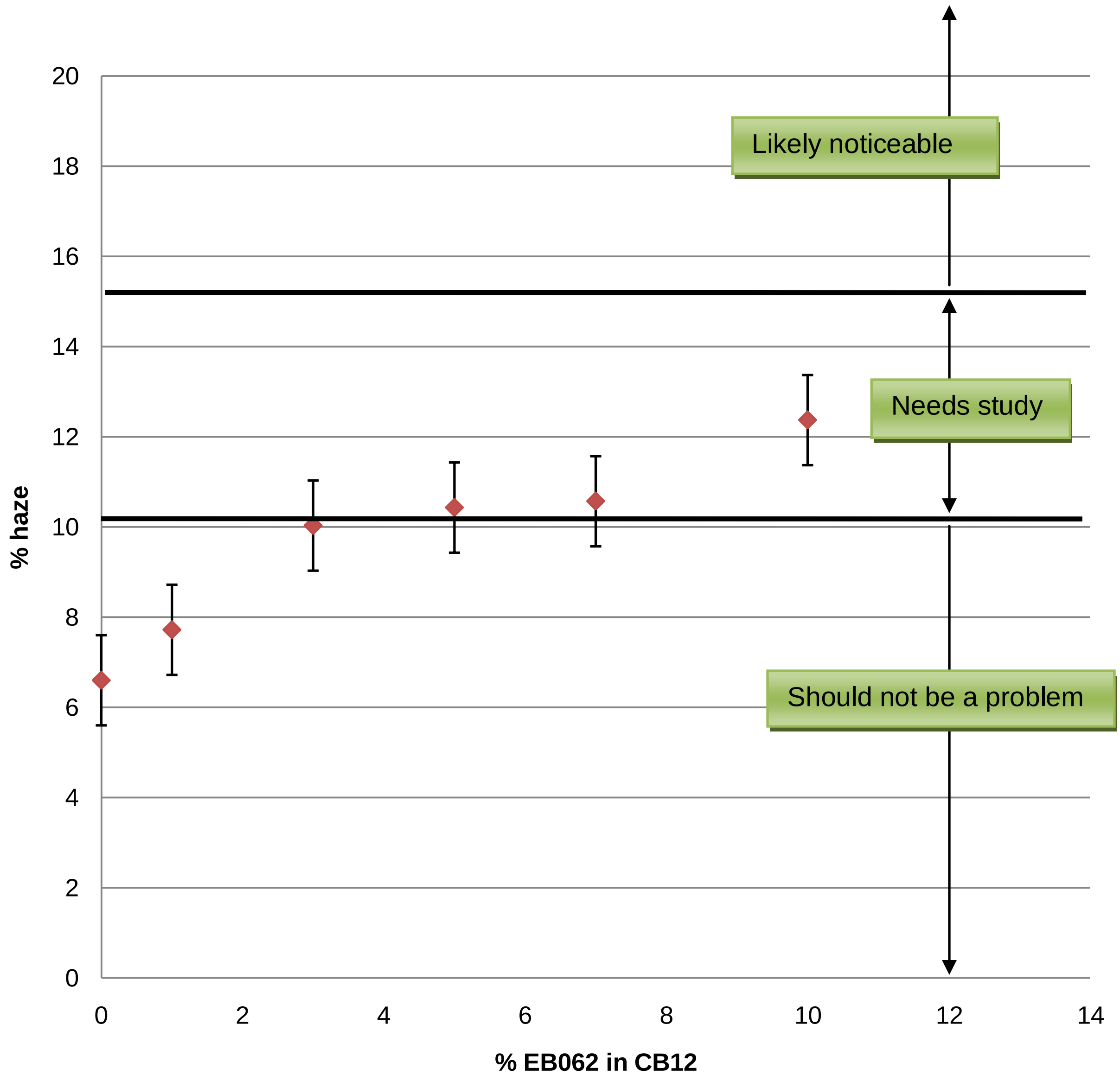

Figure 4 - % Haze in 3.2mm Plaques

L* in 3.2mm Plaques

Figure 5 below assesses L*, with L* = 0 meaning black and L* = 100 meaning white. The Protocol indicates that L* below 82 could be a problem, while for clear PET L* should be greater than 82. As shown below, all tested loadings for EB062 were greater than 82.

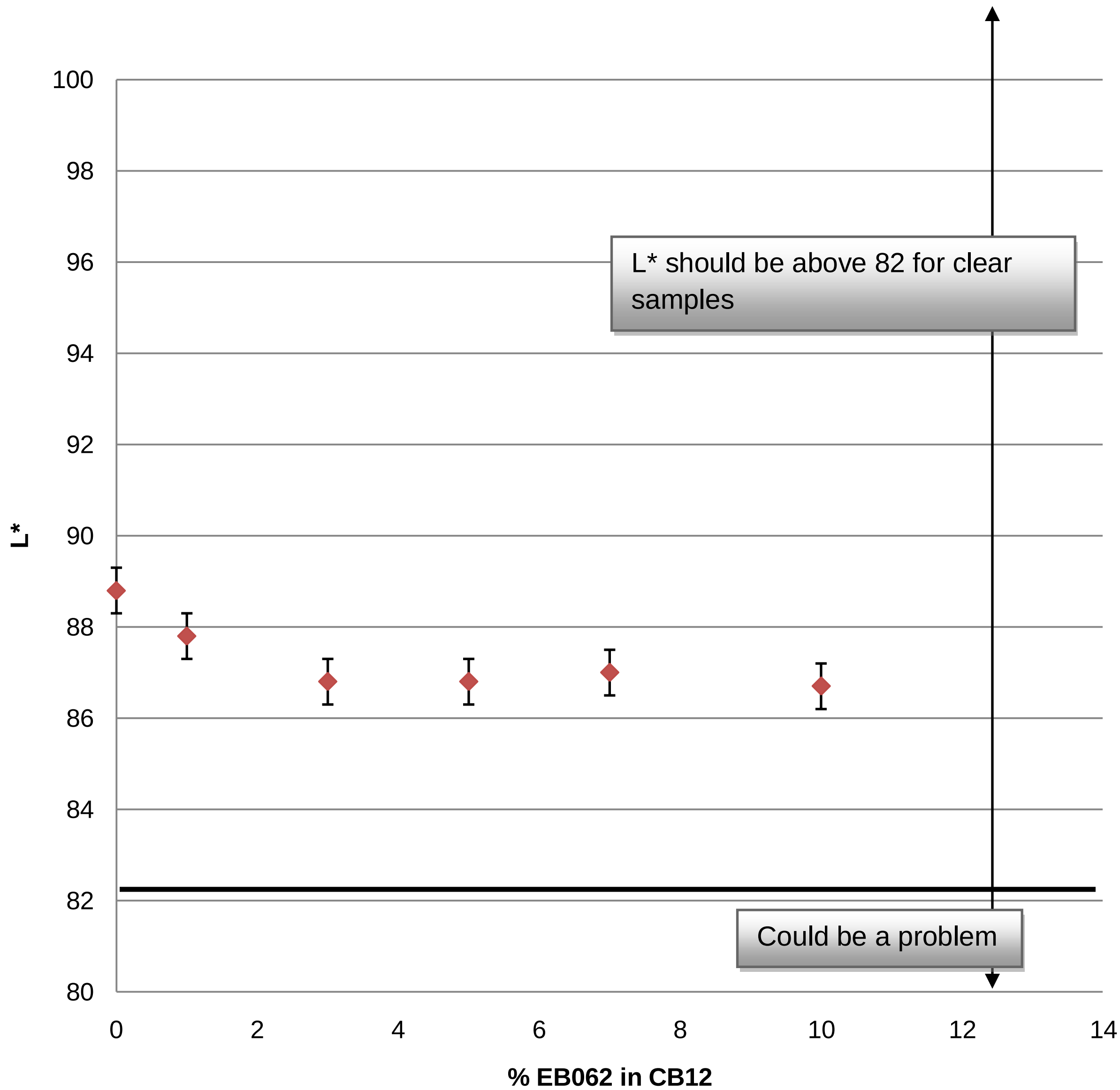

Figure 5 - L* in 3.2mm Plaques

Δb* in 3.2mm Plaques

The APR Protocol states that Δb* less than 1.5 should not be a problem, between 1.5 and 5.5 needs study, and greater than 5.5 is unsuitable for many applications. All tested EB062 plaques were below 5.5. Δb* is essentially a measure of yellowness.

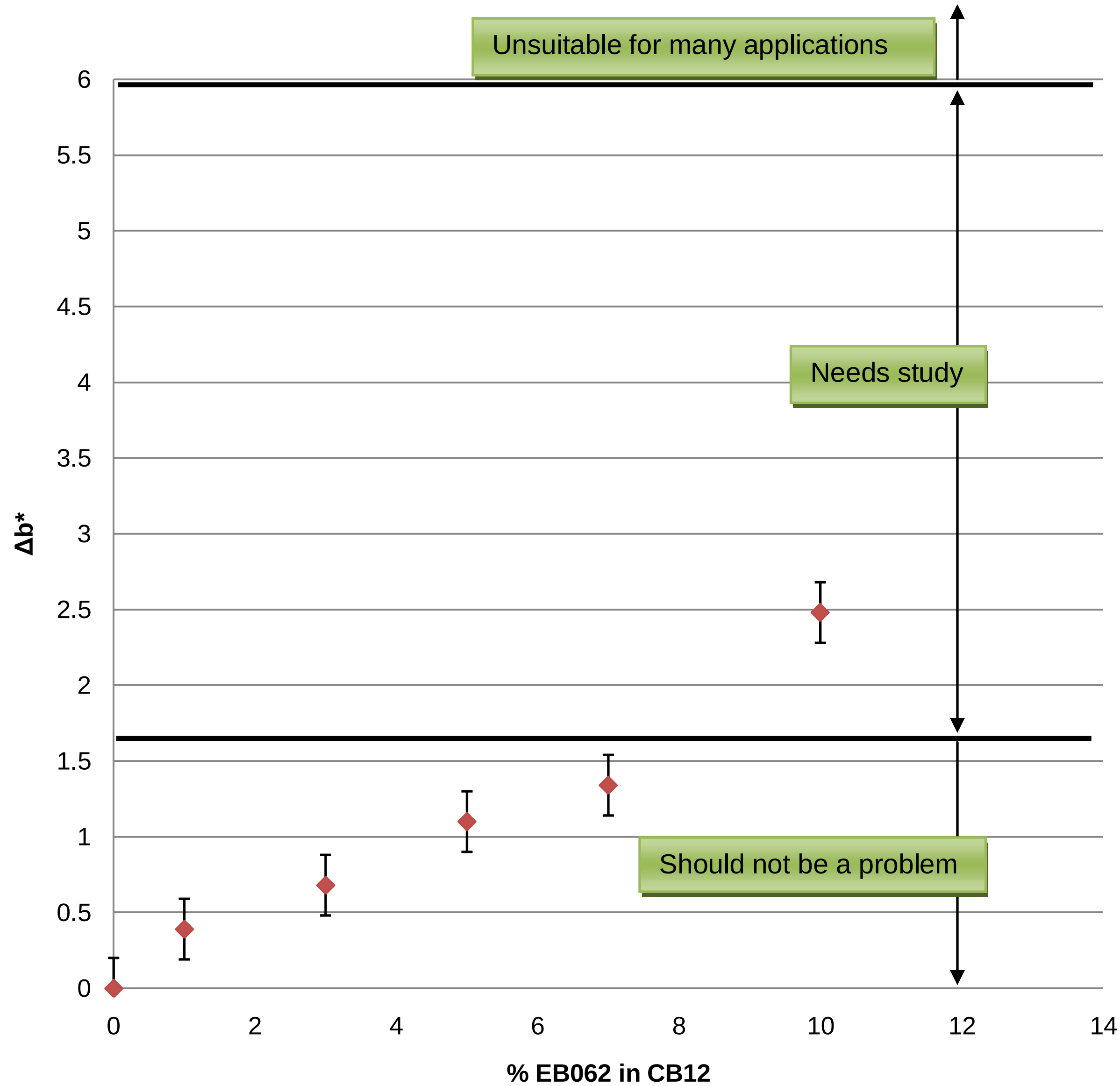

Figure 6 - Δb* in 3.2 Plaques

Summary

Following the test methods and procedures described in the APR Critical Guidance Protocol, blends of CB12 (PET) with up to 3% EB062 yielded results that fall within Guidance values. In many instances, data for CB12 loaded with 10% EB062 pass or do not deviate excessively from the guidance values. This is an important assessment given that EB062 is intended for EBM handleware applications and is expected to be present in the marketplace at less than 1% relative to PET…and may likely take 10 years to reach this level.

Dryer Evaluations

PET Drying

In addition to testing according to the APR Critical Guidance Protocol, Eastman conducted numerous internal dryer studies designed to duplicate the drying systems at many recyclers. Drying is a critical part of the recycling process. PET post-consumer recycle flake is dried at the same drying conditions as virgin PET (160°C) prior to pelletizing. PETG becomes tacky when dried higher than 70°C because it will not crystallize. Therefore, in the PET PCR drying conditions, PETG sticks to the sidewalls of the dryer and creates clumps of PCR (sometimes referred to as “popcorn”) that will not convey from the dryer. It is important to again note that EB062 is **not** PETG. EB062 **will** crystallize during the drying process and prevent tacking.

Dryer Test Protocol

The test protocol developed in the Eastman lab was designed to duplicate the dryer setting found at many recycling facilities.

1000 lbs Plug Flow Dryer Test

A 1000 lbs mass flow drying hopper was loaded with PCR flake from URRC (United Resource Recovery Corporation) and set at 160°C. The dried flake was discharged at 180 lbs/hr while the dryer was refilled with the test blend, exposing the blend to the 160°C drying temperature for 4 hrs. The dryer was kept full during the entire evaluation. The discharge orifice was 2” in diameter. The material discharged from the dryer was passed over a ½” screen to capture and collect any clumped material. When the test was concluded, the dryer was opened to observe any sticking on the walls.

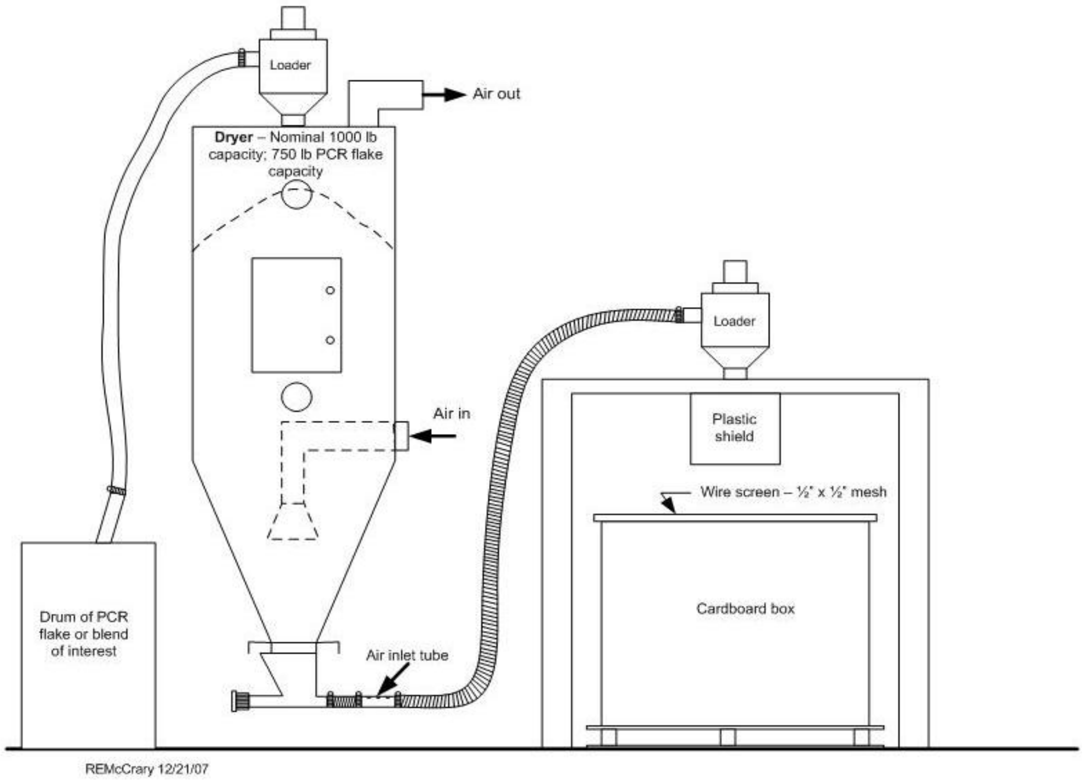

Figure 7 - PCR Dryer Test Arrangement

Figure 8 - Dryer Test Arrangement

Figure 9 - "Popcorn" Caught on Screen

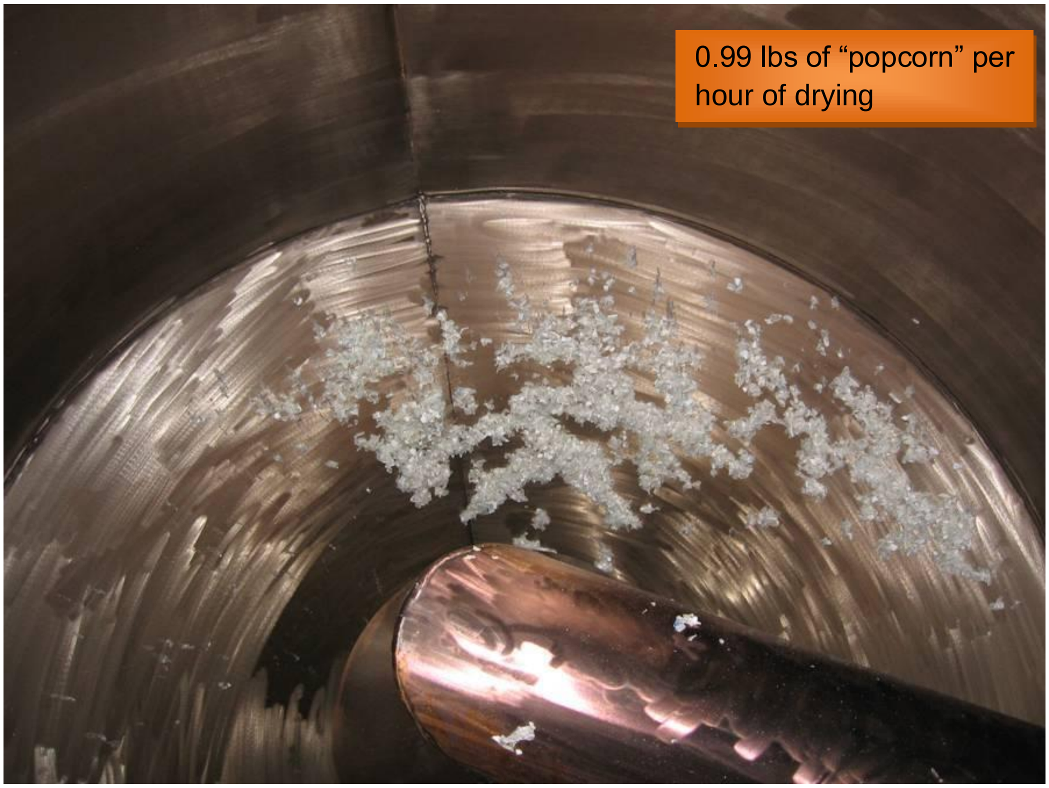

Figure 10 - PCR Flake w/1% PETG Inside of Dryer Cone after Testing

Figure 11 - PCR Flake w/5% EB062 Inside of Dryer Cone after Testing

Figures 12 and 13 below show the relative size of clumps of EB062 at 5% loading versus PETG at 5% loading.

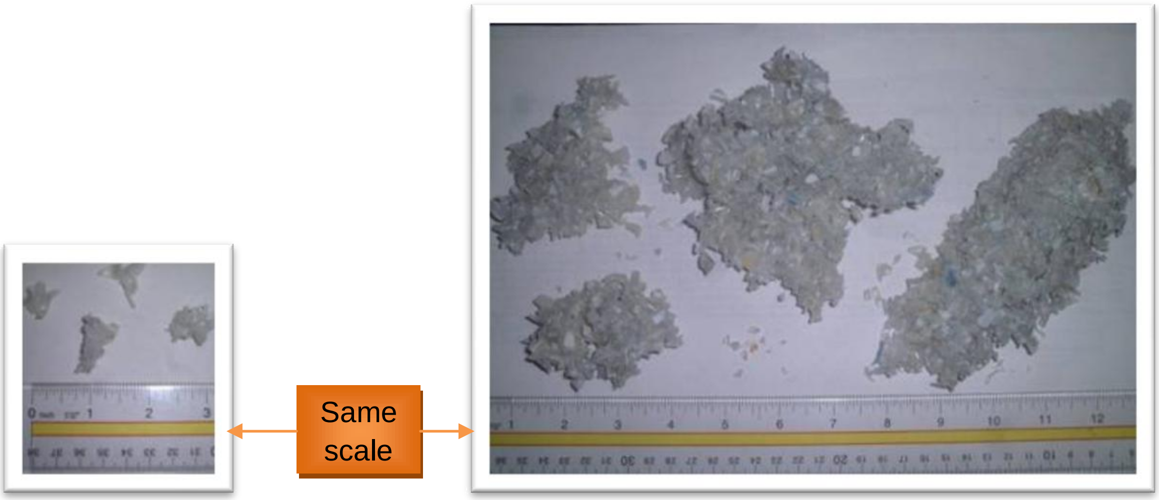

Figure 12 - EB062 at 5% Loading

Figure 13 - PETG at 5% Loading

40g Can Dryer Test

A 40g sample was placed in a can 2 inches in diameter and 3 inches deep. This can was then placed for 4 hours in an oven set at 160°C. To simulate a 12ft. tall dryer, 5 lbs of total weight were applied to the top of the can. This was done by adding 1 lb per hour to simulate flake traveling down the dryer. At the end of the 4 hours, the percentage of material that formed clumps greater than ½" in diameter was measured. Only 1% clumping was noticed at a 10% loading level.

Table 1 - 40g Can Dryer Test

% EB062 regrind in PET PCR	160°C / 4 hrs. % clumped
1	0
2	0
5	0
10	1

75 lbs Static Dryer Test

50 lbs samples were placed into a 75 lbs Conair static dryer set at 160°C. 0%, 2%, 5%, and 8% EB062 regrind flake were mixed into PET, and both PET flake (PCR) and PET pellets (CB12) were used in this test. (Pellets were used to allow the extrusion of film.) 6 lbs of weight were placed on top of the flake to simulate the additional mass of material in a 9 ft tall dryer. (Note that the pellets/flake were not moving within the bed during drying.) After 4 hours, the dryer was turned off, the 2" diameter material discharge was opened and the flow behavior was noted. The discharge material was then passed through a 0.36" screen and the total amount of

clumped material was captured and weighed. 20 mil film was extruded from the pellet samples for haze and color measurements.

Table 2 - 75 lbs Static Dryer Test

	Clumped Material in 50 lbs. (%)	Relative Flow	Film Haze (%)	Film (b*)
100% PET 0% EB062	0	Free flow	2.2	0.38
98% PET 2% EB062	0	Free flow	1.7	0.57
95% PET 5% EB062	0	Some slight tapping required	1.7	0.85
92% PET 8% EB062	0.06	Flow problems	2.1	1.03

5 ft^3 RVD Test

To make the samples, 1% and 5% EB062 regrind flake were mixed into PET PCR. A 50 lbs sample was placed into a 5 ft^3 rotary vacuum dryer (RVD) set to provide a flake temperature of 160°C. After 4 hours, the vacuum was released and the dryer was opened for observation. Any flake sticking to the walls of the dryer was noted. When the dryer cooled, the material was discharged and weighed. The weight of the discharged material was subtracted from the initial weight to determine the amount of the sample that stuck to the walls. The discharged material was also passed through a 0.36" screen and the total amount of clumped material was captured and weighed. In the table below, the "total % stuck" is the amount that stuck to the walls PLUS the amount clumped.

Table 3 - RVD Test

% material in PET PCR	Flake Temp (°C)	Total % Stuck
1% EB062	160	0.0
5% EB062	160	0.0

Dryer Study Summary

This dryer study suggests that EB062 should not cause problems in typical dryer configurations when present at forecasted loading levels (from less than 1% to 5%). This loading is significantly higher than the amount of PETG that can be tolerated. Again, EB062 is expected to be present in the marketplace at less than 1% compared to PET. 5% loading represents a 5X spiking factor.

[Note: Not all dryers have the configurations represented in this study.]

End-Use Testing

Bottle Testing

In addition to the dryer evaluation and the APR tests, Eastman also studied the impact of EB062 PCR on bottles properties in end-use testing. Five samples were made, labeled A, B, C, D, and E. Each sample was made of EB062 and bottle-grade PET (CB12). The blends were extruded into pellets and the pellets were solid stated for 5 hours at 215°C.

A	CB12
B	CB12 + 0.5% EB062
C	CB12 + 1% EB062
D	CB12 + 2% EB062
E	CB12 + 5% EB062

The blends (A-E) were pellet mixed with CB12 at a 30/70 ratio to represent the PCR blends (A-E) being added to virgin PET at 30% loading. These final blends were then molded into preforms and blown into ½ liter bottles. (A control sample of 100% CB12 was also blown.) Sidewall haze, sidewall % crystallinity, bottle permeability, 24-hour bottle headspace acetaldehyde (AA), bottle burst properties, and free-blow behavior were measured and analyzed.

The key takeaway is that bottle properties do not significantly change with the addition of up to 5% EB062.

Table 4 - Bottle Properties

Preforms of	Sidewall Crystallinity (%)	Sidewall Haze (%)	Bottle O2 Permeability (ul/pkg/day)*	Free-blow Circumference (in)	Free-blow Volume (cc)
# of replicates			1-2	5	5
100% CB12	35.1	1.33	40	14.92 ± 0.3	2091 ± 112
CB12 w/ 30% A (0%)	34.6	1.11		13.56 ± 0.5	1724 ± 108
CB12 w/ 30% B (0.5%)	36.2	1.46	41	13.19 ± 0.9	1663 ± 232
CB12 w/ 30% C (1%)	34.0	1.34	40	14.95 ± 0.5	2095 ± 142
CB12 w/ 30% D (2%)	32.4	1.37	43	13.98 ± 0.6	1939 ± 224
CB12 w/ 30% E (5%)	32.3	1.26	42	13.93 ± 0.9	1831 ± 208

* - standard deviation of ±1.6

Table 5 - Bottle Burst Properties

	% Expansion (13s/130psi)	% Expansion (burst)	Burst Pressure (psi)	Test Time (s)
100% CB12	6.0	46.1	179	28
CB12 w/ 30% C (1%)	5.4	40.4	181	27
CB12 w/ 30% E (5%)	5.1	41.6	190	29

Other End Use Testing

The table below summarizes additional testing Eastman performed with external recyclers.

Table 6 - End-Use Testing Summary

	Tests Completed	Results
Bottles	Bottles were produced from a 70/30 mixture of CB12 with solid stated simulated recycle material. The simulated material contained up to 5% EB062.	No effect on bottle stretching characteristics or properties.
Food Grade PTI	2.5% and 5% EB062 was run in an RVD food-grade process simulation.	No sticking was observed. A very small number of clumps were found.
Food Grade URRC	3.5% and 5% EB062 was run in the URRC food-grade process.	No operational problems were encountered but there was an increase in the amount of ejected discolored particles.
Fiber	5% undried EB062 was run in a fiber process (the PET was dried) at a fiber production facility and carpet samples were made.	No processing problems were encountered. Fiber passed all physical tests. Carpet passed all aesthetic tests.
Film	2.5% and 5% EB062 in PET was run to generate 0.025” film at a PET film producer.	The 2.5% ran without issue and good film was produced. The 5% caused some process/drying problems.

Conclusions

Eastar® EB062 is a copolyester manufactured by Eastman Chemical Company for use in the extrusion blow molding process to produce clear handleware containers. It is not an ISBM PET resin and is not intended to replace PET in non-handleware applications. EB062 is expected to be present in the marketplace at less than 1% compared to PET. For the amount that enters the steam, EB062 handleware can be hand or machine separated from PET as its own stream or with PVC handleware. In spite of sorting efforts, a certain amount of EB062 will enter the PET recycle stream. While EB062 does not meet the rigorous 25% and 50% loading called for in the APR's Critical Guidance Document, it can be tolerated when present at reasonable multiples of the expected market penetration.

To prevent excessively high loadings, Eastman will work with converters and brokers to ensure that EB062 PIR is directed to the high value markets that exist for it and not put into the PET PIR stream.