# Using Near-Infrared Sorting to Recycle PLA Bottles

## Introduction

Plastic bottle recycling provides value to the environment and society because of the recognized environmental and economic benefits.  Recycling diverts material from alternative waste streams such as land filling or incineration, as well as conserves natural resources and energy.  It also provides economic incentives in terms of sellable post consumer and post industrial flake.

Today, plastics are being recycled in a number of different ways.  These techniques include mechanical, chemical, or thermal recycling.  For post-consumer bottles, the current waste disposal and recovery system relies primarily on mechanical recycling.  This method involves the process of collection, sortation, and separation of the bottles into various streams for selling and downstream reprocessing.

Polyethylene terephalate (PET) and high density polyethylene (HDPE) make up a large percentage of the plastic bottles that get recycled.  In the last several years, new plastics made from renewable resources such as plant-based materials, have come onto the market. Some of these biopolymers are made from polylactic acid (PLA).  NatureWorks LLC proprietary PLA-based biopolymer is marketed under the Ingeo™ trademark.

Since PLA bottles look and feel similar to PET bottles, recyclers often consider material identification between the two difficult.  Because of this, the possibility of mixing the different materials together exists.  As a result, there is concern in the recycling community that PLA bottles, at high enough levels, would contaminate the PET recycle stream due to chemical and thermal property differences.  These differences could affect down stream processing and final product properties.  The inclusion of PLA bottles is also considered to take away value in the PET recycle stream by creating problems with sortation efficiency, accuracy, and potential yield loss.

Because PET bottles account for the majority of the clear plastic bottles that get used and recycled, there needs to be enough critical mass in the market place to justify the economics of creating an independent PLA bottle recycling operation or separate stream.

In the last decade, biopolymers have been making good progress in terms of technical improvements and market acceptance.  The growth of this industry continues to be strong, signifying society's demand for more sustainable products and market development.  For example, PLA offers a number of environmental and sustainable benefits; such as being renewable resourced based, having a lower carbon footprint, using less fossil fuel, and creating less green-house gas emissions than conventional polymers.   Biopolymers also offer the opportunity for innovation and the creation of

“green” or “cutting-edge” business development and job growth. With the advances of technology, equipment manufacturers have developed systems that can automatically sort different types of plastic. Because the recycling industry has shown concern about how to identify and effectively sort PLA bottle from PET bottles to prevent contamination, NatureWorks LLC has over the last several years evaluated the ability of a number of these sorting technologies, with positive results.[1]

In order to continue to introduce Ingeo™ biopolymer into the plastic bottle market in a responsible way, NatureWorks LLC and Primo Water Corp. conducted a commercial-scale bottle recycling evaluation to demonstrate that automated systems being used today in the recycling industry are capable of separating PLA bottles from PET bottles with good accuracy and efficiency. In this evaluation, near-infrared equipment was used since it is a common sortation technology in large recycling operations and can accurately identify many different types of polymers.

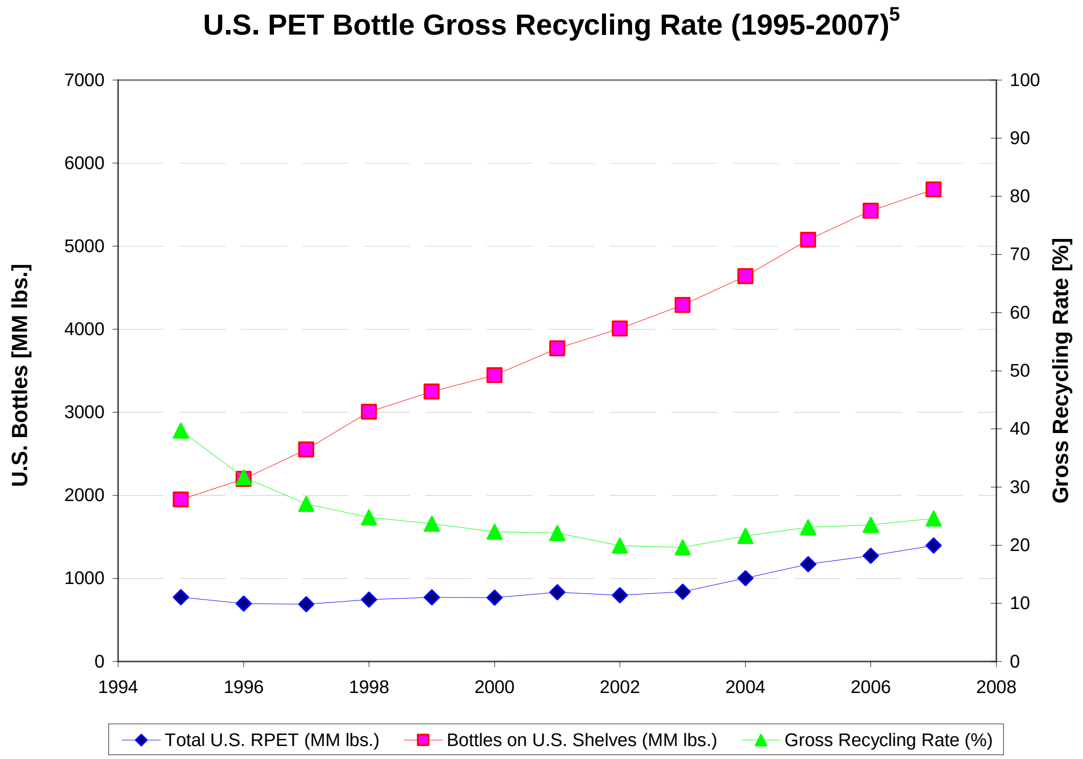
## Background

According to the United States Environmental Protection Agency (EPA), the U.S. generated approximately 254 million tons of trash in 2007, but only recovered 33.4% of this material.[2] This recovery was either through recycling or composting efforts.

As a percentage of the 254 million tons of trash, plastics make up 12.1% of this waste. Even though only 6.8% of all plastics get recovered, some types of plastic articles get recycled more than others. In particular, higher recovery rates are seen for plastic bottles. According to the 2007 EPA report, the recovery of PET soda bottles was 37%. Water bottles and HDPE milk bottles were recovered at about a 28% recycle rate.[2] For comparison, the 2007 U.S. National Post-Consumer Bottle Recycling Report estimated the total post-consumer recycle rate to be 26% for HDPE bottles and 24.6% for PET bottles.[3]

While PET and HDPE bottle recycling has shown good success due to its strong market demand, mature technology, and established collection infrastructure, collection growth has slowed in recent years and has essentially plateaued since 1997.[4] **Chart 1** illustrates these trends.[5] In the last few years, however, the PET bottle recycling rate has rebounded slightly. But because the growth of plastic PET bottles is drastically outpacing collection and recycling, more and more plastic bottles are being sent to landfills. Slow collection growth has been a major factor in declining or low recycle rates. This slow growth in PET bottle collection is a barrier to improving PET bottle recycling rates.

**Chart 1 – PET Bottle Gross Recycle Rates in the U.S. (1995-2007)**



In order to keep pace with the market demand and acceptance of a greater variety of plastic packaging and recycled-content products, recyclers will ultimately benefit if they are able to adapt, integrate to add value, and create new products and markets.[4] Recyclers who are leaders in innovation and technology, such as those who use near-infrared sortation and automation equipment, will be able to improve their recycle quality and adapt to the ever changing needs of the market.

Once volumes of Ingeo and other PLA-based bottles increase, a recycled materials markets will develop. Effective sorting technologies, such as near-infrared, will be useful for separating these bioplastics into a high-value stream. PLA can then be reprocessed for new uses through either traditional mechanical means or recovered through chemical recycling (hydrolysis), which is an efficient method of reconstituting the biopolymer for new uses.

In order to demonstrate and prove that PLA bottles can be sorted and recycled in the current recycling infrastructure, a large-scale sortation trial was conducted by NatureWorks LLC and Primo Water Corporation.

## Objectives

Since PLA is becoming more prevalent in the bottles market, the recycling industry has had questions with the sortability of PLA bottles. The concern is if PLA bottles can be sorted effectively, if at all, in the current infrastructure. Another concern is if bottles made out of PLA do get through the sorting process, what levels of contamination are of concern to the PET recycler and end-user?

NatureWorks LLC and Primo Water Corp. have collaborated on this work to demonstrate PLA bottles can be separated out of a commercial PET recycle stream using NIR sorting equipment. This was accomplished by spiking in a known amount of PLA bottles into a PET deposit stream during the sortation process. For this test, large-scale equipment and rates were used that are typical or representative of the recycling industry.

The objectives of this work were to determine the following:

- Prove that NIR equipment is able to recognize PLA's NIR signature and capable of negative sortation at typical, large scale production rates
- Evaluate using existing equipment under normal processing conditions
- Determine sorting yields and process efficiencies
- Measure the impact clear recycled PET flake (spiked with PLA bottles) has on haze and color at various let downs and plaque thicknesses
- Validate the impact clear recycled PET flake (spiked with PLA) has on commercial sheet quality (haze and color) and end-product value (sellable or not).

## Participants in Study

The following table, **Table 1**, summarizes the companies that participated in this study.

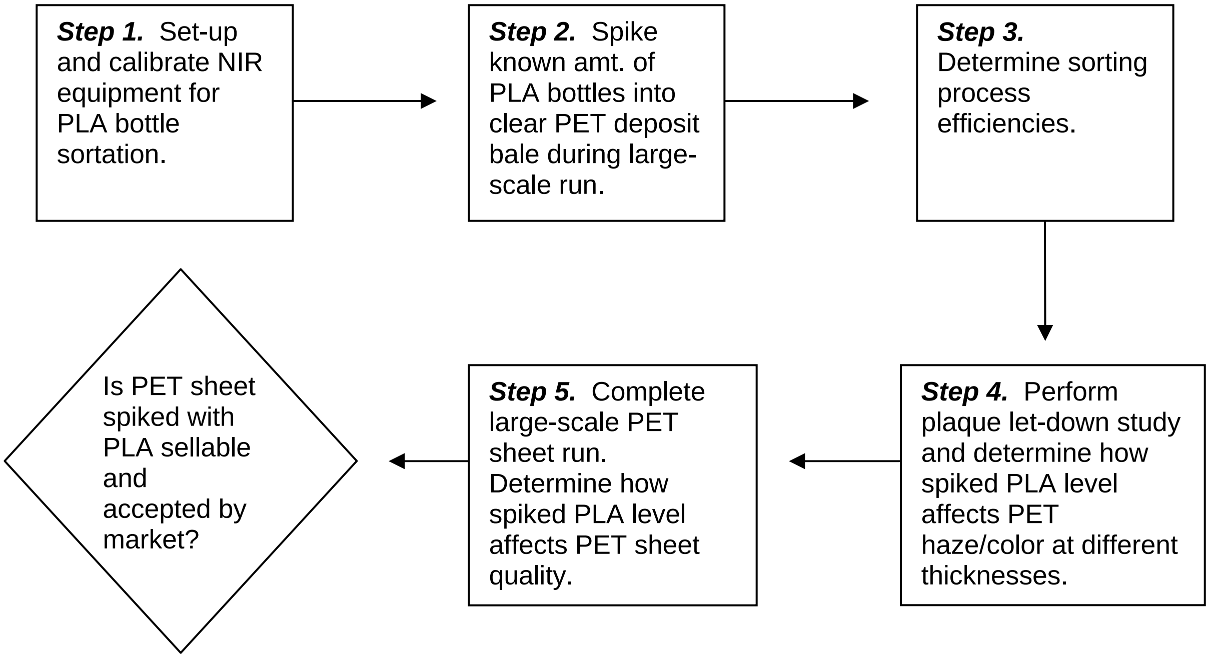
**Table 1 – Participants in NIR Sortation Study using PLA Bottles**

| Company | Role |
|---|---|
| NatureWorks LLC | Resin supplier |
| Primo Water Corporation | Water bottle supplier |
| Major PET bottle recycler, processor, and sheet manufacturer | Recycler & manufacturer |
| TITECH | Provider of sensor-based sorting equipment |
| Plastics Forming Enterprises, LLC | Independent testing, R&D lab |

## Experimental

The process flow in **Figure 1** illustrates the steps carried out for this sortation study.

**Figure 1 - Process Flow for NIR Sortation Study using PLA Bottles**



***Step 1.*** Set-up and calibrate NIR equipment for PLA bottle sortation.

***Step 2.*** Spike known amt. of PLA bottles into clear PET deposit bale during large-scale run.

***Step 3.*** Determine sorting process efficiencies.

***Step 4.*** Perform plaque let-down study and determine how spiked PLA level affects PET haze/color at different thicknesses.

***Step 5.*** Complete large-scale PET sheet run. Determine how spiked PLA level affects PET sheet quality.

Is PET sheet spiked with PLA sellable and accepted by market?

A complete load of PET bottle deposit bales (~ 35,000 lbs.) and approximately 1500 Primo water bottles without labels, (or about 75 lbs.) were used as the starting material. The PET bales used for the run were pulled from this source. A commercial scale PET bottle recycling facility was used, which was located at a major PET bottle recycler, processor, and sheet manufacturer. The optical sorter used on the sortation line was a

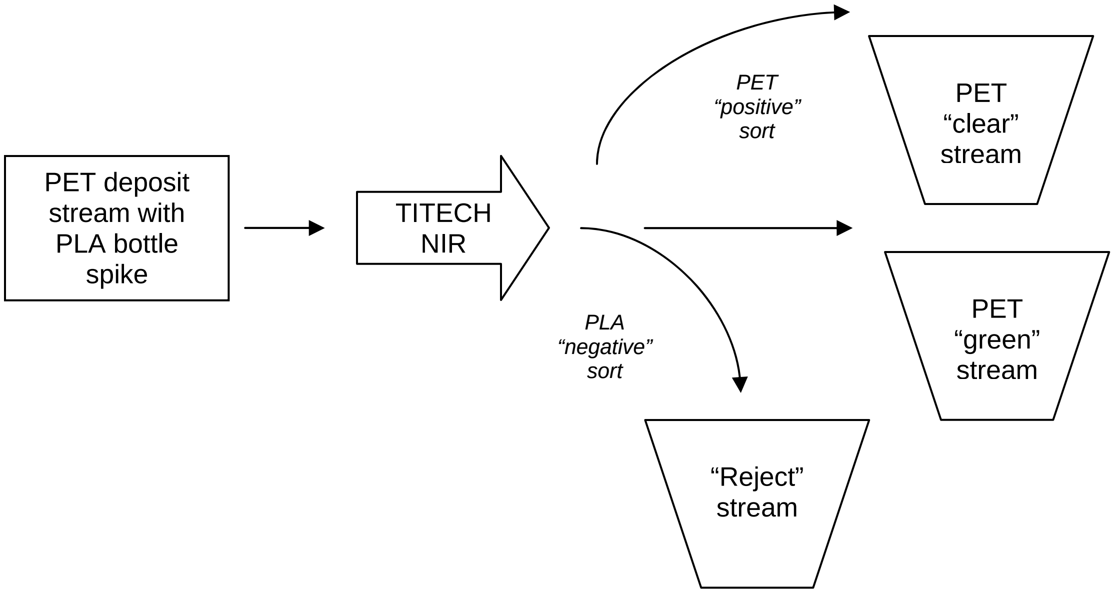
TITECH near-infrared machine with an effective width of 40 inches. The Primo water bottles were flattened beforehand, to simulate the form the bottles would be upon entering a recycling facility. A breakdown of the above process flow is explained in more detail below, starting from ***Step 1*** through ***Step 5***.

### *Step 1 – TITECH NIR Calibration*

The TITECH near-infrared sorting machine was first adjusted for PLA recognition. This process involved a TITECH technician to calibrate and fine tune the program for PLA. This was done by placing a Primo bottle (without any label) underneath the near-infrared sorting beam and recording PLA's near-infrared signature. This scan was then saved into the software's memory for addition into the material recognition library. Five bottles were scanned for the calibration test.

Before ***Step 2*** was started, a manual spike pre-test was done for 10 minutes to ensure that the software programmed for PLA recognition was actually working correctly. This separate test was done beforehand to ensure everything was working on the sortation lines. For this 10 minute pre-test, a known amount of Primo bottles were spiked into the PET sorting line by hand. Operators were stationed at locations throughout the sorting lines to collect all of the spiked Primo bottles. The sorting line was set up using clear PET as the "positive sort" stream and the Primo bottles were being "negatively sorted" to the reject stream. A third "green PET" bottle stream was also being collected and baled. The schematic in **Figure 2** illustrates the sortation set-up used for this pre-test and ***Step 2.***

**Figure 2 – NIR Optical Sortation Line Set-up**



## *Step 2 – Sortation Tests*

After the calibration and pre-test checks, three test runs were completed using various conditions to understand how the "new PLA program" would affect the efficiency of PET sortation and also gather PET base line run data. The procedure for this part of the study included the following three test runs:

- ***Test A:*** Run only PET deposit bale material through the sorting line. Run a standard PET NIR program on the TITECH sorter without Primo bottle spike.

    - Produce (2) ~ 2000 lbs. bags
    - During this time, calculate rate of the sorting line (record run time and amount produced)
    - Isolate the reject bottle bale

- ***Test B:*** Change TITECH to use "new PLA program" for bottle sorting. Run PET deposit bale material through without Primo bottle spike.

    - Produce (2) ~ 2000 lb. bags
    - During this time, calculate rate of sorting line (record run time and amount produced)

  - Isolate reject bottle bale.

- ***Test C:*** Run “new PLA program” for bottle sorting <u>with</u> Primo bottle spike in PET deposit bale.

  - Target 1% wt. PLA bottle spike (of the clear PET stream, using Test A and B’s average throughput)
  - Produce (2) ~ 2000 lbs. bags
  - Record run time and amount produced
  - Isolate reject bottle bale
  - Collect Primo bottles on the green conveyor

For **Test’s A, B, and C,** a known number of Primo bottles were hand fed into the sorting line. Hand feeding was done just after bale separation but before the TITECH near-infrared sorter. The Primo bottle spike was targeted to be 1%wt and was based on the average rate of the sorting line for the clear PET stream from Test’s A and B. **Table 2** summarizes this information.

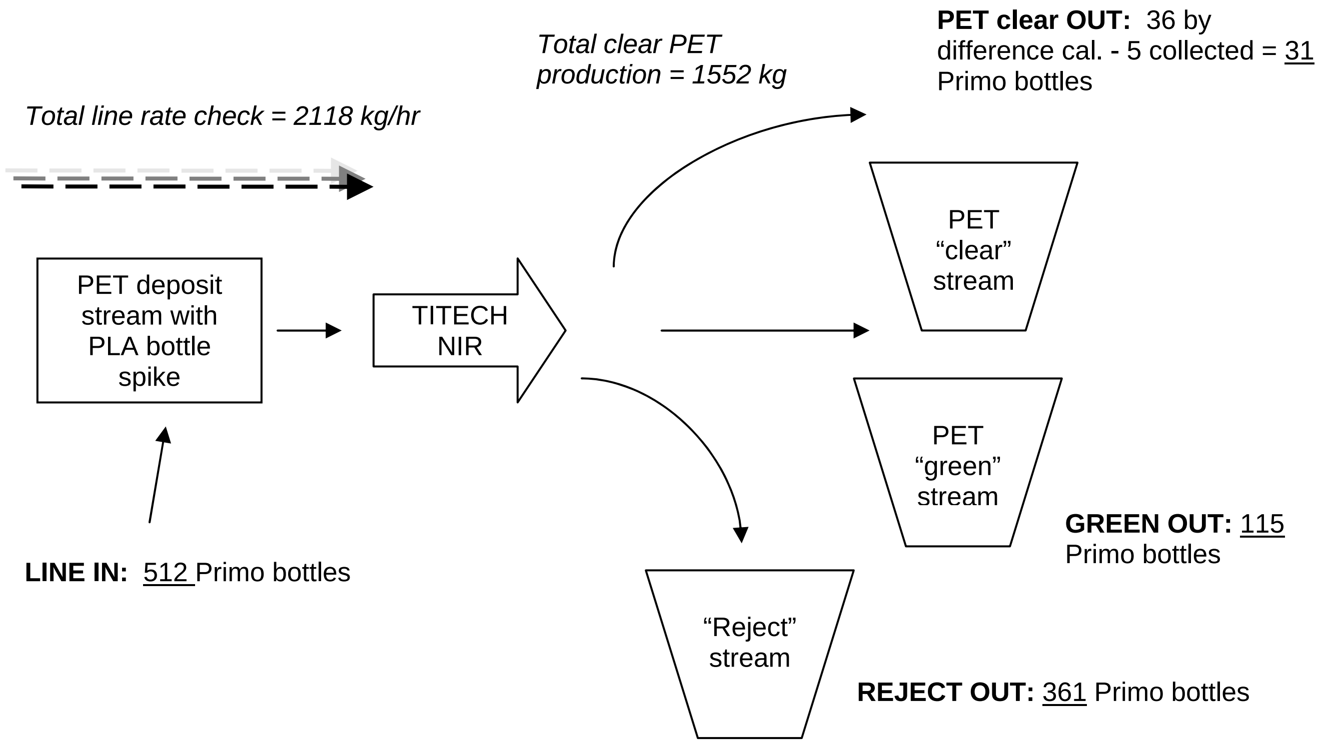
### *Step 3 – Summary of Sortation Test’s A, B, and C*

The bottles from the “reject” stream were collected, baled, and isolated to determine the removal efficiency of the Primo bottles. To do this, the isolated reject bales were broken down and the Primo bottles were identified and counted. The Primo bottles were also collected manually from the “green” stream bottle convey line and counted after the test. The supply of PET deposit bales did not contain any known quantities of PLA bottles. The output determined in this study was the known volume of PET material, along with the PLA bottles spiked into the sorting run. **Figure 3** describes the collection results from Test C, or the actual spiked Primo bottle test run with the “new PLA” TITECH program. **Table 2** describes the production results from each of the test runs and **Table 3** summarizes the TITECH NIR sorting efficiency results for the PLA bottles

**Table 2 – Production Results from Test's A, B, and C**

| Production | Test A | Test B | Test C |
|---|---|---|---|
| Total sorting line rate check (kg/hr) | 2118 | | |
| Clear PET Bag 1 collection stream (kg) | 784 | 826 | 786 |
| Clear PET Bag 2 collection stream (kg) | 788 | 780 | 766 |
| Total (kg) | 1572 | 1606 | 1552 |
| Avg. production rate (kg/hr) | 1165 | 1356 | 1693 |
| Avg. rate (kg/hr) of Test A and B | 1260 | | |
| No. of PLA bottles needed to be spiked into sorting line for Test C (bottle/min) | | | 9.25 |
| *1%wt PLA bottle spike level was targeted* | | | |
| Test C actual % PLA spike in clear PET flake *(calculated)* | | | 0.75% |

**Figure 3 – Primo Bottle Collection Results from Test C**



PPM Spike Calc.: (31 Primo bottles in clear PET flake x 22.7 g/bottle) / (1552000 g total clear PET produced)

Expected PPM level of PLA in clear PET flake = **453 ppm**

**Table 3 – TITECH NIR Sorting Efficiency Results for PLA Bottles**

| Parameter | Result |
|---|---|
| TITECH sorter effective width | 40 inches |
| Type of sorting (for PET) | Positive |
| Bale spiking level | 0.68 % |
| PLA spiking level in clear PET flake | 0.75 % |
| Total line throughput | 2118 kg/hr |
| Removal efficiency | 93 % |

## *Step 4 – Plaque Let-Down Study*

After the sortation tests in ***Steps 2 and 3***, samples of the clear PET flake (or control) and the clear PET flake spiked with PLA (453 ppm) were sent to Plastics Forming Enterprises, LLC (PFE) to perform a plaque let-down study. The purpose of this work was to determine how the spiked PLA from the sortation run affected the appearance of the clear PET flake. This test was done at three different thickness let-downs (0.063, 2, and 3 mm) to gauge how thickness, in conjunction with the PLA spike, would affect PET's appearance.

The plaque molding preparation and testing was done by PFE's laboratory and according to the washing plaque protocol established for PET bottle recycling. The following equipment and procedures were used by PFE.[6]

- Recycle System and Equipment
    - Pilot plant wash system, 4 kg batches
    - Convection ovens (surface drying)
    - Kice elutriator
    - Grinder with 3/8" screen
    - Desiccant dryer
    - Lab-scale injection molder (with a five (5) stepped plaque mold)
    - Spectrophotometer
        - Hunter Labs Colorquest II
        - Measurement: Total transmission
        - Conditions: CIELab, 10 degrees, D65
- Procedure
    - Ground incoming control and test flake (3/8" flake size), were elutriated (air separation)

- Caustic Wash: Control and test flake material were exposed to a caustic wash of 1% NaOH and 0.3% Triton X-100 (surfactant) mixed with water at 185$^{o}$F (85$^{o}$C) for 15 minutes. The liquid solid ratio was 4:1 by weight and an agitator was used at 880 rpm. These conditions represented a standard wash process used to remove dirt and label adhesive during the typical reclamation process.
- Rinse: Flakes were drained of caustic wash solution and rinsed for 2 minutes with room temperature water at the same 4:1 slurry ratio and 880 rpm of agitation to remove the caustic solution.
- Drying: Flakes were dried in a stainless steel dryer for 4 hours at 200°F (93$^{o}$C). Following the removal of surface moisture, the flakes were then air separated a second time.
- Blend: Test flake was then blended into the control flake at three different concentration levels; 0%, 25%, 50% and 100%. Also a 50/50 blend of test material and virgin PET was added as a second control. These blends were fine ground reducing the flake size to ¼". The flake samples made and used are shown in **Figure 4.**
- Plaque Molding: Prior to being made into molded plaques, the blended flake was dried in a desiccant dryer at 320°F (160$^{o}$C) for 4-6 hours to ensure all moisture was removed from the blended material.

**Table's 4, 5, and 6** summarize the plaque color data at the various test flake blend levels and across the three different thicknesses.[6] Haze and L*, a*, and b* measurements were taken on more than one specimen (n = 6) to get an average value of the data set. A blend of 50/50 clear PET test spike material and virgin PET was also included in this study to compare against the 50% blend test material.

**Figure 4 – PCR Flake Samples used for Plaque Let-Down Study (left to right – 0, 25, 50, and 100% Test Flake Blends)**

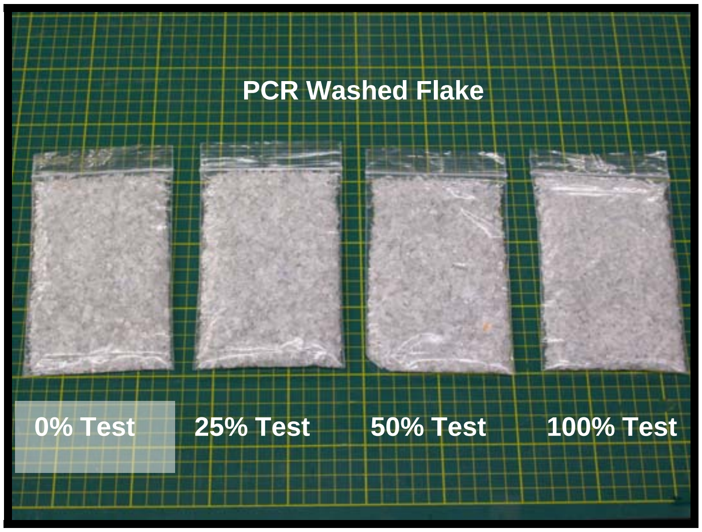


**Table 4 – 0.063 mm Plaque Color Results**

| Run # | Description | L* Values | a* Values | b* Values | L* Avg | a* Avg | b* Avg | Haze | Haze Avg |
|---|---|---|---|---|---|---|---|---|---|
| 171-03 | 0% Test | 88.46 | -1.02 | 7.31 | 88.32 | -1.07 | 7.21 | 19.56 | 15.72 |
| | | 89.28 | -1.06 | 6.91 | | | | 13.72 | |
| | | 88.95 | -1.27 | 6.93 | | | | 14.59 | |
| | | 88.88 | -1.04 | 7.41 | | | | 16.92 | |
| | | 85.46 | -0.89 | 7.71 | | | | 13.14 | |
| | | 88.87 | -1.12 | 6.96 | | | | 16.36 | |
| 171-04 | 25% Test | 88.88 | -0.96 | 6.34 | 88.86 | -0.98 | 6.32 | 15.04 | 15.46 |
| | | 88.58 | -0.99 | 6.49 | | | | 16.84 | |
| | | 88.71 | -0.92 | 6.64 | | | | 16.64 | |
| | | 88.66 | -1.03 | 6.33 | | | | 16.44 | |
| | | 89.20 | -1.01 | 6.08 | | | | 13.63 | |
| | | 89.14 | -0.94 | 6.02 | | | | 14.18 | |
| 171-05 | 50% Test | 89.08 | -1.01 | 6.49 | 89.35 | -1.01 | 6.14 | 14.26 | 15.24 |
| | | 89.59 | -0.94 | 6.00 | | | | 15.20 | |
| | | 89.64 | -1.05 | 6.03 | | | | 15.10 | |
| | | 89.44 | -1.23 | 5.79 | | | | 16.45 | |
| | | 89.29 | -0.92 | 6.33 | | | | 14.28 | |
| | | 89.05 | -0.88 | 6.17 | | | | 16.13 | |
| 171-06 | 100% Test | 89.88 | -1.01 | 6.67 | 89.35 | -0.95 | 7.37 | 15.59 | 16.20 |
| | | 89.18 | -0.91 | 7.32 | | | | 17.99 | |
| | | 89.57 | -1.02 | 7.28 | | | | 15.20 | |
| | | 89.43 | -0.97 | 7.42 | | | | 15.01 | |
| | | 89.34 | -0.92 | 7.80 | | | | 15.54 | |
| | | 88.67 | -0.89 | 7.74 | | | | 17.85 | |
| 171-05 | 50% Test with VPET | 88.99 | -0.43 | 4.47 | 89.38 | -0.39 | 4.68 | 9.74 | 10.15 |
| | | 89.53 | -0.37 | 4.57 | | | | 10.19 | |
| | | 89.52 | -0.48 | 4.37 | | | | 10.49 | |
| | | 89.31 | -0.22 | 5.43 | | | | 10.13 | |
| | | 89.20 | -0.42 | 4.66 | | | | 12.45 | |
| | | 89.74 | -0.44 | 4.56 | | | | 7.89 | |

**Table 5 – 2 mm Plaque Color Results**

| Run # | Description | L* Values | a* Values | b* Values | L* Avg | a* Avg | b* Avg | Haze | Haze Avg |
|---|---|---|---|---|---|---|---|---|---|
| 171-03 | 0% Test | 86.77 | -1.36 | 8.46 | 86.75 | -1.28 | 8.68 | 18.20 | 19.51 |
| | | 86.84 | -1.24 | 8.79 | | | | 20.45 | |
| | | 86.67 | -1.53 | 8.47 | | | | 19.28 | |
| | | 86.80 | -1.20 | 8.65 | | | | 17.34 | |
| | | 86.52 | -1.21 | 8.61 | | | | 23.93 | |
| | | 86.88 | -1.16 | 9.08 | | | | 17.88 | |
| 171-04 | 25% Test | 87.25 | -1.09 | 7.30 | 86.64 | -1.14 | 7.76 | 18.11 | 19.45 |
| | | 86.85 | -1.20 | 7.33 | | | | 16.96 | |
| | | 86.48 | -1.14 | 7.83 | | | | 20.12 | |
| | | 86.26 | -1.08 | 8.21 | | | | 20.41 | |
| | | 86.27 | -1.15 | 8.04 | | | | 22.04 | |
| | | 86.71 | -1.15 | 7.82 | | | | 19.04 | |
| 171-05 | 50% Test | 86.74 | -1.02 | 7.46 | 87.25 | -1.18 | 7.37 | 19.12 | 18.05 |
| | | 87.04 | -1.09 | 7.56 | | | | 17.65 | |
| | | 87.52 | -1.41 | 7.02 | | | | 18.36 | |
| | | 87.66 | -1.26 | 7.20 | | | | 18.87 | |
| | | 87.32 | -1.11 | 7.26 | | | | 15.79 | |
| | | 87.20 | -1.21 | 7.71 | | | | 18.48 | |
| 171-06 | 100% Test | 86.38 | -1.03 | 9.57 | 86.57 | -1.05 | 9.16 | 20.34 | 19.68 |
| | | 86.27 | -0.97 | 9.84 | | | | 19.26 | |
| | | 86.54 | -1.06 | 9.29 | | | | 18.40 | |
| | | 86.59 | -1.13 | 8.97 | | | | 19.48 | |
| | | 86.29 | -1.00 | 8.92 | | | | 22.07 | |
| | | 87.35 | -1.13 | 8.34 | | | | 18.54 | |
| 171-05 | 50% Test with VPET | 88.03 | -0.63 | 5.12 | 87.94 | -0.59 | 5.38 | 9.71 | 11.91 |
| | | 87.95 | -0.62 | 5.22 | | | | 14.08 | |
| | | 87.72 | -0.48 | 5.99 | | | | 11.69 | |
| | | 88.00 | -0.64 | 5.44 | | | | 11.79 | |
| | | 88.12 | -0.54 | 5.28 | | | | 12.55 | |
| | | 87.84 | -0.61 | 5.22 | | | | 11.63 | |

**Table 6 – 3 mm Plaque Color Results**

| Run # | Description | L* Values | a* Values | b* Values | L* Avg | a* Avg | b* Avg | Haze | Haze Avg |
|---|---|---|---|---|---|---|---|---|---|
| 171-03 | 0% Test | 82.58 | -1.53 | 11.94 | 82.44 | -1.63 | 11.81 | 23.20 | 27.16 |
| | | 82.65 | -1.58 | 11.85 | | | | 32.15 | |
| | | 82.59 | -1.67 | 11.56 | | | | 24.31 | |
| | | 81.93 | -1.64 | 11.93 | | | | 29.82 | |
| | | 82.58 | -1.59 | 11.96 | | | | 27.50 | |
| | | 82.32 | -1.74 | 11.63 | | | | 25.97 | |
| 171-04 | 25% Test | 82.17 | -1.46 | 10.73 | 82.06 | -1.49 | 10.74 | 25.91 | 26.13 |
| | | 81.65 | -1.50 | 11.09 | | | | 27.38 | |
| | | 81.73 | -1.51 | 10.96 | | | | 28.56 | |
| | | 81.75 | -1.51 | 10.82 | | | | 26.74 | |
| | | 82.23 | -1.57 | 10.51 | | | | 24.34 | |
| | | 82.81 | -1.39 | 10.35 | | | | 23.84 | |
| 171-05 | 50% Test | 82.52 | -1.57 | 10.87 | 83.13 | -1.56 | 10.17 | 24.77 | 25.52 |
| | | 83.45 | -1.50 | 10.19 | | | | 24.56 | |
| | | 83.50 | -1.66 | 9.89 | | | | 26.37 | |
| | | 83.34 | -1.77 | 9.70 | | | | 26.50 | |
| | | 82.97 | -1.55 | 10.56 | | | | 25.83 | |
| | | 83.00 | -1.33 | 9.81 | | | | 25.08 | |
| 171-06 | 100% Test | 82.39 | -1.40 | 11.67 | 81.75 | -1.25 | 12.68 | 29.08 | 27.83 |
| | | 81.84 | -1.24 | 12.16 | | | | 28.44 | |
| | | 81.70 | -1.30 | 12.63 | | | | 26.04 | |
| | | 81.35 | -1.12 | 13.11 | | | | 26.64 | |
| | | 81.72 | -1.16 | 13.05 | | | | 27.48 | |
| | | 81.49 | -1.28 | 13.44 | | | | 29.31 | |
| 171-05 | 50% Test with VPET | 85.17 | -0.90 | 6.90 | 85.73 | -0.93 | 6.99 | 14.83 | 17.43 |
| | | 84.81 | -0.84 | 7.05 | | | | 18.55 | |
| | | 85.21 | -0.91 | 7.15 | | | | 16.30 | |
| | | 85.93 | -0.87 | 7.79 | | | | 18.06 | |
| | | 86.44 | -1.06 | 6.57 | | | | 19.17 | |
| | | 86.81 | -1.01 | 6.45 | | | | 17.68 | |

### *Step 5 – Commercial Sheet Run*

Sorting, grinding, flake washing and sheet extrusion were performed on the standard PET bale above, with approximately 0.7%wt (~ 7,000 ppm) PLA bottles added to the process.

Since there was no significant difference in color and haze between the control (clear PET flake) and clear PET test material (spiked with PLA) from the lab-scale plaque let-down study in ***Step 4***, a PET sheet run was done to determine the impact the spiked PLA material had on PET sheet extrusion and product quality.  The sheet was made on an existing commercial sheet extrusion line.

In order to compare the commercial sheet results with an independent test laboratory, clean flake from both the PET control and PET test material was provide to PFE from the sheet manufacturer.  The test material was compared to the control material after the sheet manufacturer's washing process.  PFE made 3 mm thick plaques for color and haze measurements.  The estimated level of PLA spike in the PET test flake was 453 ppm.

For ***Step 5***, the following equipment and procedures were used.

- Recycle System and Equipment

    - Commercial recycle system
    - Commercial sheet extrusion system
    - Lab-scale injection molder, for plaques (PFE)

- Procedure
    - Blend: Test flake was blended into control flake at three different concentration levels; 0%, 50%, and 100%. These blends were fine ground reducing the flake size to ¼".
    - Plaque Molding: Prior to being made into molded plaques, the blended flake was dried in a desiccant dryer at 320°F (160°C) for 4-6 hours to ensure all moisture was removed from the blended material.
    - Sheet Extrusion:  Existing commercial scale equipment was used under normal PET processing conditions.
    - Sheet manufacturing:  The post-consumer recycle content (PCR), or PET test material spiked with PLA, was fed at a 70% rate with PET virgin material.  Therefore, the expected PLA flake content in the sheet is 317 ppm.
    - Sheet sampling:  Sheet roll stock was made at 0.021-0.022 inches in thickness.  Sheet samples for testing were taken on every roll, or 20-30 minutes of each roll's run.  Sheet was made and samples taken over a 6

hr. period with continuous feeding with no designated cut-off point between the control and test material.

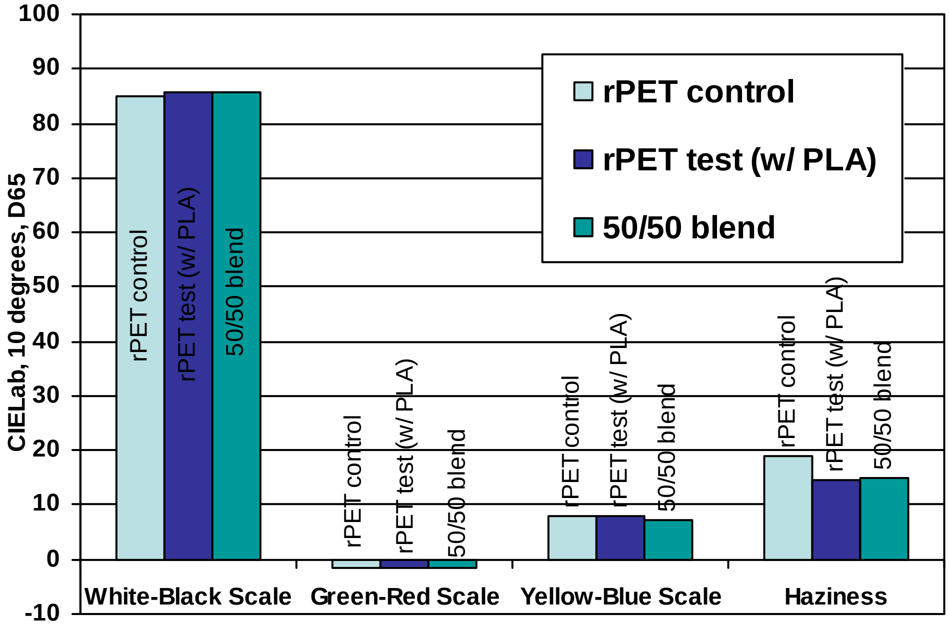
**Table 7** summarizes the plaque color and haze data at the various test flake blend levels using a 3 mm sample thickness.[7] Haze and L*, a*, and b* measurements were taken on more than one specimen (n = 6) to get an average value of the data set. A blend of 50/50 clear PET control material and the clear PET test flake spiked with PLA was also included in this study for comparison. **Figure 5** below also illustrates the 3 mm plaque data in graphical format, where L* represents the "white-black" scale, a* represents the "green-red" scale, and b* represents the "yellow-blue" scale. Haze is also included in the chart.

**Table 8** summarizes the color and haze data on the commercial sheet samples collected over a 6 hr. run. The sheet samples were sent to PFE for the measurements.[7] **Figure 6** illustrates this data in a graphical format, which plots the samples taken in time over the course of the 6 hr. run versus the color and haze measurements taken on the samples.

**Table 7 – 3 mm Plaque Color and Haze Results on Washed Flake used for Commercial Sheet Run**

| Run # | Description | L* Values | a* Values | b* Values | L* Avg | a* Avg | b* Avg | Delta Ecmc | Delta Avg | Haze | Haze Avg |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 182-03 | Control | 84.91 | -1.5 | 7.27 | 84.82 | -1.60 | 8.04 | 0.70 | 0.45 | 17.73 | 19.01 |
| | | 84.66 | -1.69 | 8.15 | | | | 0.17 | | 20.05 | |
| | | 85.28 | -1.75 | 7.72 | | | | 0.45 | | 17.24 | |
| | | 84.66 | -1.58 | 8.46 | | | | 0.41 | | 19.57 | |
| | | 84.61 | -1.5 | 8.58 | | | | 0.56 | | 20.44 | |
| 182-04 | Test | 85.61 | -1.47 | 7.55 | 85.80 | -1.47 | 7.92 | 0.35 | 0.48 | 15.57 | 14.49 |
| | | 86.22 | -1.46 | 7.73 | | | | 0.26 | | 13.11 | |
| | | 85.42 | -1.62 | 8.1 | | | | 0.30 | | 13.76 | |
| | | 85.39 | -1.38 | 8.85 | | | | 0.91 | | 15.41 | |
| | | 86.34 | -1.43 | 7.35 | | | | 0.58 | | 14.62 | |
| 182-05 | 50/50 Blend | 86.03 | -1.79 | 7.33 | 85.80 | -1.64 | 7.28 | 0.23 | 0.19 | 14.75 | 14.92 |
| | | 85.71 | -1.65 | 7.32 | | | | 0.06 | | 15.93 | |
| | | 85.5 | -1.56 | 6.92 | | | | 0.37 | | 16.2 | |
| | | 85.82 | -1.62 | 7.29 | | | | 0.04 | | 14.01 | |
| | | 85.92 | -1.59 | 7.52 | | | | 0.26 | | 13.71 | |

**Figure 5 – 3 mm Plaque Color and Haze Comparison on Washed Flake used for Commercial Sheet Run**



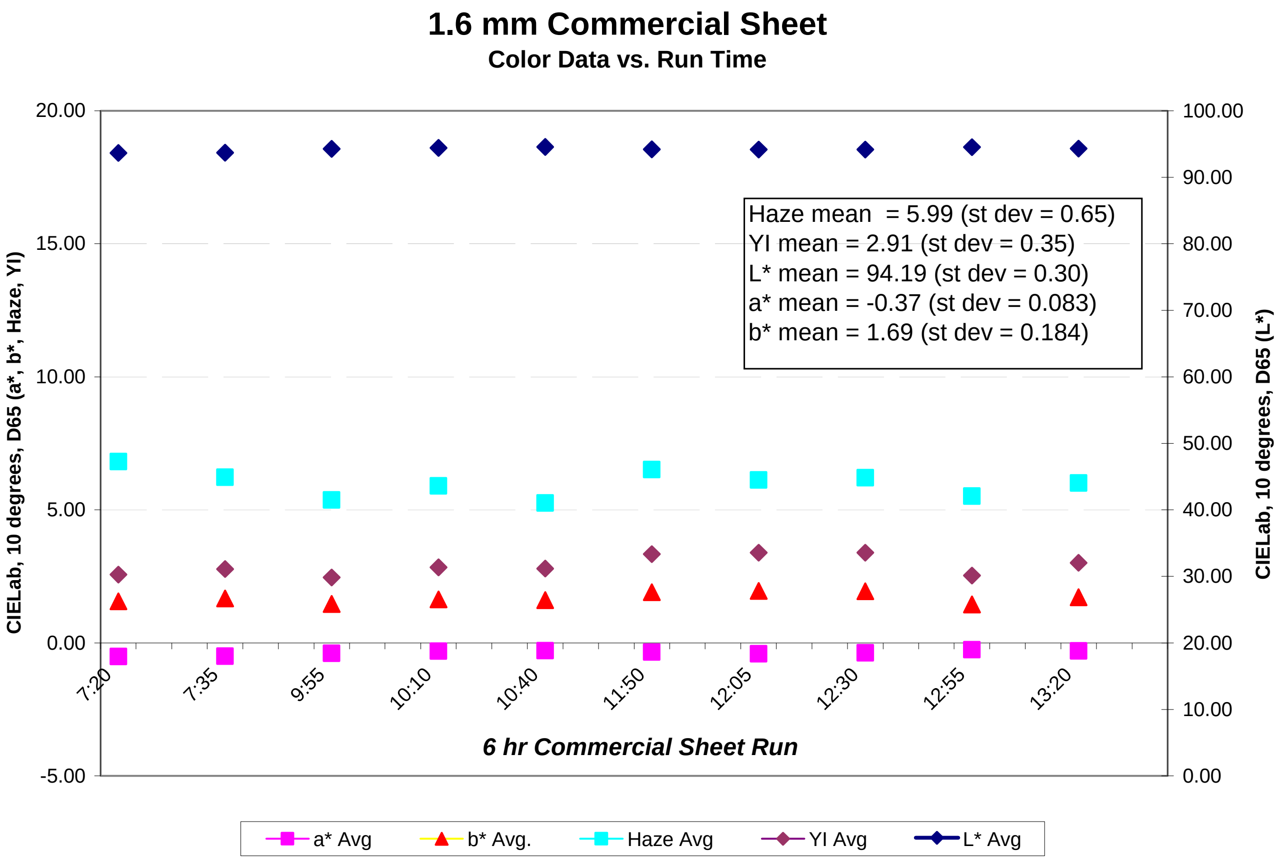
The entire commercial sheet made (70% PCR of the test flake/30% virgin PET) during the 6 hr. run was collected and the processing results showed no significant difference through the commercial sheet extrusion evaluation. In both the plaque and commercial sheet samples made, there was no significant difference between the control and the test flake. The sheet was sold, without issue, to the recycler/sheet producer’s customer.

**Figure 7** illustrates samples of the 0.021-0.022 in. sheet produced and sold. The picture compares sheet samples taken at the beginning of the sheet run, where 100% of the control rPET flake was used (left image) for the 70% PCR portion of the blend with 30% virgin PET; and from the middle and end of the sheet run, where 100% of the test rPET flake, spiked with approximately 453 ppm PLA, was used (middle and right image) for the 70% PCR portion of the blend with 30% virgin PET.

**Table 8 – 0.021-0.022 in. Sheet Color and Haze Results. 6 hr. Commercial Run (70% PCR using PET flake spiked with PLA and 30% Virgin PET). Estimated PLA Bottle Flake Content in Sheet at 317 ppm.**

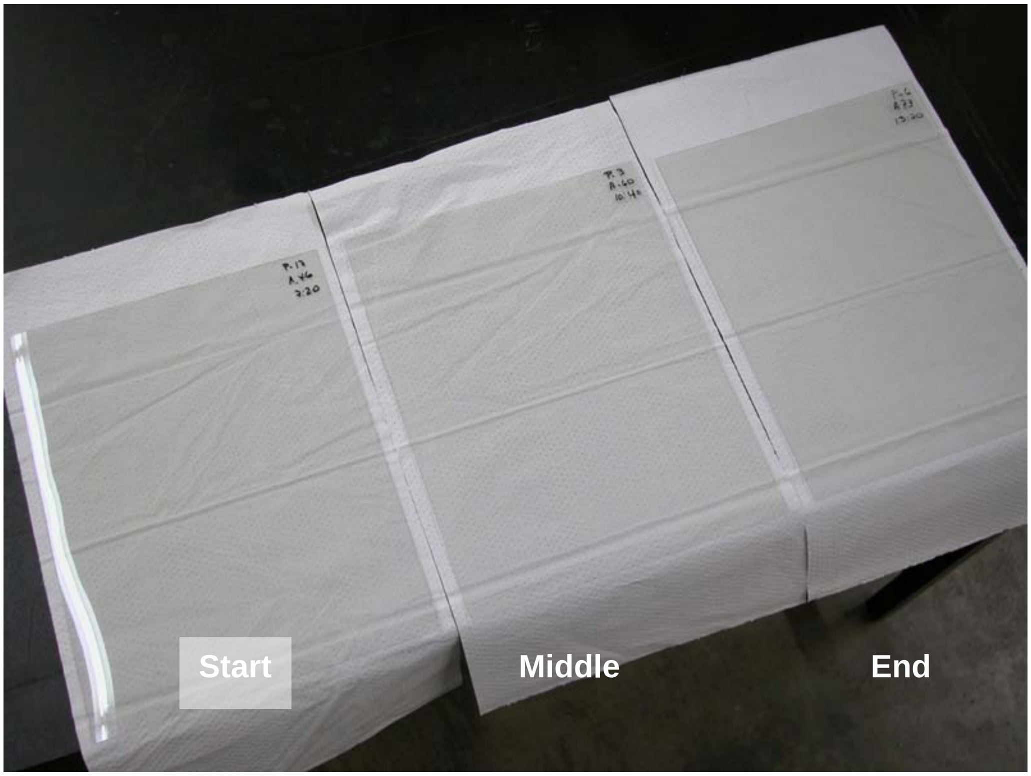
| Run # | Description | L * Values | a* Values | b* Values | L* Avg | a* Avg | b* Avg | Haze | Haze Avg | YI | YI Avg |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 7:20 | A46 | 93.68 | -0.5 | 1.54 | 93.66 | -0.51 | 1.56 | 6.03 | 6.81 | 2.53 | 2.56 |
| | | 93.64 | -0.51 | 1.57 | | | | 6.29 | | 2.58 | |
| | | 93.67 | -0.52 | 1.58 | | | | 8.11 | | 2.58 | |
| 7:35 | A48 | 93.68 | -0.51 | 1.66 | 93.71 | -0.50 | 1.67 | 6.59 | 6.22 | 2.75 | 2.78 |
| | | 93.79 | -0.5 | 1.68 | | | | 6 | | 2.8 | |
| | | 93.65 | -0.5 | 1.67 | | | | 6.07 | | 2.78 | |
| 9:55 | A54 | 94.24 | -0.39 | 1.48 | 94.26 | -0.39 | 1.46 | 5.17 | 5.38 | 2.5 | 2.46 |
| | | 94.37 | -0.37 | 1.45 | | | | 5.95 | | 2.43 | |
| | | 94.18 | -0.41 | 1.46 | | | | 5.01 | | 2.44 | |
| 10:10 | A55 | 94.47 | -0.31 | 1.61 | 94.42 | -0.31 | 1.63 | 5.57 | 5.90 | 2.79 | 2.84 |
| | | 94.41 | -0.32 | 1.64 | | | | 6.47 | | 2.85 | |
| | | 94.39 | -0.31 | 1.65 | | | | 5.66 | | 2.87 | |
| 10:40 | A60 | 94.56 | -0.28 | 1.59 | 94.43 | -0.30 | 1.61 | 5.51 | 5.26 | 2.77 | 2.79 |
| | | 94.51 | -0.3 | 1.62 | | | | 5.7 | | 2.82 | |
| | | 94.22 | -0.31 | 1.61 | | | | 4.56 | | 2.79 | |
| 11:50 | A65 | 94.17 | -0.35 | 1.94 | 94.19 | -0.34 | 1.91 | 6.13 | 6.52 | 3.39 | 3.34 |
| | | 94.19 | -0.35 | 1.94 | | | | 6.24 | | 3.39 | |
| | | 94.22 | -0.33 | 1.84 | | | | 7.18 | | 3.23 | |
| 12:05 | A67 | 94.06 | -0.42 | 1.99 | 94.15 | -0.41 | 1.95 | 5.83 | 6.13 | 3.46 | 3.39 |
| | | 94.18 | -0.4 | 1.93 | | | | 6.01 | | 3.35 | |
| | | 94.22 | -0.4 | 1.94 | | | | 6.54 | | 3.36 | |
| 12:30 | A68 | 94.12 | -0.38 | 1.94 | 94.19 | -0.38 | 1.95 | 6.32 | 6.21 | 3.38 | 3.39 |
| | | 94.16 | -0.38 | 1.98 | | | | 6.39 | | 3.45 | |
| | | 94.28 | -0.37 | 1.92 | | | | 5.92 | | 3.34 | |
| 12:55 | A70 | 94.55 | -0.27 | 1.48 | 94.59 | -0.26 | 1.45 | 5.72 | 5.52 | 2.58 | 2.53 |
| | | 94.53 | -0.26 | 1.48 | | | | 5.43 | | 2.59 | |
| | | 94.7 | -0.24 | 1.38 | | | | 5.4 | | 2.41 | |
| 13:20 | A73 | 94.3 | -0.31 | 1.74 | 94.30 | -0.30 | 1.72 | 5.88 | 6.01 | 3.04 | 3.01 |
| | | 94.34 | -0.3 | 1.72 | | | | 6.05 | | 3.02 | |
| | | 94.26 | -0.3 | 1.7 | | | | 6.1 | | 2.98 | |

**Figure 6 – 6 hr. Color and Haze Data Collection on 0.021-0.022 in. Commercial Sheet Samples**



Haze mean = 5.99 (st dev = 0.65)
YI mean = 2.91 (st dev = 0.35)
L* mean = 94.19 (st dev = 0.30)
a* mean = -0.37 (st dev = 0.083)
b* mean = 1.69 (st dev = 0.184)

**Figure 7 - 0.021-0.022 in. Commercial Sheet Samples Collected (start, middle, and end of extrusion run)**



## Summary

### *Step 1 – TITECH NIR Calibration*

- TITECH NIR equipment was able to be programmed to specifically sort PLA bottles.
- The TITECH machine adjustment to sort out the PLA did require a TITECH technician to fine-tune the program.
- The equipment was calibrated for PLA without any significant problems.

### *Step 2 – Sortation Tests*

- TITECH sorter effective width:  40 inches
- The system was set up positively sorting the PET and rejecting the PLA.
- The evaluation was done by spiking in Primo water bottles during a commercial PET recycle processing line.
- No issues were encountered when using the “new PLA program” to sort out the Primo bottles.
- The tests did not allow measurement of the effect on yield for the new PLA sorting setup.  Larger-scale tests would be needed.

### *Step 3 – Summary of Sortation Test’s A, B, and C*

- Total sorting line throughput:  4670 lbs/hr
- Bale spiking level:  0.68%
- Removal efficiency:  93%

*Step 4 – Plaque Let-Down Study*

- The estimated level of PLA spike in the PET test flake was 453 ppm.
- Sample plaques were measured across three thicknesses (0.065, 2, and 3 mm) and four different blend levels (0, 25, 50, and 100%).
- The control and test material did not show any significant difference in color and haze.

*Step 5 – Commercial Sheet Run*

- The estimated level of PLA spike in the PET test flake was 453 ppm.
- A 70% PCR level was fed with PET virgin material to make sheet. Therefore, the expected PLA bottle flake content was 317 ppm.
- Existing sheet extrusion equipment was used, under normal PET process conditions.
- The control and test rPET flake was dried normally at typical PET conditions. No drying issues were noted during the sheet run.
- Sheet samples were taken over a 6 hr. run period with continuous feeding. There was no designated cut-off point between the PET control flake and the test flake.
- The results show no significant difference through the commercial evaluation of the sheet extrusion process.
- The test results show no significant difference from the start of the sheet trial to the end of the run.
- The commercial sheet results were compared with an independent test laboratory. 3 mm plaque samples were molded and measured for color and haze.
- There was no significant difference between the PET control flake and test material in both the 0.021-0.022 in. thick sheet and 3 mm thick plaque samples.

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