EFFECTS OF GREEN REINFORCEMENT STRATEGIES ON MECHANICAL PROPERTIES OF HIGH VOLUME POLYMERS

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Effects of Green Reinforcement Strategies on Mechanical Properties of High Volume Polymers

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ABSTRACT

Green reinforcement in polymer systems have shown great promise in reducing cost, negative environmental effects, and dependency on nonrenewable resources. Both natural fillers and composite regrind can increase mechanical performance, while reducing new resin inputs for polymer based components. PVC and Polyurethane foam are widely used in North America in high volumes. Ground corn cob greatly increased the modulus of a flexible PVC system while minimally reducing maximum strength. The corn cob also showed signs of being a suitable filler in a polyurethane foam composite panel at low concentrations with minimal changes in mechanical performance. Polyurethane composite regrind illustrated great potential being used in new polyurethane composite panels with acceptable material properties. Replacing just a few percent of polyurethane and PVC with green reinforcement could reduce new production of these polymers by millions of pounds per year in North America alone.

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CHAPTER 1. INTRODUCTION

Polymer usage has been increasing for many years and is evident in everyday life. It is difficult to pass through a day without using a device that contains a polymer. Whether it is a thermoset, thermoplastic, or a composite utilizing either type of polymer, the applications are endless. Polymers are often chosen for their great balance of mechanical properties, ease of processing, and price. In design of new products, a dramatic amount of emphasis is being put forth to reduce component cost and negative environmental effects. Many components sent into service years ago have been placed in landfills across the world and are still there today given the slow rate of degradation. This is a large driving force for developing ways to recycle polymer products after their useable life is exhuasted. In addition, polymer prices have been increasing with fluctuation in crude oil, making it important for manufactures to research alternative ways to decrease the amount of polymer being used in the products.

Fillers have been a common choice to begin making polymer components more economical. In general, fillers are implemented to offset the volume of polymer used in order to reduce the cost and alter mechanical properties of the neat resin. Common fillers such as calcium carbonate, glass spheres, and titanium dioxide have been used for many years to offset resin cost, increase modulus, and alter appearance. These mineral fillers and their benefits are accepted and understood very well by the polymer industry. However, natural fillers, when used properly in polymer systems, can provide similar benefits to those of mineral fillers. The need for a natural or biobased filler has already been illustrated by the plastics market. To prove natural fillers can perform the same task as mineral fillers, they must be processed with a variety of polymer systems and tested under all different conditions to fully understand the opportunities.

Natural fillers provide many advantages over mineral fillers by being less abrasive on processing equipment, less expensive, and a renewable resource. Ground corn cob is a great option for a natural filler given it is a by-product of a process already fully established and profitable. By utilizing ground corn cob, both industries are able to capitalize on the benefits of the natural filler. Ground corn cob comes from corn seed manufacturing, after the kernels have been removed from the cob, for animal bedding, polishing, and chemical absorbent. Ground corn cob is also very low in density which can potentially allow for more resin to be offset with the same mass of a mineral filler.

Utilizing natural fillers could change many industries outside of just the polymer-manufacturing world. Utilizing natural fillers can help reduce the oil and gas dependency, infuse more dollars into the agricultural industry, and change the public's perception on the polymer industry. Reducing the oil and gas consumption can be achieved by replacing a percentage of the natural resource derived polymers with a renewable by product. This is a subject that is not going away is only becoming more important to find ways to alleviate the dependency on nonrenewable resources. Infusing dollars into the agricultural industry by utilizing current by-products is a great way to add value to a vital industry. Also, by making polymer based products cheaper and more eco-friendly will help the plastics industry public perception, which could lead to more uses unable requiring a renewable percentage.

Natural fillers seem ideal when describing the advantages acquired but many challenges arise in processing and environmental effects. During the processing of many polymer components high heat is required or generated, which can lead to degradation of the natural filler within the matrix. The degradation of the natural filler could release gases into the component reducing the quality of the specimen. Also given the hydrophilic nature of natural fillers, the filled polymer system is more likely to be affected in high moisture situations. However, considered one of the challenges of natural fillers, it may also be considered one of its greatest attributes. The ability to biodegrade is becoming a very important issue for the end of life disposal of a component. By introducing natural filler, the polymer system becomes partially biodegradable.

Natural fillers can help with offsetting polymer, but another green strategy is to recycle polymer components and reintroduce them into a new product. Reclaiming material is not a new idea and has been successful for many years in the metals industry. Some success has been found in the thermoplastic industry in reusing scrap and used polymer and composite components. However, difficulties are still being encountered in the reclaiming process of composites. It is more difficult given there are more than one material constituents to separate or blend. In addition, many composites have been made from thermosets, which are unable to be liquefied for reprocessing. One way to reclaim composite material is to grind it up (i.e. regrind) and introduce it into a new polymer system for processing. This regrind becomes a filler reducing the amount of polymer used in future manufacturing as well as solving the disposal issues. The composite industry is growing at leaps and bounds, finding a feasible way to up-cycle scraps and used composite parts would change the landscape of the composite industry. Reducing the final cost of producing composites would allow the aerospace industry to have more value in its scrap parts and production waste that could trickle down through the automotive, mass transit, household, and even consumable items. Reducing the raw material costs in each industry, while breaking down barriers where cheaper and less efficient materials are being used to save costs. The school bus industry is a great example of mass production using plywood floors for the low cost and acceptable performance. If using a recycled composite floor, the bus could have increased rider safety, improved fuel economy, longer service life, and reduced maintenance costs.

Also in a composite system introducing a regrind, if done properly, can have a positive influence on the mechanical performance. Composites of polymer and fibers are blended together and replace neat polymer. If a sufficient aspect ratio is obtained in the regrind fibers, load transfer can occur between the fiber and matrix.

With the increase in resin cost in both thermoset and thermoplastic, a green filler strategy could be utilized in both. In this study, the affects of the ground corn cob will be investigated with an injection molded flexible polyvinyl chloride (PVC), and a foamed polyurethane (PU) composite. Each polymer system is used in very high volumes and have applications suitable for fillers. Also, the ability to perform a regrind operation with the foamed PU composite will be used to illustrate a second way to offset resin cost and reduce the environmental affects. PVC is one of the highest volume engineered polymers and can be altered to the specific mechanical characteristics desired. Polyurethane is used in many automotive applications and is a great fit for the natural fillers because of a high attraction to water, as the foaming reaction can be increased by the inherent moisture in the filler.

In concept, offsetting resin with a filler or regrind has several advantages for the manufacture and user of the components. However, the processing of components may become more difficult and mechanical properties may be altered. To understand if the concepts of regrind and natural fillers are feasible in a manufacturing facility, processing of components must be proven and the mechanical properties must be evaluated. To process the natural filler and thermoplastic, the constituents should be compounded into pellet form using extrusion prior to final component processing. Introducing natural filler or regrind into a thermoset, can occur just prior to final processing by mixing liquid resin and dry filler with high speed mixers. Once acceptable specimens are processed, mechanical testing such as tensile, flexural, compression, etc. are conducted and compared to specimens processed without additives.

CHAPTER 2. BACKGROUND/LITERATURE REVIEW

Polyvinyl chloride and PU are both widely used with significant volumes of material produced each year. PVC is the third most used thermoplastic behind polyethylene (PE) and polypropylene (PP) while thermoset PU is widely used as rigid and flexible foams as wells a common adhesive [1-2]. With the vast applications of these two polymers, utilizing a filler has already been performed for many years, with fillers such as calcium carbonate and wood floor [3-5]. Fillers, whether natural, mineral, or regrind present processing and performance characteristics needed to be overcome by proper filler size, surface treatment, and filler percentage. Understanding the filler being introduced is the first step to determining the compatibility to the resin system.

2.1. The Structure of Corn Fibers

Ground corn cob would make a suitable polymer filler because of its low bulk density, availability, and cost [6]. Figure 1 illustrates the different constituents within a corn cob that is ground down into the fine paticles. Ground corn cob from BestCob LLC, [7] is sieved into categories and the particles shown in Figure 1 passed through a 80 mesh screen (178 μm) and labeled -80. The product is a very consistent mix of particles from the corn cob and could be readily incorporated into a polymer system.

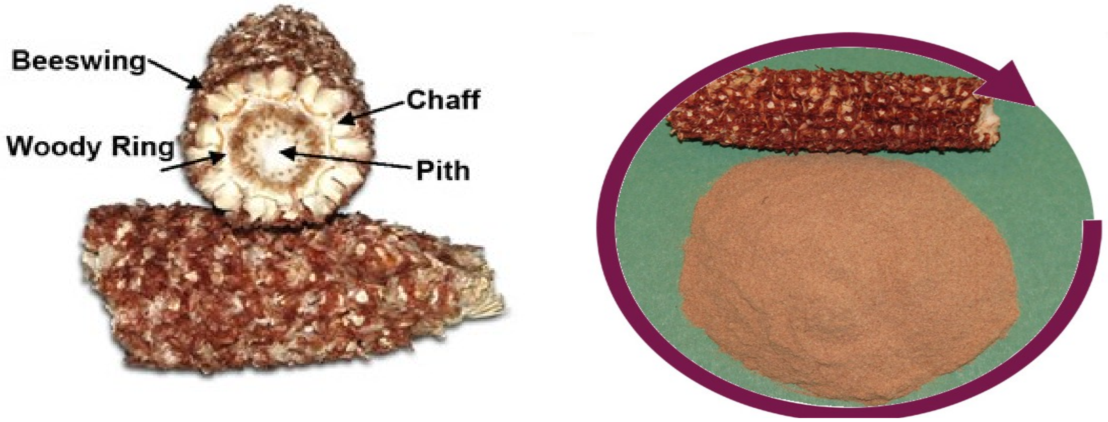

Figure 1. Corn cob physical structure and ground corn cob [7].

Currently ground corn cob is a by-product of seed production and is used in animal bedding for its ability to absorb moisture [7]. The cob consists of the pith, a woody ring, chaff, and beeswings. The ground cob is a good absorbent, because of the hydrophilic nature of the corn cob particles. The ground corn cob consists of cellulose, hemicellulose, starch, lignin, and protein, of which the cellulose is a desireable natural polymer chain given the strength that can be derived given a suitable transfer of load[8].

2.2. Surface Treatments on Natural Fibers

Given the chemical structure of ground corn cob and other cellulose based fillers, a surface treatment to improve adhesion between the matrix polymer and the filler is typically used. There have been several proven techniques to accomplish this bridging for fillers. Each different polymer type has surface treatments that have a great balance between performance, cost, and processability. The surface treatments studied here are directed to the PVC matrix since PU systems are naturally a good fit for natural fillers. Polyurethane foams utilize hydroxyl groups as part of the chemical reaction during curing. This affinity for hydroxyls will allow the ground corn cob to be a part of the reaction and increase the attraction between filler and resin[9].

2.2.1. Bleaching Treatment

Bleaching of natural fibers is a common practice in cotton industry to enhance the whiteness of the fiber. The bleaching processes breaks down components of the natural fiber while leaving the integrity of the cellulose structure. The lignin is broken down, while the pectin can be hydrolyzed [10]. By bleaching, the remaining natural fiber will contain a higher mass percentage of cellulose, leading to a more desirable filler structure.

2.2.2. Polymethylene Polyphenyl Isocyanate Treatment

Polymethylene polyphenyl isocyanate (PMPPIC) treatment has been a proven treatment used in the wood flour/PVC production of composite decking[11]. The ability to brigde the cellulose natural fiber and the polymer chains of PVC is critical for proper interaction. PVC and the cellulose phase of the natural filler are continuously linked by the PMPPIC interface [12]. With a sufficient interfacial interaction between the PVC and corn cob, a more efficient load transfer can be achieved.

2.3. Recycling Thermoset Composites

Recycling solutions in the field of thermoset polymer matrix composites (PMCs) are especially in high demand because of the inability to remelt the thermoset matrix as is possible in thermoplastic PMCs. The primary methods available in thermoset PMC recycling are mechanical recycling, thermal processing, chemical processing, and energy recovery. Mechanical recycling methods involve grinding up the composite waste and using the ground material as a filler or reinforcement in new materials. Thermal and chemical processing methods utilize chemical and heat interactions to recover fibers, polymer building blocks, and energy for future use. Energy recovery methods deal almost exclusively with the combustion of the composite in return for the energy given off during the combustion process [13,14]. The present work of this thesis focuses solely on mechanical recycling methods.

Research in the area of thermoset PMC mechanical recycling has found promising applications of the ground composite as both filler material and reinforcements in neat polymers. Ground circuit board composites, composed of fiberglass reinforcement in a thermoset matrix, are used as reinforcement in a thermoplastic matrix[15]. The study found that the ground composite drastically improved the tensile and flexural properties of the thermoplastic. Reporting increases in tensile strength, tensile modulus, flexural strength and flexural modulus of a PP composite of 28.4%, 62.9%, 87.8%, and 133.0%, respectively. Promising results have been found using ground fiberglass reinforced thermoset composites in fiber reinforced concrete

[16]. Ground fiberglass reinforced thermoset composites have also been found to provide property improvements as reinforcement in neat PU foams [17] and as a filler with no adverse effects in epoxy thermoforming molds [18].

2.4. Processing with Fillers and Regrind

Processing thermoplastic and thermoset systems with natural fillers and regrind particles often require new techniques[14,19]. Natural fillers in a thermoset system may absorb resin, affecting the mix ratio, and may need to be incorporated after the polymerization has begun. Natural fillers in a thermoplastic processing setting can degrade at the high melt temperatures of certain polymers. Regrind particles in a thermoset resin cause wet-out issues by impeding flow of resin through reinforcement. The particles act as flow damns when the reinforcement begins to filter out the particles and create a barrier. Regrind particles in thermoset or thermoplastic systems often accelerate equipment wear due to abrasive nature of regrind particles containing mineral fibers [20]. The abrasion problem of regrind particles can be reduced by large equipment tolerances or where the particles are introduced. Particles often affect viscosity, resin flow, curing rate, postprocessing shrinkage, surface finish, and part quality to name a few factors to watch during processing [19]. Not all these factors will occur in a negative manner, but have potential to alter processing for the better or worse. The ideal regrind particle size found for polyurethane foam was less than .2mm and smaller particles can be beneficial but are dependent upon the application [14].

2.4.1. Compounding

Compounding fillers with polymers has been occurring for decades and was critical in the success of many polymers. Extrusion, is a common means of compounding fillers with thermoplastic[21]. Extrusion allows two or more constituents to be metered into the extruder and homogenously mixed together. The product of extrusion, is a continuous strand of blended polymer that can be pelletized, granulated, or used as a final part. Co-rotating twin screw extrusion is an effective, reliable, and economical style of blending fillers and polymers[22]. This process is common in the wood filler composite decking industry to effectively blend a high concentration of natural filler into a polymer system [23].

2.4.2. Reaction Injection Molding

Reaction injection molding (RIM) is the process of injecting mixed thermoset resin and letting the reaction of the resin occur in a closed mold [24]. In the case of PU foam, polyol and isocyanate are mixed and forced into a closed mold[25]. The two liquid components expand during the foam reaction, filling the mold cavity and encapsulating reinforcement fibers or particles[26]. Fillers can be added before mixing or they can be encapsulated during the foaming reaction [17].

CHAPTER 3. OBJECTIVE FOR THE RESEARCH

The objective of this research is to investigate the mechanical properties and processability of filling thermoplastic polymer and thermoset composite systems. To be feasible, processing techniques need to be established and resulting products be characterized. Fillers can alter material properties and processability. In general, the greater the filling percentage, the greater the performance loss is. This is not true for all fillers and characteristics, as some material characteristics might be better or closer to the desired properties. The thermoplastic PVC polymer system was chose to illustrate the affects of a bio-based filler. A thermoset PU/fiberglass composite was targeted to illustrate the effects of introducing regrind and separately bio-based filler.

Introducing bio-based fillers into a neat polymer system was accomplished at NDSU by utilizing a twin-screw extruder and pelletizer. The pellets can then be utilized for industrial extrusion, injection, and compression molding. With the filled polymer, mechanical testing and important processing parameters were gathered, information necessary for processing and component design. Processing parameters gathered from observations of extruding and injection molding as well as melt flow index.

Introducing filler into a composite brings a new set of challenges compared to the filling of a neat polymer. The fiberglass reinforced PU foam board is a great candidate for filler, due to its low fiber volume fraction and applications. By salavaging the trimmings and scrap product for regrind or utilizing natural filler, a significant amount of resin could be offset. The fiberglass reinforced PU foam board will be processed using reaction injection molding. The reaction injection molding process will remain the same, only the material layup will consist of regrind and natural filler particles, offsetting the resin volume.

Regrind PU panels will be processed and tested to determine the ideal filler quantity, size, and reduction method. Mechanically characterizing the panels in flexure, tensile, and compression will indicate the tradeoffs for filler percentage and properties. There will be a filler threshold when processability will become difficult and the composite will no longer be an acceptable product.

Natural filled PU panels will be mechanically characterized at different filler loadings. The natural filler will not require material reduction or particle size research since the received product requires no additional steps.

CHAPTER 4. EXPERIMENTAL PROCEDURES

This section outlines the overall experimental procedures. The various materials used, tests performed and the instruments used to measure the physical and mechanical properties of the specimens are summarized.

4.1. Materials

The materials used for processing the different composites can be divided into polymers, reinforcement, and fillers. The two different polymers used were PVC grade Georgia Gulf 8652 and a proprietary PU foam from SpaceAge Synthetics Ltd. of Fargo, ND. The PVC is a flexible grade that is great for low loading and high toughness applications. The proprietary PU foam from SpaceAge Synthetics consisted of two parts, polyol and isocyanurate, to create a rigid polyurethane matrix. When properly mixed together the thermoset polymerization and a side reaction creating a gas to create the foam occurs.

The reinforcement was only used with PU resin and consisted of 350g/m^2 fiber glass continuous filament mat and 610g/m^2 woven fiber glass. Fillers used consisted of -80 ground corn cob from Best Cob Ltd of Rock Falls, IL and regrind particles of post processed PU/fiber glass from SpaceAge Synthetics Ltd. A PMPPIC from Polyscience Inc. was used as a surface treatment for the -80 ground corn cob to improve the interaction with the PVC.

4.2. Surface Treatments

Surface treatments were performed on the -80 ground corn cob to increase the interfacial bond to the PVC matrix. A bleaching treatment was performed to the fibers before compounding and also coating treatment of PMPPIC were performed prior to introduction into the PVC matrix. These are two separate final treatments intended to improve interaction with the PVC.

The bleaching treatment consisted of corn fibers immersed into 70 °C distilled water with 0.5 ml pure acetic acid and 1 g sodium chlorite for half an hour. After 2 h bleaching, the corn fibers were filtered. The bleached fibers were dried in an oven for 24 h at 80 °C.

The coating of PMPPIC consisted of diluting the PMPPIC in toluene to create a solution. The corn cob fibers were added and mixed with a magnetic mixer. Once the toluene evaporated to the point of no longer mixing, the moist fibers were drained further and dried in a convection oven.

4.3. Chemical Analysis

The chemical analysis data of untreated corn fiber, and bleached corn fiber were obtained from the nutrition lab in Department of Animal and Range Sciences, NDSU. These included dry matter testing, neutral detergent solution and acid detergent solution characterization, and starch spectrophotometry. These measurements allowed for the quantification of percentage dry matter, as well as percentage cellulose, hemicellulose, lignin, starch, and ash. Dry matter was determined according to AOAC standard 930.15, in samples were massed at room temperature, heated at 100 °C for 24 h, cooled in a desiccator, and then massed a second time. Neutral detergent fiber, acid detergent fiber, and acid detergent lignin analysis were performed using an $\text{ANKOM}^{200/220}$ Fiber Analyzer according to methods in USDA Agricultural Handbook No. 379[27].

4.4. Material Processing

The processing of the composite materials was performed in a laboratory environment. Composite processing was conducted with precision equipment that can be easily scaled up for industrial use. Processing the PVC was performed with a twin screw extruder and an injection molder. The processing of the PU composite was conducted with a small temperature-controlled, hand-operated press. When conducting research of this nature it is important to keep in mind the scalability of the processes used and how they could be implemented into industry. If the processing is too expensive or difficult for high throughput, it becomes less feasible and unlikely to have an effect on the way polymers are manufactured.

4.4.1. Extrusion

To melt blend the PVC and -80 corn cob, a co-rotating twin screw extruder (Leistritz Micro-18/GL-40D) was used seen in figure 2. Machine settings were altered to allow for easily handled extrusions for pelletizing. A filler loading was chosen for this thesis to be 20% by mass. This filler percentage was chosen for the good balance of economic benefits and minimal negative processing and performance issues. The screws each were set to 225 revolutions per minute, producing a dwell time of 30-40 seconds through the temperature profile from hopper to die of 165°C, 168°C, 171°C, 171°C, 171°C, 176°C, 176°C, and 176°C. The heating schedule used was the manufactures recommendations for processing this particular PVC.

Figure 2. Leistritz Micro-18/GL-40D extruder.

4.4.2. Injection Molding

Test Specimens were processed using an injection (Technoplas, Inc. SIM 5080) seen in figure 3. Two styles of specimens were processed for testing. The PVC and PVC blends had a dwell time around 1-2 minutes through a temperature profile from hopper to nozzle of 165°C, 168°C, 171°C, 174°C, and 176°C. Very similar to the extrusion process the manufactures recommended temperatures were used for processing. Dog bone and straight bars were processed to allow material characterization to be performed.

Figure 3. Technoplas, Inc. SIM 5080 injection molder.

4.4.3. Reaction Injection Molding

The PU composite processing for this study was done on a small scale using a Carver Hot Press pictured in Figure 4. The composite panels were made by mixing polyol and isocyanate together, which react to form the PU foam, and pouring the mixture into a mold containing the bottom continuous filament mat (CFM) preform and glass loft filter material, which allows separation between the top and bottom glass layer. Once the mixture was poured onto the filter material, a top CFM mat was placed on top of the mixture. The cavity was then covered and placed into the press at 70 MPa, where the panel was left to cure for one hour. The panel created, was similar to a Thermo-Lite panel produced by SpaceAge Synthetics Ltd. PU composite panels were labeled with four digits describing the density, glass layup, and surface finish. The first two digits represent the lbs/ft^3 of the panel, the third digit denotes the glass layup, and the final digit signifies how many surfaces are sanded. If the third digit is 0 the panel contains loft material in the center and sandwich between $350g/m^2$ continuous filament mat. If the third digit is 5 the panel contains loft material in the center and sandwich between $350g/m^2$ continuous filament mat and $610g/m^2$ woven fiberglass on both sides.

Figure 4. Carver hot press used for processing composite panels.

The mixing ratios of the polyol and isocyanate were calculated with the regrind filler to maintain a constant global density throughout the testing. For the nonwoven panels a global density of 360 kg/m^3 was targeted and labeled as a 2400. The global density was increased for the woven panels to ensure full wet out of the woven fibers. The target global density for the woven was 450 kg/m^3 and labeled 2850. Table 1 shows the ratios of isocyanate, polyol, and regrind filler used in the nonwoven panels. Table 2 shows the ratios used for the woven panels. When pouring from a mixing cup into the mold it was found that an additional 10 wt% was necessary to compensate for resin left in the cup during pour. Given the short pot life of the PU, little time can be taken in removing all resin from the mixing cup.

Table 1. Example of Nonwoven PU Composite Panel Resin Inputs for Four Filler Percentages

	0wt%	5 wt%	10 wt%	15 wt%
Isocyanate (g)	222.8	211.6	200.5	189.4
Polyol (g)	185.7	176.4	167.1	157.8
Regrind (g)	0	20.5	40.8	61.3

Table 2. Example of Woven PU Composite Panel Resin Inputs for Four Filler Percentages

	0wt%	5 wt%	10 wt%	15 wt%
Isocyanate (g)	205.2	214.4	184.6	174.4
Polyol (g)	171.0	178.6	153.9	145.3
Regrind (g)	0	18.8	37.6	56.4

Higher filler percentages were not trialed as poor wetout became an issue with the woven reinforced panels. The woven fiberglass acted as a tight filter and greatly impeded the flow of resin once the filler particles began collecting. It would take more foam to wetout a higher filler loaded panel, defeating the purpose of utilizing filler at higher concentrations.

4.5. Density Test

The densities of all samples made were determined using a Mettler Toledo 33360 density determination kit at room temperature. The densities were calculated using the following equation $\rho=(A/P)\bullet\rho0$, where ρ is the density of the sample of fibers or composites, A is the weight of sample in air, P is the buoyancy of the sample in distilled water, andρ_0 is the density of distilled water at the given temperature.

4.6. Mechanical Characterization

This section provides details on all mechanical tests performed in this study; several types of tests were conducted to characterize the systems. Tensile, flexural, and compression testing was conducted on the polyurethane composite panels, Thermo-Lite. Tensile, melt flow, and dynamic mechanical analysis (DMA) testing was conducted on the PVC systems to understand mechanical and flow behavior of the systems. All of these material characteristics are extremely important to understand what loading percentage is acceptable and how to design a component utilizing these green fillers. Before a filler is utilized in either thermoplastic or thermoset industry, a full characterization would occur with these tests being the some of the very first performed to get a good understanding of the performance.

4.6.1. Tensile

Tensile testing was performed on the PVC specimens according to ASTM Standard D412[28] on a 5-specimen sample set using an Instron model 5567 load frame. The speed of the crosshead was 5.0, 50.0, and 100.0 mm/min. Each test was performed until tensile failure occurred. The maximum load was recorded and the specific tensile strength was calculated for each sample set. The dogbone specimens tested had an approximate cross section in the gauge section of 4mm x 10mm.

In-plane tensile testing was performed on the PU composite specimens to find the tensile strength and modulus of the Themo-Lite materials tested in the plane of the board. ASTM D1037[29] was the standard followed. It was found that the specimen geometry required an adjustment to ensure proper failure in the gauge section of the specimens for some Thermo-Lite series. Proper failure was accomplished by narrowing the gauge section to 1 inch instead of the prescribed 1.5 inch. Figure 5 shows a specimen and instrumentation attached during in-plane tensile testing. All the tensile testing was conducted in the 0° direction of panel with 5 test specimens for each group. These specimens all had a gauge section of 25mm x thickness which is slightly narrower than recommended by the standard to ensure failure in the gauge section. A test rate of 5min/min was used for the Thermo-Lite specimens.

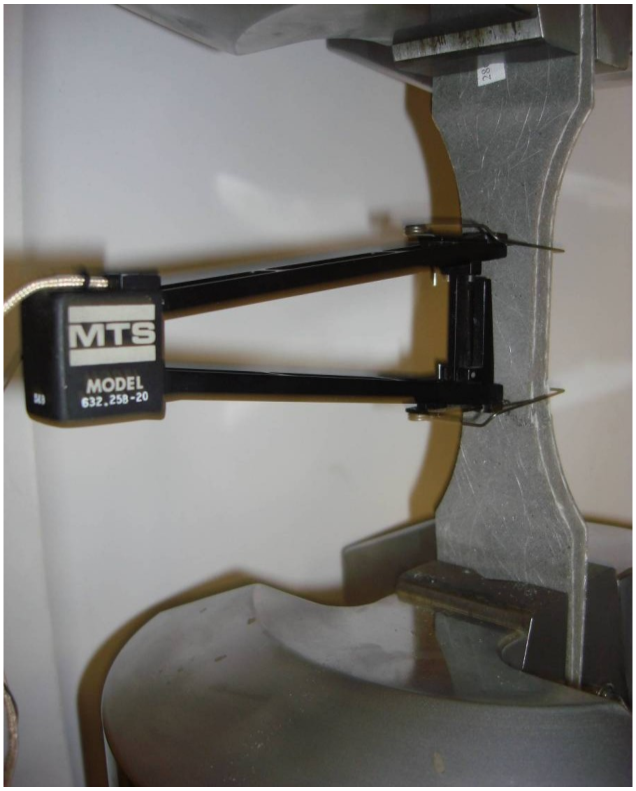

Figure 5. PU composite in-plane tensile test with extensometer.

4.6.2. Flexural Testing

Four point bend testing was performed to find the flexural strength and modulus of the Themo-Lite materials tested perpendicular to the plane of the board and at the 0° fiber direction with 5 specimens in each group. ASTM C393[30] was the standard followed; the specimen length/thickness ratio was chosen to be 32, and quarter point loading was employed. Figure 6 shows a four point bend flexural specimen during testing. The specimens were all cut to be twice as wide as the thickness as suggested in the standard and tested at a rate of 15mm/min.

Figure 6. PU composite four point bend flexure test.

4.6.3. Out-of-Plane Compression Testing

Out-of-plane compression testing was performed to find the compressive strength and modulus of the Themo-Lite materials perpendicular to the plane of the board. ASTM C365[31] was the standard followed; strength values were recorded at 2.5 percent strain. Figure 7 shows an out-of-plane compression specimen during testing. Since the Thermo-Lite doesn't have a definitive compression failure, 2.5 percent strain is used as this is common recording point with similar materials. Five 50mm x 50mm x thickness test specimens were tested for each group of Thermo-Lite. These specimens were tested at a rate of 0.5mm/min.

Figure 7. PU composite out-of-plane compression test.

4.6.4. Melt Flow Index

To determine the effects natural fillers have on the melt flow index (MFI) in a PVC matrix, ASTM D1238[32] and a Tinius Olson AD987 Audio was used. This very basic test determines the amount of polymer that is pressed through a set orifice with a given temperature and pressure over time. This test was conducted twice for both the neat PVC and filled PVC.

4.6.5. Dynamic Mechanical Analyizer

A DMA Q 800 was used to determine glass transition temperature and dampening coefficient. The testing parameters use were a dual cantilever support with a ramp rate of 3°C/min from -100°C to 100°C at a frequency of 1 hertz. The test was conducted three times for both filled PVC and neat PVC.

4.6.6. DSC

Differential scanning calorimetry (DSC) measures temperature and heat flow associated with thermal transitions within the material. These thermal transitions allow the determination of the glass transition temperature of the PU foam which results in a distinct change in heat capacity. A TA Instruments Q1000 DSC was used on neat PU and PU filled with ground corn cob. The DSC testing performs three cycles: heat, cool, and heat. The DSC samples were tested from -50°C to 200°C at 20°C/min, followed by a cooling cycle to -50°C as fast as possible, and finally heated to 200°C at 20°C/min. From the curves generated, two glass transition temperatures, Tg, can be observed at different temperatures. The first Tg results from the first heat cycle and second Tg comes from the second heat cycle. Generally the second Tg is a higher temperature since a higher degree of cure has been achieved after the first heat cycle. This will illustrate the effects the absorbent filler will have on the polymer reaction, by altering the first Tg via quality of cure.

CHAPTER 5. RESULTS AND DISCUSSIONS

This chapter presents and interprets the results obtained in this study. The focus of this research was to determine if either of the fillers are a viable option to reduce the amount of polymer used for a component. The mechanical and processing characteristics discussed in the results could be used as a guideline for designing the qualities desired from such a system. The feasibility of any of the three systems depends on the mechanical performance results and how they affect the design of product. If a thicker component needs to be designed to overcome the ill effects of filler, the cost savings may be relinquished by the additional thickness. However, not every application is designed to the full strength and may be additionally benefited by the increase in modulus that can occur from a filled system.

5.1. PVC Corn Cob

The testing results on the ground corn cob filled PVC are discussed in detail below. The results provide information needed for processing and designing a component utilizing a corn cob filler.

5.1.1. Chemical Analysis

The chemical analysis performed on the ANKOM$^{200/220}$ Fiber Analyzer determines the mass percentage of cellulose, hemicellulose, lignin, starch, ash, and protein in the dry -80 ground corn cob and bleached -80 ground corn cob, listed in Table 3. The bleaching process of the ground corn cob was performed to reduce constituents of starch and protein, which allows for a greater percentage of the desired cellulose and hemicellulose. The bleaching process increased the dry mass percentage by 5 percent for both the cellulose and hemicellulose, which should allow the polymer system to capitalize on the mechanical strength of the cellulose and hemicellulose. Note, due to the testing method and equipment not all constituents are accounted for and are listed as other in table 3, but illustrates the improvement in cellulose content.

Table 3. Corn Cob Chemical Analysis Dry Mass Percentages

	-80	**Bleached -80**
Cellulose	24.27%	30.19%
Hemicellulose	27.24%	32.90%
Lignin	4.3%	4.2%
Starch	9.9%	6%
Ash	3.9%	3.2%
Protein	3.4%	2.8%
Other	26.99%	20.71%

5.1.2. Density Tests

The neat and filled density of the PVC systems are catorgized below in table 4. The density increased 4% with 20% ground corn cob blended into the polymer. Neither the bleaching or surface treatment significantly altered the density of the filled system. With such a small variation in density the specific strength and modulus, which is the strength and modulus divided by the density, a design based on weight would not need to be altered.

Table 4. Density of PVC Systems

	PVC	PVC – 20% -80	PVC – 20% Bleached -80	PVC -20% PMPPIC -80
Average Density (g/cm^3)	1.150 ±0.001	1.196 ±0.001	1.197 ± 0.002	1.198 ±0.002

5.1.3. Melt Flow Index

Melt flow index was conducted on the neat PVC and the untreated -80 corn cob filled PVC at 20% filler content. Melt flow information in table 5 is critical for processors of the polymer as it can dictate the pressure, temperature, and the cycle time needed for injection molding. The melt density was approximately 6% higher in the filled system which is very similar to the solid density difference. The viscosity of the filled system is 200% higher when introducing 20% filler. As followed by the higher viscosity, the mass flow rate and volume rate are greatly reduced in the filled system with drops of 64% and 66%, respectively. The Celcius/kg is the suggested testing parameter for a flexible PVC, it states the polymer is heated to 175C with a 5kg mass used to create pressure for flow.

Table 5. Melt Flow Index Results

	PVC	**PVC -20% -80**
Celcius/kg	175/5	175/5
Melt Density (g/cc)	1.0691	1.1309
Viscosity (Pa-sec)	9845.2	29120.8
Flow Rate (g/10)	2.6985	0.9645
Volume Rate (cc/10)	2.5235	0.8525

5.1.4. Tensile Properties

The tensile test results of strength, modulus, and max strain are shown below in Figures 8, 9, and 10, respectively. The tensile strength of the neat PVC was 6.8 MPa with the filled systems all following below the neat resin. The filler content was 20% by mass for all three systems. The untreated ground corn cob filled system had the highest strength utilizing the ground corn cob with 6 MPa. The bleached ground corn cob produced the lowest strength of 4.9 MPa and the surface modified ground corn cob was slightly higher at 5.1 MPa.

The tensile strength is lower with the filled systems because the filler replaced load carrying polymer chains, meaning the stress transfer between matrix and fiber was very poor. The PMPPIC fibers performed the worst and poor stress transfer could be from the plastizer used in the PVC played a role in the inability to create an effective interface. The PMPPIC treatment did not have the same effectiveness as with rigid PVC in research by Maldas and Kokta

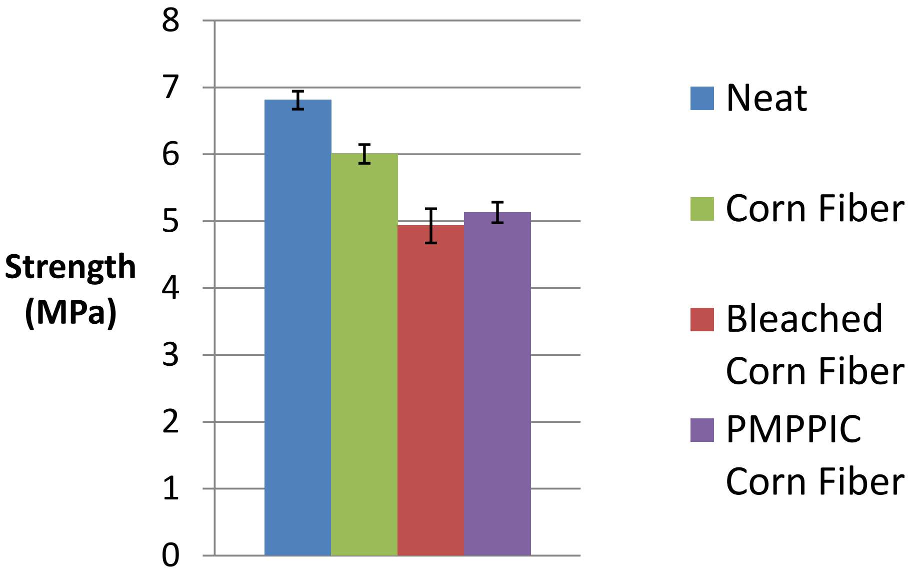

Figure 8. Tensile strength of PVC systems.

The tensile modulus was increased by 126%-135% when introducing all forms of the 20% ground corn cob. Fiber treatments had no statistical influence on the modulus seen in figure 9. The significant increase in tensile modulus can be attributed to restriction of polymer chain movement. Since the PVC chains are unable to slide along each other they are forced to carry the load quicker than when they could move.

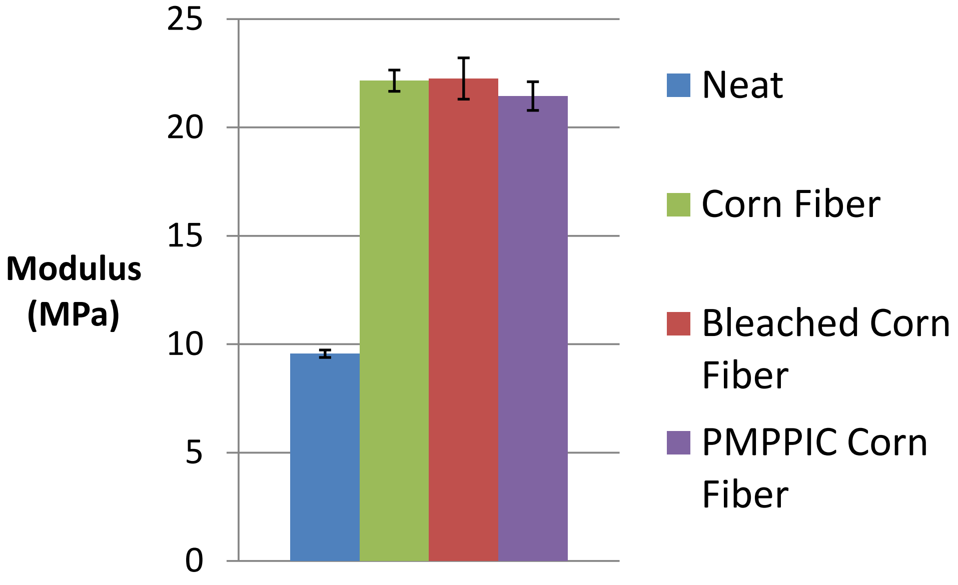

Figure 9. Tensile modulus of PVC systems.

Strain to failure of the tensile test helps determine the resilience to failure and the toughness of a system, seen in figure 10. Inverse to the modulus, the neat PVC has a much greater strain to failure since the flexible PVC is meant to deform in most applications. The chemical treatments had little effect on the strain to failure of the tensile specimens. The strain to failure for the filled systems were much lower since polymer chain movement is restricted by the filler and deformation comes in the form of breaking polymer chains.

The alterations in tensile properties must be considered when designing with a filled PVC, making it difficult to consider it for a direct swap where the flexible PVC is currently being used. Affecting both the ultimate strength, modulus, and strain to failure reduces the amount of energy absorbed during a tensile loading event.

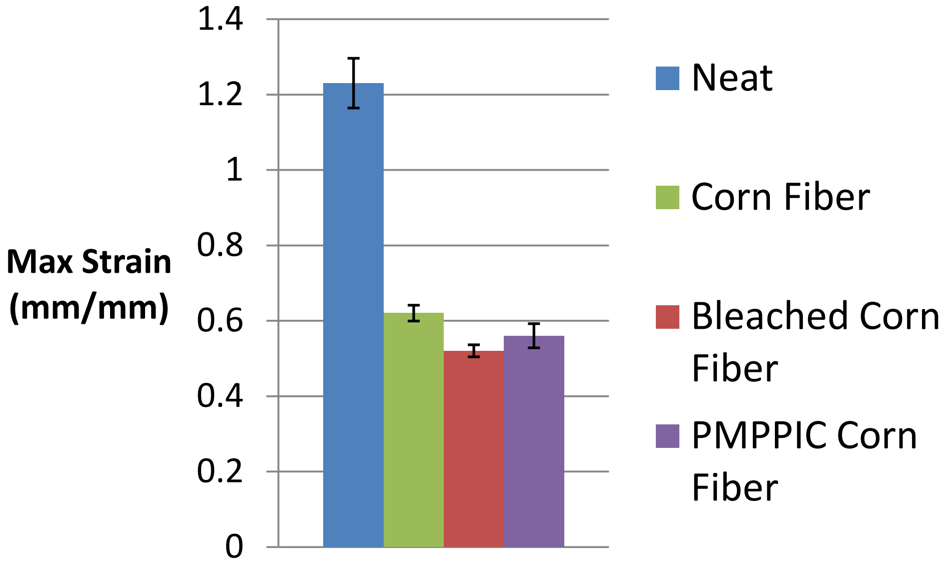

Figure 10. Tensile strain to failure of PVC systems.

5.1.5. Dynamic Mechanical Analyzer

The DMA results were generated for the neat and filled PVC systems from a temperature range of ± 100 C. Figures 11 and 12 illustrate the plotted responses of the testing conducted.

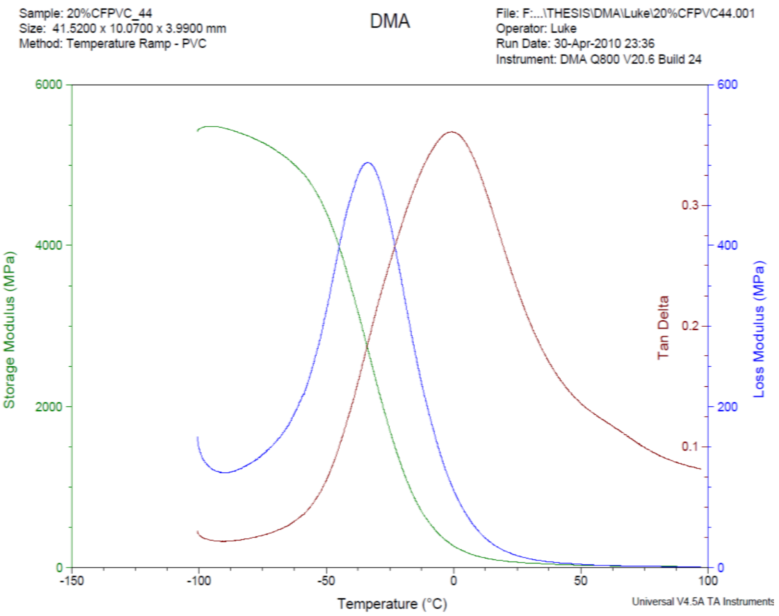

Figure 11. DMA curve 20% filled PVC.

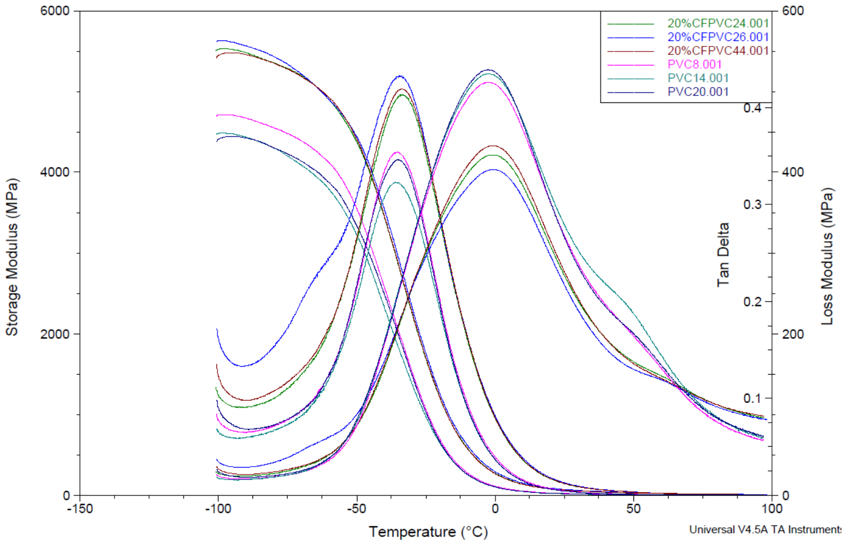

Figure 12. DMA curves for all PVC specimens.

The important information to take from the graphs are the damping coefficient and glass transistion temperature (Tg) of the PVC. Damping Coefficient indicates how efficiently a material loses energy to molecular rearrangements and internal friction. The damping coefficient is often a characteristic to select flexible PVC. Damping Coefficient was taken from the peak of the Tan Delta and decreased by 19% with the filled system. The filled system will take longer to get back to equilibrium than the neat flexible PVC. This makes the filled system less desirable for packaging or protective layer.

Table 6. Damping Coefficient of PVC

	Neat	**20% -80**
Damping Coefficient	0.43	0.35

The glass transition temperature decreased by only 4.5% with the filled system. Glass transition temperature was taken from the peak of Loss Modulus. The ground corn cob had little influence on glass transition temperature and could be considered a nonfactor. The glass transition temperature is very important to understand for thermoplastic systems as they are often used above and below this point. Flexible PVC will encounter -30 to -35 degrees Celsius in its applications and will have significant change in strength and modulus at this temperature range. The glass transistion temperature should clearly be considered in using this flexible PVC and the same considerations should be given to the filled system.

Table 7. Glass Transition Temperature of PVC

	Neat	**20% -80**
Glass Transition Temperature, Tg (°C)	-35.4	-33.8

5.2. PU Corn Cob

The ground corn cob filled PU foam composite panels were mechanically characterized by tensile, compression, flexural, and DSC and compared to unfilled PU foam composite panels. These four tests will give a good indication as to how compatible the ground corn cob is with the PU resin. Mechanical performance is illustrated from the tensile, compression, and flexural testing, while the DSC tests can indicate if the filler affected the cure. For each of the tests four graphs were populated to show strength and modulus of each configuration. The woven fiberglass reinforced polyurethane panels and the nonwoven fiberglass reinforced polyurethane panels each had four different filler concentrations of 0, 5, 10, and 15%.

5.2.1. Tensile Properties

The woven fiberglass reinforced PU foam panels showed a decrease in tensile properties compared to the neat panel. The drop in tensile strength was expected with increasing the discontinuity of the matrix of the composite with the filler. The drops in modulus in the filled systems were more drastic with less dependance on filler percentage.

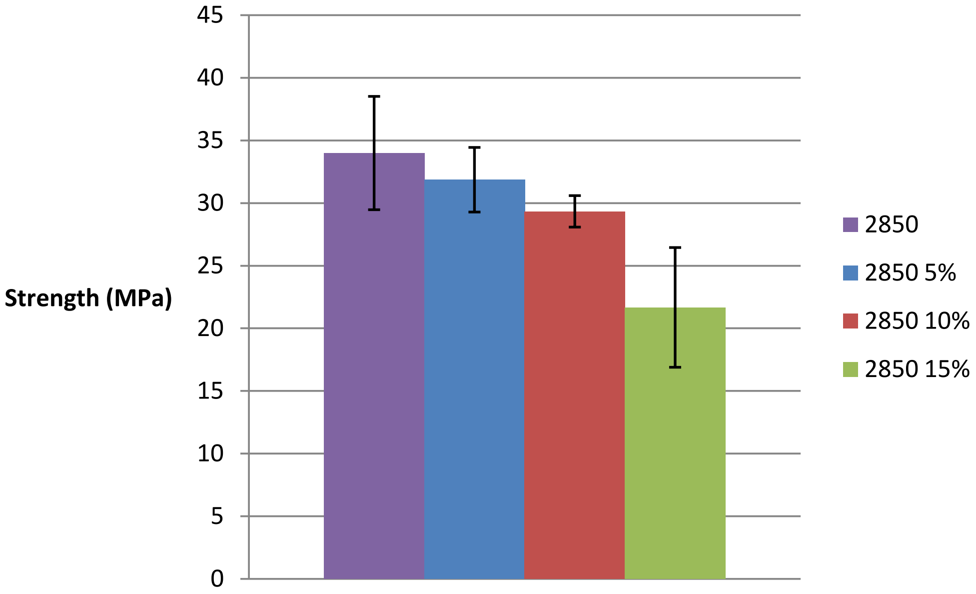

Figure 13. Tensile strength of 12.7mm thick woven PU composite.

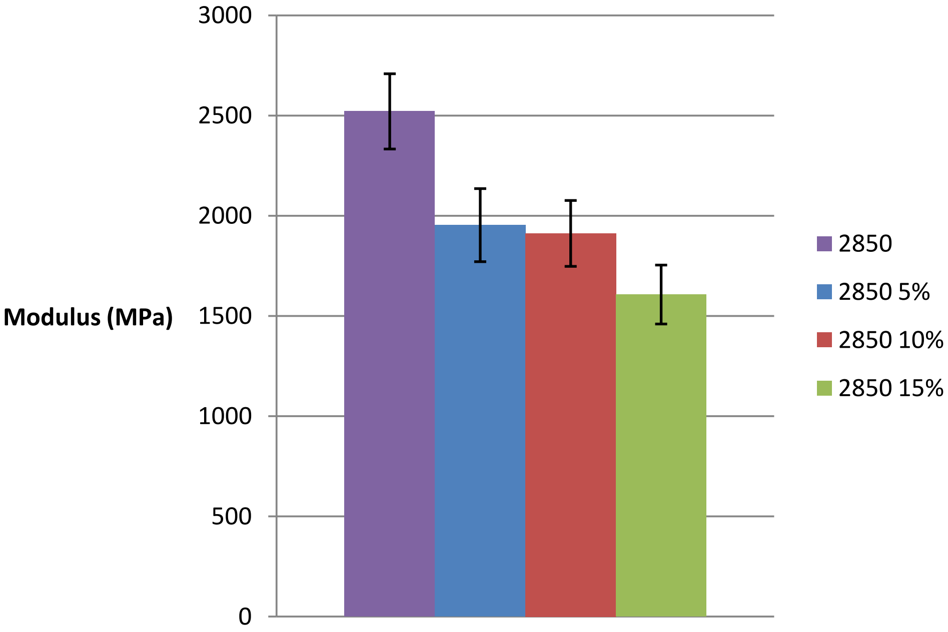

Figure 14. Tensile modulus of 12.7mm thick woven PU composite.

The nonwoven panels showed similar trends to the woven samples, but with larger percentage decrease. Since the nonwoven specimens have less fiberglass reinforcement the performance of the matrix has a larger role. The effects of the ground corn cob appear to have a stronger negative effect on the tensile strength and modulus of the PU foam.

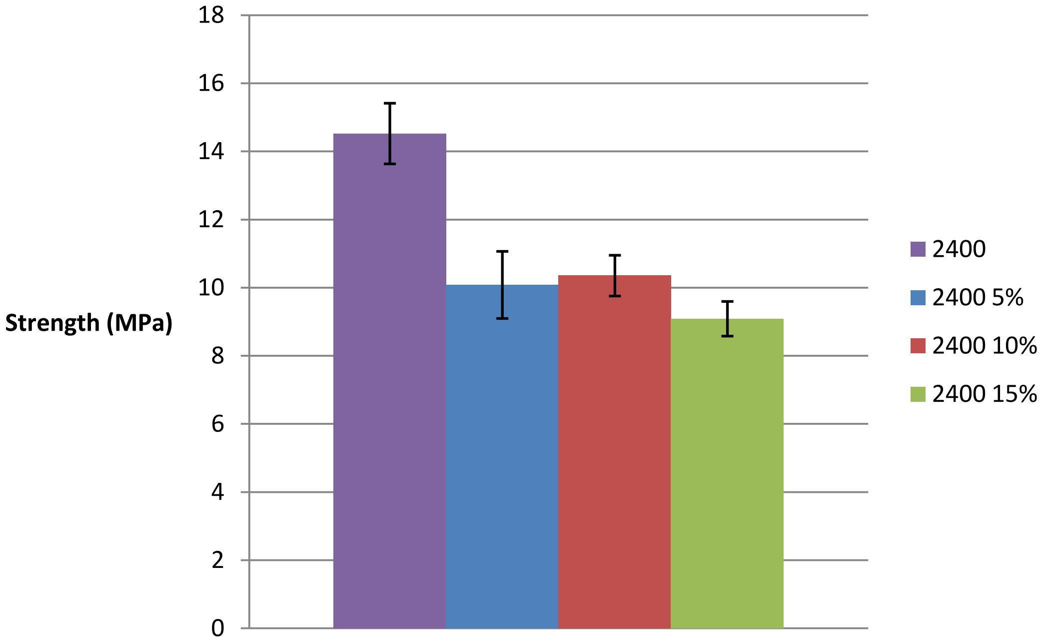

Figure 15. Tensile strength of 12.7mm thick nonwoven PU composite.

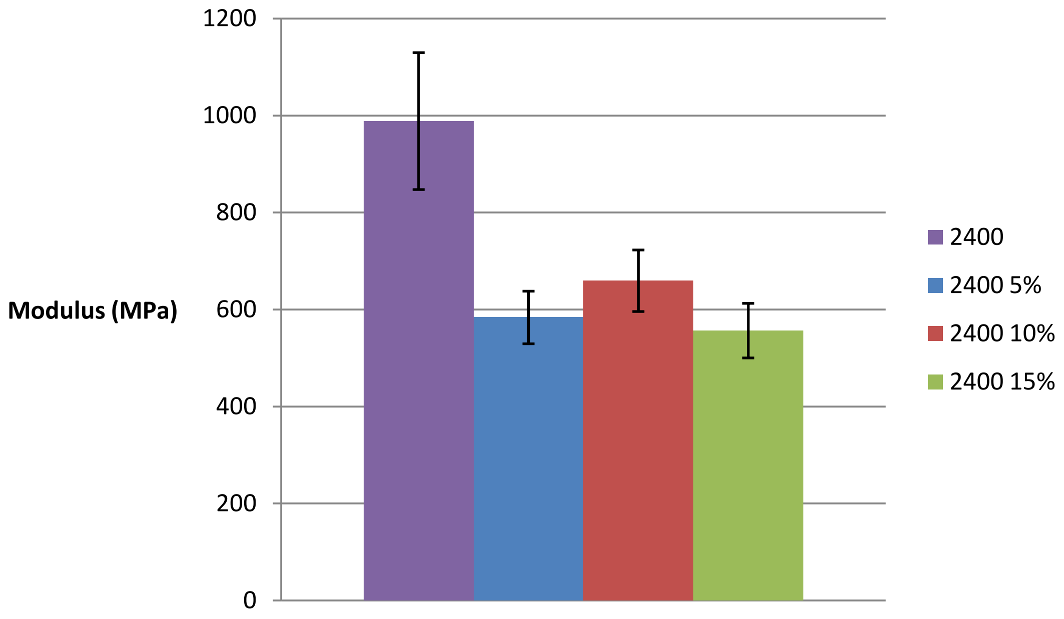

Figure 16. Tensile modulus of 12.7mm thick nonwoven PU composite.

The results indicate that the percentage of ground corn cob has an effect on the tensile performance and the ability to transfer load through the fiberglass. Unlike the filled PVC specimens, an increase in tensile modulus is not seen due to the fact that the modulus is predominantly determined by the fiberglass loading and not the neat polymer. Restricting polymer chain movement has less of a factor in the modulus as efficient load transfer between fiberglass and matrix, due to significantly higher modulus of glass fibers compared to PU foam.

5.2.2. Flexural Properties

Similar to the tensile properties, the flexural strength and modulus decreased with the addition of ground corn cob. Unlike the tensile testing, there is not a consistent drop for each filler percentage, but rather a big drop observed in all specimens. A larger decrease in strength was seen in the woven panels due to the failure mechanism. With the woven fiberglass near the top and bottom surfaces, a compressive failure occurs on the top surface. The PU foam determines the ultimate strength as a nearly pure foam interface is the buckling point along the woven fiberglass. It appears that the filler has detrimentally reduced the strength of the PU foam matrix.

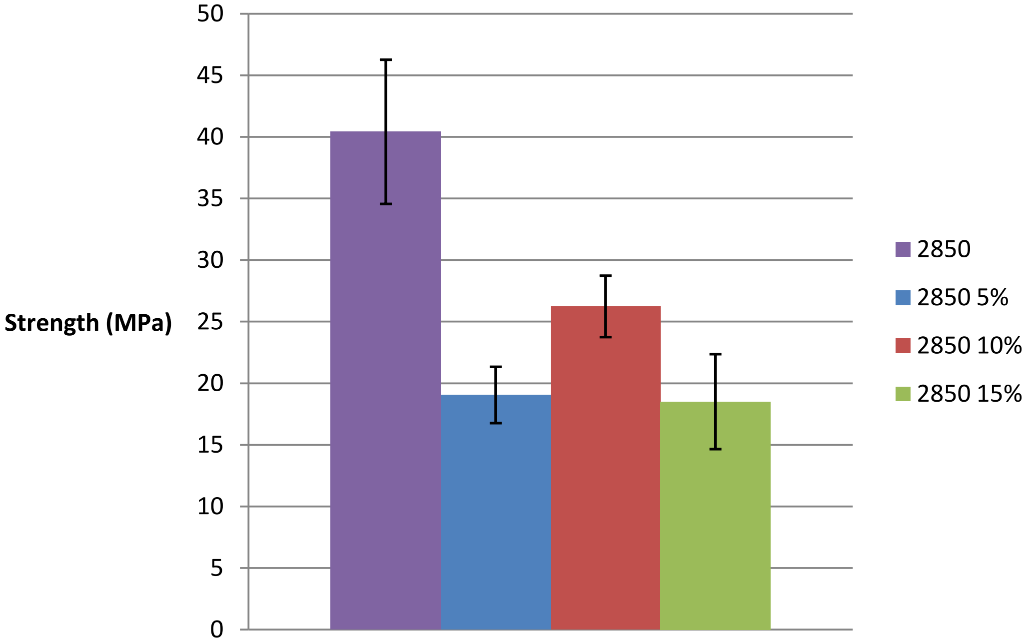

Figure 17. Flexural strength of 12.7mm thick woven PU composite.

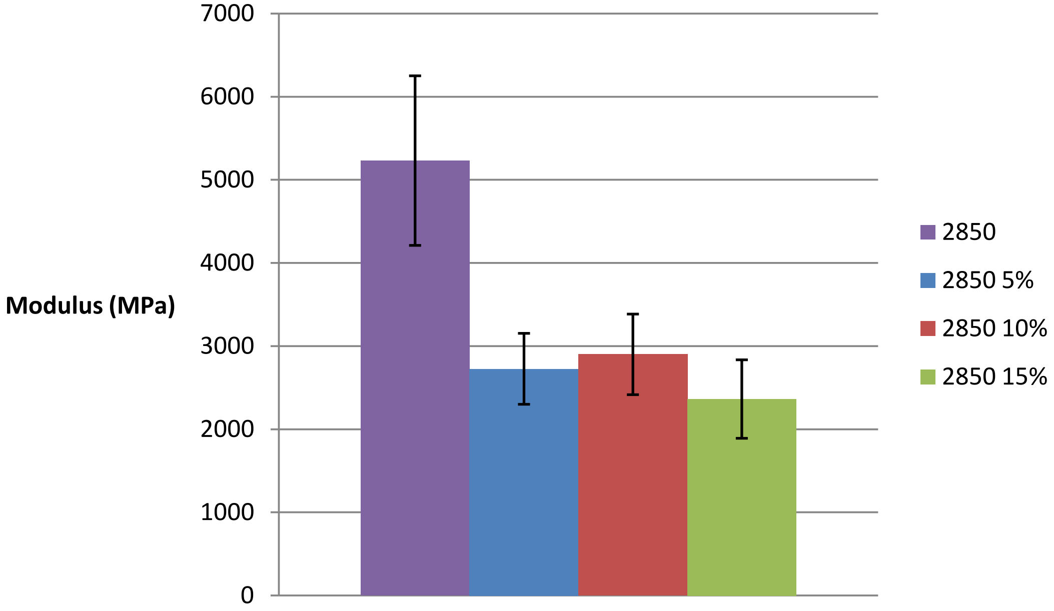

Figure 18. Flexural modulus of 12.7mm thick woven PU composite.

The nonwoven panels showed significant reduction in flexural strength and modulus with the addition of ground corn cob. As with the woven specimens there is not a linear decrease in properties, but instead a large decrease with similar values between filled systems. The tensile and flexural results indicate the possibility of a poor polymer reaction in the PU foam reducing mechanical properties.

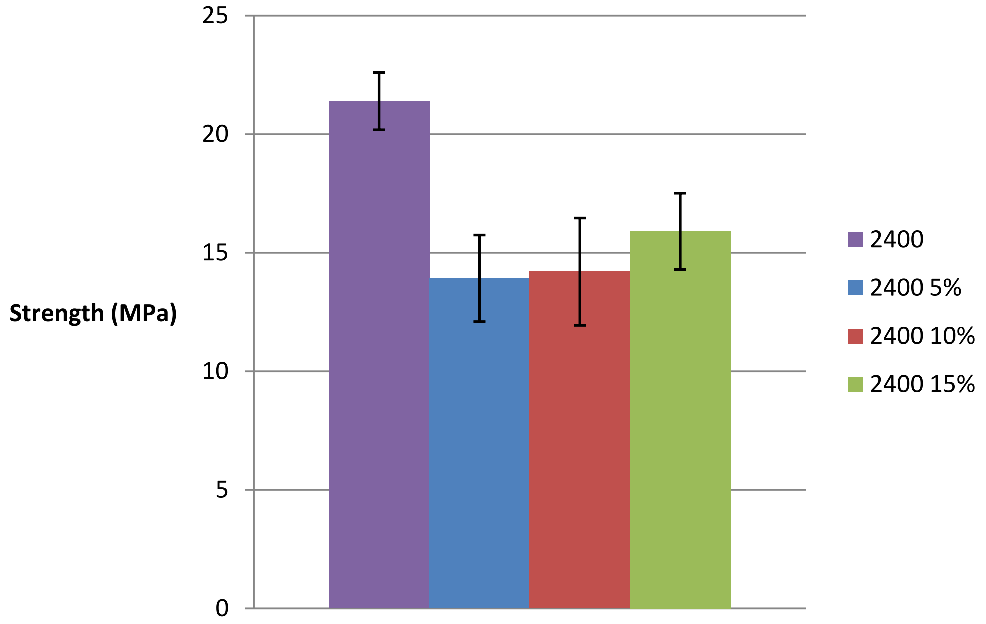

Figure 19. Flexural strength of 12.7mm thick nonwoven PU composite.

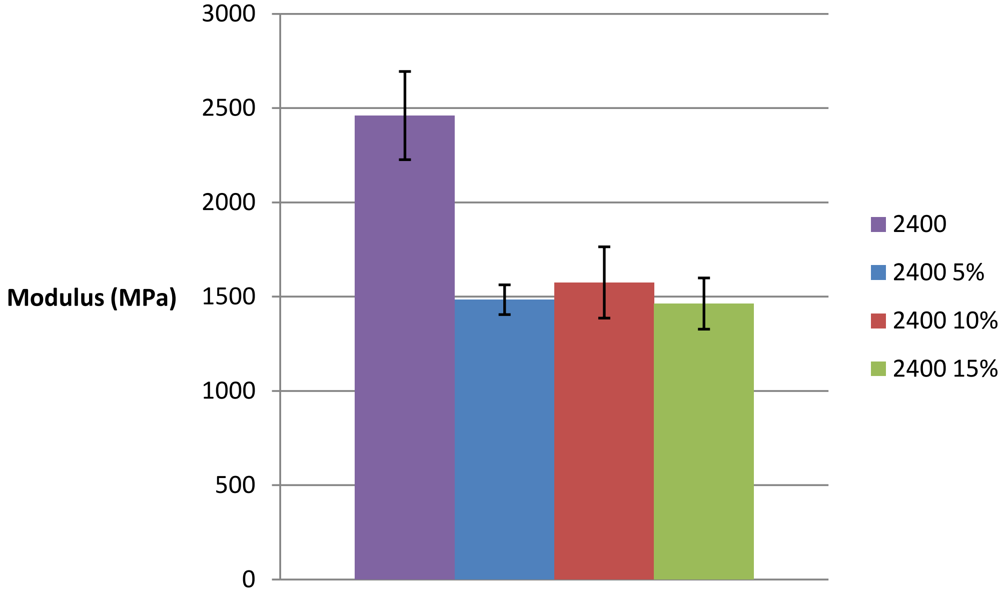

Figure 20. Flexural modulus of 12.7mm thick nonwoven PU composite.

5.2.3. Compression Properties

The compression testing results indicate little influence on the physical presence, as fillers commonly increase compression modulus to restriction of molecular motion. However it seems that little effect was witnessed in figures 21 and 22 at 5 and 10% for the woven panels with a significant drop in strength and modulus at 15% filler.

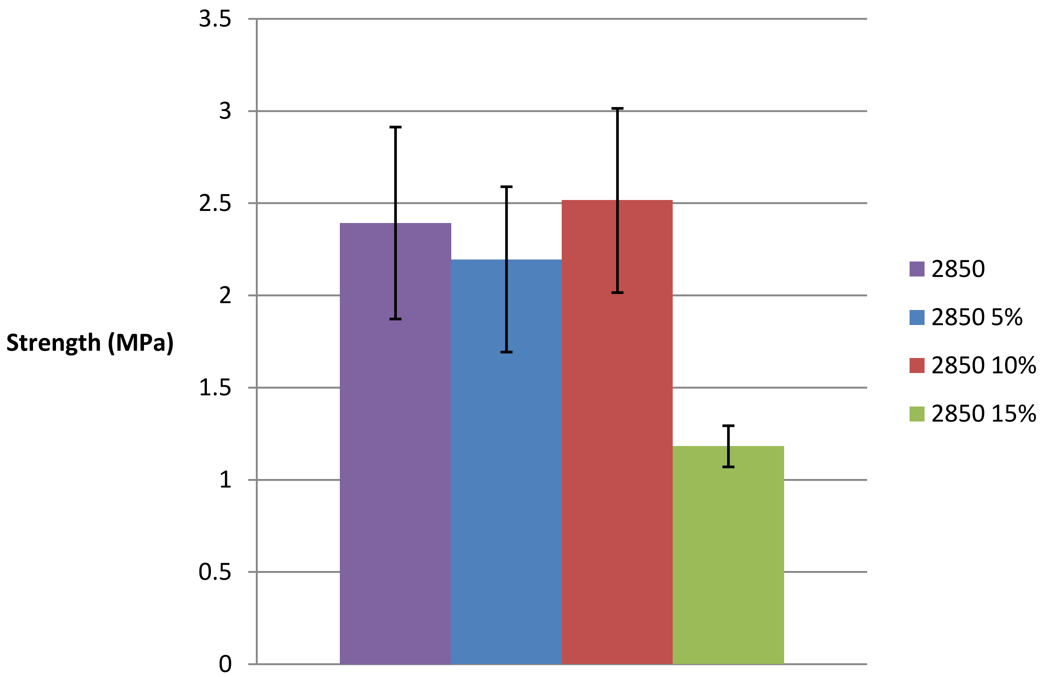

Figure 21. Compressive strength of 12.7mm thick woven PU composite.

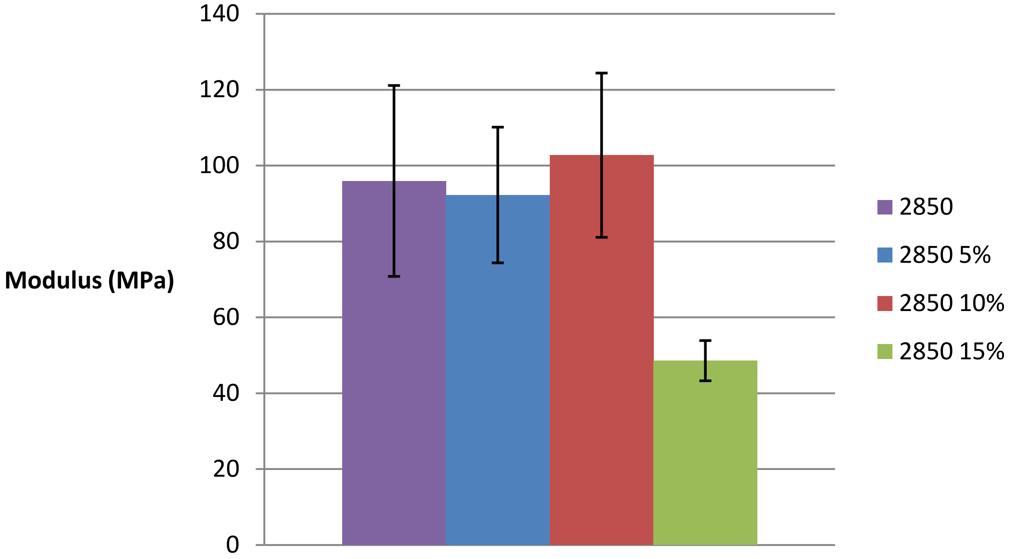

Figure 22. Compressive modulus of 12.7mm thick woven PU composite.

Similarly, 5 and 10% in the nonwoven panels perform as expected with both the neat 2400 and 15% having significantly lower values for strength and modulus in figures 23 and 24. The drop does not translated to the performance in flexural or tensile, since the fiberglass reinforcement has a greater influence on those properties and compression properties are predominantly a function of the polyurethane. Compression properties can drop significantly if the mixture of iso and poly are not kept close to ideal.

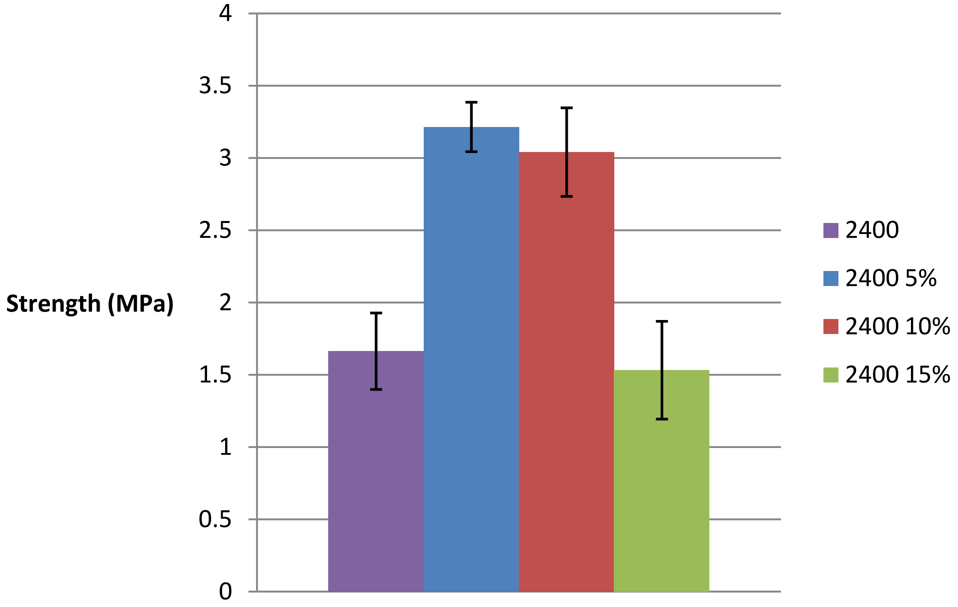

Figure 23. Compressive strength of 12.7mm thick nonwoven PU composite.

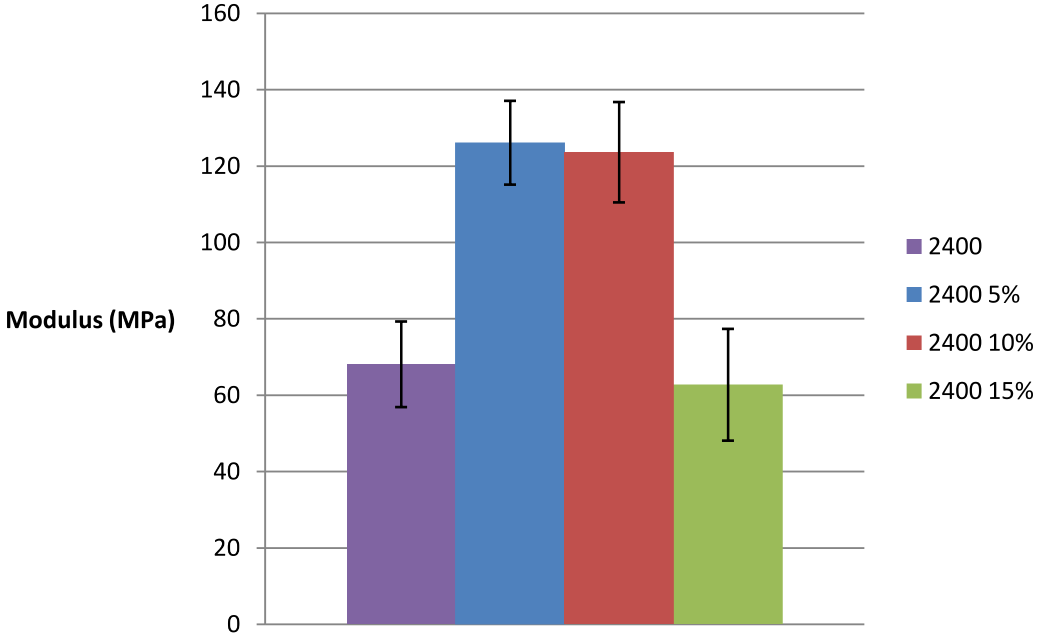

Figure 24. Compressive modulus of 12.7mm thick nonwoven PU composite.

5.2.4. DSC Properties

The DSC results were used to indicate the effects of corn cob filler on the glass transition temperature. The glass transition temperature can be used as an indicator of cure, the amount of polymerization that has occurred. When comparing the same resin, the higher the first glass transition temperature the higher percentage cure achieved during the initial reaction. The second glass transition temperature indicates the final Tg after a full cure is reached and no further polymerization will occur. Table 8 below lists the Tg1 and Tg2, which are the first and second glass transition temperatures from the DSC results.

The specimens labeled 2850 and 2400 are the neat PU composite panels and the specimens with 5CC, 10CC, 15CC are 5, 10, and 15% corn cob filled, respectively. The DSC results show a reduction in the first Tg with the increase in ground corn cob. This indicates the corn cob had a negative effect on the PU foam cure. If the ground corn cob absorbed polyol prior to being mixed with the isocyanurate, the ground corn cob would then had the opportunity to absorb polyol and make the mixture be polyol light. This helps explain the significant drop in compression performance of the highly filled system and that the neat 2400 specimens might have had a poor mixture being identified in the lower compression values and a lower Tg than a filled system of 5%. Also, by absorbing polyol their will be less foaming occurring since the water is carried in the polyol, which is reacted with the isocynaurate to generate CO_2. This will reduce the wetout capabilities of the resin by reducing the amount of gas created.

Table 8. Glass Transition Temperature Neat and Filled PU Composite

Specimen	Tg1 (ºC)	Tg2 (ºC)
2850	88.17	138.96
2850 5CC	78.19	142.41
2850 10CC	70.81	135.68
2850 15CC	64.29	133.33
2400	81.55	136.45
2400 5CC	85.65	150.45
2400 10CC	72.17	152.32
2400 15CC	67.39	152.53

During processing at the higher filler percentage panels, the surface had a tacky feel, indicating a poor reaction. The panels also had a softer surface and were more easily scratched or dented. These observations also indicate a poor reaction occurring with the higher filler percentages.

5.3. PU Regrind

PU regrind panels are filled with post processing and production scrap from the production of similar panels. The tensile tests were analyzed and used to find the tensile modulus and the tensile strength of the specimens. Tensile properties were used as a screening characteristics, assuming it would illustrate the worst case scenario of adding fillers. Figures 25-28 exhibit the mean tensile modulus and mean tensile strength of the woven and nonwoven panels at various loadings and particle sizes. The far left column represents the control Thermo-Lite, which had no regrind. The next set of columns to the right of the control represents the 5 wt.% loading of the regrind. Within that set, the particle size decreases from left to right. The last set of columns on the far right represent the 15 wt.% loadings of regrind with the particle size again decreasing from left to right. The bars on top of the columns represent the standard deviation of the tests.

Figure 25 demonstrates that the mean tensile modulus of the nonwoven was the greatest at the screen size range of 0.251 – 0.152mm with a 15 wt.% loading. The 4.75 – 0.853mm screen size range performed the worst, which was fairly predictable since particles of this size will tend to form stress concentrations in the material. The tensile modulus of the non-woven panels do tend to increase from 5 wt.% to the 15 wt% in most of the loadings, however in these cases the standard deviations overlap deeming the increase statistically insignificant. When looking at the mean tensile modulus plot of the nonwoven series, nearly all of the specimens have overlapping standard deviations, other than the 4.75 – 0.853mm screen size range. This means statistically, the regrind is not affecting the tensile modulus of the composite at 5 wt.% or 15 wt.%, which is very promising for future manufacturing. The regrind actually slightly increases the tensile modulus at certain particle sizes.

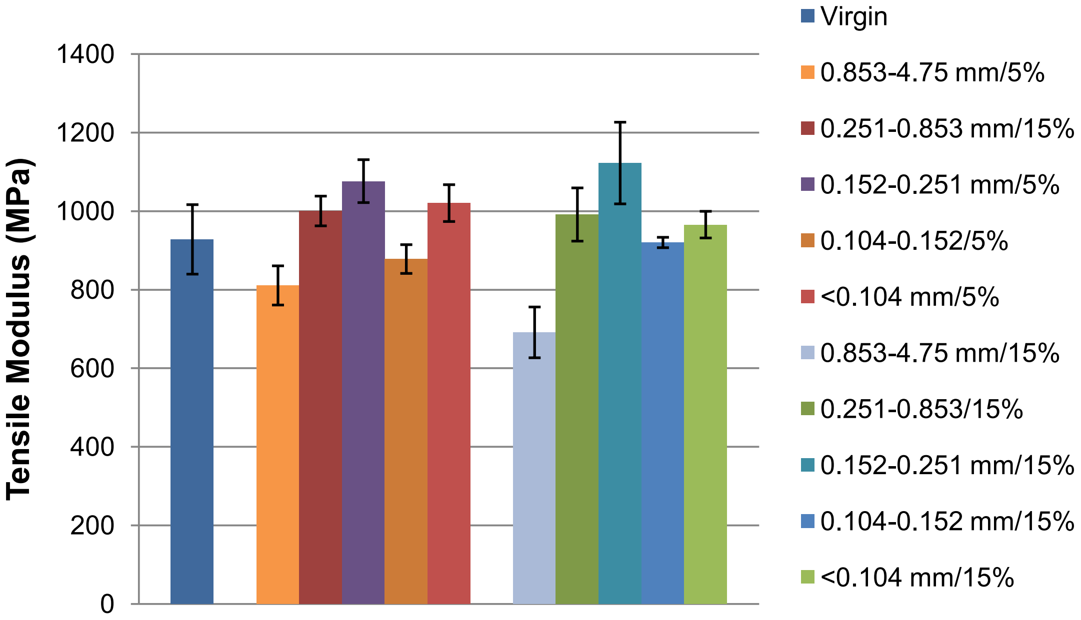

Figure 25. Mean tensile modulus of 2202 series test specimens.

Figure 26 demonstrates similar patterns that the tensile modulus plots showed for the non-woven panels. However, instead of the tensile strength slightly increasing from 5 wt.% to 15 wt.%, the tensile strength seemed to slightly decrease. However, nearly all the results other than the 4.75 – 0.853mm screen size range lie within the standard deviation of the neat material. This reiterates the trend that statistically, the regrind does not seem to be playing a significant role in the tensile properties of the composite up to a 15 wt.% loading.

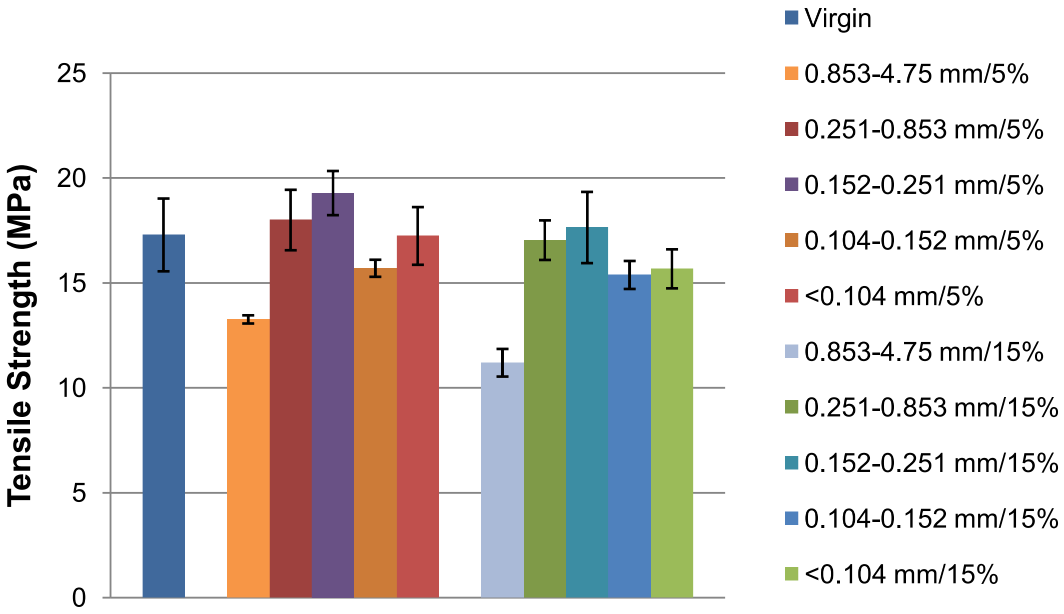

Figure 26. Mean tensile strength of 2202 series test specimens.

With regards to the tensile data for the woven panels, Figures 27 and 28 demonstrate even less of an impact of the regrind on the tensile properties. This is likely due to the fact that in the woven series, the vast majority of the load is transferred to the woven reinforcement leaving the matrix material less of a factor in determining the overall material properties. Tables 9 and 10 show the results of these tensile tests.

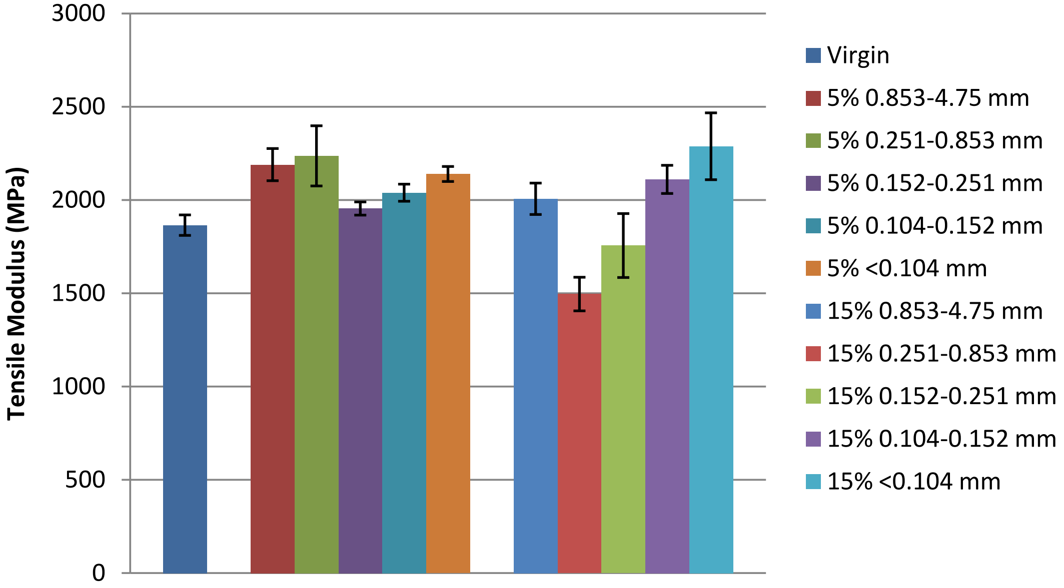

Figure 27. Mean tensile modulus of 2852 series test specimens.

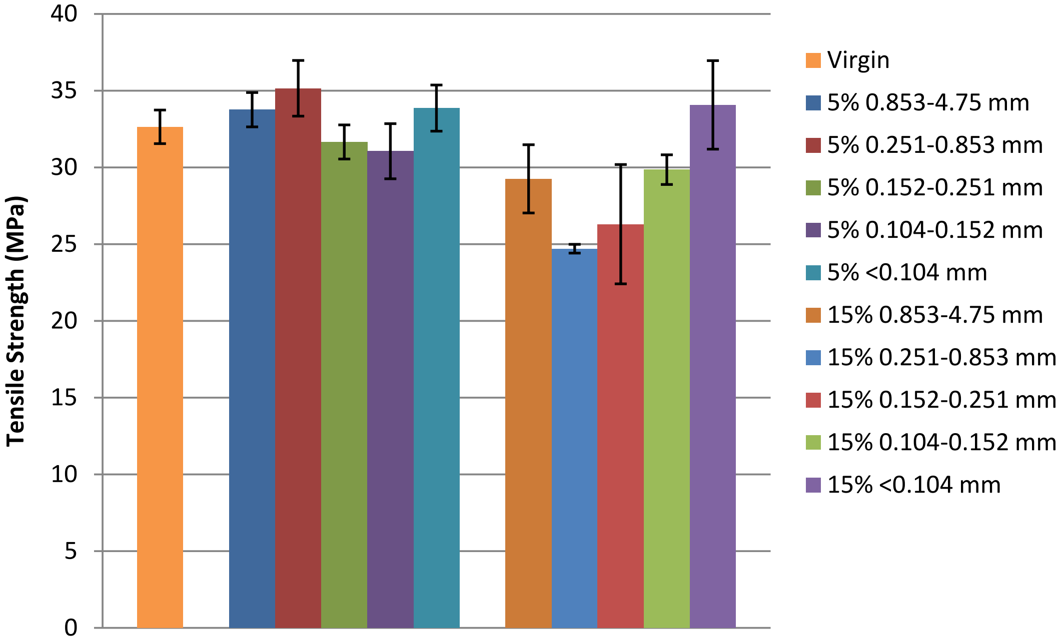

Figure 28. Mean tensile strength of 2852 series test specimens.

Table 9. Test Results of 2202 Series Panels for the Particle Size/Loading Study

	Neat	5% 0.853-4.75mm	5% 0.251-0.853mm	5% 0.152-0.251mm	5% 0.104-0.152mm	5% <0.104	15% 0.853-4.75mm	15% 0.251-0.853mm	15% 0.152-0.251mm	15% 0.104-0.152mm	15% <0.104
***E* (MPa)**	928 ±88	811 ±50	1000 ±38	1076 ±55	878 ±37	1020 ±47	691 ±65	991 ±68	1122 ±104	920 ±13	965 ±34
***σ* (MPa)**	17.3 ±1.7	13.3 ±0.2	18.0 ±1.4	19.3 ±1.1	15.7 ±0.4	17.2 ±1.4	11.2 ±0.6	17.0 ±0.9	17.6 ±1.7	15.4 ±0.7	15.7 ±0.9

Table 10. Test Results of 2852 Series Panels for the Particle Size/Loading Study

	Neat	5% 0.853-4.75mm	5% 0.251-0.853mm	5% 0.152-0.251mm	5% 0.104-0.152mm	5% <0.104	15% 0.853-4.75mm	15% 0.251-0.853mm	15% 0.152-0.251mm	15% 0.104-0.152mm	15% <0.104
***E* (MPa)**	1865 ±55	2189 ±86	2236 ±161	1954 ±35	2039 ±46	2139 ±40	2006 ±84	1496 ±90	1756 ±171	2110 ±75	2288 ±179
***σ* (MPa)**	32.6 ±1.1	33.8 ±1.1	35.1 ±1.8	31.7 ±1.1	31.0 ±1.8	33.9 ±1.5	29.2 ±2.2	24.7 ±0.3	26.3 ±3.9	29.8 ±1.0	34.1 ±2.9

5.3.1. Lab Scale Processing for Equipment Selection Tests

Unlike the ground corn cob, the regrind needs to be reduced to a size suitable to incorporate. Similar to what Zia, Bhatti, and Bhatti reported the particle size most desired for this regrind was less than .25mm, with increasing difficulty with smaller particles becuase of increased surface area to wetout. Companies sourced to evaluate different means of material reduction to the targeted sizes found in the particle size testing. In order to identify the most effective commercial grinder, a study was conducted evaluating grinders from three companies using lab scale processing. The three companies evaluated were Reduction (RE), Cumberland

(CL), and Rapid (RA). Both woven and non-woven panels were processed with 5 wt%, 10 wt%, and 15 wt% loadings and were then tensile tested. Figures 29-32 show the tensile modulus and strength plots of the tested panels. While examining either the woven or nonwoven data, it is very difficult to discover any obvious trends. Any differences in results between grinders usually falls within the standard deviation of the results, and no clear trends can be found from one wt.% set to the next.

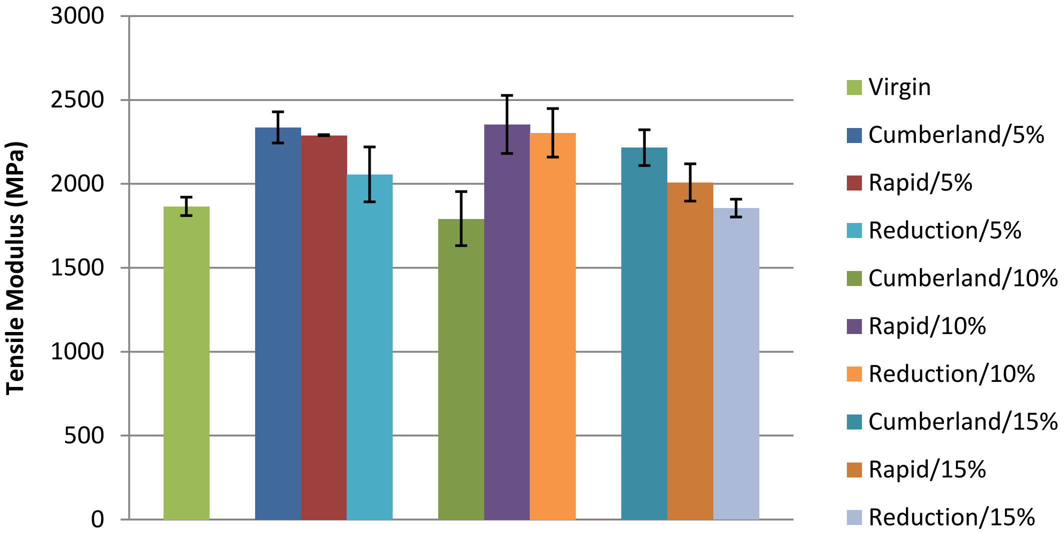

Figure 29. Tensile modulus comparison of 2852 series commercial regrind panels.

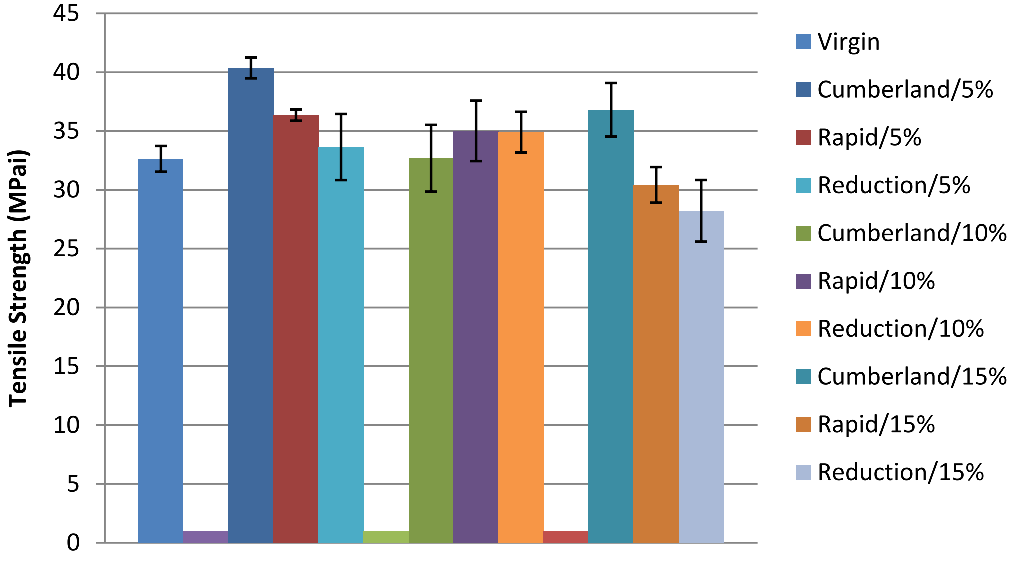

Figure 30. Tensile strength comparison of 2852 series commercial regrind panels.

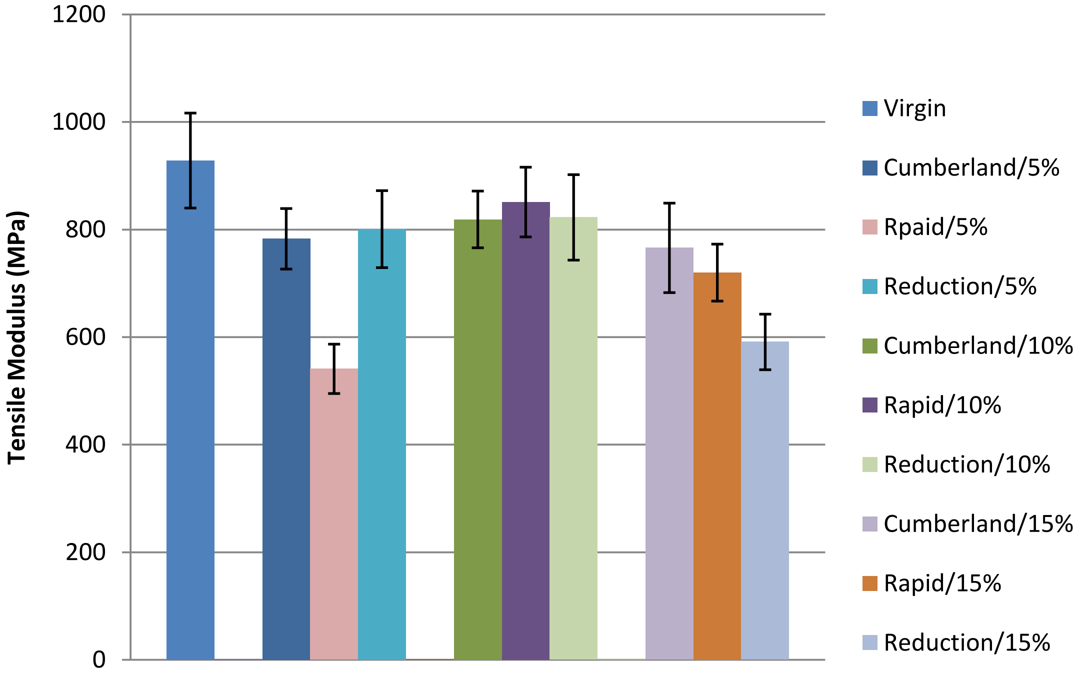

Figure 31. Tensile modulus comparison of 2202 series commercial regrind panels.

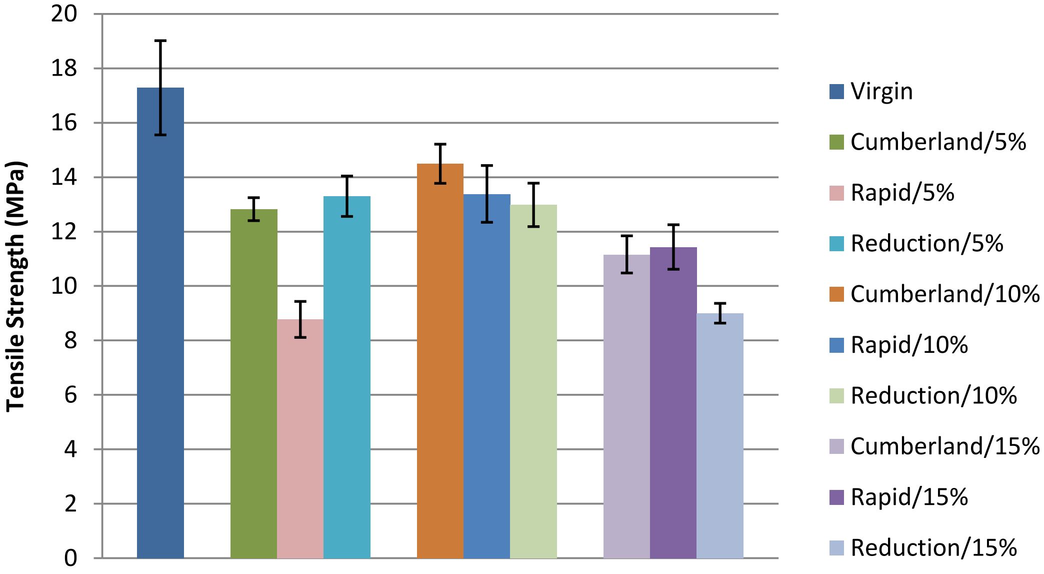

Figure 32. Tensile strength comparison of 2202 series commercial regrind panels.

The conclusion of the study is that no grinder clearly outperforms the other, so the best option is to go with the grinder that produces the most processable regrind at the best price. Tables 11 and 12 show the results for this study. With no commercial grinder significantly outperforming it was determined that full scale processing of both a granulated material and a pulverized regrind was necessary to understand the processability of the different regrinds.

Table 11. Test Results for the 2202 Series Panels for the Commercial Grinder Study

	V	*CL 5%*	*RA 5%*	*RE 5%*	*CL 10%*	*RA 10%*	*RE 10%*	*CL 15%*	*RA 15%*	*RE 15%*
E* *(MPa)	928 ±88	783 ±56	540 ±46	801 ±72	819 ±53	851 ±65	822 ±79	766 ±83	720 ±53	591 ±52
σ* *(MPa)	17.3 ±1.7	12.8 ±0.4	8.8 ±0.7	13.3 ±0.7	14.5 ±0.7	13.4 ±1.0	12.9 ±0.8	11.2 ±0.7	11.4 ±0.8	9.0 ±0.4

Table 12. Test Results for the 2852 Series Panels for the Commercial Grinder Study

	V	*CL 5%*	*RA 5%*	*RE 5%*	*CL 10%*	*RA 10%*	*RE 10%*	*CL 15%*	*RA 15%*	*RE 15%*
E* *(MPa)	1865 ±55	2335 ±93	2289 ±3	2056 ±164	1792 ±161	2353 ±173	2303 ±145	2215 ±107	2007 ±111	1855 ±53
σ* *(MPa)	32.6 ±1.1	40.4 ±0.9	36.4 ±0.5	33.6 ±2.8	32.7 ±2.8	35.0 ±2.6	34.9 ±1.7	36.8 ±2.3	30.4 ±1.5	28.2 ±2.6

5.3.2. Full Scale Material Testing

After the production of the full scale panels, the panels were sanded on both sides to provide a more even and consistent surface for quality test specimens. With the panels sanded, specimens for mechanical testing were cut out using a 3 axis computer numerical control (CNC) router. The test specimens were also given a minimum of 7 days to fully post cure before measuring and testing occurred.

Tensile, flexural, and compression specimens were taken from all panels, with the tensile specimens being of the same dimensions as the lab scale specimens. While flexural, compression, and tensile properties were checked for all materials, it was also determined that a select panel should be tested for a wide range of mechanical properties to ensure that the regrind did not have detrimental effects on less studied mechanical properties.

5.3.3. Full Scale Processing Equipment Selection Tests

The preliminary testing of the full scale processed panels were limited to tensile, flexural, and compression. Also only one concentration of regrind was used, since the desired information was to determine which regrind processes and mechanically performs the best. Figures 33–38 illustrate the strength and modulus of the three regrind panels processed in the nonwoven series. From the test results there again is not a regrind that statically out performs the other. However, it was noted that the Cumberland 3/8” regrind processed the best with the least amount of dry spots and airborne fines. The difference between the Cumberland 3/8” and 3/16” is the size of holes in the screen of the grinder. The material is continually shearing until it reaches a particle size small enough to fall through the 9.5mm (3/8”) or 4.75mm (3/16”) diameter hole. The Reduction pulverizer uses a gap between to rotating plates to determine the size of the particle. The Rapid granulator was not utilized any further as the process was the same as the Cumberland but more expensive.

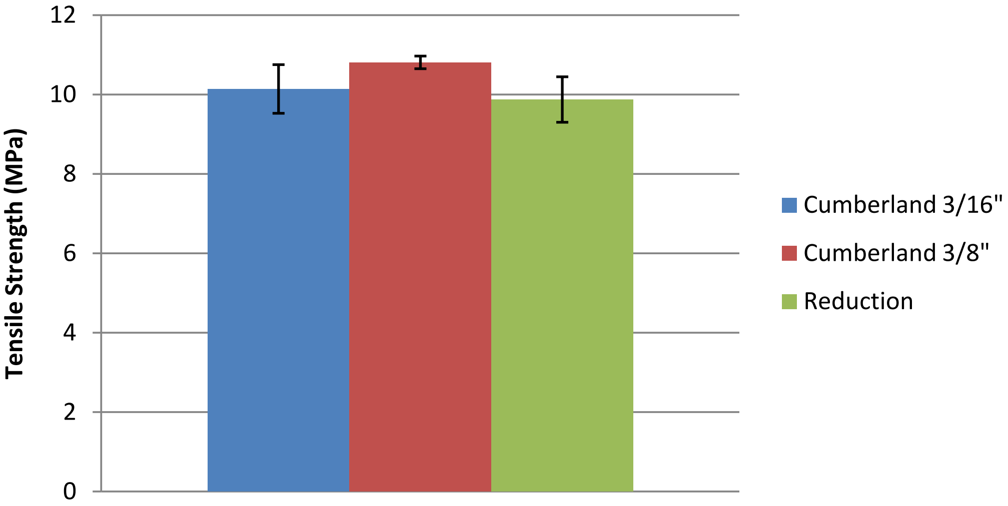

Figure 33. Tensile strength of 2202 with 15% regrind filler.

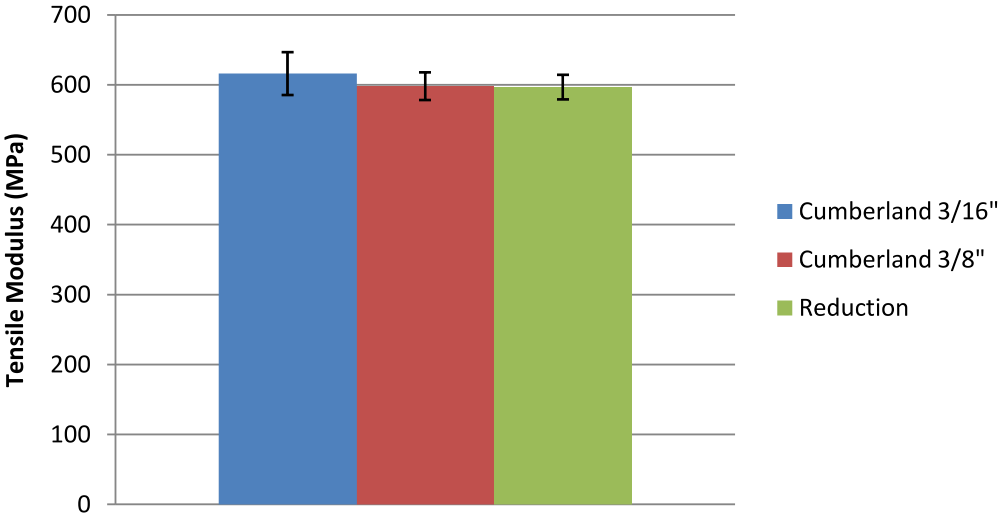

Figure 34. Tensile modulus of 2202 with 15% regrind filler.

In figures 35 and 36 the flexural strength of the Reduction pulverized material performed the worst with a slight dip in flexural strength. The flexural modulus of all three were very similar with overlapping standard deviations.

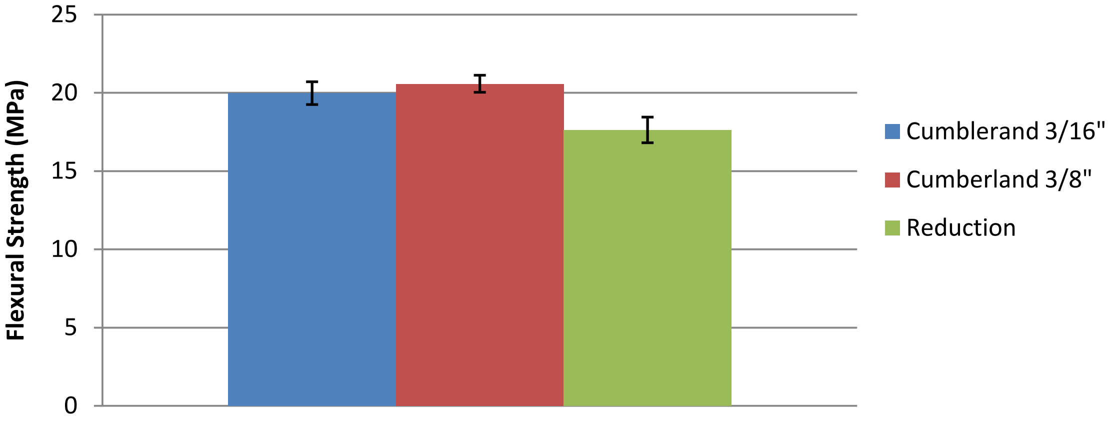

Figure 35. Flexural strength of 2202 with 15% regrind filler.

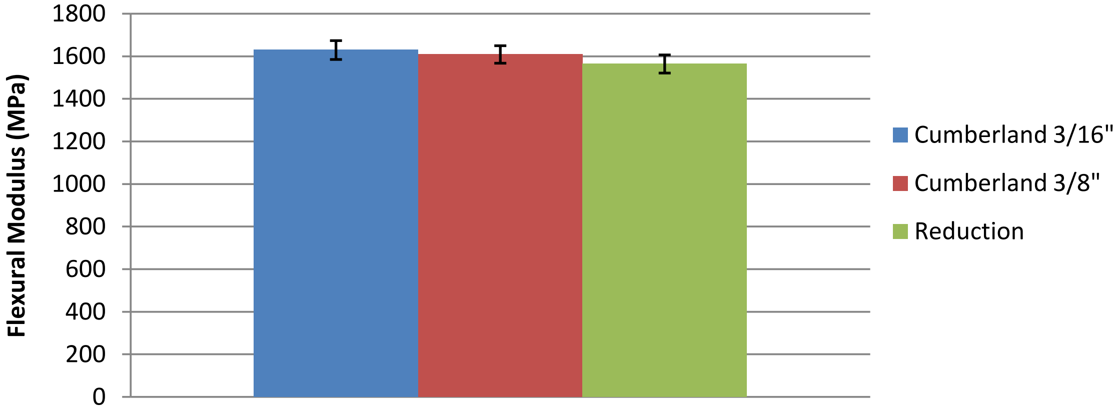

Figure 36. Flexural modulus of 2202 with 15% regrind filler.

In figures 37 and 38 the compression strength is slightly lower with the finer screen sized Cumberland but neglible when considering the standard deviations. The compression modulus is again very close with the average being slightly lower for the smaller screen sized Cumberland.

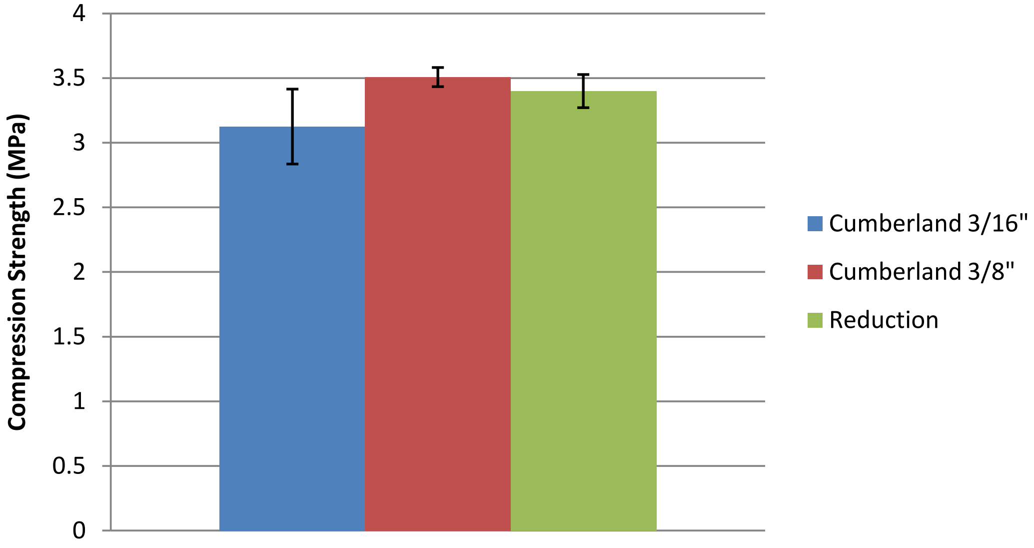

Figure 37. Compression strength of 2202 with 15% regrind filler.

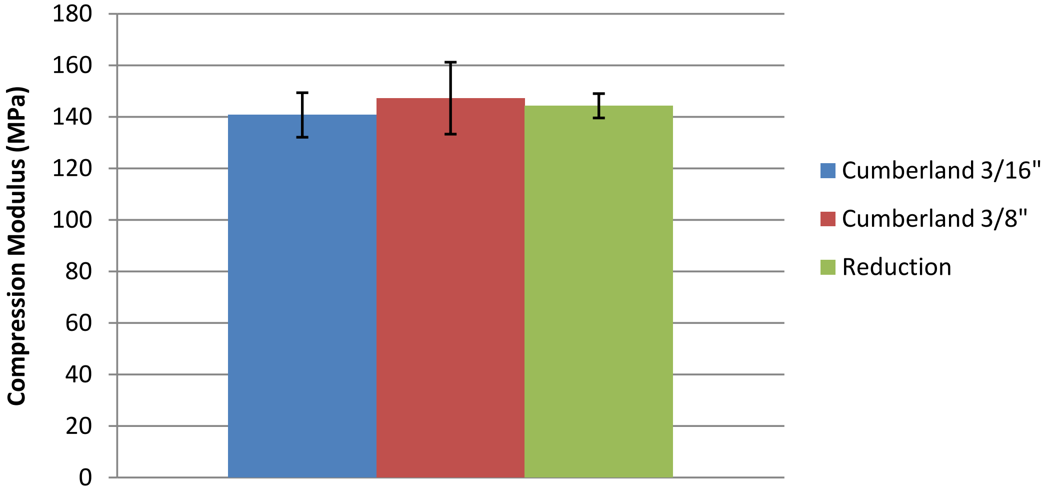

Figure 38. Compression modulus of 2202 with 15% regrind filler.

Tables 13-15 list the mechanical properties that are graphically displayed in the previous charts. The results may not show that a particular regrind is more suitable then the next, but it can be seen that consistent results were produced and the sensitivity to regrind is minimal.

Table 13. Tensile Results of Preliminary Full Scale Testing 2202 with 15% Regrind Filler

	Tensile Testing	
	Modulus (MPa)	**Strength (MPa)**
Cumberland 3/16"	616.2±30.6	10.1±0.6
Cumberland 3/8"	598.1±19.8	10.8±0.2
Reduction	596.9±17.6	9.9±0.6

Table 14. Flexural Results of Preliminary Full Scale Testing 2202 with 15% Regrind Filler

	Flexural Testing	
	Modulus (MPa)	**Strength (MPa)**
Cumberland 3/16"	1629.2±44.3	20.0±0.7
Cumberland 3/8"	1608.5±41.1	20.6±0.5
Reduction	1563.7±42.6	17.6±0.8

Table 15. Compression Results of Preliminary Full Scale Testing 2202 with 15% Regrind Filler

	Compression Testing	
	Modulus (MPa)	**Strength (MPa)**
Cumberland 3/16"	140.7±8.6	3.10±0.3
Cumberland 3/8"	147.2±14.0	3.5±0.1
Reduction	144.3±4.7	3.4±0.1

Figures 39–44 illustrate the strength and modulus of the three regrind panels processed in the woven series. From the test results there again is not a regrind that statically outperforms the other in all categories. However, it was noted that the Cumberland 3/8” regrind processed

the best with the least amount of dry spots and fines in the woven as it did with the nonwoven series. The Cumberland 3/16” showed a slight advantage over the 3/8” and Reduction samples, but the advantage does not appear to be statically great enough to warrant pursuing the 3/16” or Reduction pulverizer. The tensile strength and modulus of the panels are extremely close with the standard deviations overlapping.

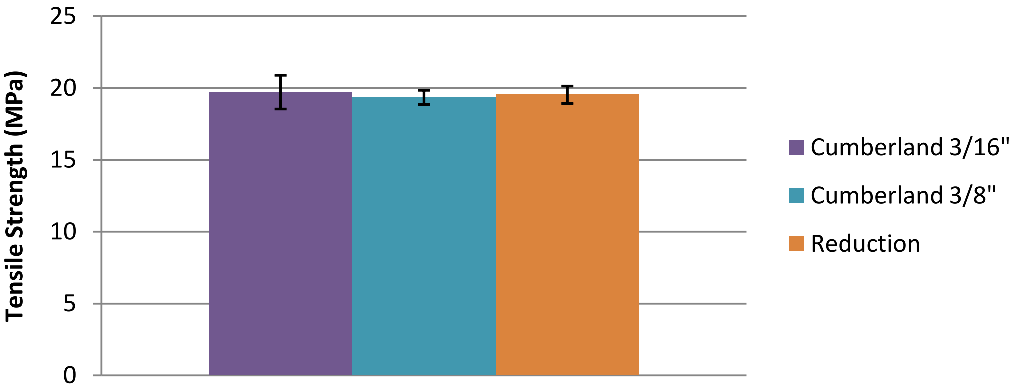

Figure 39. Tensile strength of 2852 with 15% regrind filler.

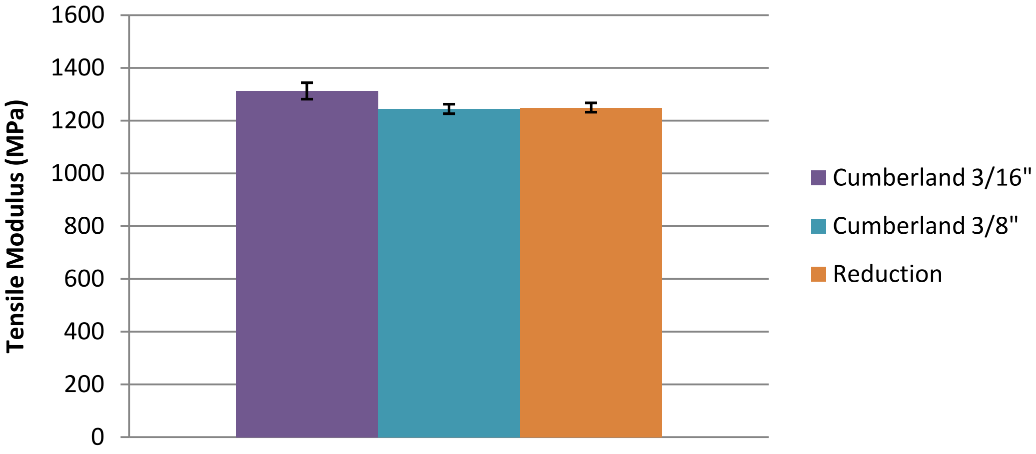

Figure 40. Tensile modulus of 2852 with 15% regrind filler.

In figures 41 and 42 the flexural modulus and strength are both higher with the finer screen sized Cumberland regrind. The modulus of the specimens were much closer then the strength values. The flexural modulus of the Cumberland was 22% higher than the Reduction pulverizer regrind.

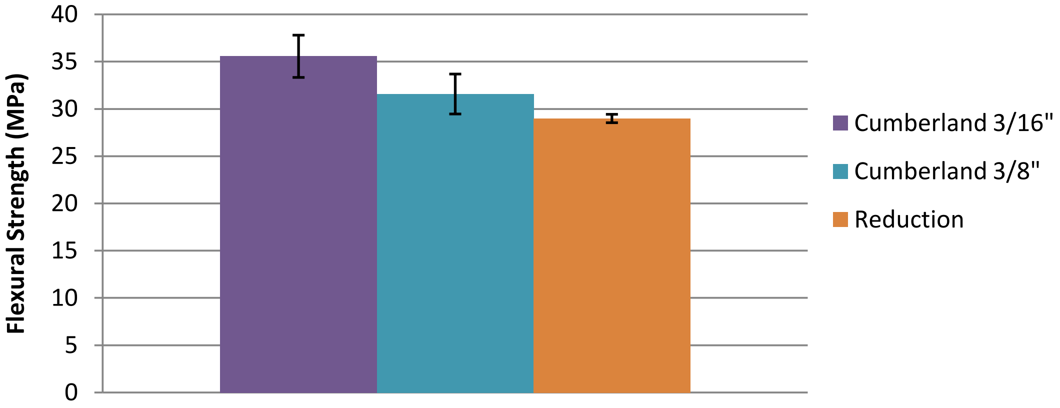

Figure 41. Flexural strength of 2852 with 15% regrind filler.

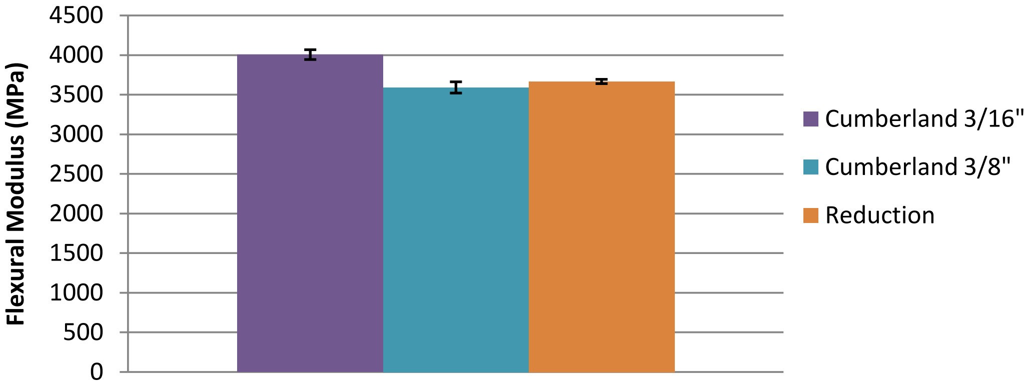

Figure 42. Flexural modulus of 2852 with 15% regrind filler.

As expected in figures 43 and 44 the compression category had very little difference. Neither the modulus or strength indicated a specific regrind that was significantly improved, however the consistency of the results seemed to be closer with Reduction regrind.

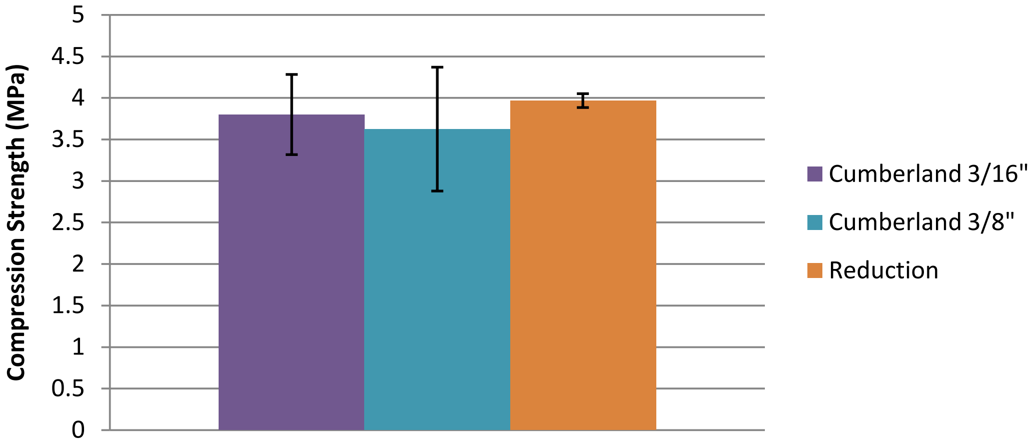

Figure 43. Compression strength of 2852 with 15% regrind filler.

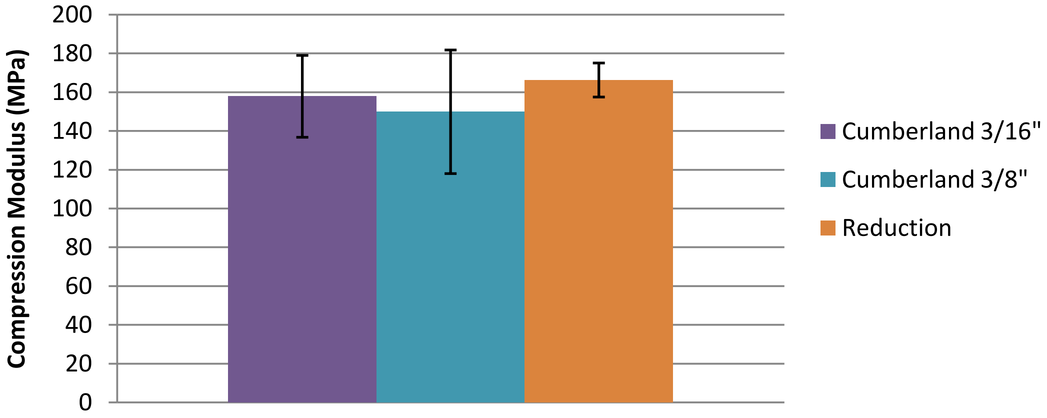

Figure 44. Compression modulus of 2852 with 15% regrind filler.

Tables 16-18 list the mechanical properties that are graphically displayed in the previous charts. Again no distinct conclusion based on properties could be achieved. Similar to the nonwoven series boards there was a great deal of consistency and very little difference between regrinds.

Table 16. Tensile Results of Preliminary Full Scale Testing 2852 with 15% Regrind Filler

	Tensile Testing	
	Modulus (MPa)	**Strength (MPa)**
Cumberland 3/16"	1311.9±31.2	19.7±1.2
Cumberland 3/8"	1243.6±18.0	19.3±0.5
Reduction	1249.0±17.6	19.5±0.6

Table 17. Flexural Results of Preliminary Full Scale Testing 2852 with 15% Regrind Filler

	Flexural Testing	
	Modulus (MPa)	**Strength (MPa)**
Cumberland 3/16"	4003.5±62.0	35.6±2.2
Cumberland 3/8"	3589.4±70.9	31.6±2.1
Reduction	3664.6±27.1	29.0±0.4

Table 18. Compression Results of Preliminary Full Scale Testing 2852 with 15% Regrind Filler

	Compression Testing	
	Modulus (MPa)	**Strength (MPa)**
Cumberland 3/16"	157.9±21.1	3.8±0.5
Cumberland 3/8"	149.9±31.9	3.6±0.7
Reduction	166.3±8.8	4.0±0.1

The test results of the preliminary full scale processing illustrated that mechanical properties were almost identical for the different equipment styles tested. Therefore, it was determined that panel processability using the regrind was the most important factor. It was deduced that the Cumberland 3/8" screen granulator provided the most effective means of reducing regrind that created quality material finish on production equipment. The remainder of the full scale processing only uses this regrind.

5.3.4. Full Scale Processing Production Qualification

Utilizing the regrind produced from the Cumberland granulator and a 3/8" screen, panels were produced ranging in regrind percentage, density, thickness, and reinforcement. Tables 19–24 contain the test results for tensile, flexural, and compression testing along with a comparison to unfilled specimens. The tables list the regrind boards using a material code of the form xxxx–Rxx, where the first four digits denote the board series, the R denotes regrind, and the final two digits denoting the percentage of regrind. The panels tested represent the most common thicknesses produced at SAS in the high, low, and mid range of densities achievable using production equipment to fully understand the regrind affects on the product line. The biggest discrepancy was seen in the out of plane tensile modulus. The drastic increase of 158% could be attributed to the new open weave glass that was sourced for the regrind. This would also help explain the increase in flexural performance, as the failure mode is similar. The point of failure is along the fabric interface line for both out of plane tensile and flexural. With the open weave, it is possible to have better wet-out and continuity of matrix through the interface. These factors could easily improve the performance of these properties, but would need further examination to fully understand the affects of the open weave fabric.

Tensile test results presented in table 19 show trend of a decrease in strength and modulus on most panels. The 0 series does not have woven fiber glass and a substantial amount of the load is carried by the polyurethane foam. The slight drop in properties is acceptable given the little use of the composite panels in tensile loading situation. Tensile properties are a create screening tool, but for this product the tensile results are imperative to retain full strength.

Table 19. Tensile Test Results for Final Processing of 0 Series Panels

Thickness	Regrind Board	Tensile Testing		Standard Thermo-Lite Board	
		Modulus (MPa)	Strength (MPa)	Strength (MPa)	Modulus (MPa)
25.4mm	1802-R10	491.9±16.9	9.2±0.7	13.7	744.8
	1802-R15	537.6±33.0	9.1±0.7	13.7	744.8
	2602-R10	928.2±30.3	14.9±0.3	15.4	855.2
	3402-R10	880.8±30.0	14.1±0.2	17.1	965.5
	3402-R15	899.5±17.9	14.2±0.5	17.1	965.5
19mm	1802-R10	568.7±54.4	9.4±0.4	12.6	657.2
	2602-R10	721.6±50.4	12.4±0.5	14.3	765.5
	3402-R10	886.8±47.1	14.6±0.5	16.1	875.9
12.7mm	2002-R15	518.5±35.3	8.3±0.6	11.9	595.2
	2002-R10	524.5±29.0	8.1±0.5	11.9	595.2
	2602-R10	594.6±31.9	9.9±0.3	13.2	677.2
	3402-R15	746.4±11.3	12.8±0.6	15.0	786.2
	3402-R10	792.1±22.0	13.5±0.2	15.0	786.2

Flexural results shown below in table 20 represent the strength and modulus of the 0 series panels. The flexural results are arguably the most important characteristics of the Thermo-Lite as it is the most common loading scenario. The filled systems showed little affect on the strength or modulus. The 0 series panels when used in industry rarely reach maximum flexural strength due to the large deflection required for failure. Since the filler did not alter the modulus significantly the "feel" of the panel would not be changed in service.

Table 20. Flexural Test Results for Final Processing of 0 Series Panels

Thickness	Regrind Board	Flexural Testing		Standard Thermo-Lite Board	
		Modulus (MPa)	Strength (MPa)	Strength (MPa)	Modulus (MPa)
25.4mm	1802-R10	1505.5±57.7	15.9±1.0	16.5	1241.4
	1802-R15	1554.4±25.5	15.5±1.5	16.5	1241.4
	2602-R10	1859.6±14.8	25.2±0.3	21.3	1441.4
	3402-R10	2009.4±28.0	29.0±1.4	26.2	1641.4
	3402-R15	2020.9±24.9	29.3±0.5	26.2	1641.4
19mm	1802-R10	1250.1±40.0	16.5±0.3	17.0	1310.3
	2602-R10	1596.2±34.3	23.8±0.8	21.9	1517.2
	3402-R10	1877.9±28.2	28.2±1.1	26.7	1717.2
12.7mm	2002-R15	1505.5±57.7	15.9±1.0	18.8	1434.5
	2002-R10	1554.4±25.5	15.5±1.5	18.8	1434.5
	2602-R10	1859.6±14.8	25.2±0.3	22.3	1586.2
	3402-R15	2009.4±28.0	29.0±1.4	27.2	1793.1
	3402-R10	2020.9±24.9	29.3±0.5	27.2	1793.1

Compression results shown in table 21 do not have any glaring differences between filled and neat boards. Since the filler is of the same material it appears little to no increase in compression modulus is seen and seems to have a similar response to a compressive load.

Table 21. Compression Test Results for Final Processing of 0 Series Panels

Thickness	Regrind Board	Compression Testing		Standard Thermo-Lite Board	
		Modulus (MPa)	Strength (MPa)	Strength (MPa)	Modulus (MPa)
25.4mm	1802-R10	112.5±8.0	2.6±0.1	2.7	113.1
	1802-R15	106.5±11.7	2.3±0.2	2.7	113.1
	2602-R10	236.1±28.7	5.6±0.4	5.1	203.4
	3402-R10	318.4±22.8	8.0±0.1	7.4	294.5
	3402-R15	318.0±23.5	7.7±0.4	7.4	294.5
19mm	1802-R10	101.4±8.0	2.0±0.1	2.7	113.1
	2602-R10	193.8±7.7	4.7±0.2	5.1	203.4
	3402-R10	276.6±26.4	6.6±0.1	7.4	294.5
12.7mm	2002-R15	89.6±10.1	2.2±0.3	3.3	135.9
	2002-R10	102.7±7.0	2.5±0.1	3.3	135.9
	2602-R10	164.6±8.7	4.1±0.2	5.1	203.4
	3402-R15	216.7±7.3	5.2±0.6	7.4	294.5
	3402-R10	455.0±26.9	8.4±0.4	7.4	294.5

The tensile results shown in table 22 are from the 5 series panels consisting of a higher glass loading with woven fiberglass. The majority of the load is carried by the fiberglass and the ill effects of the regrind appear to be drowned out in the strength and modulus. As long as good wet-out is achieved it appears the filler has little effect on the ultimate strength or modulus.

Table 22. Tensile Test Results for Final Processing of 5 Series Panels

Thickness	Regrind Board	Tensile Testing		Standard Thermo-Lite Board	
		Modulus (MPa)	Strength (MPa)	Strength (MPa)	Modulus (MPa)
25.4mm	2252-R10	1188.0±41.9	20.7±1.3	23.5	1275.9
	2252-R15	1085.2±53.0	17.2±1.6	23.5	1275.9
	2652-R10	1689.3±65.5	28.4±0.8	24.5	1351.7
	3252-R10	1193.6±87.0	19.1±1.1	25.9	1462.1
	3252-R15	1481.1±49.2	23.8±1.8	25.9	1462.1
19mm	2452-R10	1546.9±54.0	27.0±1.1	30.2	1503.4
	3452-R10	1689.3±65.5	28.4±0.8	32.6	1689.7
12.7mm	2852-R10	5498.8±76.5	41.3±1.6	37.4	1765.5
	2852-R15	2181.1±72.8	40.4±0.8	37.4	1765.5
	3252-R10	2144.4±48.9	37.7±3.4	38.3	1841.4
	3652-R10	2408.8±83.7	44.1±3.1	39.2	1917.2
	3652-R15	5579.4±175.9	41.6±1.2	39.2	1917.2

Table 23 portrays the flexural strength and modulus of the 5 series panels. It shows an increase in strength and modulus was achieved in most filled systems. This could be attributed to the different open weave fiber glass used to improve wet-out. The woven has better fiber alignment and a better interaction at the plane of woven fiberglass increase the ultimate strength and modulus.

Table 23. Flexural Test Results for Final Processing of 5 Series Panels

Thickness	Regrind Board	Flexural Testing		Standard Thermo-Lite Board	
		Modulus (MPa)	Strength (MPa)	Strength (MPa)	Modulus (MPa)
25.4mm	2252-R10	3333.9±102.4	27.9±1.8	26.1	2448.3
	2252-R15	3310.0±106.4	29.2±2.3	26.1	2448.3
	3252-R10	3999.3±149.3	45.9±4.0	27.2	2503.4
	3252-R15	3824.3±75.9	47.7±2.6	27.2	2503.4
19mm	2452-R10	3141.0±80.3	35.5±1.5	29.4	2793.1
	3452-R10	3598.6±82.8	52.1±2.6	30.6	2841.4
12.7mm	2852-R10	4426.4±142.9	45.4±3.6	33.0	3144.8
	2852-R15	4619.5±85.6	46.2±1.0	33.0	3144.8
	3252-R10	4650.5±133.8	55.9±2.2	33.4	3165.5
	3652-R10	4629.5±100.2	68.0±2.0	33.9	3186.2
	3652-R15	4694.5±189.7	63.6±4.2	33.9	3186.2

The increase in compression strength and modulus is also attributed to the open weave fiberglass used. This again provided better through-the-thickness foam growth, strengthening the panels in the out of plane compression. Cost of the open weave fiberglass has prohibited it from being used in current production, but with offsetting the cost of resin a superior product can be produced with a filler.

Table 24. Compression Test Results for Final Processing of 5 Series Panels

Thickness	Regrind Board	Compression Testing		Standard Thermo-Lite Board	
		Modulus (MPa)	Strength (MPa)	Strength (MPa)	Modulus (MPa)
25.4mm	2252-R10	109.0±6.1	3.4±0.3	2.4	104.1
	2252-R15	261.6±5.8	4.7±0.0	2.4	104.1
	2652-R10	198.9±11.1	6.3±0.4	3.6	149.7
	3252-R10	271.0±7.3	9.8±0.2	5.4	217.9
	3252-R15	292.4±7.7	10.4±0.1	5.4	217.9
19mm	2452-R10	142.1±4.6	4.7±0.0	2.7	115.9
	3452-R10	266.9±7.0	9.8±0.2	5.7	229.0
12.7mm	2852-R15	145.0±11.3	5.2±0.2	3.3	138.6
	3252-R10	182.7±11.1	7.2±0.2	4.5	183.4
	3652-R10	234.9±18.6	9.8±0.4	5.7	229.0
	3652-R15	222.1±27.0	9.1±0.9	5.7	229.0

In looking at the results of the final processing it becomes a lot of data to absorb and can be broken down to averages. Table 25 summarizes the previous 24 panels with percentages changes in properties for the panels. Tensile properties of the regrind boards endured a minor drop in strength and modulus for both woven and nonwoven series for all thicknesses and densities. This is to be expected as it was explained early that tensile loading can heighten the affects of fillers. Flexural properties seen a similar change but to the better for the nonwoven series and a drastic increase for woven series. A 53% increase in strength was witnessed for woven series boards. This change is most likely not from the regrind but the different woven fabric used. Further investigation is being done to determine if the alteration in glass was the driving factor for this increase and why. As for the compression strength and modulus of the regrind boards very little change was noticed. Compression results stayed very close to the standard Thermo-Lite product results, which makes sense the a similar material is being compressed. Many composites using fillers increase in compression properties due to the different and often harder material being the filler. In the regrind case, the material constinuents are the same and only changing the continuity.

Table 25. Percentage Summary of 24 Full Scale Panels

	Tensile Strength	Flexural Strength	Compression Strength
Woven 10%	-6.6%	53.7%	74.3%
Woven 15%	-3.4%	53.2%	66.6%
NonWoven 10%	-18.8%	4.4%	-6.5%
NonWoven 15%	-24.0%	1.2%	-16%

In addition to the broad test matrix for tensile, flexure, and compression, one 25.4mm thick 2652 series panel was tested for additional information on edgewise compression, interlaminar shear, and out-of-plane tensile. These mechanical properties were then compared to those of a similar standard production panel. The additional tests were simply performed to ensure that the regrind does not drastically affect secondary mechanical properties not tested for in the main production panel test matrix.

Table 25 lists the results of the additional mechanical tests as well as the properties of standard Thermo-Lite. It can be seen that the results from these additional tests fall into the same range as those of the standard product. Thus concerns of secondary property degradation with the addition of regrind were alleviated.

Table 26. Full Set of Test Results for 2652 Series Panel

	Regrind Board 10%		Standard Thermo-Lite	
25.4mm 2652	Modulus (MPa)	Strength (MPa)	Modulus (MPa)	Strength (MPa)
In Plane Tensile	1241.4±40.0	21.4±1.4	1363.4	25.5
Out of Plane Tensile	185.5±15.9	3.6±0.6	71.7	3.6
Flexural	3329.7±222.8	33.1±3.1	2836.6	31.4
In Plane Compression	204.1±13.8	4.8±0.3	162.1	4.0
Edgewise Compression	677.9±78.6	10.4±2.9	720.7	12.9
Interlaminar Shear	53.8±4.3	4.0±0.1	46.2	4.0

CHAPTER 6. CONCLUSIONS AND FUTURE RECOMMENDATIONS

Incorporating fillers into a polymer system can be done to reduce cost, alter appearance, alter mechanical and electrical properties, and/or meet end use design requirements. The processing and characterization of the filled systems was the main objective of this research. Cost reduction and environmental effects are attractive reasons to why this research is important. The industrial application of this thesis has great potential with the millions of tons of PU and PVC consumed in North America alone.

Introducing natural fillers into PVC have been done for many years with great success with wood fibers. PVC has a low melt temperature reducing the amount of degradation occurring during common compounding and processing. Reducing the degradation during processing helps reduce the amount of offgasing from the natural filler lowering the chance for voids and surface blemishes. Flexible PVCs are commonly used as seat covers, electrical cords covers, and packaging components. The flexible PVC chosen for this research would be an efficient packaging material to protect a component by damping vibrations and absorbing impacts.

Utilizing ground corn cob as a filler for a flexible PVC has potential for the proper application. With little effect on ultimate strength, damping coefficient, and Tg without chemically treating the filler. The chemical treatments to the ground corn cob showed no advantages with the flexible PVC and are not worth the additional cost associated with treatments.

Processing the filled flexible PVC required little change from the neat resin. In the extruding process it was actually easier with the increase in melt viscosity allowing the extradite to hold together better. During the injection molding process there also appeared to be no issues filling the specimen geometry.

Utilizing ground corn cob as a filler for a PU foam composite panel showed significant decrease in mechanical performance. The PU foam composites panels are intended to be a low density semi structural panel, which makes a low density natural filler attractive. However when introducing the ground corn cob, resin is quickly absorbed into the filler altering the polymer reaction. The absorption of resin into the filler not only negatively affects the polymer reaction by absorbing liquid the fillers are no longer a low density filler, but a soft and dense filler.

The production of PU foam composite panels inherently produces scrap from post processing panels. This scrap is commonly discarded to a landfill which cost the producers and negatively affects the environment. Utilizing the scrap as regrind is a growing trend in the composite industry. The regrind is essentially a filler, reducing resin consumption and disposal costs. The acceptable percentage of regrind was determined by processability and mechanical performance.

It was determined that a shredder and granulator were necessary to reduce scrap material to the optimal size and consistency. The two pieces of equipment enable the reduction trimmings, bleedout, and full panels for the reintroduction into new panels. Much of the screening of regrind particle size and concentration were performed on lab scale production and select options were trialed in full scale screening with production panels.

After considering the results from the production panel tests and the expectations of mechanical property retention with the addition of regrind, it was concluded that the addition of 10 to 15% regrind by resin weight is acceptable. Including this percentage of recycled material does not cause a statistically significant reduction in the most important mechanical properties of compression and flexure, and causes acceptably small reductions in secondary mechanical properties. Since 10 to 15% regrind can be introduced while maintaining density and performance, 10 to 15% of the cost of the polyurethane foam could be replaced with the minimal cost of the regrind. Polyurethane regrind would make a great filler for a low density semi structural polyurethane composite panel.

Future recommendations for filling flexible PVC with ground corn cob include alternative fiber treatments and application specific goals. Alternative fiber treatments could possibly increase the strain to failure of the filled system to increase the toughness of the flexible PVC. Flexible PVC components need very specific material requirements of toughness, damping coefficient, modulus, electrical conductivity, and processing techniques. The introduction of a filler into a flexible PVC could affect all aspects of required of the polymer making it very difficult to do a direct swap utilizing the same base flexible PVC for a component and should be considered a new system.

Filling PU foam composite panels can be achieved in many ways. Future recommendations to focus on regrind of polyurethane scraps and not ground corn cob. With the ground corn cob comes many issues during processing with the inherent nature of the filler to absorb resin. However the introduction of regrind eliminates the fear of resin uptake. Further understanding of the affects of regrind on the performance of the PU foam composite panel in service of application before introducing into full production should be performed. Also, investigating high filler percentages without targeting comparable composite performance as a neat panel. A highly filled composite panel could be targeted at nonstructural applications using polyurethane more for the insulative properties.

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