OPTIMAL PROCESS DESIGN FOR RECYCLED PET BLENDS IN INJECTION MOLDING

by

PERRY H. JOHNSON

A Senior Project submitted

In partial fulfillment

of the requirements for the degree of

Bachelor of Science in Manufacturing Engineering

California Polytechnic State University

San Luis Obispo

Graded by:__________________ Date of Submission:________________

Checked by:_________________ Approved by:______________________

Table of Contents

ABSTRACT

This case study on optimization of blends with process variables shows how application of advanced tools of Design of Experiments can simultaneously optimize a mixture formulation and processing conditions, taking advantage of complex interactions in the system. The effect of recycled PET and virgin PET on tensile strength and stiffness was studied. The optimal amount of mixture components to produce maximum recycled-content products is determined. As the results of doing systematic experimentation, using mixture experiments, the quality of recycled plastic products can be improved and becomes more robust to variations at the optimal thermal operating settings. This is done through numerical optimization approaches that are available, using a mixture-process-variable simplex experimental setup to generate a predictive model of mechanical response. Then manipulating the predictive models is completed to find the "sweet spot" for both mixture and process variables.

INTRODUCTION

The last few decades has witnessed significant increase in world population. This has caused considerable increase in the demand for low cost living conditions, which in part leads to dramatic increase in the consumption of plastics. Past statistics show that worldwide annual production of plastics is over 100 million tons per year [1]. There is approximately 3 million tons of plastic waste produced from that, of which environmental agencies report around 80% reaching landfill sites [2]. The practical solution is to recycle or reuse the plastic that has already been produced. In fact, recycling plastic has many advantages:

- Using a resource that would otherwise be wasted
- Reducing or preventing the amount of waste going to landfill
- Reducing the costs involved in the disposal of waste, which ultimately leads to savings for the community
- Providing employment
- Protecting natural resources
- Reducing pollution

Over the last many years, the focus of plastic recycling has changed. Earlier, the focus was on educating and encouraging the public and industry to recycle. As the necessity and incentives to reduce the volume of waste materials entering our landfills sunk into the populace's minds, market forces became such that millions of pounds of plastic waste heading for the landfill now had some value. The question then turned to one of how to collect this material and convert it into a marketable raw material. The economies dictate that recycled materials are the more expensive engineering resins, such as polycarbonate, nylon, and PET [3]. In some cases, the cost of recyclable materials also exceeds the cost of raw materials due to processing and transportation costs. In the United States, the recovery of postconsumer plastics for 2009 was approximately 7% (U.S. EPA 2011) [4]. So it is desirable to find uses for recycled plastic material that can be justified by having a similar cost to a virgin material's alternative solution.

Plastics that can be recycled are called thermoplastic polymers. Some typical examples of thermoplastic polymers are polyethylene terephthalate (PET), polypropylene, polyethylene, polycarbonate, etc. Plastic material selection for many materials (plastics, metals, etc.) can be a highly complex process if not properly approached particularly when using recycled plastics. Its methodology ranges from a high degree of subjective intuition in some areas to a high degree of sophistication in others. When selecting an additive for a mixture, it is important to take into account also the potential side-effects it may have on other properties. In some cases, the cost of the system will be reduced, but at a penalty in other directions such as mechanical properties that can influence performance of the fabricated product [5]. Any attempt to compare mixed plastic with other conventional materials (metal, wood, glass, etc.) on a straight property-for-property or

a straight cost-for-cost basis is doomed to failure from the very start. There are just too many different types of grades and formulations grouped under the overall heading of mixed plastic (Figure 1) [6].

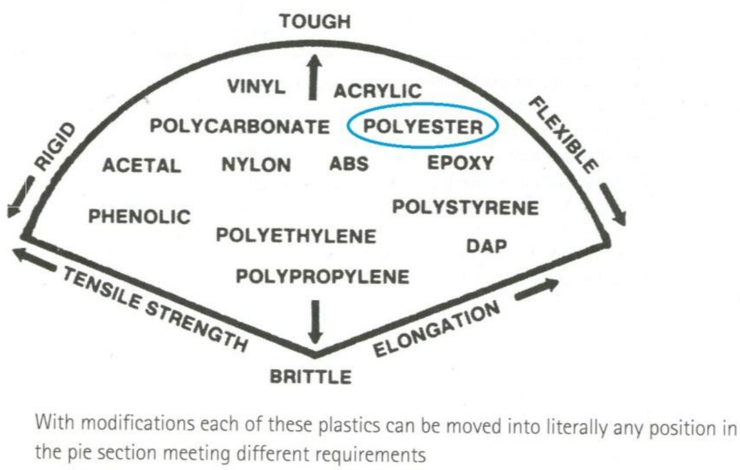


Figure 1

Certainly, polyester (PET) is one of thermoplastic polymers that is easily recycled and molded. The main driving force responsible for the increased recycling of post-consumer PET is its widespread use, particularly, in the beverage industry which has made PET the main target for plastic recycling. In this particular study, the two sources of material is virgin PET and recycled post-consumer PET coming from various bottles. The scrap PET is in flake form, but in a heterogeneous deposit soiled by many types of PET bottle (mainly clear). The chance presence of contaminants generates some problems such as cleavage of chains, a reduction in molecular weight, and a decrease in intrinsic viscosity leading to a decrease in mechanical properties of material [7]. But to reduce scope in conducting research later developed, it is assumed that the PCR (post-consumer resin) has very little contamination, thus how the material was purified (selected externally) is not considered. The process that this study is concerned with is injection molding, which is an advantageous due to the deliberate stretching of the molecular chains that happens when melted plastic is extruded; so inherent strengths of the chains are more nearly realized than they are in their relaxed configurations. Thermal effects in processing are especially important because they dictate crystallinity attained, which is significant to strength [8].

Even though producing recycled plastic products can reduce environmental impact and cost of the product, quality of the product should be also considered. Many companies hesitate to use regrind and postconsumer resins (PCRs) because of the extensive testing required to identify plausible uses and processing parameters [9]. The problem with using these low cost (self-produced) raw materials is their supposed fluctuating processing characteristics and the variability in mechanical properties [9]. Currently, many companies process either 100% virgin material or virgin material with a small percentage of regrind from industrial processing. In many cases, the regrind supply exceeds established thresholds, resulting in downcycling or

landfilling of significant quantities of regrind [9]. As discussed earlier, thermal processing conditions as well as mixture conditions affect quality characteristics, so a method that characterizes recycled plastic by both processing parameters and mixture state could dramatically increase the supply of acceptable recycled plastic over the generic threshold approach. This project aims to demonstrate the economic and technical feasibility of using a optimal ratio of recovered recycled PET plastic (RPET) to virgin PET pellets in a specific injection molding process. This is done in part by providing polynomial equations that can predict the mechanical response based on machine settings and blend state, conceived through a designed experiment.

Designed experiments are a very powerful tool to quantify the effect of the factors on the response in production processes, or, ultimately, to determine which factor level combinations provide optimal output quality. A key feature of designed experiments is that the researcher varies the factor levels systematically, according to a certain experimental plan, which is developed in the Design section. Thus, the Yield Stress and Young's Modulus are the two selected quality objectives. Thus this paper works to analyze Yield Stress and Young's Modulus results based only on three thermal processing parameters of a binary mixture design of PET/RPET in Injection Molding.

Powerful desktop computer tools now make it easy to optimize paint formulations. Aided by the computer, statistically based Design of Experiments (DOE) — a proven method for making breakthrough improvements in cost and performance — can be applied. The latest versions of dedicated DOE software exhibit more versatility than ever to create optimal designs that handle any combination of mixture components and processing factors (Design Expert by Stat-Ease is used). These computer programs easily manipulate almost any number of responses in powerful optimization routines that reveal "sweet spots" — the operating windows that meet all specifications at minimal cost.

BACKGROUND

PET Plastics

In order to more fully understand internal problems that may arise in processing, more specific knowledge of PET is required. Thermoplastic material is composed of carbon and hydrogen atoms joined together forming long-chain high-molecular-weight products. These features determine the mechanical properties such as density, stiffness, tensile strength, flexibility, hardness, brittleness, elongation and creep characteristics [10]. The molecular arrangements largely depend on the process characteristics used for manufacturing of PET products. In PET longer the main chain, the greater the number of atoms, and consequently, the greater the molecular weight.

The crystalline term is used to describe a thermoplastic (TP) of a highly ordered structure with sharp melt points. They do not therefore soften gradually as the temperature increases but tend to remain hard until a given quantity of heat has been absorbed, at which point they rapidly change to a low viscosity melt. The mechanical properties are greatly influenced by this melt flow action. They are anisotropic in flow, shrinking less in the direction of flow than transverse to it [11]. Crystalline behavior identifies its morphology; that is the study of the physical form or structure of a material. They are usually translucent (glassy) or opaque, which the PET is, and generally have higher softening points than amorphous plastics. Since commercially perfect crystalline polymers are not produced, they are identified technically as semi-crystalline TP's. The crystalline TP's normally have up to 80% crystalline structure and the rest is amorphous, with recycled content being more amorphous.

As the consequence – the crystallinity of injection molded parts can be influenced by processing conditions like mold temperature or melt temperature. When the mold temperature is higher, the crystallinity degree of molded parts is also higher. This feature of injection molded parts directly influences their mechanical properties – parts of higher crystallinity degree have higher values of hardness, tensile strength, wear resistance and better dimensional stability.

A wide range of applications is possible because of the excellent balance of properties PET possesses and because the degree of crystallinity and the level of orientation in the finished product can be controlled. Polyethylene Terephthalate does not reach its optimum properties until the level of crystallinity is raised by special processing and/or the molecules are oriented [12]. For packaging applications PET is used because it combines optimal processing, mechanical, and barrier properties. Very few other materials offer such a range of processing and property variables. In general, Polyesters (PET) are easy to process. Nevertheless, specific

properties should be noted such as an extrusion temperature of 480°F [13] can be the basis for early process design (more property information is provided in Appendix).

If small amounts of moisture are present, PET resins are inherently sensitive to degradation in the melt. This degradation process, which results in a lowering of the molecular weight (breakdown of the polymer chain), is known as hydrolysis. To prevent this effect, the moisture content of the granules should be maintained at a low level if at all possible. In general, to obtain good parts of constant quality during production, PET grades should be dried to a level below 50ppm (parts per million). Reaching the desired moisture level typically requires drying at 212-248°F during 4-6 hours. Higher temperature settings should be avoided to prevent material discoloration. If there are no heating hook-ups for the mold, running several shots until it has heated up sufficiently is an alternative.

Injection Molding

The term Injection Molding is an oversimplified description of a quite complicated process that is controllable within specified limits. Melted or plasticized plastic material is injected by force into a mold cavity. The process is one of the most economical methods for mass production of simple to complex products. Three basic operations exist. They are the only operations in which the mechanical and thermal inputs of the injection equipment must be coordinated with the fundamental behavior properties of the plastic being processed. They are heating of the plastic to molding temperature, inject it, and then cooling (or solidify) the product in the mold [14]. The programming of different injection speeds and pressures during the forward travel of the screw or plunger greatly aids in filling cavities properly. The clamp tonnage of a machine must have sufficient locking force not to cause the parting of the hold halves; it resists the force of melted plastic moving at high pressures into the mold halves. If the mating surfaces of the mold are forced apart, even a few thousands of an inch (depending on type of plastic), fluid plastic will flow out and produce flash. A diagram of a typical injection molding machine is shown below along with a co-rotational screw that can be visualized as replacing the plunger/ram system shown (Figure 2,3 [14]).

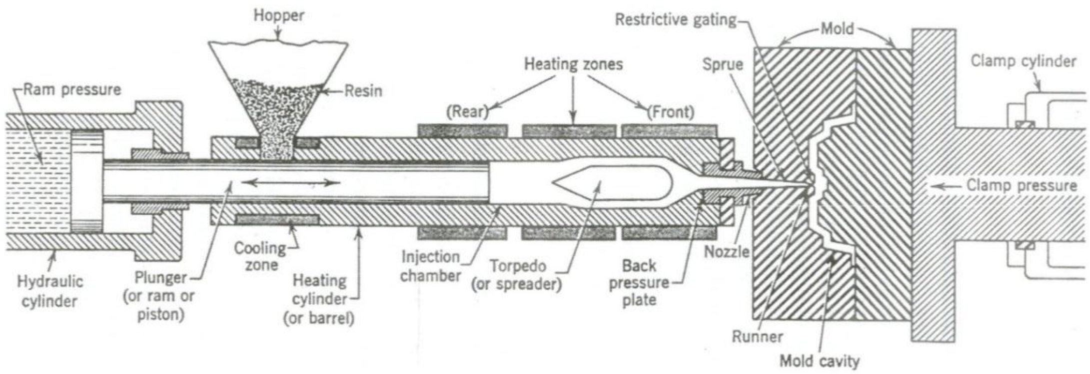


Figure 2

Figure 3

Design of Experiments

Traditional experimental design is focused on several factors that are varied in the form of treatments, but in this study there is special consideration to be given for blend states. Having a blend means that the components cannot be varied independently because they, in combination, make up the whole mixture. On the other hand, process variables can and will be changed in conjunction with blend states. This is something that needed looking into as past classroom experience was not sufficient. So, to make this project more approachable, a previous statistically significant design of this type of experiment was researched. It was carried out by an investigator who looked into mixture design with the inclusion of process variables. John Cornell had developed an adequate mathematical transformation of the variables leading to polynomial models that describe the influence of the composition on the material flow properties using an optimum experimental set [15]. The aim of these polynomial models is not to study in depth the physical phenomena, but to apply them as practical models for the optimization of blend composition. Details of how the experimental analysis will be produced in further sections, but it can be noted that the thermal processing variables are nozzle, front barrel, and rear barrel temperatures.

Design of Experiments analysis provides a solid estimate of the value of independent variables in every possible combination by varying the values of all the factors in parallel. This approach determines not just the main effects of each factor, but also the interactions between the factors. But the statistical knowledge required to perform DOE and generate polynomials from scratch can be an obstacle to its use. Design-Expert is easy to use program from Stat-Ease that will be used to complete DOE analysis.

Economic Justification

Setting aside environmental concerns, the economic success or failure of plastics recycling relies on two variables: the cost of the raw materials used to make virgin plastic, petroleum and natural gas, and the cost of recycling versus the cost of disposal, which fluctuates based on a city's proximity to recycling centers and the price to dump in local landfills. Nonetheless, the cost of recycling a bottle versus making a new one simply varies, depending where the bottle is and what the unpredictable price of oil happens to be.

It is found that a manufacturer of injection molded parts that used their own blend of recycled resin was actually incredibly successful, using methodology somewhat similar to this study. AGS Technology from Schaumburg, Illinois lowers the cost of the plastic-injection molded parts it manufacturers by using recycled plastic as its raw material [16]. They are an ISO/TS 16949-certified injection molder that uses its proprietary recycled plastics to produce high-quality molded components, primarily for the automotive and durable goods industries (Ford, Chrysler, and Chevrolet). Due to the recycled raw material cost being substantially less than virgin materials, the company is able to pass on to its customers significant cost savings on the resultant molded components.

AGS Technology formulates its own raw material, matching its properties to corresponding virgin resins and to customer specifications. Traditional plastic compounders convert recycled plastic into a raw material for injection molding applications by grinding the recycled plastic and then extruding it into pellets, which is how recycled content can sometimes exceed the cost of virgin content. Conversely, AGS Technology molds its formulated plastic regrind directly and bypasses the expensive extruding/pelletizing operation from an outside source. Often, molders who might have saved 10 percent in raw material costs by using recycled plastics see these cost savings evaporate because of the manufacturing problems they will experience while processing them. When a molder runs parts with virgin material, the runs are consistent. But with recycled material, there is more variability, so the scrap rate may go up. These types of things would have incurred costs and eliminated any potential savings. So therefore it is beneficial to optimize the recycled blends, reducing variability.

"Plastics News," a trade magazine, lists the recent price of PET virgin bottle resin pellets between 103 and 105 (104 avg.) cents a pound, compared to only 66 to 74 (70 avg.) cents a pound for PET recycled bottle flake [17]. For a brief example, we can say that about 250,000 lbs. of material is used for a certain job per year, and it uses an arbitrary 50-50 blend. The cost differential between the recycled material and the corresponding virgin resin is $0.34 per lb. That's an annual savings of $42,500 for the customer ($260,000 for plain virgin PET, and $217,500 for the 50-50 blend), without sacrificing any quality standards because the performance

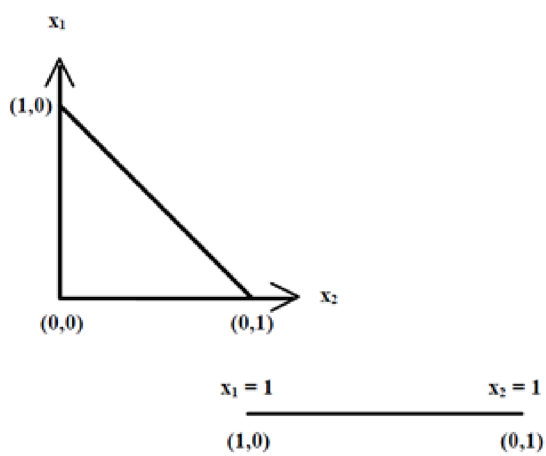
characteristics have been optimized. What could be a better testimonial to the economic benefit of using recycled plastics?

Christopher Racelis, AGS President, explains that their “business model is that we’re a low-cost producer for ‘shoot-and-ship’ parts, using recycled plastics as our raw material.” The products that they manufacture are material-intensive, so the bulk of the cost—it could be 50, 60, 70 percent—is in the raw material. So “the best way to remove cost from the part is to mold the most cost-effective raw material. And that is recycled plastic,” Racelis mentions [16].

DESIGN

DOE Basis

The ultimate goal of this experiment is to reveal (and be able to predict) at which point is the mixture the strongest (or suitable) based on a treatment of process settings. This is also looked into on a basis of maximizing the recycled content, in order to become mechanically comparable to virgin characteristics, which provides reduced cost depending on how demanding an application is. As defined by Cornell (1990) [15], process variables are factors in a mixture experiment that do not form any portion of the mixture, but whose levels, when changed, affect the blending properties of the ingredients. Process variables can vary independently from the mixture variables. To model the response of the combined experiment, a combination is made of the model in the process variables and the mixture model, where “q” is the number of components and “z” is an individual process factor. As a result the factor space reduces to regular (q-1) dimensional simplex. For this experiment, q=2 so it is a straight line, $x_1+x_2=1$. x_1 is PET and x_2 is RPET.



Simplex factorspace for a 2 component mixture

Figure 4

The resulting experimental region forms a simplex, a geometric figure with one more vertex than the number of dimensions. In two dimensions, a simplex is a triangle. In three dimensions, a simplex is a tetrahedron, and so on, but in this experiment the limit is to one dimension for mixtures (without the inclusion of process variables).

Simplex Lattice Solution Space

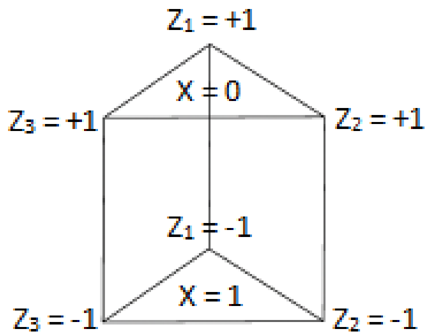


Figure 5

Figure 5 shows the experimental region, or "mixture space," for the two unconstrained components in addition to the process variables. The tops of the resulting prism (a simplex) represent the maximum allowed content for a specific component. At any point in the region, the ingredients total to 100%, in addition to the three two-factor arrangements. A design used to explore the whole factor space is the "{q,z} simplex lattice design". Each mixture variable varies by equally spaced values from 0 to 1 (Scheffe) [18]:

$$x_i = 0, 1/m, 2/m, ..., 1; \text{ for } i = 1, 2, \ldots, q$$

To add statistical power, 6 blend states were created by stating that blends are made by fifths (m=5, q=2). The term "m" is simply referring to how spread out the blend design is. In this design all possible treatment combinations with these proportions for each mixture variable are used, so the blends are as follows:

(x_1,x_2):
(1,0); (4/5,1/5); (3/5,2/5); (2/5,3/5); (1/5,4/5); (0,1)

As a matter of fact, it is theoretically possible that blends of more than three samples could be more informative. Also, and more likely, it is possible that considering other proportions than 50-50 or thirds in the binary blends, yields better designs. Therefore, design of experiments can be likened to design of bridges: when in doubt, build it stout. In other words, if you're not sure what to do, invest in a higher-order polynomial. The added blends needed to fit the bigger models may reap great benefits through discovery of unexpected interactions. The {q,1} simplex lattice design is appropriate for first order models, while the {q,2} and {q,3} are suited respectively for a second order and special cubic canonical polynomial mixture model. This experiment is a {2,3} Simplex Lattice.

Creating a Mathematical Model

The outcome of a statistically significant DOE is a polynomial model that can be used to predict the response at any combination of tested variables. As you can see from the derivation below, the models for crossed mixture-process designs can be very cumbersome, even for a relatively simple study like the one done on the PET/RPET.

Models for analyzing data from mixture-process variable experiments are usually obtained by combining traditional Scheffe type models for the mixture variables with response surface models for the process variables as outlined in Cornell's work [15]. For example, a common mixture-process variable model is obtained by crossing the second-order Scheffe model,

$$E(Y) = \sum_{k=1}^{q} \beta_k x_k + \sum_{k=1}^{q-1} \sum_{l=k+1}^{q} \beta_{kl} x_k x_l,$$

where q is the number of mixture components and x_k is the proportion of component, k, in the mixture, with a main-effects-plus-two-factor-interactions model in the process variables,

$$E(Y) = \alpha_0 + \sum_{k=1}^{m} \alpha_i z_i + \sum_{k=1}^{m-1} \sum_{l=k+1}^{m} \alpha_{ij} z_i z_j,$$

where m is the number of process variables and z_i represents the setting of the i^{th} process variable. The combined model can be written as:

$$\begin{aligned} Y = & \sum_{k=1}^{q} \gamma_k^0 x_k + \sum_{k=1}^{q-1} \sum_{l=k+1}^{q} \gamma_{kl}^0 x_k x_l \\ & + \sum_{i=1}^{m} \left[\sum_{k=1}^{q} \gamma_k^i x_k + \sum_{k=1}^{q-1} \sum_{l=k+1}^{q} \gamma_{kl}^i x_k x_l \right] z_i \\ & + \sum_{i=1}^{m-1} \sum_{j=i+1}^{m} \left[\sum_{k=1}^{q} \gamma_k^{ij} x_k + \sum_{k=1}^{q-1} \sum_{l=k+1}^{q} \gamma_{kl}^{ij} x_k x_l \right] z_i z_j + \varepsilon \end{aligned}$$

In this expression, the terms

$$\sum_{k=1}^{q} \gamma_k^0 x_k + \sum_{k=1}^{q-1} \sum_{l=k+1}^{q} \gamma_{kl}^0 x_k x_l$$

correspond to the linear and non-linear blending properties of the mixture components. Each term

$$\left[\sum_{k=1}^{q} \gamma_k^i x_k + \sum_{k=1}^{q-1} \sum_{l=k+1}^{q} \gamma_{kl}^i x_k x_l \right] z_i$$

contains the linear effect of the i^{th} process variable z_i on the components' blending properties, and terms of the form

$$\left[\sum_{k=1}^{q} \gamma_k^{ij} x_k + \sum_{k=1}^{q-1} \sum_{l=k+1}^{q} \gamma_{kl}^{ij} x_k x_l \right] z_i z_j$$

describe the interaction effect of process variables z_i and z_j on the blending properties (derivations credit [19]), which produces the 21 term polynomial (Y = Yield Stress, E = Young's Modulus):

Predictive Model

$Y \text{ or } E = \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2$
$+ \beta_{11} x_1 z_1 + \beta_{12} x_1 z_2 + \beta_{13} x_1 z_3 + \beta_{21} x_2 z_1 + \beta_{22} x_2 z_2 + \beta_{23} x_2 z_3$
$+ \beta_{121} x_1 x_2 z_1 + \beta_{122} x_1 x_2 z_2 + \beta_{123} x_1 x_2 z_3 + \beta_{112} x_1 z_1 z_2 + \beta_{113} x_1 z_1 z_3 + \beta_{123} x_1 z_2 z_3 + \beta_{212} x_2 z_1 z_2$
$+ \beta_{213} x_2 z_1 z_3 + \beta_{223} x_2 z_2 z_3 + \beta_{1212} x_1 x_2 z_1 z_2 + \beta_{1213} x_1 x_2 z_1 z_3 + \beta_{1223} x_1 x_2 z_2 z_3$

This predictive model will be used to generate response surface graphs, which make interpretation much easier than looking at all the coefficients. It is a linear crossed with quadratic model, which proves to be robust. However, upon dissecting the equation, notice that the first line contains only mixture components (x-variables). It represents the blending properties, averaged over the various process conditions. The second line of the equation reveals the linear effect of process factors (z_1, z_2, and z_3), which shifts the mean response at any given combination of mixture components. The last lines of the equation represent interactions between process factors and the mixture. When these complex interactions are present, the shape of the response surface changes as process conditions are varied.

Since blend states are hard to change factors during experimentation (injection molding works with one compound at a time), Block Designs for mixture experiments are employed. Groups of mixture blends are assumed to differ from other groups or blocks by an additive constant ("fifths"). A design is said to block orthogonally with respect to the blending properties of the components if the estimates of the blending properties in the fitted model are uncorrelated with and are unaffected by the effects of the blocks. The order in which experiments are should be randomized though to avoid influence by uncontrolled variables such as tool wear, and ambient temperature. These changes, which often are time-related, can significantly influence the response. If the run order is not randomized, the DOE may indicate factor effects that are really due to an uncontrolled variable that just happened to change at the same time.

The experimental design is shown below (48 runs, with two replications):

Treatment	Run Order	Z1	Z2	Z3	Blend (%PET)	Y (MPa)	E (GPa)
1	3	-1	-1	-1	100		
2	2	1	-1	-1	100		
3	6	-1	1	-1	100		
4	7	1	1	-1	100		
5	5	-1	-1	1	100		
6	8	1	-1	1	100		
7	1	-1	1	1	100		
8	4	1	1	1	100		
1	3	-1	-1	-1	80		
2	7	1	-1	-1	80		
3	2	-1	1	-1	80		
4	8	1	1	-1	80		
5	5	-1	-1	1	80		
6	1	1	-1	1	80		
7	6	-1	1	1	80		
8	4	1	1	1	80		
1	1	-1	-1	-1	60		
2	7	1	-1	-1	60		
3	6	-1	1	-1	60		
4	8	1	1	-1	60		
5	3	-1	-1	1	60		
6	4	1	-1	1	60		
7	2	-1	1	1	60		
8	5	1	1	1	60		
1	3	-1	-1	-1	40		
2	7	1	-1	-1	40		
3	6	-1	1	-1	40		
4	5	1	1	-1	40		
5	2	-1	-1	1	40		
6	8	1	-1	1	40		

7	1	-1	1	1	40		
8	4	1	1	1	40		
1	6	-1	-1	-1	20		
2	2	1	-1	-1	20		
3	5	-1	1	-1	20		
4	1	1	1	-1	20		
5	7	-1	-1	1	20		
6	8	1	-1	1	20		
7	3	-1	1	1	20		
8	4	1	1	1	20		
1	8	-1	-1	-1	0		
2	4	1	-1	-1	0		
3	3	-1	1	-1	0		
4	1	1	1	-1	0		
5	2	-1	-1	1	0		
6	6	1	-1	1	0		
7	5	-1	1	1	0		
8	7	1	1	1	0		

Injection Molding Parameters

All parameters other than blend state and temperature (nozzle, front/rear barrel) that are used run the injection molding process will be held constant for as long as possible, and are determined on site due to variances in machine design.

Cylinder temperature settings

As a standard, a flat or slightly increasing temperature profile should be applied from the feeding section to the nozzle of the cylinder. These are general indications that may require specific adaptation, but the actual parameters will be set as follows according to initial PET research:

z_1 (nozzle temperature) = 500°F (low, -1), 520°F (high, +1)
z_2 (barrel temperature, front) = 470°F (low, -1), 490°F (high, +1)
z_3 (barrel temperature, rear) = 475°F (low, -1), 495°F (high, +1)

These temperatures are necessary to produce well crystallized PET parts.

Injection rate

The preferred injection rate should be high to fill the mold quickly so as to obtain best surface quality and maximum chain orientation. A high injection rate is also favorable to avoid poor parting line quality and premature freezing of the gate during filling. In the case of visible defects in products, the injection rate will be reduced, provided that the defects are not caused by insufficient venting.

Holding pressure and time

Holding pressure is preferably 40-60% lower than injection pressure. This compensates for volumetric shrinkage for solidifying and crystallizing melt. The holding pressure must be sufficiently high to prevent sinks in the thickest section of the product, but on the other hand, it should not be set so high that the product starts to flash or residual stresses are built-in. Holding time should be prolonged proportionally as wall thickness and gate sizes increase. The faster the rate of cooling, the more retention there is of the frozen orientation.

Plasticizing screw speed and back pressure

Depending on the screw diameter, speed should be set in 50 to 100 rpm range, whilst maintaining a 250-300psi backpressure. Plasticizing times should fall well within limit set by the actual cooling time.

Mold geometry

The mold to be used is a Master Unit Die (MUD) for ASTM Type I test specimen (for tensile testing), which contains two separate test specimen cavities in the mold (Figure 6), for averaging two responses. Also included are ejection pins and plate (Figure 7).

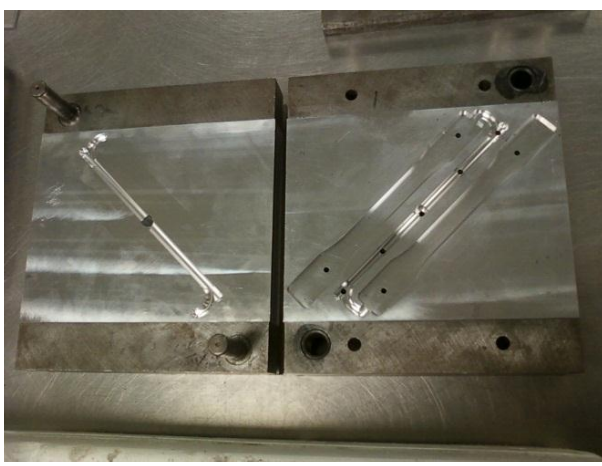


Figure 6

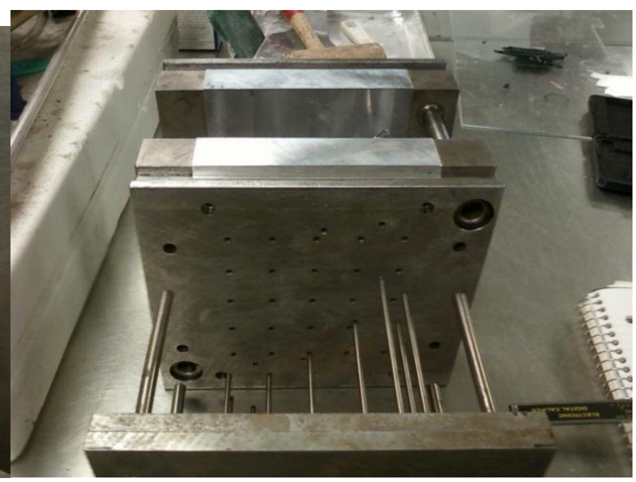


Figure 7

METHODOLOGY

The general process taken in this experiment can be summarized by the flow chart given below by Figure 8:

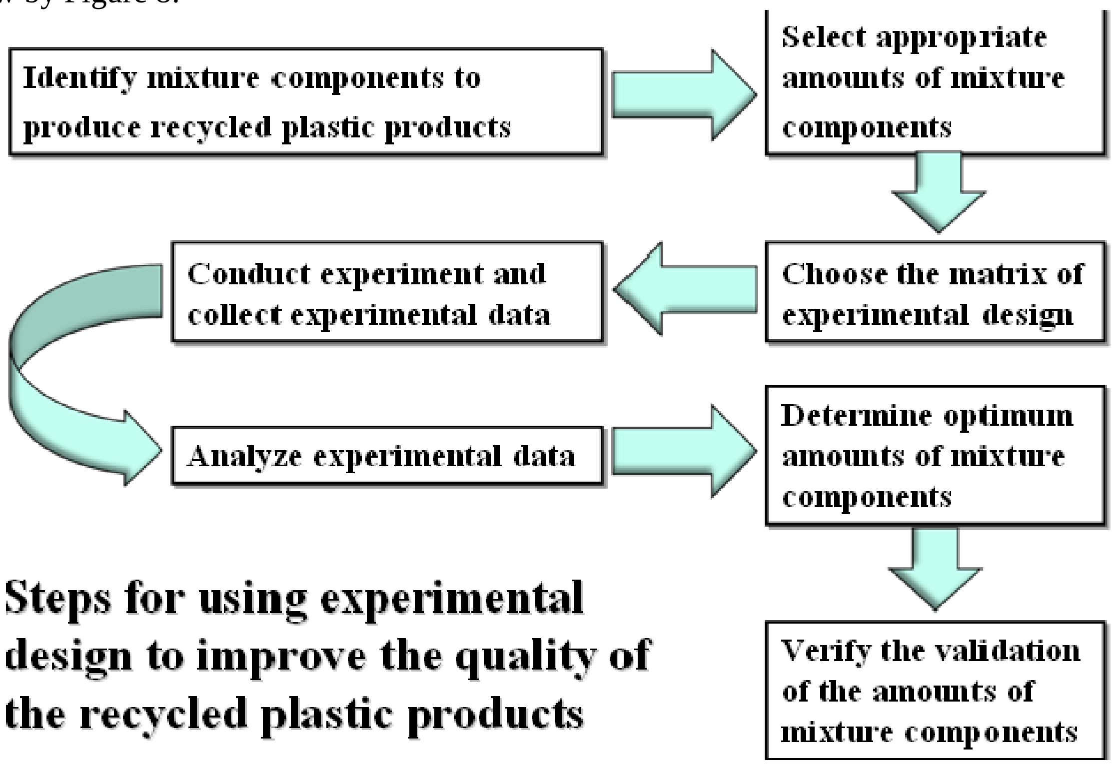


Figure 8

<u>Material Adequacy</u>

Virgin PET

This plastic was in pellet form, stored in a cabinet that was not temperature controlled, but was encased in its original packaging box in a near atmospherically separated condition with the help of its sealed plastic bag. PET’s hygroscopic nature may have detrimental effects on processing due to unknown historical conditions.

Recycled PET

This plastic was in flake form, and was delivered by train to a large commercial resin silo under ambient conditions at an off-campus facility. This RPET was washed and transferred to an AEC Whitlock OS series blender via vacuum lines then to a Con Air crystallizer for 45 minutes with an air temperature of 311°F. The RPET mixture was sent to a Con Air carousel drier for 4 hours operating at 284°F with a -104°F dew point to achieve a final moisture content rating of less than 50 ppm.

Experimental Runs

Before performing the full experiment, thermal process settings were checked out and concluded as indeed correct on the Morgan Press, a vertical injection molding machine located at the Manufacturing Engineering Department labs at Cal Poly. Testing of a few blends under varied injection pressures and heating temperatures proved that both resin stocks were compatible.

Experimental runs were performed at Cornucopia Plastics in Paso Robles, CA on a Nissei 60-ton 3-ounce shot size injection molding machine. All labor (except batching) was performed by Eric, an operator of the machine, and supervised by Frank Burke, the Operations Manager at Cornucopia Plastics. The machine set up can be seen below in Figures 9 and 10:

Figure 9

Figure 10

The tensile strip mold was fitted snugly into the universal tool attachment (MUD molds are quite common for injection molding machines). Initial temperature settings were set, and the machine operator, Eric, found the right process variables to keep constant with virgin PET while binary blends were being made. Each blend was a constant 3.5 pounds. The process parameters that could be given are:

Screw RPM: 38% of maximum (was increased to 42% on last two blends to get full shots)
Injection Pressure: 300psi (always constant)
Shot size: constant (increased on last two blends to get full shot, due to flake size, not weight)
Cooling time: 8 seconds (always constant)

At each blend, temperature settings were adjusted to either low or high levels according to the experimental plan, with randomization, which meant that intermediate breaks happened to let barrel and nozzle zones reach equilibrium states. In addition, many shots were made initially to heat up the mold since there were no hook-ups available. In all, 48 shots were retrieved, which contained two tensile strips each, were gathered although many more shots were taken as adjustments; so 96 data points could be presented.

<u>Gathering of Data</u>

Tensile testing

After separating all tensile strip specimens from their sprues and runners, they were gathered to perform tensile tests in the Materials Engineering student laboratory at Cal Poly (Figure 11). The machine being used in accordance with ASTM D638-10 “Standard Test Method

for Tensile Properties of Plastics" was the Instron 3369 universal testing machine shown in Figure 12. The ambient temperature was 72°F at 25 mm/min grip separation.

Figure 11

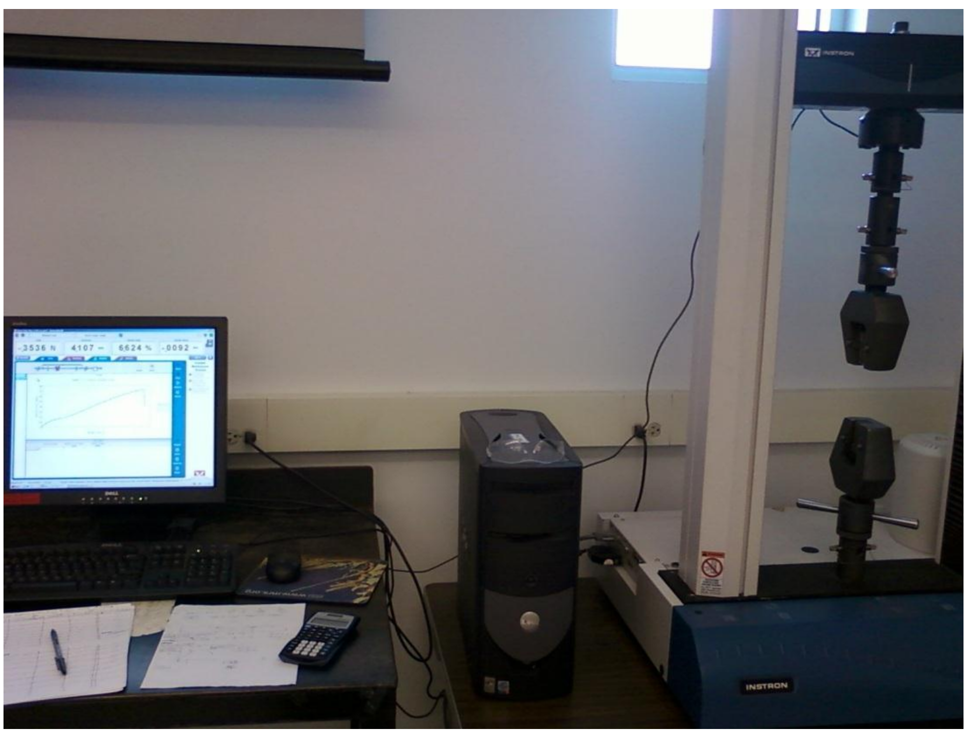


Figure 12

To ensure the integrity of the tests, straightness in grips was checked during every set-up, the load associated with gripping (~30-50N) was not balanced because this is a real force acting on the test strip, and nominal length, width, and thickness were measured for each grouped

mixture (not assuming same dimensionality) and tested with those appropriate values (see Figure 13 and Figure 14).

Specimen Lengths:

- (1,0) = 62.0 mm
- (4/5,1/5) = 62.6 mm
- (3/5,2/5) = 62.6 mm
- (2/5,3/5) = 62.6 mm
- (1/5,4/5) = 62.6 mm
- (0,1) = 62.6 mm

Specimen Widths:

- (1,0) = 12.64 mm
- (4/5,1/5) = 12.80 mm
- (3/5,2/5) = 12.70 mm
- (2/5,3/5) = 12.80 mm
- (1/5,4/5) = 12.75 mm
- (0,1) = 12.85 mm

Specimen Thicknesses:

- (1,0) = 3.19 mm
- (4/5,1/5) = 3.21 mm
- (3/5,2/5) = 3.19 mm
- (2/5,3/5) = 3.20 mm
- (1/5,4/5) = 3.19 mm
- (0,1) = 3.21 mm

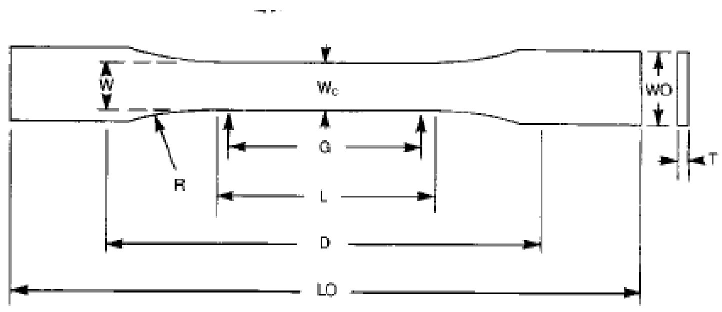


Figure 13

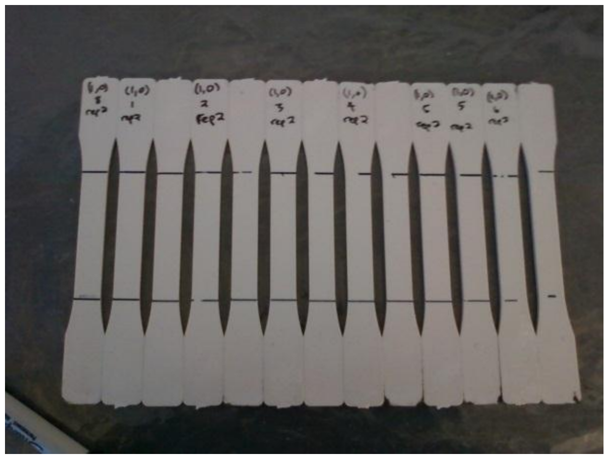


Figure 14

To determine values of Yield Stress and Young's Modulus, horizontal lines were drawn on stress-strain graphs at Yield Points and slopes was measured, respectively. An example stress-strain graph is shown below in Figure 15.

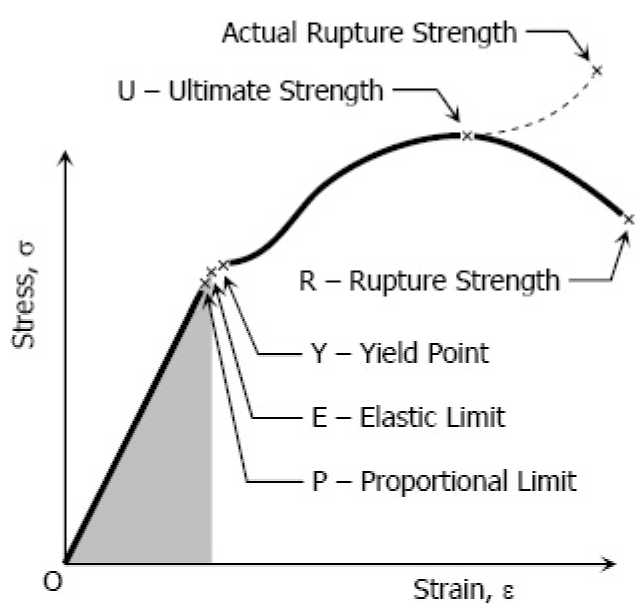


Figure 15

Figure 16 shows an example plot with data from the (4/5, 1/5) blend of treatments 3 and 4 (each has two replications, so read as specimen #'s 5 and 6 for treatment 3, and specimen #'s 7 and 8 for treatment 4). The rest of the collected graphs are located in the Appendix.

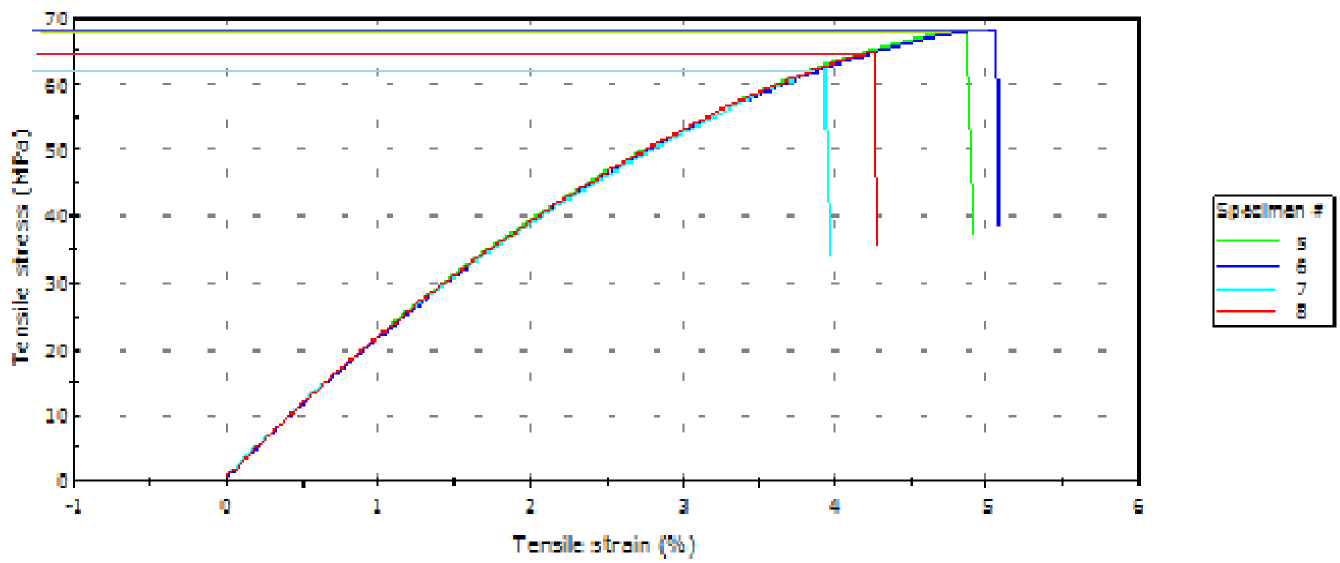


Figure 16

RESULTS

The collected data from 96 tensile strips shows 48 responses, each showing two mechanical quality characteristics, Yield Stress, and Young's Modulus. It should be noted that treatment 6 was ignored due to it being an extreme outlier (it was found that the graphical estimation methods to determine the two properties were not adequate for that point).

Treatment	Run Order	Z1	Z2	Z3	Blend (%PET)	Y (MPa)	E (GPa)
1	3	-1	-1	-1	100	74.75	1.3746
2	2	1	-1	-1	100	69.55	1.3721
3	6	-1	1	-1	100	74.9	1.905
4	7	1	1	-1	100	69.7	2.09
5	5	-1	-1	1	100	60	2.09
~~6~~	~~8~~	~~1~~	~~-1~~	~~1~~	~~100~~	~~35~~	~~2.4141~~
7	1	-1	1	1	100	73.25	1.2453
8	4	1	1	1	100	54.25	1.2461
1	3	-1	-1	-1	80	68.9	1.67
2	7	1	-1	-1	80	63.9	1.815
3	2	-1	1	-1	80	67.94	1.85
4	8	1	1	-1	80	63.455	1.82
5	5	-1	-1	1	80	61.75	1.895
6	1	1	-1	1	80	57.25	1.775
7	6	-1	1	1	80	65.8	1.815
8	4	1	1	1	80	59.45	1.905
1	1	-1	-1	-1	60	50	1.655
2	7	1	-1	-1	60	59.95	1.64
3	6	-1	1	-1	60	61.2	1.7
4	8	1	1	-1	60	58.25	1.69
5	3	-1	-1	1	60	60.25	1.555
6	4	1	-1	1	60	62.5	1.68
7	2	-1	1	1	60	53.25	1.73
8	5	1	1	1	60	64	1.625
1	3	-1	-1	-1	40	59.81	1.32
2	7	1	-1	-1	40	48.8	1.37
3	6	-1	1	-1	40	46.2	1.46
4	5	1	1	-1	40	53.47	1.455
5	2	-1	-1	1	40	58	1.33
6	8	1	-1	1	40	52.2	1.435
7	1	-1	1	1	40	53.3	1.485
8	4	1	1	1	40	60	1.425
1	6	-1	-1	-1	20	53.99	1.155
2	2	1	-1	-1	20	53.5	1.19
3	5	-1	1	-1	20	52.5	1.175
4	1	1	1	-1	20	45.15	1.13
5	7	-1	-1	1	20	45.25	1.175
6	8	1	-1	1	20	54.32	1.19
7	3	-1	1	1	20	54.325	1.13
8	4	1	1	1	20	51.875	1.15

1	8	-1	-1	-1	0	54.34	1.055
2	4	1	-1	-1	0	44	1.045
3	3	-1	1	-1	0	54.8	1.01
4	1	1	1	-1	0	53	1.01
5	2	-1	-1	1	0	52.575	1.01
6	6	1	-1	1	0	56.15	1.06
7	5	-1	1	1	0	40.4	1.085
8	7	1	1	1	0	53.4	1.015

After all the effort that goes into planning, running, and analyzing a designed experiment, it is very exciting to get the results of your work. There is a tendency to eagerly grab the results and rush out to production and say, "We have the answer! This will solve the problem!" BEFORE doing that, confirmations runs are needed to verify the outcome (but at the end, time and money costs cut this final step). Good software packages will provide a prediction interval to compare the results within some degree of confidence. Remember that in statistics absolutes are never dealt with – there is always uncertainty in recommendations.

ANOVA Legend: (A = x_1, B = x_2, C = z_1, D = z_2, E = z_3)

Response 1 (Yield Stress)

Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	2464.91	20	123.25	6.29	< 0.0001	significant
Linear Mixtur	*1712.60*	*1*	*1712.60*	*87.36*	*< 0.0001*	
AB	*51.69*	*1*	*51.69*	*2.64*	*0.1165*	
AC	*32.86*	*1*	*32.86*	*1.68*	*0.2068*	
AD	*30.74*	*1*	*30.74*	*1.57*	*0.2216*	
AE	*29.10*	*1*	*29.10*	*1.48*	*0.2340*	
BC	*0.13*	*1*	*0.13*	*6.670E-003*	*0.9355*	
BD	*14.02*	*1*	*14.02*	*0.72*	*0.4055*	
BE	*5.97*	*1*	*5.97*	*0.30*	*0.5858*	
ABC	*18.60*	*1*	*18.60*	*0.95*	*0.3390*	
ABD	*38.55*	*1*	*38.55*	*1.97*	*0.1727*	
ABE	*1.81*	*1*	*1.81*	*0.092*	*0.7637*	
ACD	*53.08*	*1*	*53.08*	*2.71*	*0.1119*	
ACE	*39.68*	*1*	*39.68*	*2.02*	*0.1667*	
ADE	*1.46*	*1*	*1.46*	*0.074*	*0.7873*	
BCD	*40.58*	*1*	*40.58*	*2.07*	*0.1622*	
BCE	*2.84*	*1*	*2.84*	*0.14*	*0.7067*	
BDE	*2.33*	*1*	*2.33*	*0.12*	*0.7329*	
ABCD	*16.25*	*1*	*16.25*	*0.83*	*0.3710*	
ABCE	*47.82*	*1*	*47.82*	*2.44*	*0.1304*	
ABDE	*3.42*	*1*	*3.42*	*0.17*	*0.6796*	
Residual	509.70	26	19.60			
Lack of Fit	*94.72*	*9*	*10.52*	*0.43*	*0.8999*	*not significant*
Pure Error	*414.98*	*17*	*24.41*			
Cor Total	2974.61	46				

The Model F-value of 6.29 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case, Linear Mixture Components are indeed significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model.

The "Lack of Fit F-value" of 0.43 implies the Lack of Fit is not significant relative to the pure error. There is a 89.99% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- it is desirable to have the model fit.

- R-Squared = 0.8287
- Adjusted R-Squared = 0.6968
- Predicted R-Squared = 0.5611
- Adequacy Precision = 10.006

The "Predicted R-Squared" of 0.5611 is in reasonable agreement with the "Adjusted R-Squared" of 0.6968. "Adequacy Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 10.006 indicates an adequate signal. This model can be used to navigate the design space. Significance is also shown by approximate normality in Figure 17.

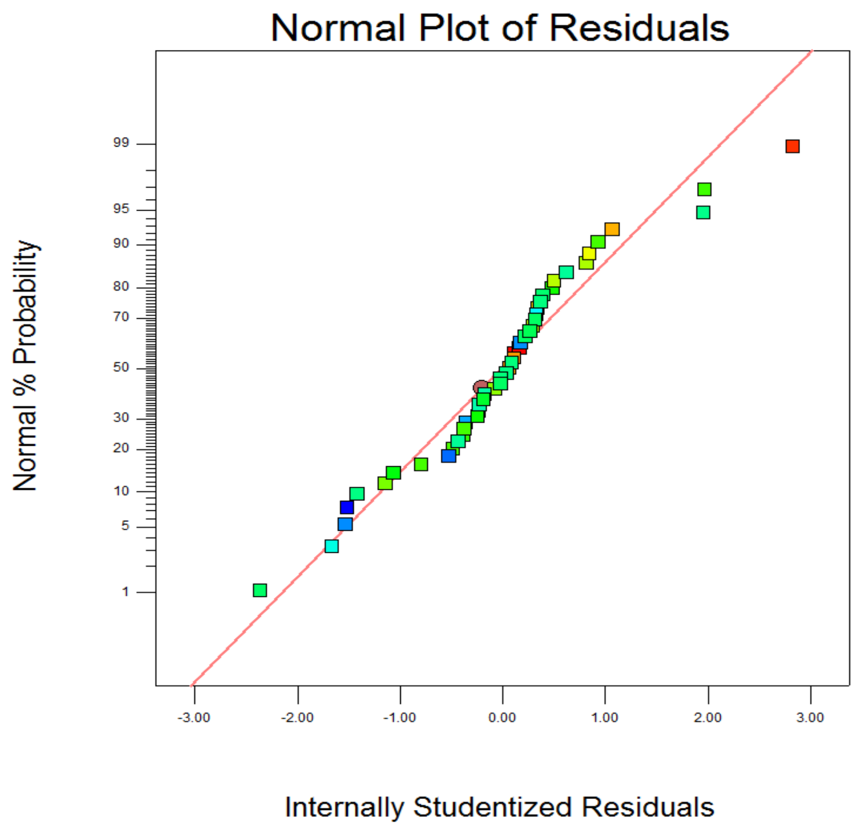


Figure 17

Response 2 (Young's Modulus)

Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	3.65	20	0.18	4.51	0.0002	significant
Linear Mixtur	*3.02*	*1*	*3.02*	*74.61*	*< 0.0001*	
AB	*0.059*	*1*	*0.059*	*1.46*	*0.2380*	
AC	*2.860E-003*	*1*	*2.860E-003*	*0.071*	*0.7923*	
AD	*6.064E-004*	*1*	*6.064E-004*	*0.015*	*0.9035*	
AE	*0.059*	*1*	*0.059*	*1.47*	*0.2362*	
BC	*0.013*	*1*	*0.013*	*0.32*	*0.5746*	
BD	*1.727E-003*	*1*	*1.727E-003*	*0.043*	*0.8378*	
BE	*0.025*	*1*	*0.025*	*0.61*	*0.4430*	
ABC	*4.484E-005*	*1*	*4.484E-005*	*1.109E-003*	*0.9737*	
ABD	*3.509E-005*	*1*	*3.509E-005*	*8.681E-004*	*0.9767*	
ABE	*0.046*	*1*	*0.046*	*1.14*	*0.2964*	
ACD	*0.032*	*1*	*0.032*	*0.80*	*0.3793*	
ACE	*3.690E-003*	*1*	*3.690E-003*	*0.091*	*0.7649*	
ADE	*2.971E-003*	*1*	*2.971E-003*	*0.074*	*0.7884*	
BCD	*1.395E-003*	*1*	*1.395E-003*	*0.035*	*0.8541*	
BCE	*0.012*	*1*	*0.012*	*0.31*	*0.5840*	
BDE	*5.242E-003*	*1*	*5.242E-003*	*0.13*	*0.7216*	
ABCD	*0.030*	*1*	*0.030*	*0.73*	*0.4006*	
ABCE	*8.118E-003*	*1*	*8.118E-003*	*0.20*	*0.6577*	
ABDE	*1.594E-003*	*1*	*1.594E-003*	*0.039*	*0.8441*	
Residual	1.05	26	0.040			
Lack of Fit	*0.14*	*9*	*0.015*	*0.29*	*0.9693*	*not significant*
Pure Error	*0.91*	*17*	*0.054*			
Cor Total	4.70	46				

The Model F-value of 4.51 implies the model is significant. There is only a 0.02% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case, Linear Mixture Components are indeed significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model.

The "Lack of Fit F-value" of 0.29 implies the Lack of Fit is not significant relative to the pure error. There is a 96.93% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- it is desirable to have the model fit.

- R-Squared = 0.7763
- Adjusted R-Squared = 0.6043
- Predicted R-Squared = 0.3266
- Adequacy Precision = 6.574

The "Predicted R-Squared" of 0.3266 is not as close to the "Adjusted R-Squared" of 0.6043 as one might normally expect. This may indicate a large block effect (blend affected) or a possible problem with the model and/or data. Things to consider are model reduction, response transformation, outliers, etc. "Adequacy Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 6.574 indicates an adequate signal. This model can be used to navigate the design space. Significance is also shown by approximate normality in Figure 18.

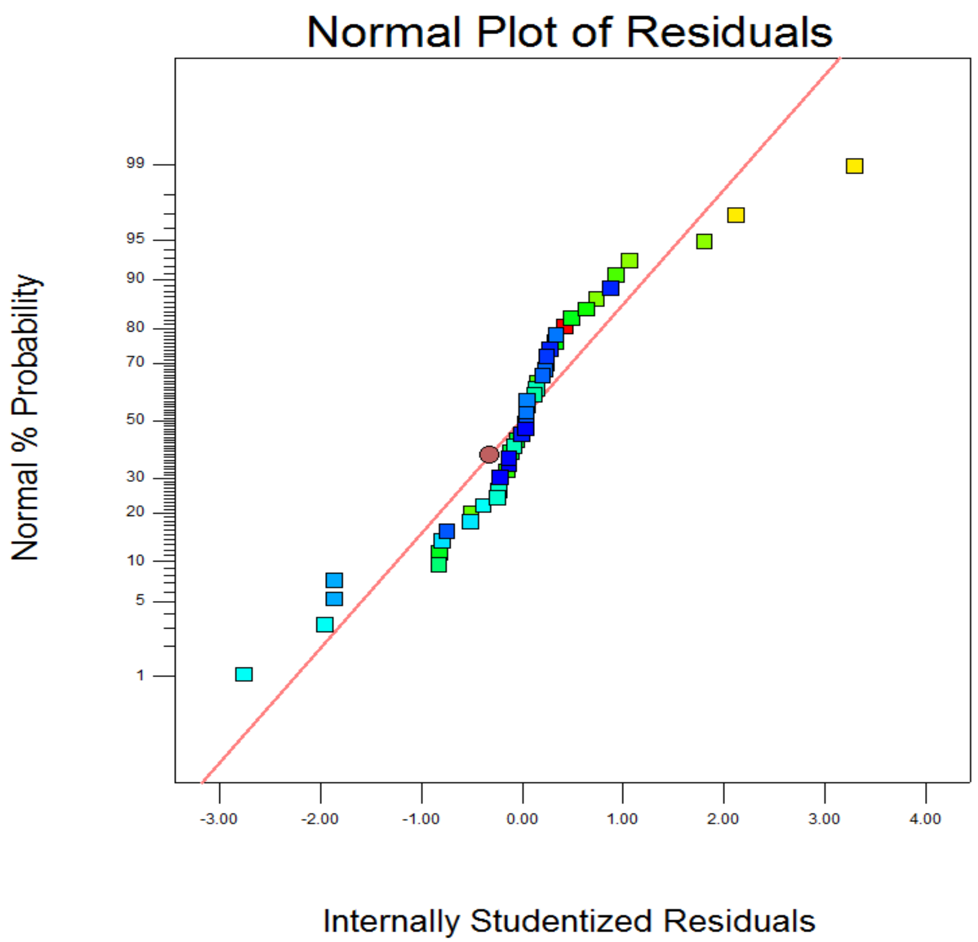


Figure 18

Solution To Predictive Model

$Y = 71.43x_1 + 51.16x_2 - 17.45x_1x_2$
$- 4.21x_1z_1 + 4.35x_1z_2 - 2.68x_1z_3 - 0.20x_2z_1 + 2.07x_2z_2 + 1.18x_2z_3$
$+ 12.03x_1x_2z_1 - 18.27x_1x_2z_2 + 2.87x_1x_2z_3 - 5.46x_1z_1z_2 + 5.01x_1z_1z_3 + 0.88x_1z_2z_3 + 2.41x_2z_1z_2$
$- 0.93x_2z_1z_3 + 0.83x_2z_2z_3 + 9.70x_1x_2z_1z_2 - 20.71x_1x_2z_1z_3 - 5.16x_1x_2z_2z_3$

$E = 1.86x_1 + 0.94x_2 + 0.59x_1x_2$
$- 0.039x_1z_1 + 0.019x_1z_2 + 0.12x_1z_3 + 0.062x_2z_1 + 0.023x_2z_2 + 0.075x_2z_3$
$+ 0.019x_1x_2z_1 - 0.017x_1x_2z_2 - 0.46x_1x_2z_3 - 0.13x_1z_1z_2 - 0.048x_1z_1z_3 + 0.040x_1z_2z_3 - 0.014x_2z_1z_2$
$- 0.062x_2z_1z_3 - 0.0391x_2z_2z_3 + 0.41x_1x_2z_1z_2 + 0.27x_1x_2z_1z_3 - 0.11x_1x_2z_2z_3$

In this model, the blends are percentages and process variables are coded as high or low values. Using this method, adequate predictions (within tested range of temperatures) can be made due to significance of the model shown in the previous section.

Response Surface Graphs

Response 1 (Yield Stress)

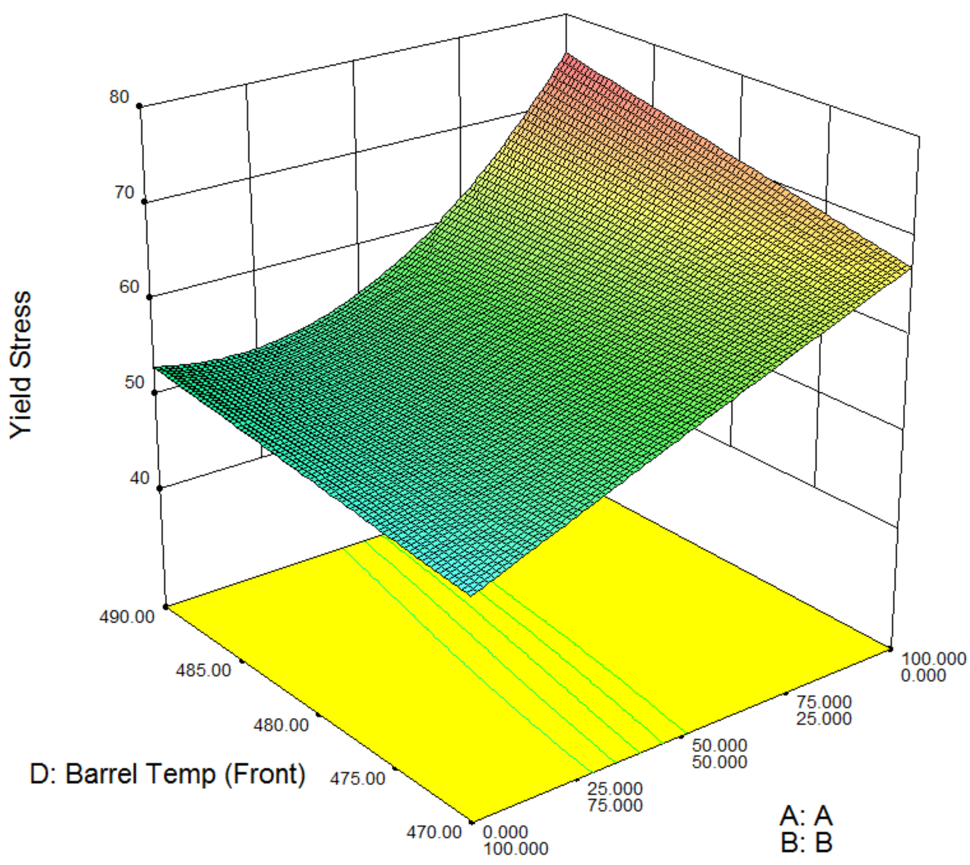


Figure 19

Based on the largest interaction in the prediction polynomial that involves process variables, $-18.27x_1x_2z_2$ (ABD), the interaction with blend state can be seen graphically in Figure 19. With a lower front barrel temperature (z_2, D), Yield Stress acts linearly relative to PET/RPET content, and with a higher front barrel temperature, Yield Stress acts exponentially relative to PET/RPET content.

Response 2 (Young's Modulus)

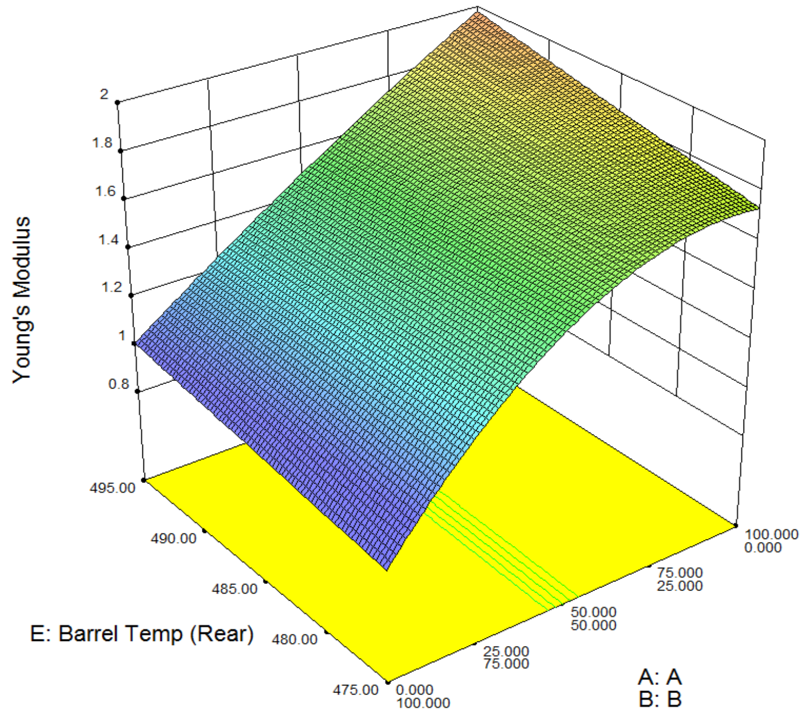


Figure 20

Based on the largest interaction in the prediction polynomial that involves process variables, $-0.46x_1x_2z_3$ (ABE), the interaction with blend state can be seen graphically in Figure 20. With lower rear barrel temperature (z_3, E), Young's Modulus acts in a steep concave manner relative to PET/RPET content, and with a higher rear barrel temperature, Young's Modulus acts in a linear fashion relative to PET/RPET content.

<u>Optimization</u>

Upon maximizing Yield Stress, maximizing Young's Modulus, and maximizing RPET content simultaneously, the best solution (there are many solutions to gain these maximums) is rated as 0.556, responding with: 42.844% PET and 57.156% RPET as the best mixture, processed at 520°F at the nozzle, 490°F in the front of the barrel, and 475°F at the rear of the barrel. The Yield Stress and Young's Modulus predicted responses are 60.2276 MPa and 1.57526 GPa, respectively. It must be noted that maximizing both mechanical properties *and* the RPET content creates a severe bias towards the recycled content, hence it must also be understood that optimizing without that parameter will always have a prediction saying 100% PET is favored.

CONCLUSION

Recycling plastic can reduce consumption of energy, non-renewable fossil fuels use, as well as global emissions of carbon dioxide. The effect of recycled PET and virgin PET on tensile strength and stiffness was studied. The optimal amounts of mixture components to produce recycled plastic products are determined. As the results of doing systematic experimentation, using mixture experiments, the quality of recycled plastic products can be improved and becomes more robust to variations at the optimal operating settings. The results have proven that the manufacturer can use these settings of recycled PET and virgin PET to produce quality products with low cost (quality depends on source as some recycled content qualities can be very high) and environmental impact reduction.

This case study on optimization of blends with process variables shows how application of advanced tools of DoE can simultaneously optimize a mixture formulation and processing conditions, taking advantage of complex interactions in the system. Response surface graphics, which can be produced in association with prediction formulas, make it easy to find the peak performance. If it is necessary to juggle many responses to keep products in specification, numerical optimization approaches are available to manipulate the predictive models and find the "sweet spot" for both mixture and process variables. DOE helps our customers set their processing conditions to achieve the required levels of strength with our materials. Careful analysis shows them which process conditions they need to focus on holding precisely and which ones they don't need to worry about. DOE also provides significant statistical information that helps demonstrate its validity.

The ultimate benefit comes from discovery of operating windows that satisfy all customer specifications most economically. The economic justification is spearheaded by a designer who wishes to optimize their material with reduced environmental impact; this study shows that there can be uses of recycled resin outside of its initial purpose, gaining a second life as a new mechanically strong material. If this is accomplished, you and your company will gain a competitive advantage and generate big profits.

THANKS

The author would like to thank the Faculty of Engineering at California Polytechnic State University for supporting the research connected with this paper, specifically Dan Waldorf, the Technical Advisor for this Manufacturing Engineer's senior project. In addition, the author would like to thank Frank Burke of Cornucopia Plastics for donating time, labor, and machinery to complete this work. An Industrial Technology Professor, Keith Vorst, needs to be thanked as well due to his donation of the tensile strip MUD mold.

RESOURCES

1. Hannequart, JP. Good Practice Guide on Waste Plastics Recycling: A guide by and for logal regional authorities. Association of cities and regions for recycling (ACRR), Belgium: 2004.
2. B. L. Fletcher and M. E. Mackay, "A model of plastics recycling: does recycling reduce the amount of waste", Resources, Conservation and Recycling, col. 17, pp. 141-151, 1996.
3. Staniulis, A. Method for lowering component costs by direct injection molding of recycle plastics. *Annual Recycling Conference*, 255-257, 260. 1998.
4. Environmental Protection Agency, U.S. (2011, January 25). *Common Wastes & Materials - Plastics*. Retrieved from http://www.epa.gov/osw/conserve/materials/plastics.htm
5. Rosato, D. V., & Rosato, D. V. Selecting Plastic and Process. *Reinforced Plastics Handbook* (3 ed., pp. 831-835). Oxford: Elsevier. 2004
6. Rosato, D. V., & Rosato, D. V. Selecting Plastic and Process. *Reinforced plastics handbook* (3 ed., pp. 826-827). Oxford: Elsevier. 2004.
7. Torres, N., Robin, J., & Boutevin, B. Study of thermal and mechanical properties of virgin and recycled Polyethylene terephthalate before and after injection molding. *European Polymer Journal*, *36*(10), 2075-2080. 2000.
8. F. A. Mantia. Handbook of Plastics Recycling, Shrewsbury, UK: Rapra Technology, 2002.
9. Kuswanti, C., Xu, G., Koelling, K., Stuart, J., Qiao, J., et al. An engineering approach to plastic recycling based on rheological characterization. *Journal of Industrial Ecology*, *6*(3-4), 125-135. 2003.
10. Khan, Z. , Kamaruddin, S. , & Siddiquee, A. Feasibility study of use of recycled high density polyethylene and multi response optimization of injection moulding parameters using combined grey relational and principal component analyses. *Materials & Design*, *31*(6), 2925. 2010.
11. Rosato, D. V., & Rosato, D. V. Plastics. *Reinforced plastics handbook* (3 ed., pp. 109). Oxford: Elsevier. 2004.
12. Rosato, D. V., Rosato, D. V., & Rosato, M. V. Plastic Property, polyester. *Plastic product material and process selection handbook* (pp. 40-80). Kidlington, Oxford, UK: Elsevier. 2004.
13. Rosato, D. V., Rosato, D. V., & Rosato, M. V. Injection Molding. *Plastic Product Material and Process Selection Handbook* (pp. 192-220). Kidlington, Oxford, UK: Elsevier. 2004.
14. Rosato, D. V., Rosato, D. V., & Rosato, M. V. Plastic Process, polyester. *Plastic product material and process selection handbook* (pp. 85-86). Kidlington, Oxford, UK: Elsevier. 2004.
15. Cornell, J. A. *Experiments with mixtures: designs, models, and the analysis of mixture data* (2nd ed.). New York: Wiley. 1990.
16. Bachman, K. (2010, May 15). *Injection molder slashes part costs using recycled plastics*. Retrieved from http://beta.machinedesign.com/article/ags-technology-slashes-injection-molded-part-costs-by-making-its-own-raw-material-from-recyc.
17. PlasticsNews. *Resin pricing*. (2011, June 2). Retrieved from http://www.plasticsnews.com/resin-pricing/all-resins.html

18. Scheffé, H. Simplex-centroid designs for experiments with Mixtures, Journal of Royal Statistical Society. Series B, 25, 235-263. 1963.
19. De Ketelaere B. & Goos P. & Brijs K. "Prespecified Factor-Level Combinations in the Optimal Design of Mixture-Process Variable Experiments Abstract: Designed experiments allow researchers to explore and understand the relation between set," Working Papers 2011001, University of Antwerp, Faculty of Applied Economics. 2010.

APPENDIX

PET Properties

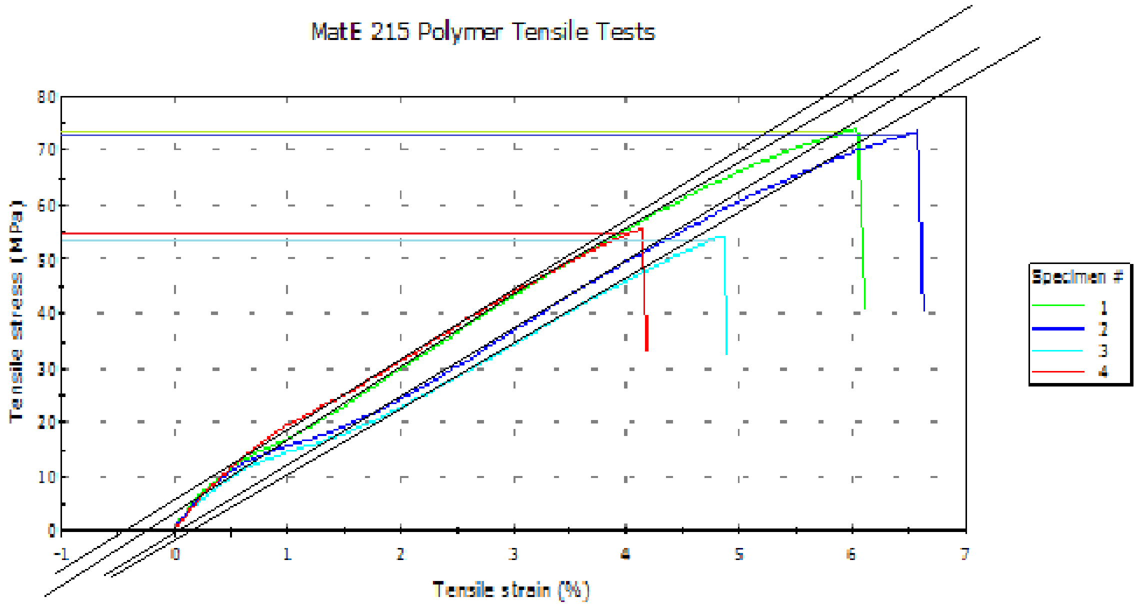
Resin data[b]	*Specific gravity* ($g\,cm^{-3}$)	*Density* ($lb\,ft^{-3}$)	*Specific volume* ($in^3\,lb^{-1}$)	*Specific volume* ($cm^3\,g^{-1}$)	*Extrusion temperature* (°F)	*Specific heat* ($BTU\,lb^{-1}\,°F^{-1}$)	*Water absorption in 24 h* (%)	*Maximum water content allowable*
ABS, extrusion	1.02	64.0	27.0	0.980	435	0.34	0.25	
ABS, injection	1.05	65.0	26.0	0.952		0.40	0.40	0.20
Acetal, injection	1.41	88.0	19.7	0.709		0.35	0.25	
Acrylic, extrusion	1.19	74.3	23.3	0.839	375	0.35	0.30	
Acrylic, injection	1.16	72.0	24.1	0.868		0.35	0.20	0.08
CAB	1.20	74.6	23.1	0.833	380	0.35	1.50	0.15
Cellulose acetate, extrusion	1.28	80.2	21.6	0.781	380	0.40	2.50	
Cellulose acetate, injection	1.26	79.0	21.9	0.794		0.36	2.40	0.20
Cellulose proprionate, extrusion	1.22	76.1	22.7	0.821	380	0.40	1.70	
Cellulose proprionate, injection	1.22	75.5	22.9	0.828		0.40	2.00	0.25
CTFE	2.11	134.0	13.1	0.473		0.22	0.01	
FEP	2.11	134.0	12.9	0.465	600	0.28	<0.01	
Ionomer, extrusion	0.95	59.6	29.0	1.050	500	0.54	0.07	
Ionomer, injection	0.95	59.1	29.2	1.060		0.54	0.20	
Nylon-6	1.13	70.5	24.5	0.886	520	0.40	1.60	0.15
Nylon-6,6	1.14	71.2	24.3	0.878	510	0.40	1.50	0.15
Nylon-6,10	1.08	67.4	25.6	0.927		0.40	0.40	0.15
Nylon-6,12	1.07	66.8	25.9	0.935	475	0.40	0.40	0.20
Nylon-11	1.04	64.9	26.6	0.962	460	0.47	0.30	0.10
Nylon-12	1.02	63.7	27.1	0.980	450		0.25	0.10
Phenylene oxide based	1.08	67.5	25.6	0.926	480	0.32	0.07	
Polyallomer	0.90	56.2	30.7	1.110	405	0.50	0.01	
Polyarylene ether	1.06	66.2	30.7	0.940	460		0.10	
Polycarbonate	1.20	74.9	23.1	0.832	550	0.30	0.20	0.02
Polyester PBT	1.34	83.6	20.7	0.746			0.08	0.04
Polyester PET	1.31	8.18	21.1	0.746	480	0.40	0.10	0.005
HD polyethylene, extrusion	0.96	59.9	28.8	1.040	410		<0.01	
HD polyethylene, injection	0.95	59.3	29.1	1.050		480	<0.01	
HD polyethylene, blow molding	0.95	56.9	28.8	1.040	410		<0.01	
LD polyethylene, film	0.92	57.44	30.1	1.090	350		<0.01	
LD polyethylene, injection	0.92	57.4	30.1	1.090		400	<0.01	
LD polyethylene, wire	0.92	57.4	30.1	1.090	400		<0.01	
LD polyethylene, ext. coating	0.92	57.1	30.0	1.090	600		<0.01	
LLD polyethylene, extrusion	0.92	57.4	30.1	1.087	500			
LLD polyethylene, injection	0.93	58.0	29.8	1.075		425		
Polypropylene, extrusion	0.91	56.8	30.4	1.100	450		0.03	
Polypropylene, injection	0.90	56.2	30.7	1.110		490	<0.01	
Polystyrene, impact sheet	1.04	64.9	26.6	0.963	450		0.10	
Polystyrene, gp crystal	1.05	65.5	26.2	0.943	410	425	0.03	
Polystyrene, injection impact	1.04	64.9	26.6	0.968		440	0.10	
Polysulfone	1.25	77.4	22.3	0.807	650	680	0.30	0.05
Polyurethane	1.20	74.9	23.1	0.834	400	400	0.10	0.03
PVC, rigid profiles	1.39	86.6	19.9	0.720	365		0.02	
PVC, pipe	1.44	87.5	19.7	0.714	380		0.10	
PVC, rigid injection	1.29	83.6	21.0	0.756		380	0.10	0.07
PVC, flexible wire	1.37	85.5	20.2	0.731	365			
PVC, flexible extruded shapes	1.23	76.8	22.5	0.814	350			
PVC, flexible injection	1.29	80.5	21.4	0.776		300		
PTFE	2.16	134.8	12.9	0.464			<0.01	
SAN	1.08	67.4	25.6	0.927	420	470	0.03	0.02
TFE	1.70	106.1	16.3	0.589		610	0.01	
Urethane elastomers	0.83	51.6	33.5	1.210	390	400	0.07	0.03

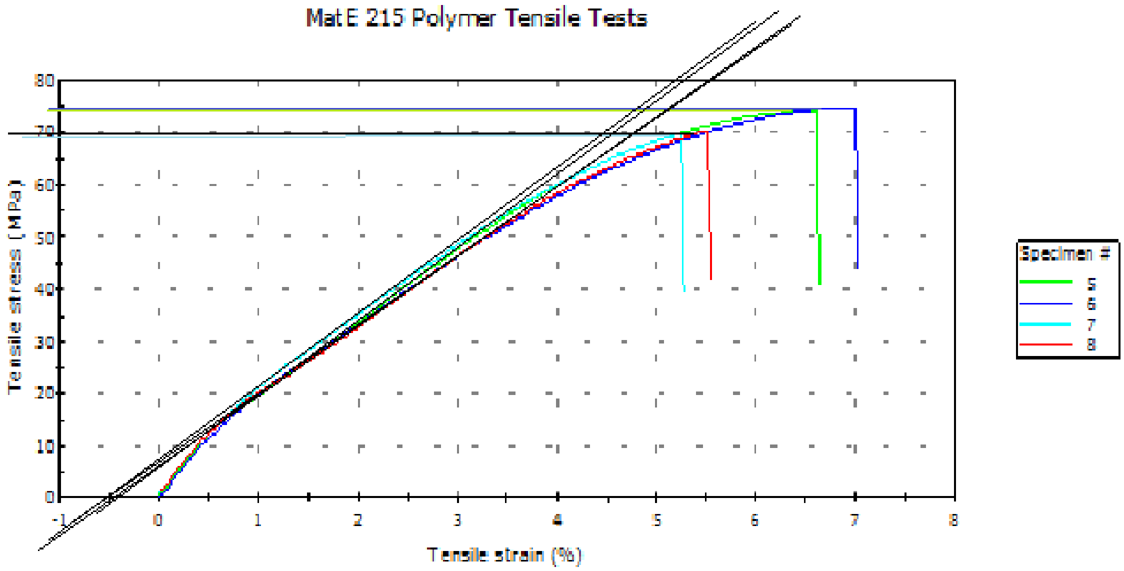
[a] Specific information on all machine settings and plastic properties is initially acquired by using the resin supplier's data sheet on the particular compound or resin to be used.

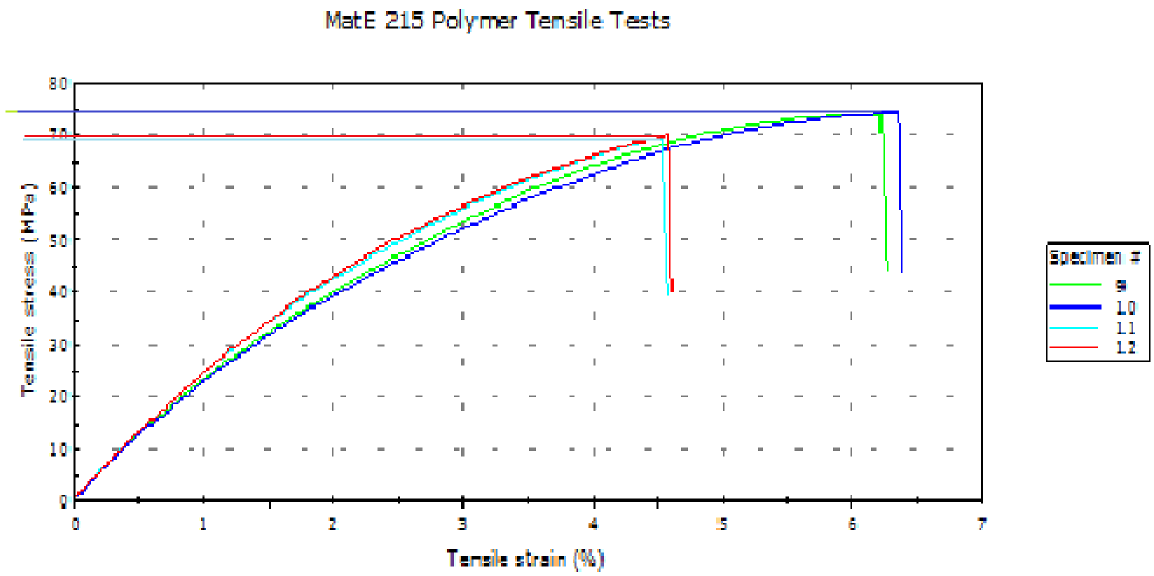
[b] These are strictly typical average values for a resin class; consult your resin supplier for values and more accurate information.

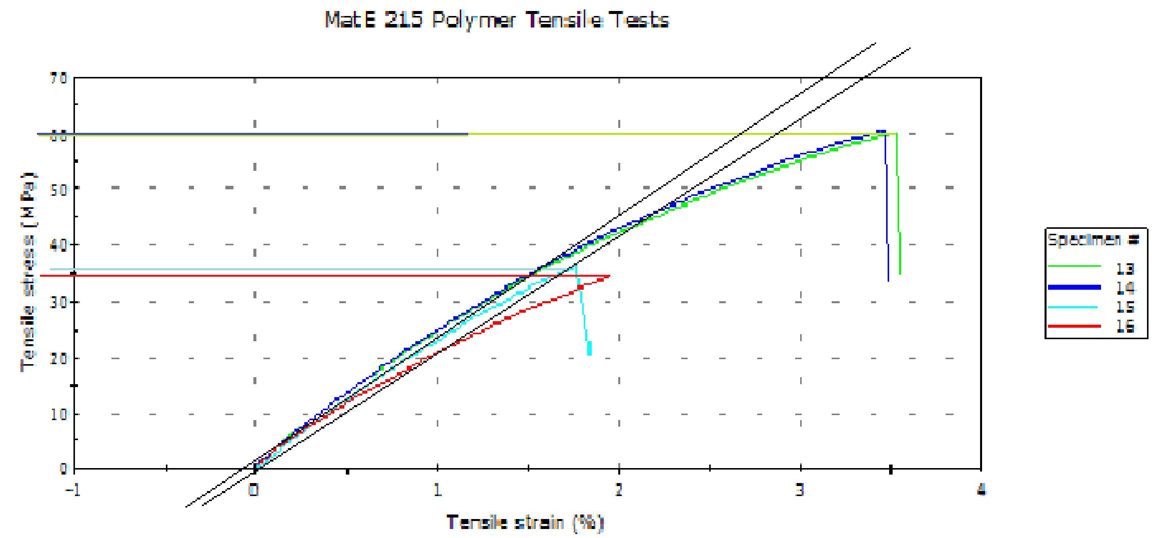
Tensile Testing (testing done in pairs of replicates)

(1,0) [First testing batch starts with treatment #7, as "specimen #1"]

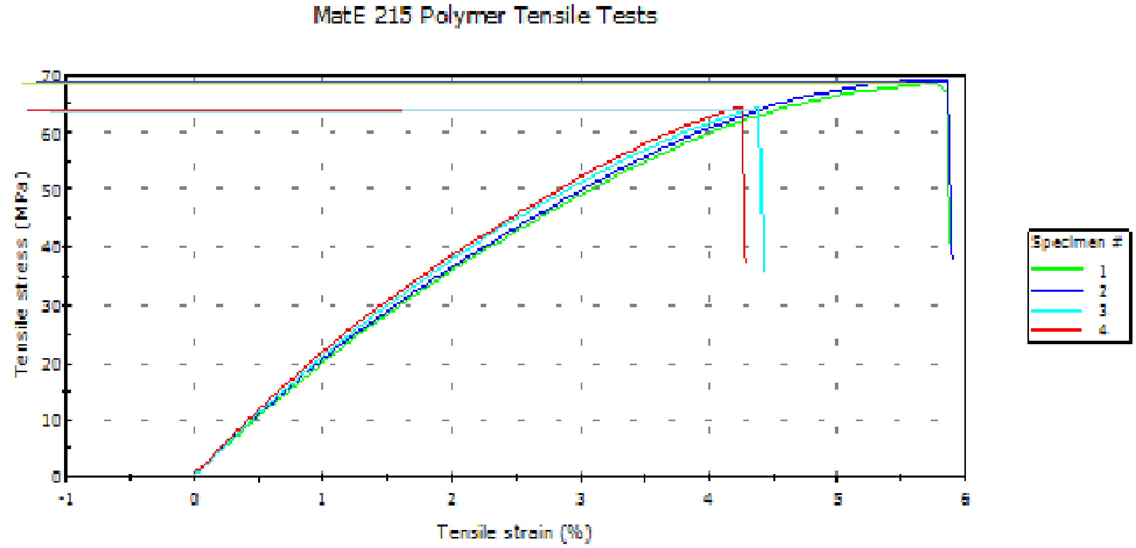


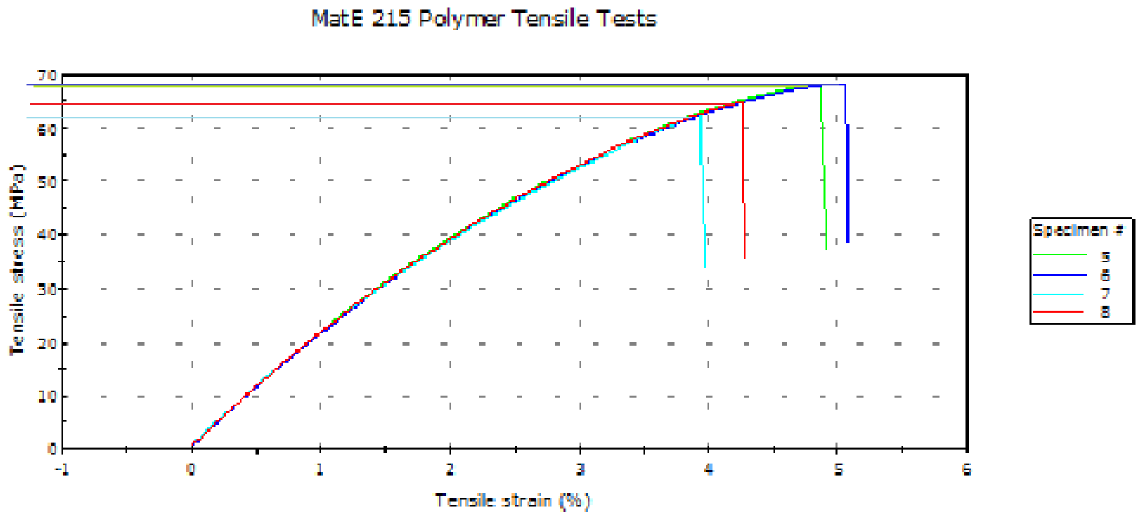


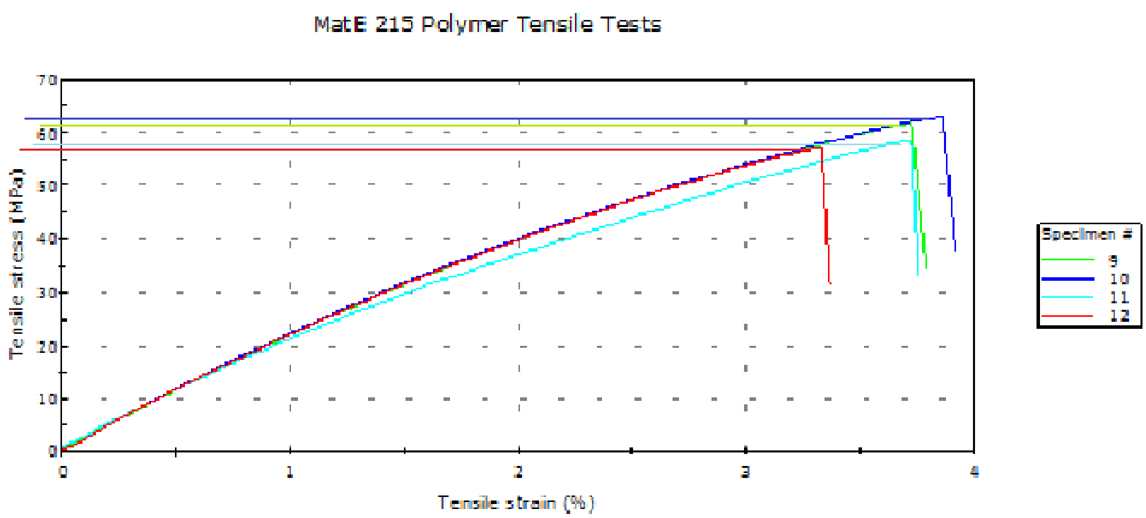

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
9
10
11
12

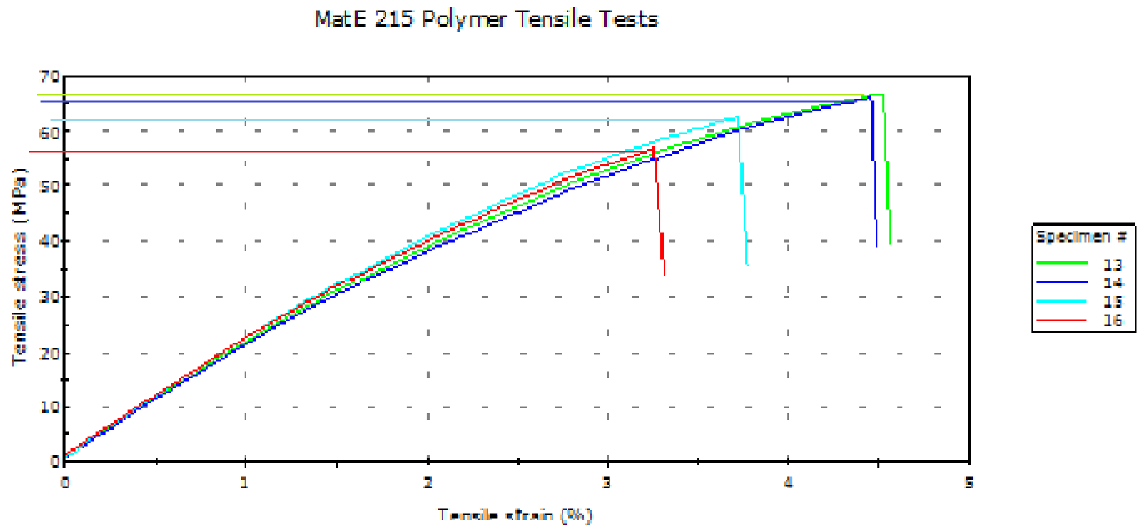

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
13
14
15
16

(4/5,1/5)

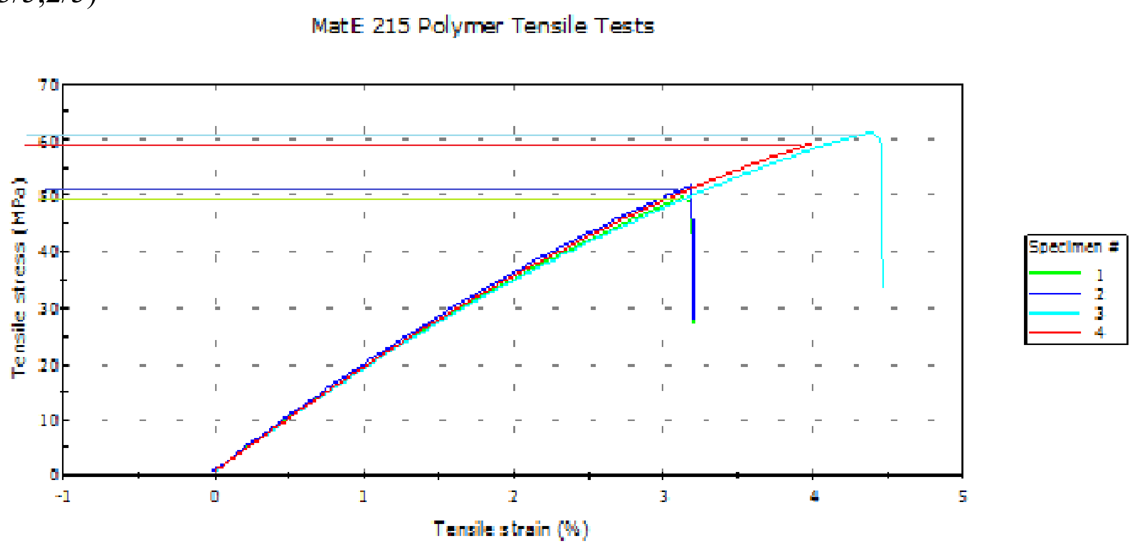

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
1
2
3
4

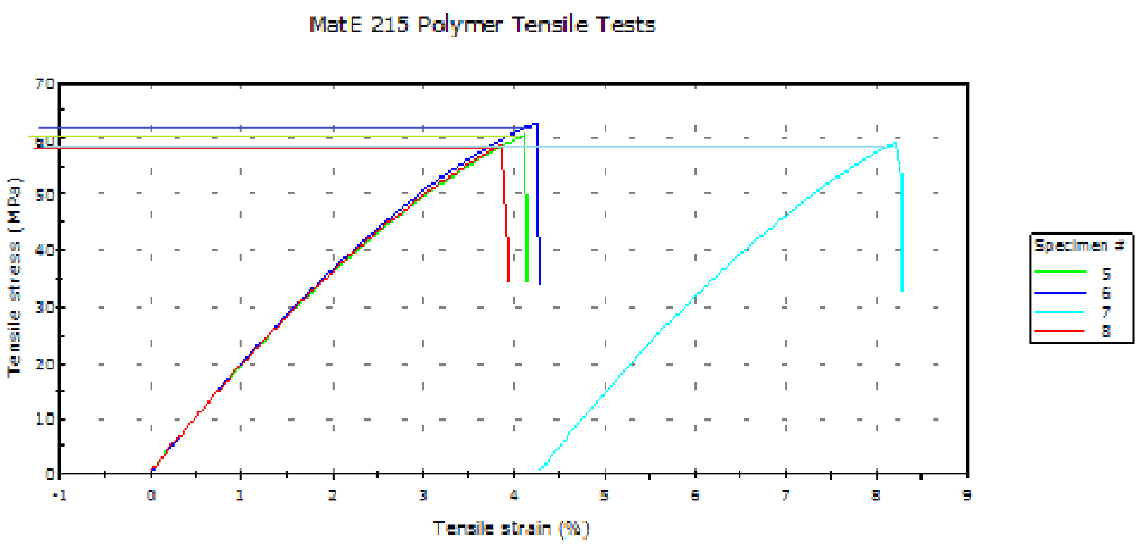

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
5
6
7
8

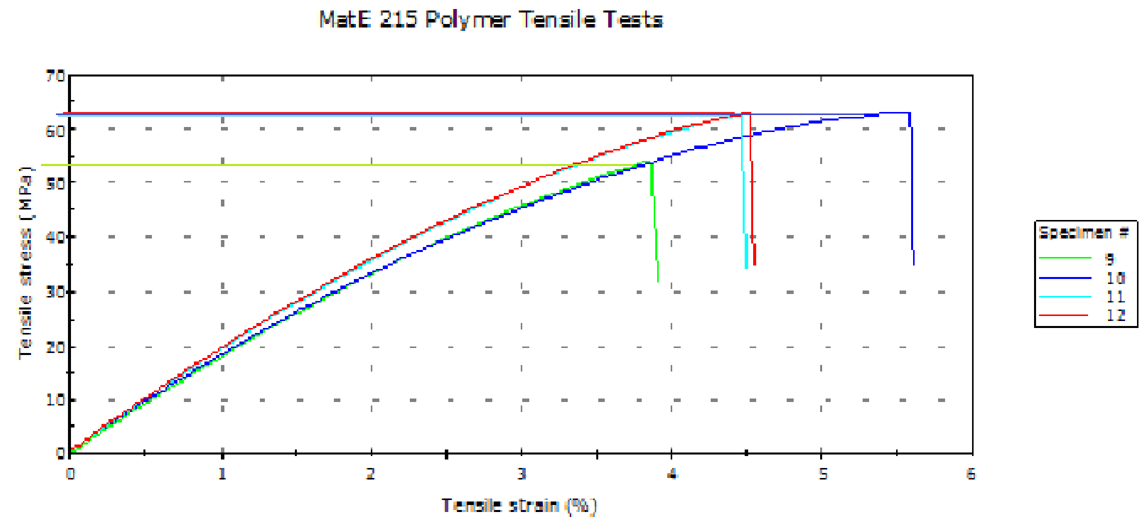

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
9
10
11
12

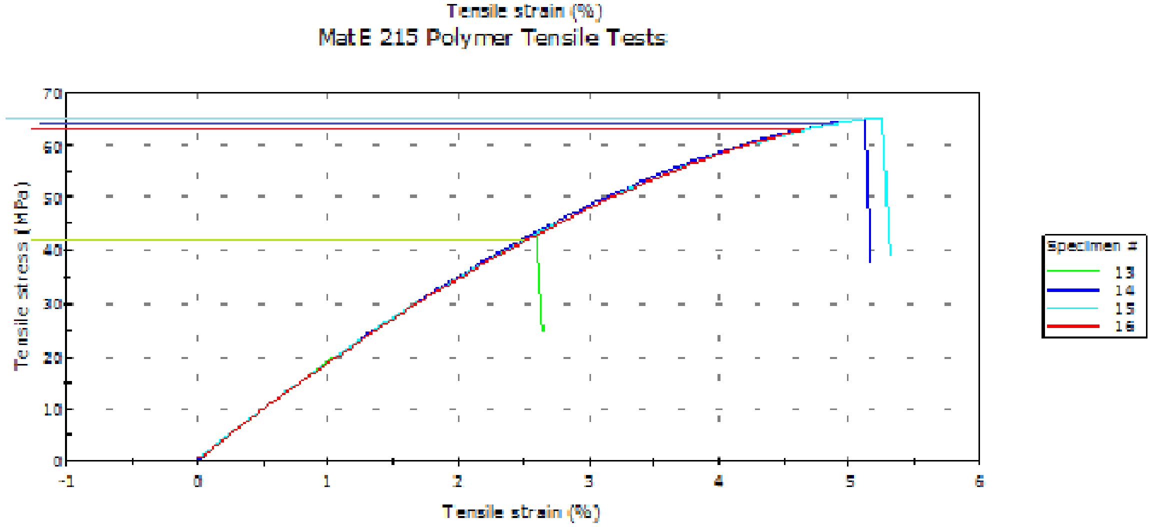

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
0
10
20
30
40
50
60
70
0
1
2
3
4
5
Specimen #
13
14
15
16

(3/5,2/5)

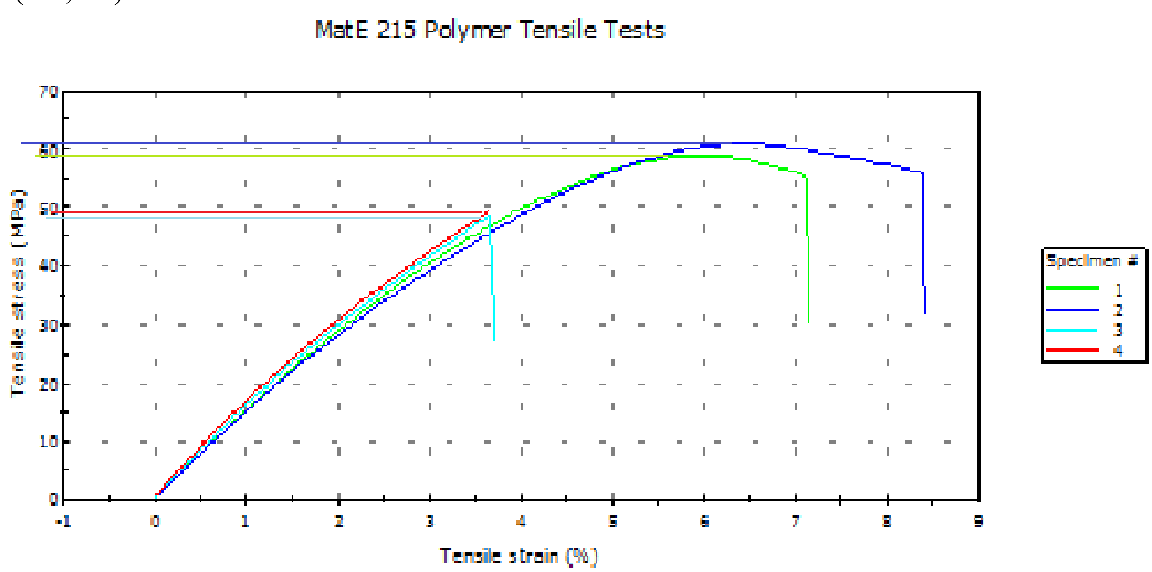

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
0
10
20
30
40
50
60
70
-1
0
1
2
3
4
5
Specimen #
1
2
3
4

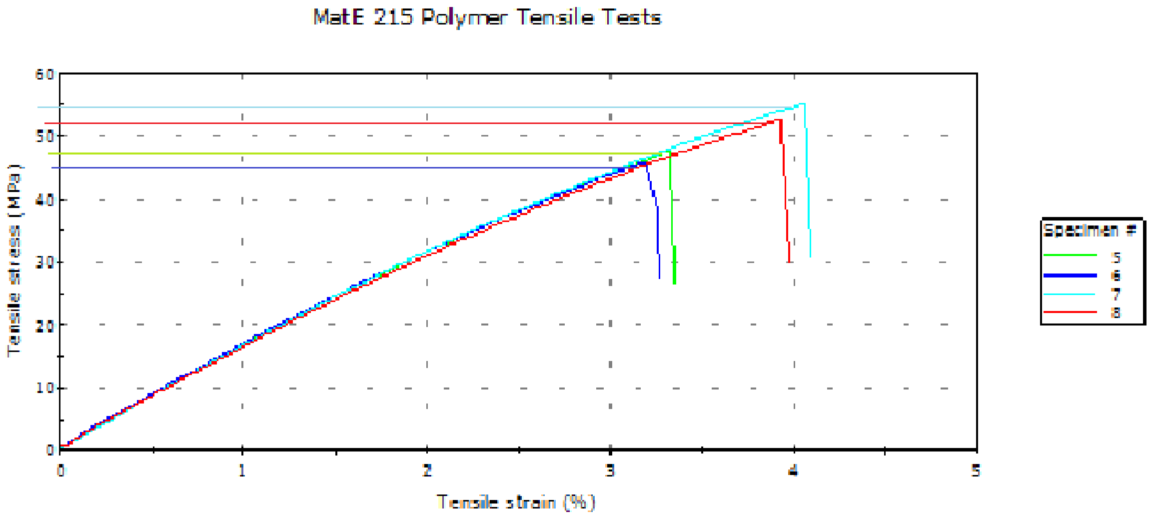

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
0
10
20
30
40
50
60
70
-1
0
1
2
3
4
5
6
7
8
9
Specimen #
5
6
7
8

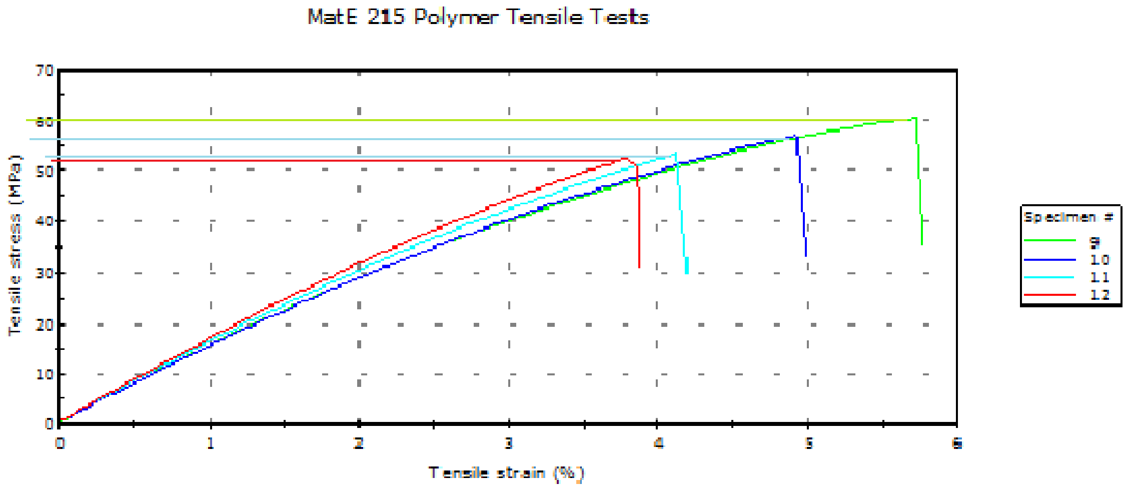

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
9
10
11
12

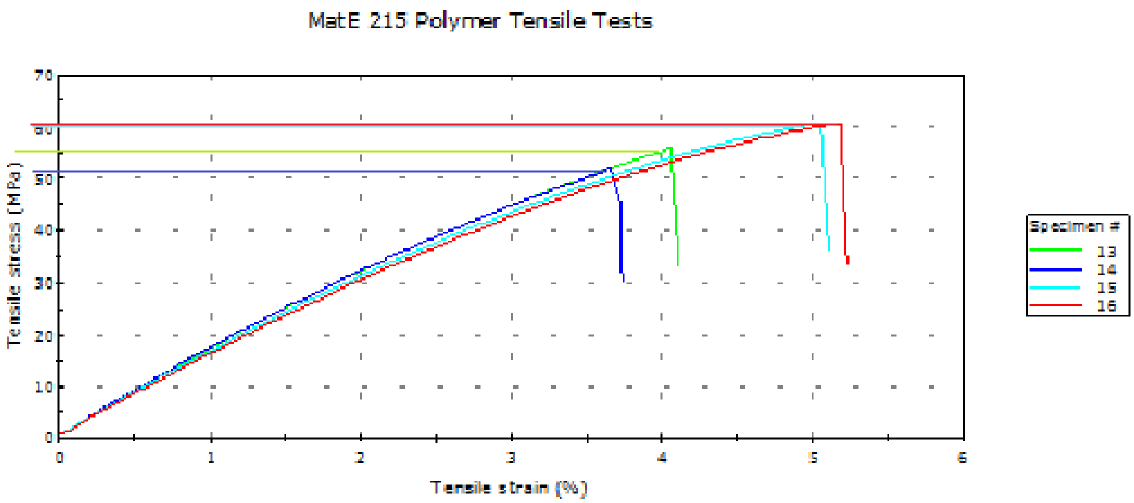

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
13
14
15
16

(2/5,3/5)


MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
1
2
3
4

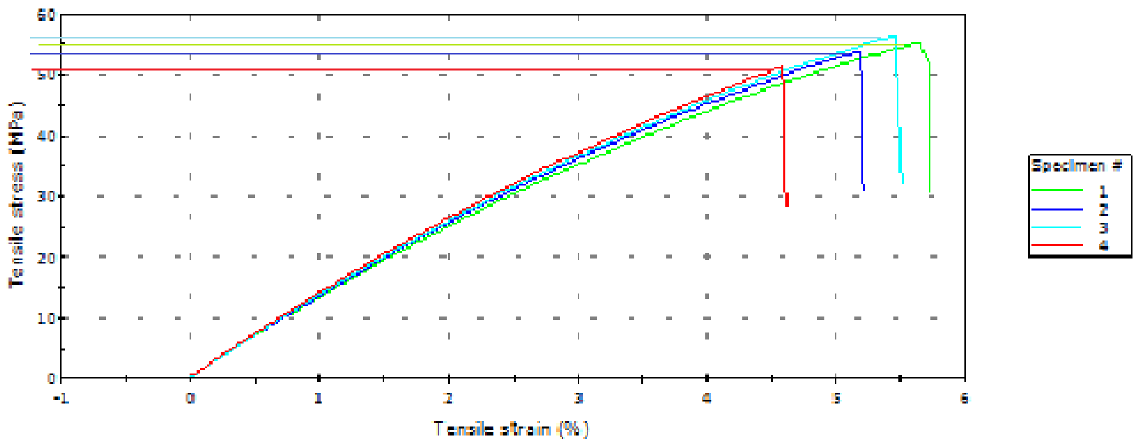

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
5
6
7
8


MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
9
10
11
12

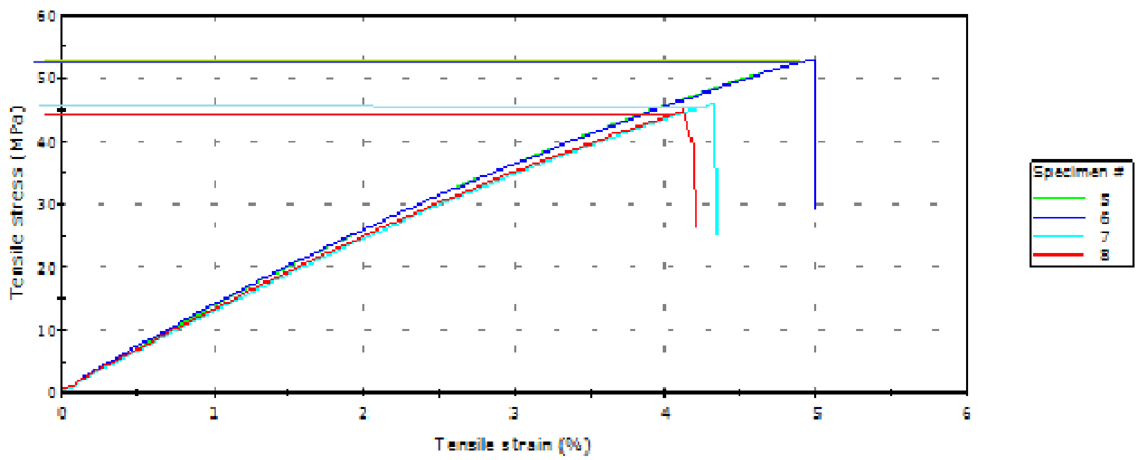

MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
13
14
15
16

(1/5,4/5)

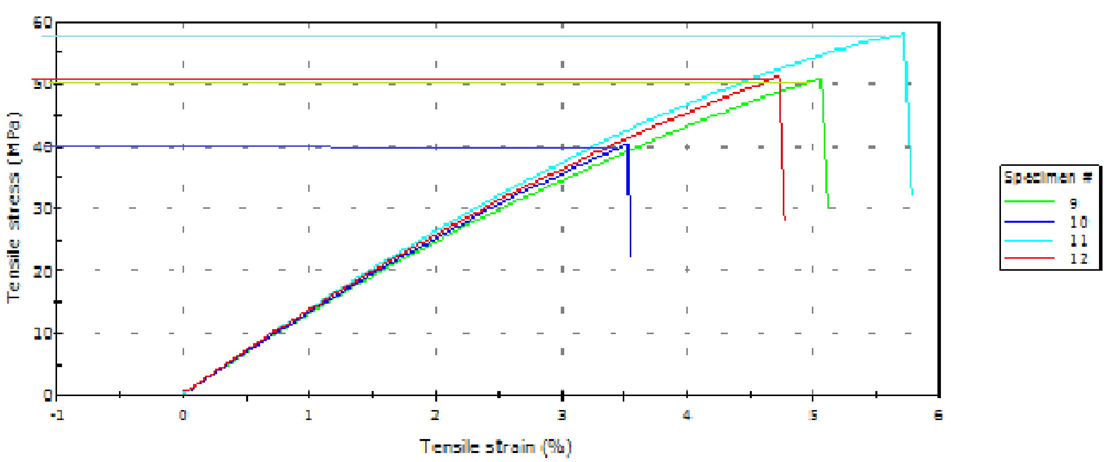
MatE 215 Polymer Tensile Tests

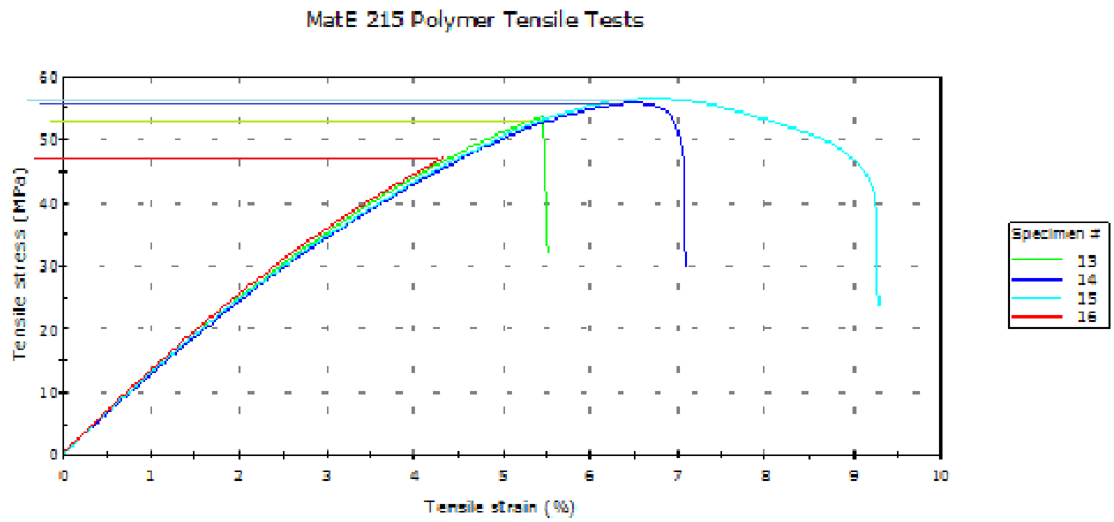


MatE 215 Polymer Tensile Tests

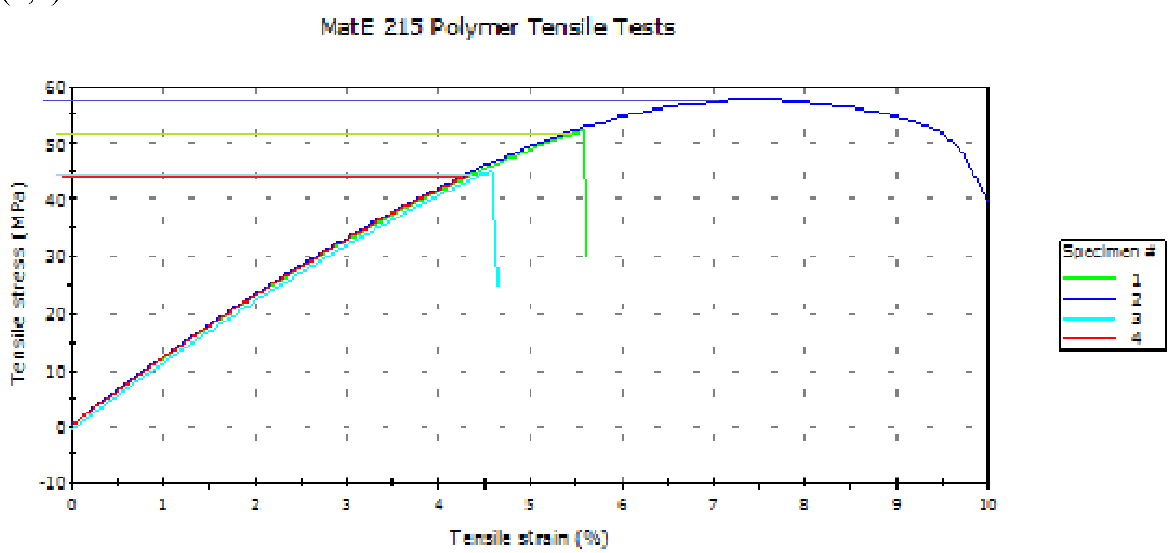


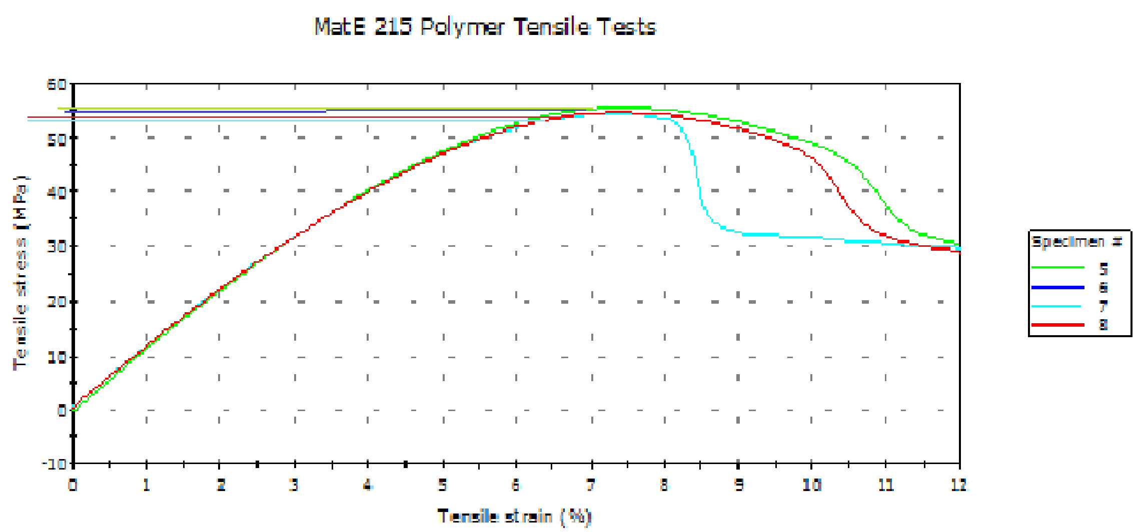
MatE 215 Polymer Tensile Tests

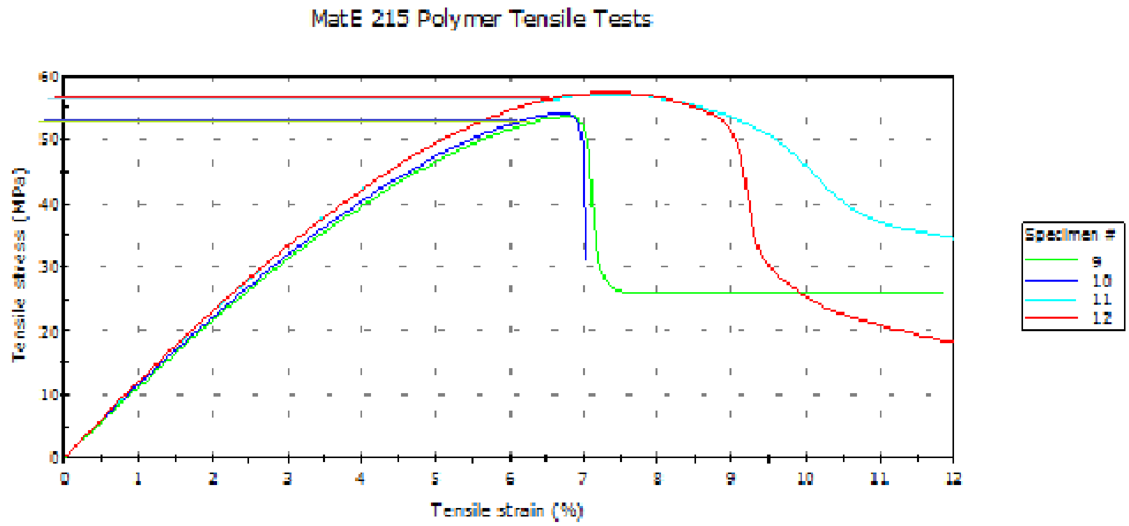



MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
13
14
15
16

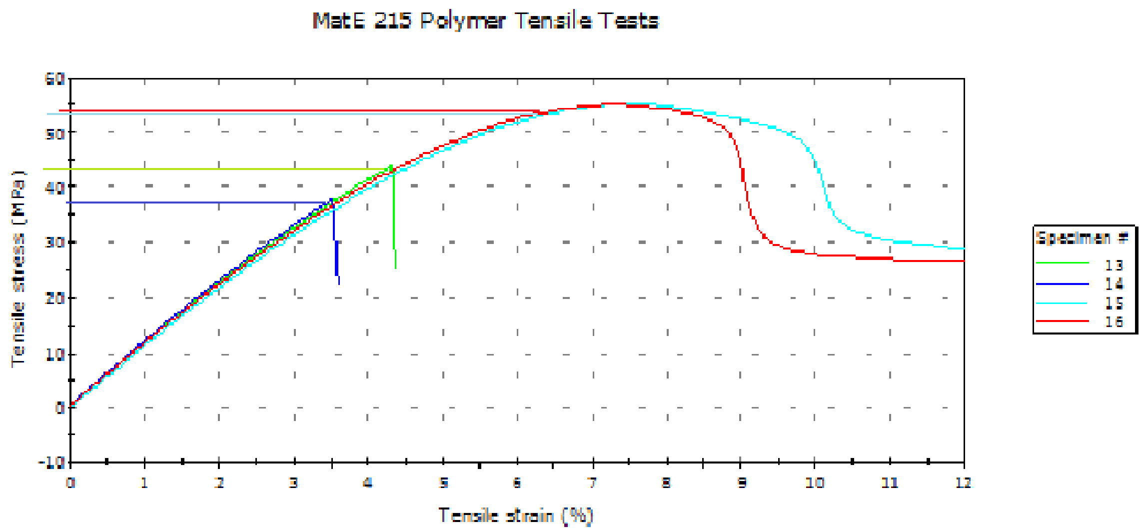
(0,1)


MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
1
2
3
4


MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
5
6
7
8



MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
9
10
11
12



MatE 215 Polymer Tensile Tests
Tensile stress (MPa)
Tensile strain (%)
Specimen #
13
14
15
16