Increasing Sustainability: PCR Resin Use in Film Packaging
Sponsored by Procter & Gamble

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Introduction

This past year, I have worked alongside four undergraduate University of Michigan students to complete a project through the Multidisciplinary Design Program (MDP), sponsored by consumer goods company Procter & Gamble. Joining from three different departments across the College of Engineering, this project allowed us each to showcase our individual technical knowledge and shared commitment to increasing sustainability. The goal of this project is to contribute to P&G's Ambition 2030 goals of incorporating more recycled content into packaging applications. We helped achieve this by developing a portfolio of polymer characterization and testing methods to create a resin fingerprint for post-consumer recycled (PCR) film grade polyethylene. The developed test methods were then organized into our first deliverable: a flow chart or "decision tree" that would allow future users to follow our protocols for resin analysis. The data from our technical testing methods was then used to create a heat map, our second deliverable, for determining what PCR could be used in P&G and Glad JV applications. Our final deliverable was to compile all this data obtained from our analytical methods into a comprehensive technical data sheet for all the analyzed resins.

P&G's Ambition 2030 goals are ambitious goals aimed at reducing the company's reliance on new petroleum plastics, limiting their environmental footprint, and enabling responsible consumption. Of these goals, the two most relevant to our project are consumer packaging will be 100% recyclable or reusable and that P&G will reduce virgin petroleum plastic use in packaging by 50%. By integrating post-consumer recycled (PCR) content into packaging this will contribute to a circular economy, reduce the amount of virgin petroleum plastic used and validate their claim that packaging is recyclable.

While there has been a large increase in the amount of recycled rigid plastics being used in the consumer goods industry, film plastics remain an area where plastic recycling is very limited. In fact, less than 10% of films are recycled. However, plastic film packaging is used widely across the consumer goods industry, from toilet paper to diapers to menstrual products. Its light and flexible nature make it a great material for protecting sensitive products without adding much additional weight. One of the major difficulties with recycling film packaging is film packaging is typically multi-material (common examples include PE/nylon/EVOH coextrusions, PE/PET/OPP laminations and PE/met laminations). This makes it much harder to melt down and reuse, because polymer contamination can have negative effects on the physical performance and processing ability of the material. In addition, as these films travel from the store to in-home use, from the recycle bin to the material recovery facilities, unwanted particles can accumulate, and these films become heavily contaminated. This contamination can include other polymer contamination, dirt, grit, food particles, paper, and chemical contamination. This contamination can lead to changes in performance and optical properties, presenting many challenges when introducing recycled materials into packaging.

Our project seeks to identify these issues and make choosing and integrating PCR materials into packaging an easier, more desirable process. We were able to do this by analyzing current PCR resin pellets on the market and determining what contaminants are present in the resin material and consequently what effect they would have on the mechanical properties of the film. Our methods and results are explained in this report, with specific details removed for confidentiality.

Project Summary

Objective — The MDP Procter & Gamble Team aims to allow quick characterization and evaluation of post-consumer recycled (PCR) polyethylene film by developing resin fingerprinting methods with the following deliverables:

- Decision Tree: recommend test method for efficient and accurate fingerprinting
- Heat Map: evaluate resin against packaging needs and requirements across business units
- Materials Recovery Facility (MRF) Recommendations: ensure improved resin quality

These deliverables will provide P&G with the tools to quickly analyze new incoming PCR materials and evaluate what packaging applications the material would be acceptable in. An outline of our project plan can be seen in Figure 1.

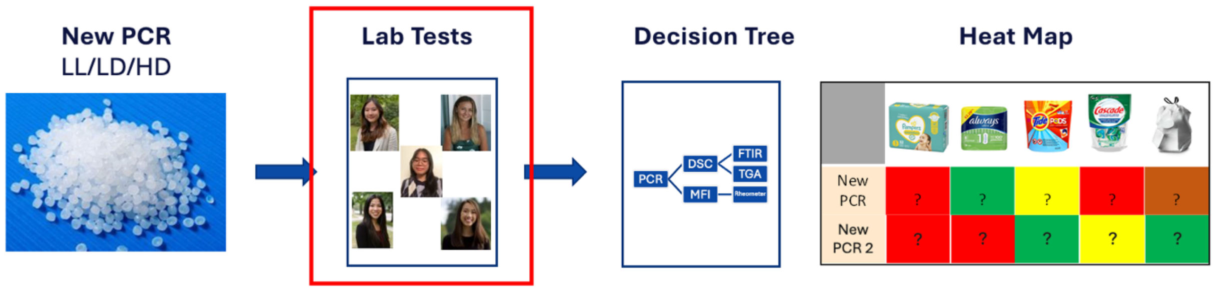

Figure 1. *The MDP team administers a series of lab tests on new PCR samples. The data collected from those trials is used to motivate two major deliverables: the decision tree and heat map.*

The bulk of our project was spent in the lab testing step, becoming familiar with the different analytical tools and detailing test methods for analyzing the pellet and film properties. We were able After creating detailed test methods, we were able to pass the experimental work off to a team at P&G to continue testing additional resins.

Scope — Within the scope of our project is developing test methods for our analytical techniques, identifying correlations between these tools, and making data-driven recommendations to upstream processes. These results will be used to draw conclusions that will be displayed in our three deliverables. While these conclusions can be used to determine where these PCR materials may be successful our methods should not be used to determine exact values. The constraints of our project are that we will only be analyzing the seven PCR film samples provided by P&G and their selected suppliers. A comprehensive analysis of all PCR resins on the market is outside of the scope of our project. Our work is also constrained by the proprietary information regarding the processing of PCR samples. Outside the scope of our project would be applications of our work to the greater recycling process, developing upstream PCR sorting technology or the physically integrating of PCR into P&G product packaging.

Project Organization

The project itself was organized into four phases: pellet characterization, film characterization, the FlexLoop™ sidebar and a consumer understanding study. These phases correspond to the stages of a PCR's lifetime, from its pellet form to the film to the hands of the consumer. The work done during each phase is detailed below.

Pellet Characterization – In this phase, we looked to analyze the polymer and other contaminants present in each PCR resin. We were able to do this by creating our own polymer blends using virgin resin and comparing those properties to the PCR. We have analyzed structure, composition, rheological, thermal, and optical properties of control and PCR pellet samples and developed test methods on LC/MS, FTIR, DSC, TGA, Parallel Plate Rheometer, MFI, and Colorimeter.

Film Characterization – In this phase, we analyzed how the resins performed on the blown film line, as they were blown from their pellet form into a film. We had two different thicknesses of film, corresponding to P&G packaging requirements. Looking at the films themselves, we analyzed their mechanical properties using tensile and dart tests. We have collaborated with teams at P&G to continue looking at their heat-sealing properties and further investigate polymer/chemical contaminants using IR and optical microscopy.

FLEXLOOP™ Sidebar – In this sidebar, we used our expertise to help improve FLEXLOOP™, a process developed by P&G to increase the removal of chemical species from recycled materials. Typically, recycled materials undergo a surface washing to be prepped for reuse. This can remove the species from the surface, but neglects to remove any unwanted species that may have migrated into the bulk of the material. FLEXLOOP™ uses a series of solvent extractions to pull out those permeable species from the bulk of the material, giving a much cleaner and safer PCR that can be used in more high sensitivity applications. However, this process was previously being modelled on the lab scale, and the company is working to scale this process up. Therefore, P&G was very interested in having our help in understanding the mechanisms of these permeable species and identify areas for process improvements.

Consumer Understanding – In this final phase of our project, we aimed to understand consumers' perceptions of PCR, assumptions about recycled materials and identify areas of greatest concern. While the majority of our project was very technical, I was very interested seeing how our work would impact real people in packaging applications. By understanding the consumer perspective, we could work to generate consumer-driven recommendations for PCR implementation and ensure success, not only on the technical side.

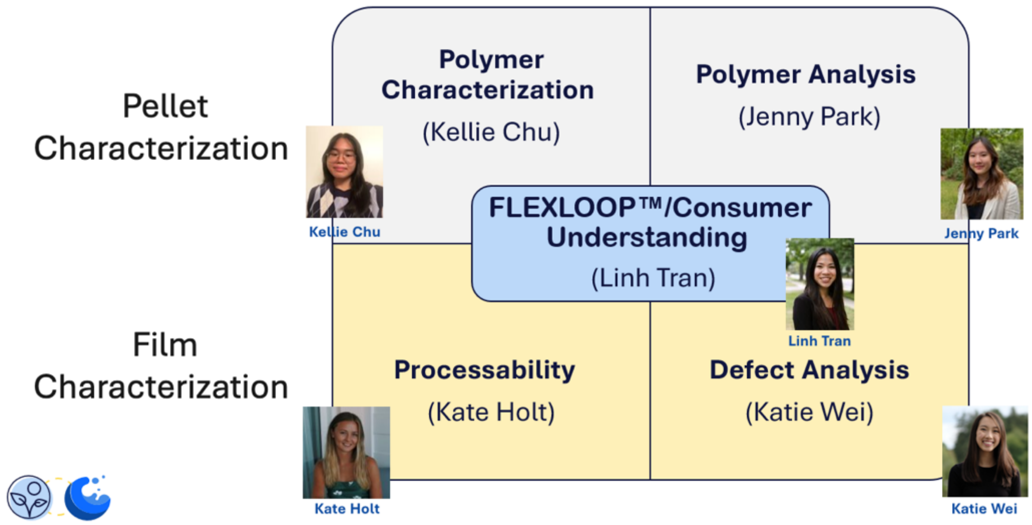

Figure 2. Distribution of work between MDP team members.

Individual Work

In this section I will detail the individual work I completed on the FlexLoop™ process improvements and consumer understanding study.

FlexLoop™

FlexLoop™ is a less complex pathway to obtain low-chemical contamination recycled flexible films. Going one step further than traditional mechanical recycling, that only does a basic surface washing step, this process uses a series of solvent extractions to reduce contamination levels to nearly the same levels virgin film, without the high amounts of energy needed to completely depolymerize the plastic. The table below outlines the problem of common chemical contaminants in PCR films and the efficacy of FlexLoop™.

Table 1. *Common chemical contaminants present in PCR films are reduced to near-virgin levels using the FlexLoop™ process. LOQ or "limit of quantification" signifies an amount lower than what is detectable by our analytical techniques.*

	Typical Virgin Film	Typical Film PCR	Typical Film PCR After FLEXLOOP™
Pesticides	< LOQ	~20 X LOQ	< LOQ
Alkylphenols	**< ~100 X LOQ**	**~6,000 X LOQ**	**< ~20 X LOQ**
Bisphenol A	**< LOQ**	**~6,000 X LOQ**	**< ~20 X LOQ**
Dioxins/PCBs	< LOQ	~400 X LOQ	< ~2 X LOQ

Phthalates	< LOQ	~700 X LOQ	< ~2 X LOQ

However, as you can see from the chart, the two species that seem to persist most aggressively are alkylphenols and bisphenol A -- specifically 4-tert-pentyl-phenol, isononyl-phenol and bisphenol A. These species exist in high concentrations in polyethylene, persist even after a surface washing and are classified as endocrine disrupters, having the potential for negative health effects. For my work with FlexLoop™, I aimed to understand the mechanisms of these three species, how they migrated into and out of the film and how we can improve this extraction process.

Contaminant Quantification – I used liquid chromatography/mass spectroscopy (LC/MS) to quantify the trace amounts of these species of interest (SOI). The liquid chromatograph uses a mobile phase and column to separate the components of a solution and determine their identity by the order in which it comes out of the column. Mass spectroscopy measures the mass-to-charge ratio of the sample to quantify the amount of a known sample. I was able to use this technique with both pellet and film samples, using a solvent to extract the SOI from the solid sample to a liquid for analysis.

Problems Encountered – While these species of interest are seen virtually everywhere, there are very inconsistent levels of these contaminants in everyday recycled film. Therefore, to be able to look specifically at the mechanisms of these species, I needed to generate a control film with a known amount of each of these SOI. However, getting these species to diffuse into virgin film proved to be a challenge. I created batches of solutions laden with SOI and allowed virgin plastic film to be mixed in for up to a week. However, each time there was an indetectable amount of these species in the film. This meant that we would not be able to generate our own control film, but also that these species are likely not diffusing into the film naturally and may be embedding themselves in the films during processing.

Additionally, we were very interested in being able to model the movement of these species as they are being extracted from the film. We modelled the FlexLoop™ process in a 4L round bottom flask, heated with a hot plate and fit with a stirring attachment. The stir bar was used to rapidly agitate the solvent and film samples for maximum extraction. By taking samples of the solvent at intervals during the FlexLoop™ process, we could graph how much of these three species was being removed and at what time. We would expect this to movement to match that of classical Fickian diffusion. However, this is not what was seen during my studies and work is continuing to be done at P&G to understand the mechanisms of this process.

Results – The main reason for studying the FlexLoop™ process was to identify potential areas for process improvements, where the company could potentially reduce costs and energy during scale up. To reduce energy costs, I wanted to analyze the relevance of the mass transfer coefficient and determine whether stirring was essential to the extraction process. The mass transfer coefficient determines the factors in a solvent extraction system that control the rate the solute is transferred from one phase to another, in this case the factor being mechanical stirring. To analyze this, I conducted a study of two identical PCR samples being run through the FlexLoop™ process. One sample underwent high-speed stirring while the other had no stirring at all. I then measured the amount of species extracted over time and compared the two curves. I was able to determine from this study that there was very little change in extraction over time between the stirred and un-stirred samples, shown in the graph below. All three species exhibited similar behavior. Therefore, a viable option for reducing energy costs would be to eliminate or slow the stirring during extraction.

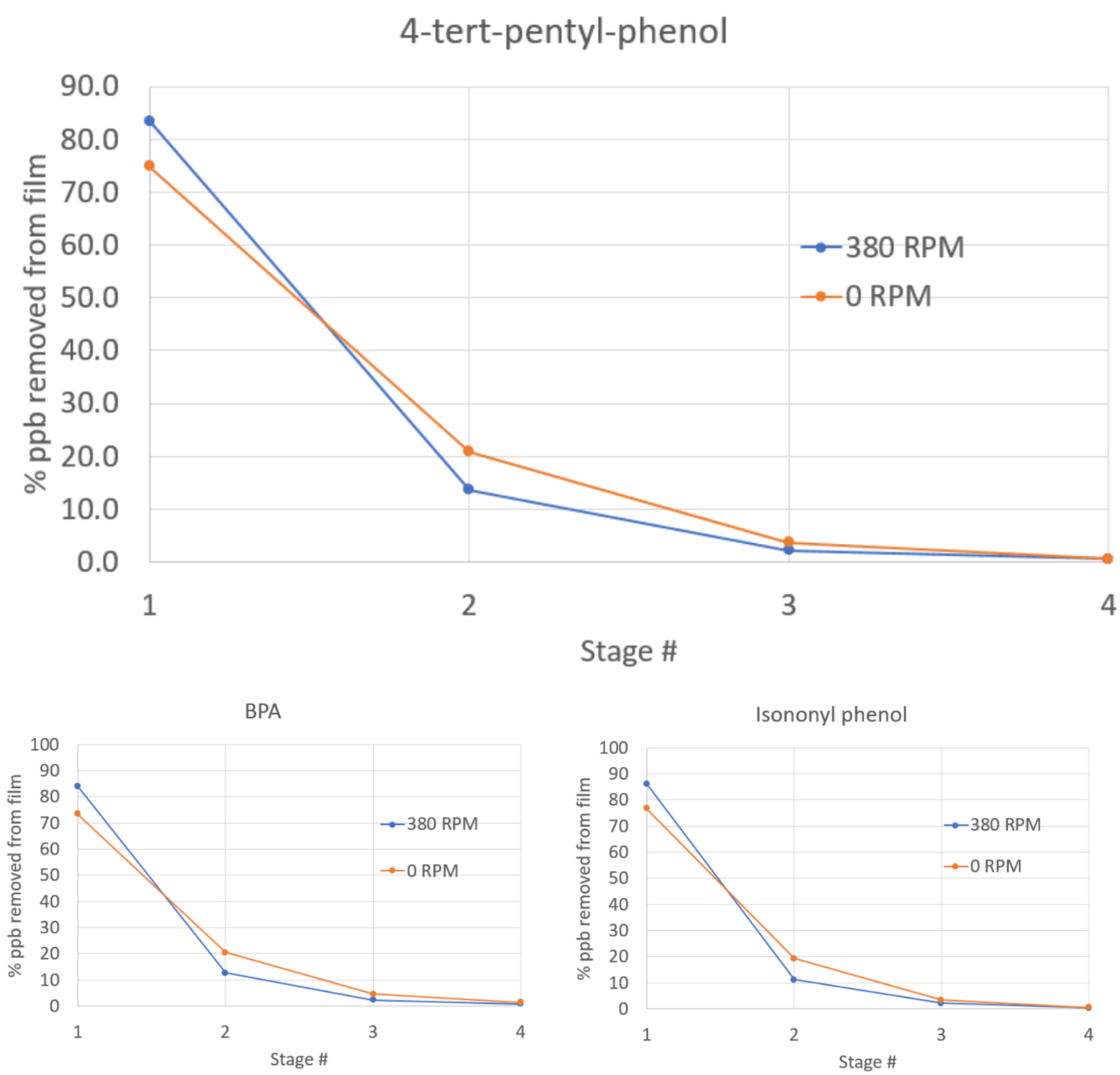

Figure 3. *Removal over time of 4-tert-pentyl-phenol remains unchanged despite changes in stirring speed. Nearly identical behavior is seen for isononyl-phenol and bisphenol A.*

The next potential area for improvement I identified was cleaning the used solvent for reuse. This would reduce material costs and minimize waste from the process. One option would be to use flash distillation to remove all unwanted species. However, this would require additional machinery and may have high energy costs. Another option I identified was using a solid adsorbent, like activated charcoal, to remove any unwanted species. This option is relatively inexpensive and requires minimal energy input. As shown in Table 2, I was able to model this successfully on the lab bench, using leftover solvent from three of the four stages of a previous FlexLoop™ run.

Table 2. *Adsorbent effective at removing SOI from used extraction solvent.*

Conc (ng/mL)	4-tert-pentyl-phenol		Bisphenol A		Isononyl phenol	
Stage 1	507	57	42. 5	<LOQ	498	<LOQ
Stage 2	570	<LOQ	88.5	<LOQ	548	<LOQ
Stage 4	548	<LOQ	91.8	<LOQ	453	<LOQ

Discussion - FlexLoop™ is very effective at reducing chemical contamination in film and resin samples. Studying the mechanisms of these trace contaminants, however, proves to be quite a challenge and could require additional testing.

Consumer Study

Along with the work I was doing in the lab, I became increasingly interested in seeing the consumer side of P&G. While the work we were doing may one day impact the products that reach consumer's homes, I wanted to have the opportunity to discuss with consumers what their thoughts were about recycled packaging. I decided to pursue an additional study that would allow me to talk to consumers face-to-face and understand their assumptions and perceptions about PCR materials. These results help to contextualize the technical work we did and generate consumer-driven recommendations for PCR implementations.

Methods – With the help of P&G's product research team, we sent out an interest survey and I selected seven respondents to meet with me for a 45-min interview. All participants had self-identified as eco-conscious and were often looking for alternatives to single-use plastic. Each interview was split into three discussions: a claims assessment, a material acceptance survey, and a material check.

Claims Assessment – The main goal of this discussion was to see how language in advertisements influenced how consumers perceived a product. I selected a series of 10 packaging claims that related to PCR or recycled materials and asked consumers the following questions:

> What does this mean to you?
> How does this affect your opinion of the product?
> How important is this to you?

Which would you MOST be interested in seeing on pack? Why?
Which would you LEAST be interested in seeing on pack? Why?

The full list of the claims discussed can be found in Appendix A. The claims that fared the best were ones with clear, actionable claims. Consumers responded very positively to claims with specific amounts and locations from where the material was sourced from, such as "Made from 100% ocean bound plastic." When companies shared their values through their claims, it gave them confidence in the company's mission and about purchasing their products.

Claims that fared the worst were ones with very general or vague wording. This gave consumers a lot of doubt as to the actual meaning of the statement and reduced their confidence in the company's values. They also reacted negatively to claims with unfamiliar logos or certifications, as these were deemed unclear in what they signified.

In conclusion, consumers are looking for transparency. They want to know actual amounts of PCR and from where it is being sourced. It's important to find ways to eliminate the uncertainty of recycled packaging and provide tangible sources that consumers will resonate with.

Material Acceptance – There are two main categories that fall under the term post-consumer recycled: post-household and post-commercial. Post-household is what you typically think of with recycled materials, final packaging that's been used, tossed into a recycling bin, picked up from the curb and brought to a recycling facility. Post-commercial, on the other hand, is only ever brought to a commercial setting. This is typically packaging used in transportation of final goods, like back-of-the-store stretch wrap or boxes. Post-commercial materials are generally cleaner and have less contamination than post-household, as they are not exposed to as much usage.

When asking the panelists what their impressions of these two terms were, most had a strong understanding of what post-household materials were. They were able to easily conceptualize what it would look like and where it would come from. Post-commercial, however, had a much more mixed understanding. This term was unfamiliar to post, and most panelists could not identify where it would come from or look like. After providing them with a surface level definition of both terms, I allowed them to use their own opinions and generalizations to choose which PCR they think would be a better fit for a variety of different household goods. The results of that discussion are displayed in Table 3.

Table 3. *Categories of household goods and what materials they considered to be a better fit.*

Category	Post-Household	Post-Commercial
Laundry Products (Liquid detergent, fabric softener, scented beads)	Strong fit	Moderate-weak fit
Shower Products (Shampoo/conditioner, body wash)	Strong fit	Moderate-weak fit

Skin Products (Lotions, make-up)	Strong fit	Moderate-weak fit
Bags (Grocery, trash, shopping)	Moderate-weak fit	Strong fit
Personal Care Products (Deodorant, grooming)	Strong fit	Moderate-weak fit
Household Cleaners (Multipurpose sprays, floor cleaner)	Strong fit	Moderate-weak fit
Personal Health Care (Vitamins/supplements, powder, Metamucil)	Moderate-weak fit	Moderate-weak fit
Feminine Protection (Pads, tampons)	Moderate-weak fit	Moderate-weak fit
Dish Care (Dish soap, dishwashing tablets)	Strong fit	Moderate-weak fit
Baby Care Products (Diapers, wipes)	Moderate-weak fit	Moderate-weak fit

In general, panelists preferred post-household materials for most household packaging applications. One stated "keep that cycle going," indicating that by recycling a certain packaging for the same application contributed to their understanding of the circular economy. Another stated, "[post-consumer] not necessarily worse, just where my head initially went," reiterating how consumers are more familiar with the term post-household and therefore seem more comfortable with it having it being used in their packaging. Four out of the seven panelists reasoned that post-commercial materials would be a worse fit overall, as it may be exposed to harsher chemical, pesticides, or exhaust fumes during transportation. Three panelists accurately identified the opposite, that post-commercial materials would be cleaner, easier to obtain in abundance and more efficient to use.

From this discussion, it's clear that there is no advantage to specifying post-house or post-commercial when advertising PCR materials. It did, however, show that eco-conscious consumers are comfortable with PCR materials in almost all packaging applications. The three categories of most concern to consumers were baby care, feminine care, and personal health care. These product categories being the closest to the body, it makes sense that most consumers would be more cautious about what might be in the packaging, as opposed to maybe a cleaning product.

Material Check – For the last piece of our discussion, I showed two physical mockups of a Pampers diaper package, one made from 100% virgin film and the other with 50% PCR material. Other than the material itself, the packages were identical. I allowed the panelists to touch and hold both packages and tell me what differences they noticed and which item they preferred.

Two of the panelists preferred the PCR packaging, stating that it was thicker and less "plastic-y". They also stated they liked the matte finish of the recycled packaging, that it was visually "more eye-catching" and felt more natural, whereas the shine on the virgin film felt "unnecessary and unnatural". Four of the panelists stated they had no preference between the two but would prefer to buy the recycled packaging if there was a visual indication or label on the printing. Most said they would buy the PCR packaging if there was no difference in price or count. The last remaining panelist expressed hesitation with buying the PCR packaging. They had said it has an unpleasant "plastic-y, vinyl-y feel" and would be most concerned with whether the recycled package could be recycled again.

From this final discussion, it's clear that a visual claim indicating the recycled content of the packaging material would be important for consumer's decision to purchase. Over half of the participants stated that an obvious visual indication, such as a label, would encourage them to buy the recycled version over the original.

Team Deliverables

In this section, I will detail the work and deliverables completed by the entire P&G MDP team.

Decision Tree — To create a resin fingerprint, the team constructed a preliminary decision tree to assign a ranking to the technical tests as seen in Figure 2. The decision tree consists of four gates: preliminary characterization, processing and rheology, mechanical and optical properties, and aging and stability. Within each of these gates, there is a hierarchy of the instruments. Analyzing the data from these instruments, we ordered them within each gate based on the utility of data we were able to collect and efficiency of the test method.

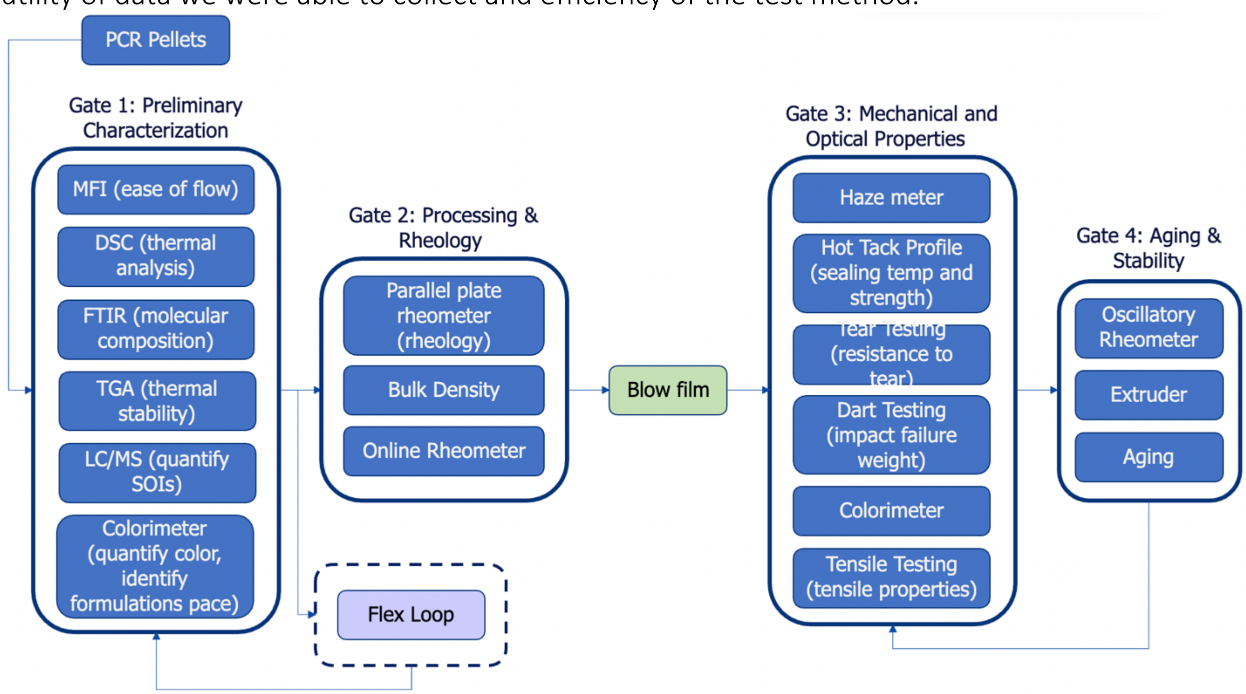

Figure 3. *Decision tree for evaluating PCR resins.*

Starting with the preliminary characterization, these are relatively low cost, low effort tests that can be highly indicative of the resin's success further down the line. If the resin cannot pass a given threshold in this gate, specifically SOI threshold values, we would recommend integrating FlexLoop™ to further clean the resins sample and remove stubborn contamination and residue. We would then recommend that the cleaned samples return to the beginning of Gate 1 and the tests be repeated. Gate 2 focuses on processing and rheology testing that will give insight into the resin's success on the blown film line. Past that gate, the resins would then be blown into a film and sent to Gate 3, where the film's mechanical and optical properties would be analyzed. Finally, to ensure the film properties can be successful on-shelf and do not degrade over time, the films will undergo aging and stability testing in Gate 4, where after a given period the mechanical and optical properties would be reanalyzed to see if any changes are seen. With the decision tree and the detailed test methods provided, future researchers can follow the steps we outlined to efficiently analyze an incoming PCR and determine its success in a packaging application.

Heat Map — To quickly determine which PCR is suitable for use in specific P&G packaging applications, we have created a preliminary heat map that indicates property deviations between PCR and control film: 80% LDPE, 20% LLDPE at a 50-micron gauge.

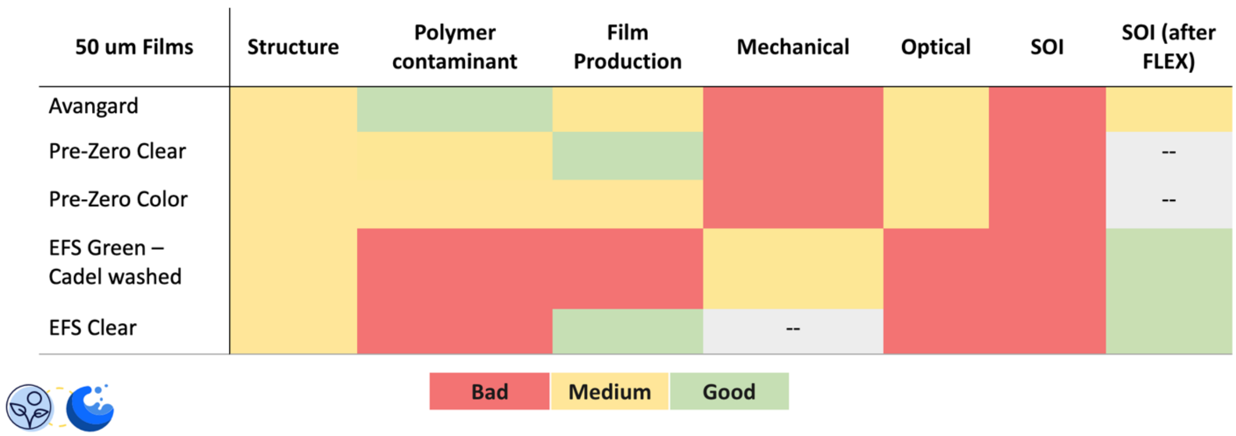

Figure 4. *Heat map for 50 um films, representative of P&G applications.*

Seven PCR resins from various suppliers were characterized and evaluated on their material properties, processability, and performance. Sample details are listed below in Table 2. These samples were compared to generated control blends of virgin polyethylene resins with specific amounts of polymer "contaminants" added. From our testing of these materials, we have developed detailed test methods for a variety of instruments and trained others at P&G on these test methods. As they continue testing additional PCR materials, they will expand their portfolio of recycled materials and improve integration of these materials.

Table 2. *Control and PCR resins and films tested to obtain complete portfolio of material and resin properties.*

Samples	Control	PCRs

Pellet		• LLDPE spiked with HDPE (0.5 - 10wt. %) • 80/20 LLDPE/LDPE spiked with PP (0.5 - 10wt.%) • LLDPE/LDPE ○ 100/0, 95/5, 90/10, 85/15, 80/20, 70/30	• PCR #1 • PCR #2 • Post-commercial #1 • Post-commercial #2 • Post-household #1 • Post-household #2 • Post-household #3
Film	10um	• 100% LLDPE	• 100% PCR • 50/50 PCR/LLDPE • 25/75 PCR/LLDPE
	50um	• 80/20 LLDPE/LDPE	• 100% PCR • 50/50 PCR/ (80/20 LLDPE/LDPE) • 25/75 PCR/ (80/20 LLDPE/LDPE)

Pellet Characterization — Contaminants that have been identified are polypropylene (PP), high density polyethylene (HDPE), nylon, EVOH, PET, legacy pigments, cellulose, bisphenol A (BPA), 4-tert-pentyl-phenol, and isononyl phenol. All PCRs were found to have >80% LLDPE. Rheological analyses were performed using parameters like zero-shear rate viscosities, shear thinning indices, melt strength to predict processability.

Film Characterization — On the pilot blown film line, we produced monolayer films: 2 types of control film and 7 types of PCR film. To test the hypotheses from rheological analyses, processability was analyzed during the blown film production process. We conclude that rheology is very predictive of processability; from the rheological data, we predicted which of our given films would process the best, and that was true based on the film production process analysis.

Mechanical tests were conducted on the monolayer film to quantify the mechanical properties, heat sealing properties, and defects. Raw data, instead of averages, were used to capture the variation in performance across samples. We will test multilayer films to investigate correlation between monolayer and multilayer films extrapolate the performance of multilayer films. While ImageJ is a useful tool for image analysis and quantifying defects, it is difficult to standardize the thresholding across all the images. we recommend investigating alternative thresholding methods should be researched methods to better characterize the size and aspect ratio of contaminants.

Finally, Colorimeter and Rhopoint Hazemeter were used to characterize composition and optical properties of films. Films are evaluated on the following parameters: lightness, haze, sharpness, opacity, waviness, and clarity.

FLEXLOOP™ Sidebar — FlexLoop™ is a process developed to improve the removal of chemical contaminants from PCR films, using solvents to extract species of interest (SOI) from the surface and bulk of the film which are then detected with Liquid Chromatography/Mass Spectroscopy (LC/MS). To scale up this process from the lab bench, we worked to develop a constitutive understanding of the process and the behavior of these species, as well as explore opportunities for optimization. While the behavior of these species contradicts traditional models of diffusion, FlexLoop™ proves to be useful in reducing SOI content in PCR resins and films.

Technical Data Sheets – The data obtained during all our testing has been compiled into comprehensive data sheets. These are to be using by P&G business units to easily determine the PCR's material properties and determine whether they would be successful in their desired packaging application.

Conclusion

This was a very fascinating project that challenged both my technical and critical thinking skills. I not only was able to enhance my research and lab skills, but I was also able to look at research from a whole new framework. Looking at this huge problem of plastic waste, it was really rewarding to work on a project that would help to tackle it in a very real way. To work with a company with millions of consumers throughout the globe, I know that the work I and my team was able to complete will impact the lives of many for years to come. P&G will continue to use the testing methods we created to expand their portfolio of recycled flexible films and meet their sustainability goals.

Appendix A


MADE FROM
RECYCLED
MATERIAL


BOTTLE NOW
MADE FROM
100%
OCEAN BOUND
PLASTIC*


BOTTLES
NOW MADE FROM
100%
RECYCLED
PLASTIC


I'M MADE
WITH
RECYCLED
PLASTIC*


POST
CONSUMER
RECYCLED
COMMITTED TO REDUCING
PLASTIC WASTE


100%
RECYCLED

	
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