

PROCESSING CHARACTERISTICS AND PROPERTIES OF GLASS FIBER
REINFORCED COMPOSITES FROM POST CONSUMER CARPETS

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PROCESSING CHARACTERISTICS AND PROPERTIES OF GLASS FIBER
REINFORCED COMPOSITES FROM POST CONSUMER CARPETS

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SUMMARY

Currently a large volume of post consumer carpet is discarded and landfilled every year. The amount is around 2.3 million tons in 2002 and is estimated to be about 3.5 million tons in 2012. Efforts made to recycle the post consumer carpets so far have diverted little carpet from landfills due to economic reasons. It is crucially important to convert post consumer carpet into products that are low cost and have large volume demand. The major components in carpets are nylon 6, nylon 66, and polypropylene. These plastic materials may provide a low-cost alternative in the manufacture of glass fiber reinforced thermoplastics, which can be used to make pallets, highway guardrails and other products. The focus of this thesis is to develop approaches for the manufacturing of low cost glass fiber reinforced composites from post consumer carpet.

In this research, two processes are used to make glass mat thermoplastic (GMT) sheets from post consumer carpets (PCC) by compression molding: (1) webs of shredded carpet fibers are debulked and then compression molded with glass mats, and (2) shredded carpet fibers are melt processed into plastic pellets and then compression molded with glass mats. Two types of PCC are used, one with nylon 6 face fibers and the other with polypropylene face fibers. The shredded PCC are analyzed, including composition and degradation analysis by TGA and DSC tests, and melt flow characteristics of the pellets by the melt flow rate (MFR) test. The composites fabricated are tested for their physical and mechanical properties such as flexural properties and impact resistance. The results of the tests are compared with those of commercial GMT products and those of composites from glass-filled

extruded PCC boards. The influence of processing conditions, compositions and fiber length on the properties of those PCC composites is studied.

CHAPTER 1

INTRODUCTION

The first goal of this research is to develop the process of making low cost glass mat reinforced thermoplastic (GMT) compounds from PCC. Generally when high product standards for a recycled plastic product are specified, a separation process with high selectivity is required. It not only causes technical difficulties in processing and it also increases the cost of recycling. If the steps of recycling process are reduced and the property problems due to impurities are avoided, it could make the process a more cost-effective and attractive business option. Fiber acts as the load carrier in fiber-reinforced composites. The improvement of mechanical properties of the matrix will not proportionally improve the performance of the whole composite. Zhang et al[1] have investigated the effect of impurities brought from recycling carpet in the matrix on the mechanical properties of the composite. The long glass fibers in glass mat thermoplastics are more important than the purity of matrix based on results from tensile tests and impact tests. In addition, like most of fiber-reinforced composites, the presence of long glass fiber dominates the performance of the composites, and thus the GMT composite with recycling polypropylene was expected to have similar mechanical properties as glass fiber-reinforced virgin polypropylene. This has been confirmed by the results of Zhang et al[2].

The second goal of the research is to compare flexural properties and impact resistance of GMT from post consume carpet (PCC) with glass fiber reinforced compounds made by extrusion compounding of long glass fibers 12.7 mm ~ 25.4 mm with PCC pellets. The glass fiber concentration and process conditions as well as their influence on the properties of the composites are investigated.

CHAPTER 2

LITERATURE REVIEW

2.1 Carpet Construction

Generally, carpet consists of face yarn, primary backing fabric, bonding component and secondary backing fabric.

(1) Face yarn

Most carpet manufactured in U.S.A. is made of one of six pile fibers: nylon, polypropylene, acrylic, polyester, wool or cotton[3]. The major fibers for commercial carpet are nylon, polypropylene (PP) and wool. Nylons (synthetic polyamide), commonly nylon 6 or nylon 66, are extensively used for carpet and account for 60 percent of all carpet face fibers. About 33 percent of all carpet face fiber is polypropylene. Pile yarn or fibers represent up to 85 percent of the total material cost of the carpet, making this the primary cost factor.

(2) Backing system

The primary backing fabric, the applied bonding adhesive and the secondary backing comprise the backing system. The primary backing and secondary backing are typically made of woven or non-woven polypropylene fabrics. The yarns or fibers are secured into the primary backing by applying bonding adhesive, synthetic latex or vinyl. The secondary backing is usually attached to provide stability to the carpet structure. Styrene butadiene latex rubber (SBR) mixed with CaCO_3 filler is the most frequently used backing compound in tufted carpet. The typical broadloom carpet contains approximately 10% polypropylene as backing material, 9% SBR adhesive, and about 35% CaCO_3 as filler[4].

2.2 Carpet Waste Sources

Generally, carpet waste can be tracked from two sources: pre-consumer and post consumer carpet waste (PCC). Pre-consumer carpet waste comes from carpet manufacturing in the form of trimmed edges and shear lint. The irregular shape cut-off carpet from carpet fitting process in automotive industries is another source of pre-consumer carpet waste. PCC waste usually refers to either commercial carpets or residential carpets removed from homes or business sites.

For the PCC, in addition to the multi-component construction mentioned above, the dyes and coatings as well as other contaminants accumulated during the use, such as dirt, food scraps and all kinds of fragment, should be considered.

Both nylon 6 and nylon 66 are common polymers used as fibers in the form of thermoplastic. SBR and CaCO_3 are not valuable materials. The attempts to recycle PCC have been closely related to recovering nylon 6 and nylon 66 from PCC.

2.3 Carpet Waste Recycling

According to the data collected by U.S. EPA (U.S. Environmental Protection Agency) approximately 2.3 million tons of carpet was disposed in the United States in 2002. But only 1.22 percent of total discarded carpet was either recycled or reused and less amount of carpet was incinerated; the rest went into municipal solid waste (MSW) landfills[5]. The discarded carpet accounts for over 1 percent of all MSW by weight, or about 2 percent by volume. Landfilling is by far the most common disposal method for MSW. Land is one of the most valuable resources for sustainable human development. Waste disposal by using landfills has been thought an increasingly severe threat to the environment preservation for future generations. Growing concerns about MSW disposal capacity have been raised during the last decade. In order to reduce consumption of land by discarded carpets and divert them from the

landfills, increasing efforts have been made by various groups to promote reclamation and recycling of discarded carpet waste.

In January 2002, carpet and fiber manufacturers signed the National Carpet Recycling Agreement, Memorandum of Understanding for Carpet Stewardship (MOU), along with several state governments and non-governmental organizations. This agreement established national goals for the years 2002-2012 to increase the amount of reuse and recycling of post consumer carpet and reduce the amount of waste carpet going to landfills. Comparing with the achievement of 2002, it is still a long way to reach the goal of 20 to 25 percent recycling rate with 40% landfill diversion by 2012. The Carpet America Recovery Effort (CARE) was created as a third-party organization to be responsible for tracking the progress.

In December 1990, European carpet manufacturers joined in Germany to form GuT, an environmental association of the carpet industry aiming to enhance user and environmental friendliness through the entire life cycle of carpet installation. The organization with the European carpet industry established the company Carpet Recycling Europe (CRE) and created a 3-year project Recycling of Carpet Materials (RECAM) in 1996. RECAM concluded that around 1.6 million tons of carpet waste was sent to landfills or incinerators in west Europe every year[6].

2.4 Technologies for Post Consumer Carpet (PCC) Recycling

2.4.1 Collection, Sorting and Separation of PCC

For the purpose of diverting the PCC from the landfills, in general, collecting and sorting are necessary before recycling the components of carpet into useful products. Currently PCC is collected from the major retail stores and then transported to the sorting places, where the collected PCC goes through either manual sorting or automatic sorting depending on the requirement for selectivity of the recycling

products. Collecting and sorting is a capital- and labor- intensive process and involves addressing related logistical issues. After being sorted, PCC is subjected to further separation of their multi-components. Usually the sorted carpets are shredded into strips that are dismantled to form a mixture of fibers and backing materials. Then a substantial portion of the fibers is separated from the backing materials by using a separation system. For instance, the carpet shreds are transported over a vibrating screen to sift out additional backing and latex, and then through a rotating granulator to chop the fiber material into very fine pieces. An air separation system removes additional contaminants (backing, latex, dust) by the density differentiating principle. CENSORTM centrifuge technology is also an example of gravity separation system[7]. It uses separation liquid and centrifugation to separate the components with different density.

It is a relatively complex process to extract the useful components from PCC and get them ready for further recycling. Recently the decrease in tipping fees in some states has challenged the effort for diverting the PCC from landfills through recycling. It is of critical importance to develop cost-effective strategies and technologies for collecting, sorting and separation. Meanwhile, exploring new technologies and potential market for recycled products from waste carpets is a driving force to promote the recycling business. A number of approaches have been tried and developed to recover valuable raw materials from PCC.

2.4.2 Recycling Carpet Face Fibers

This option is most commonly used with nylon 6 that has more value than polypropylene, polyester, wool or blends. Caprolactam can be chemically separated from recycled carpet face fibers and reused to produce plastics products, including new carpet face fiber.

2.4.2.1 Recovering Nylon from PCC

Griffith et al[8] has presented a new process for separating and recovering nylon from PCC. The process consists of three stages: (1) Common carpet with 50wt% nylon was dissolved in 88wt% formic acid. After filtering polypropylene, SBR latex and small amount of acid and water, a solution with 2.31wt% nylon concentration was obtained. (2) By using super-critical fluid (SCF) CO₂ as anti-solvent, the nylon precipitated out of the solution at pressures between 84 and 125 bar at 40°C and was collected. (3) Formic acid and CO₂ anti-solvent was separated by adjusting the temperature and pressure and was recycled.

The nylon recovered was spherical in shape and range in size from less than 1 to 20µm. By using environmentally friendly CO₂ as super-critical fluid, the process solved the problem of separating formic acid and water rather than simply diluting formic acid solution by water to precipitate nylon. The process also shows the convenience to separate the formic acid from CO₂ anti-solvent. The feedstock that only contains specific nylon is required to ensure the purity of the product recycled, and the further separation of nylon 6 and nylon 66 is necessary in some cases. This may increase the cost of the recycling and weaken the commercial feasibility to recover nylon from PCC. In addition, the PCC is different from the new carpet due to the presence of multi-components contaminated in its service life. The impurities significantly complicate the recycling process. Further purification after general separation is necessary to obtain relatively pure crude material from PCC.

2.4.2.2 Recovering Caprolactam by Chemical Depolymerization

The technology of recycling nylon 6 fiber to caprolactam has been known since the 60's. It is reasonable and profitable to develop the approaches of recycling nylon carpet to caprolactam. One way to achieve the purpose is to recover the nylon 6

containing material, like what Griffith et al[8] did, or separate nylon 6 fiber from PCC as the first stage and then depolymerize nylon 6 fibers into caprolactam; the other attractive approach is to start with the PCC without separation to obtain caprolactam. Cobin et al[9] presented the process to reclaim caprolactam from nylon 6 carpets. The PCC scraps containing nylon 6 fiber materials and backing materials from separators were fed into a depolymerizing reactor in the presence of phosphoric acid as catalyst and superheated steam (230°C to 350°C) to produce the volatile caprolactam monomer and other waste. The pure caprolactam suitable for reuse for nylon fiber then would be obtained by distilling. This process avoids the cost of separation but it involved superheated steam and repeating distillation that call for energy supply and in turn the recycling cost; the acid catalyst could stay in the recovered caprolactam as impurity and also invite the negative impact on environment when the process is commercialized.

Braun et al[4] developed a laboratory scale approach to depolymerize nylon into caprolactam by hydrolysis. The chipped nylon 6 carpet extrudate was charged into the stainless steel reactor in the presence of nitrogen and was heated up to approximately 180°C under a constant pressure of 310-345 kPa (45-50 psi). After the system reached 180°C the pressure was increased to a desired level. Samples were collected with 2-6 g/min steam at 0-1,379 kPa (0-200 psi), and 300-340°C. In the best run of total eight experiments, 95% yield of crude caprolactam was obtained and its purity was 94.4% and resulted in an overall 89.7% yield of caprolactam. It is different from what was done by Cobin et al[9] by using higher pressure instead of acid catalyst and it saved the step of segregation of face fiber from backing. But the material used in their experiments is pelletized nylon 6 pre-consumer carpet and the impurities of the PCC might cause undesired effect on the recycling product if PCC is used; secondly, the

dimers and ammonia formed in the reaction require further purification of the crude caprolactam.

2.4.2.3 Recovering Caprolactam by Thermal Depolymerization

Depolymerization of nylon with the help of steam and catalysts such as phosphoric acid leads to high yield of salts and traces of phosphoric acid in the recovered caprolactam, which compromises the quality of the fibers made. A few approaches have been investigated to produce caprolactam by thermal degradation of nylon 6 materials. Czernik et al[10] investigated the catalysis of the thermal degradation of nylon 6 with α -alumina supported KOH in a fluidized bed reactor at 330°C and 360°C with a yield of 85% caprolactam. Mukherjee and Goel[11] obtained 90.5% caprolactam with catalyst 1% NaOH under vacuum. Bockhorn et al[12] investigated the thermal depolymerization of carpet fibers containing about 70% nylon 6 and 30% polypropylene with acid catalysis (10 wt% orthophosphoric acid) and base catalysis (60 mol% NaOH and 40 mol% KOH), separately. These catalyzed pyrolysis approaches provide promise to recycle the synthetic face fibers of the PCC but the processes require thorough separating nylon 6 carpet from other carpets.

2.4.2.4 Recovering Caprolactam by Depolymerizing Nylon 6 Carpet in Twin Screw Extruder

Muzzy et al[13] studied the process design issue for depolymerizing nylon 6 carpet in a twin-screw extruder. The shredded carpet was depolymerized in a twin-screw extruder reactor with counter rotating non-intermeshing geometry in the presence of eutectic catalyst. The process was going under 150 rpm, 345°C, 13,381 kg/h (carpet feed rate) and 2,950 Horsepower. After quenching and neutralization, toluene extraction, water extraction and repeated distillation, the process yielded 98.4% pure caprolactam.

Evans et al[14] have worked on recovering caprolactam by a thermocatalytic process performed in an extruder but also tried to convert the organic residue to hydrogen in a catalytic fluid bed reformer. The hydrogen could be used in fuel cell to generate electricity. The problems left after successful proof of concept for twin-screw reactor were impurity level improvement and economics of catalyst recovery.

2.4.3 Recycling Carpet Backing

This option is similar to that for recycling vinyl backed tiles. Once separated, the tile is shredded and added to stream to make the backings.

2.4.4 Recycling Carpet Without Separation

This process directly uses the shredded PCC with additives applied in some cases to make new products or improve the property of final products. This option is most commonly a secondary recycling process. The new products are generally used for parking barriers, lumber alternatives, and automotive parts.

(1) Thermoplastic Compounds Molded With Carpet Waste

Zhang et al[1] have conducted injection molding and compression molding using carpet waste. Edge trim carpet waste consisting of nylon 66 from manufacturing, separated polypropylene from carpet waste and polypropylene shear lint from cutting tufted carpet loops were studied. The edge trim carpet waste and separated polypropylene were debulked, ground, dried and injection molded into samples for tensile tests and Izod impact tests. The mechanical properties were compared to those of typical virgin polypropylene. The results indicated that injection molding is feasible for recycling carpet waste and the properties of edge trim are acceptable for many possible applications. In compression molding process, the three types of waste materials in the form of compact sheets were interleaved with glass mat after debulking and preheated between 220°C and 260°C followed by less than 10 min at

350-1,050 kPa pressure applied. The mold was cooled under pressure to room temperature. The impact data of these compression-molded glass mat reinforced thermoplastics (GMT) from carpet waste showed that they were comparable to the GMT using virgin polypropylene. The study also concluded that inclusion of 20wt% of glass mat resulted in a significant improvement in the impact strength of shear lint samples.

Similarly, the composites made from PCC by injection molding and compression molding was investigated[15]. First, using a NGR A-Class Type 55 VSP Repelletizing system followed by converting into pellets with a Reiter pelletizer pelletized the shredded nylon 6 PCC. The pellets were then injection molded by Sumitomo SG-75 reciprocating screw injection-molding machine with 75 tons of clamp force and 70 cm³ shot capacity. The pellets also were milled to powder and interleaved with glass mat in the mold to prepare a GMT laminate by first 25-min-heating at 260°C under nominal pressure and then staged cooling (5-min 0.5 MPa, 5-min 1.0 MPa and 10-min 1.5 MPa) on a 75 ton Wabash press with cold platens. The tensile testing and flexure testing were conducted. The results presented that laminates reinforced with 30wt% glass mat had mechanical properties comparable with commercial GMT.

(2) Used as Reinforcement for Concrete and Soil

Wang[16] has investigated the utilization of recycled fibers from textile waste for concrete and soil reinforcement. The fiber-reinforced concrete (FRC) takes advantage of low-cost raw materials and opens a chance to recycle PCC without separation. This study has demonstrated that up to 2.0vol% of carpet waste fiber reinforcement can effectively improve the shatter resistance, toughness, and ductility of concrete, which is good for bettering the durability and reliability of infrastructure. And also, the tests

on the carpet waste for soil reinforcement presented the increase in triaxial compressive strength and residual strength of soil.

2.5 Proposed Work

In this research, two processes are used to make GMT sheets from PCC by compression molding: (1) webs of shredded carpet fibers are debulked and then compression molded with glass mats, and (2) shredded carpet fibers are melt processed into plastic pellets and then compression molded with glass mats.

Two types of PCC are used, one with nylon 6 face fibers and the other with polypropylene face fibers. The composition of shredded PCC is estimated and thermal degradation is investigated by TGA and DSC tests. The melt flow characteristics of the pellets are measured by the melt flow rate (MFR) test. The composites fabricated are tested for their physical and mechanical properties such as flexural properties and impact resistance. The results of the tests are compared with those of commercial GMT products and injection molded fiber-reinforced composites from PCC. The influence of processing conditions, compositions and forms of glass fiber reinforcement on the properties of GMT is studied.

CHAPTER 3

CHARACTERIZATION OF POST CONSUMER CARPET

3.1 Composition of Post Consumer Carpet

Wellman, Inc supplied two types of post consumer carpet used in this research. One is with polypropylene face fiber (PP PCC) and the other with Nylon 6 face fiber (N6 PCC). The carpet has been sorted by face fiber using a near infrared (NIR) sensor to identify the face fiber. The carpet was then shredded, packed into a bale and shipped. Most carpets manufactured in U.S.A consist of face fibers, backing materials and styrene butadiene latex rubber (SBR) mixed with filler CaCO_3 . For PCC, it may also have some contaminants such as dirt that was accumulated during the period of service. It was expected that PP PCC had major composition of polypropylene plus SBR and CaCO_3 ; and N6 PCC mainly had nylon 6 in addition to polypropylene, SBR and CaCO_3 . In order to make a rough analysis of the percentage of composition of the PCC, Differential Scanning Calorimetry (DSC) tests were conducted.

3.1.1 Separation of Shredded PCC

Single nylon 6 fiber and single polypropylene fiber were selected from shredded carpet. Polypropylene backing fabric was selected. SBR was obtained by sorting the solid drops from shredded carpet through the small mesh size sieve. CaCO_3 was expected to be with the SBR. The shredded carpet also was converted into a powder using a Wiley mill for tests.

3.1.2 Determination of Enthalpy of Fusion and Crystallinity of the Compositions of Shredded PCC

The Differential Scanning Calorimetry (DSC) technique measures a thermal event associated with heat flow rate as a function of time and temperature. It provides

quantitative information about melting and phase transitions of materials. Test method ASTM E 793 is used to determination of the enthalpy (heat) of fusion (melting) and crystallization by DSC.

In this research, DSC test was conducted to identify the enthalpy of fusion of compositions of the post consumer carpet and their crystallinity. The tests were run on TA DSC Q100 at a heating range from 0°C to 260°C and the temperature ramp was set at 10°C/min with nitrogen purged at rate of 50 ml/min.

3.1.3 Estimation of Weight Fraction of the Composition

In a sample consisting of multiple crystallized components, the heat absorbed during melting by a component can be calculated by following equation:

$$H_{ma} \times w_{tot} = H_{ma}^* \times w_{tot} \times \%w_a \times cf_a \quad (3.1)$$

where

H_{ma} = Specific enthalpy of fusion of component a in the sample, J/g

H_{ma}^* = Specific enthalpy of fusion of single component a with hypothesized

100% crystallinity, J/g

$\%w_a$ = weight fraction of component a in the sample

w_{tot} = weight of the sample, g

cf_a = crystallinity of component a

After rearrangement of equation (3.1), weight fraction of component a in the sample can be found from the following equation.

$$\%w_a = \frac{H_{ma}}{H_{ma}^* \times cf_a} \times 100\% \quad (3.2)$$

The crystallinity fraction (cf_a) of polymer from PCC can be determined from the specific enthalpy of fusion of polymer tested, and that for the polymer crystallinity:

$$cf_a = \frac{H'_{ma}}{H^*_{ma}} \times 100\% \quad (3.3)$$

where

H'_{ma} = specific enthalpy of fusion of the single component a , J/g

H^*_{ma} = specific enthalpy of fusion of pure with hypothesized 100%

Crystallinity, J/g

With substitution of equation (3.3) into (3.2), weight fraction of component a can be simplified as following equation:

$$\%w_a = \frac{H'_{ma}}{H^*_{ma}} \times 100\% \quad (3.4)$$

H_{ma} and H'_{ma} can be obtained from the results of DSC tests on the mixture of shredded carpet including component a and single component a .

Specific enthalpy of fusion of nylon 6 is 189 J/g[17], and specific enthalpy of fusion of polypropylene is 188 J/g[17].

3.1.4 Results and Discussion

(1) N6 PCC

Values of specific enthalpy of fusion of single nylon 6 face fiber and single PP backing fabric are shown in Figure 3.1 and Figure 3.2. Figure 3.3 shows that two melting peaks occurred when the mixture of shredded N6 PCC was heated and melted. Compared with the melting point temperature in Figure 1 and Figure 2, separately, Figure 3.3 indicates that the shredded N6 PCC consisted of exactly two crystallized polymer compositions, namely, nylon 6 face fiber and PP backing. The estimated weight fractions of the components of N6 PCC by equation (3.4) are listed in the Table 3.1. The results of calculated crystallinity fraction using equation (3.3) are listed in the Table 3.2.

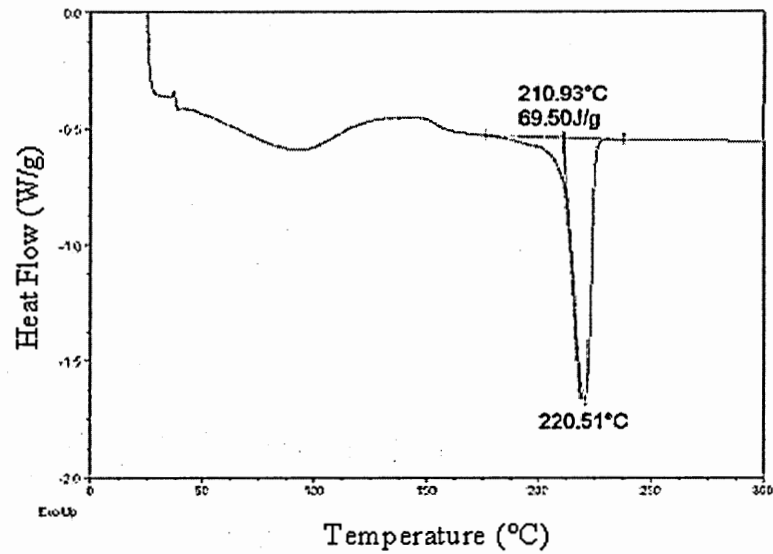


Figure 3.1 First heat cycle DSC test curve of single nylon 6 face fiber from shredded N6 PCC

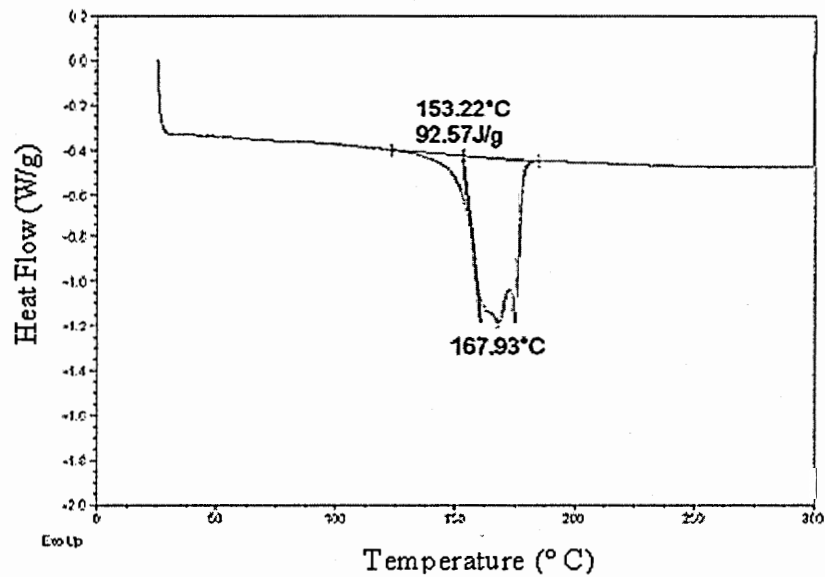


Figure 3.2 First heat cycle DSC test curve of single PP backing fabric from shredded N6 PCC

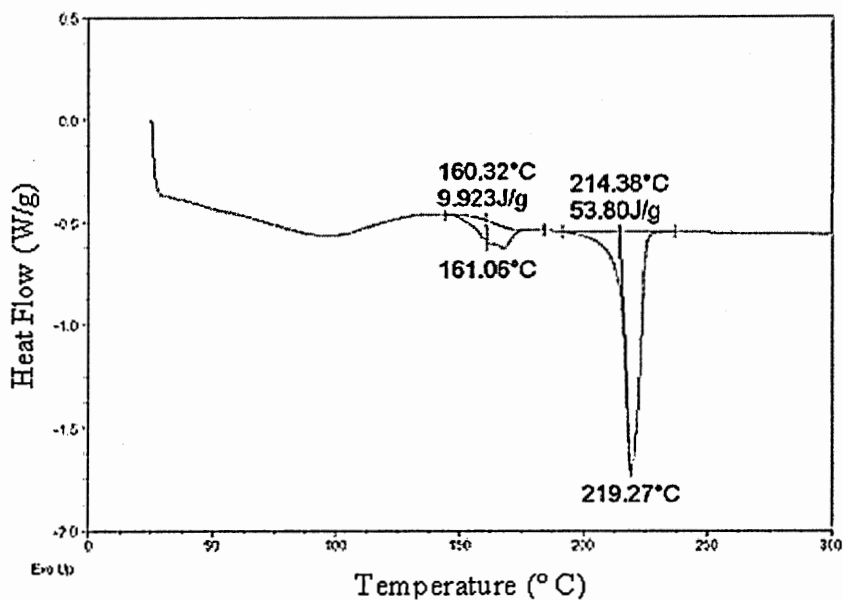


Figure 3.3 First heat cycle DSC Test curve of mixture shredded N6 PCC

Table 3.1 Estimated weight fraction and crystallinity fraction of composition of N6 PCC

Component in N6 PCC	Nylon 6 face fiber	Polypropylene backing material
H_{ma}^* (J/g)	189[17]	188[17]
H_{ma} (J/g)	69.50	92.57
H_{ma} (J/g)	53.80	9.923
Crystallinity fraction (%)	37	49
Weight fraction (%)	77	11

(2) PP PCC

In a similar way, a composition analysis was done on PP PCC. DSC tests were run on single PP face fiber and backing material as well as ground mixture of shredded PP PCC. Figure 3.4, Figure 3.5 and Figure 3.6 are the results of DSC tests. The crystallinity fraction of these two components is shown in Table 3.2. Powder sample from ground shredded PP PCC was tested on DSC and its first heat cycle is shown in Figure 3.6. There is a melting peak around 220°C. Since pure PP PCC only contains crystallized polymer materials: polypropylene face fiber and backing material, it is estimated that the melting peak was possibly caused by nylon fibers contaminated during batch processing. The crystallinity of face fibers and that of backing materials are very close. Because only one single melting peak appeared at around 167°C in the DSC curve of shredded PP PCC, the average of enthalpy of fusion of PP backing fabric and face fiber materials is taken as the general value of enthalpy of fusion of the PP materials in PP PCC. This average value is used to estimate the polypropylene weight fraction in PP PCC. Analogously, the general crystallinity fraction of PP materials in PP PCC was done by the same approach. According to possible contaminated source of nylon 6, the enthalpy of fusion of single nylon 6 fiber in N6 PCC (H'_m), 69.50 J/g was also applied to this PP PCC case. The weight fraction of the component of PP PCC calculated by equation (3.4) is listed in Table 3.2.

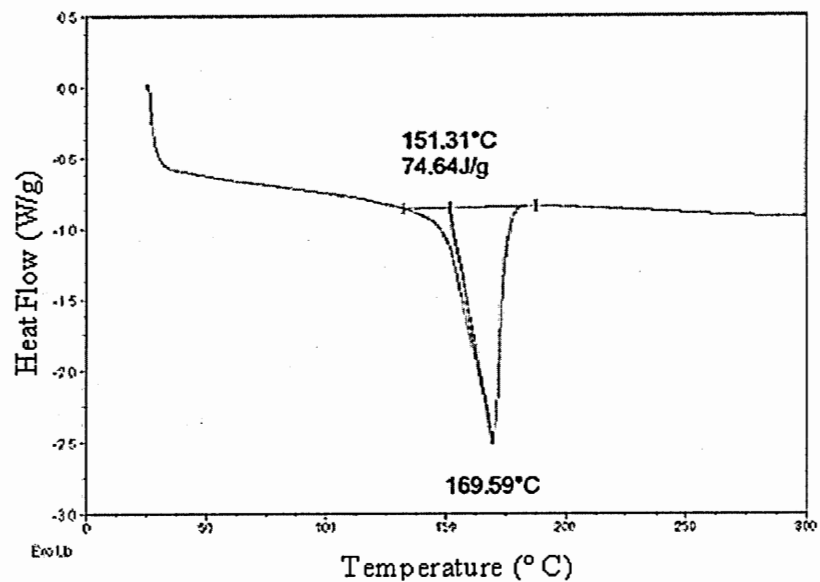


Figure 3.4 First heat cycle DSC curve of single PP face fiber from shredded PP PCC

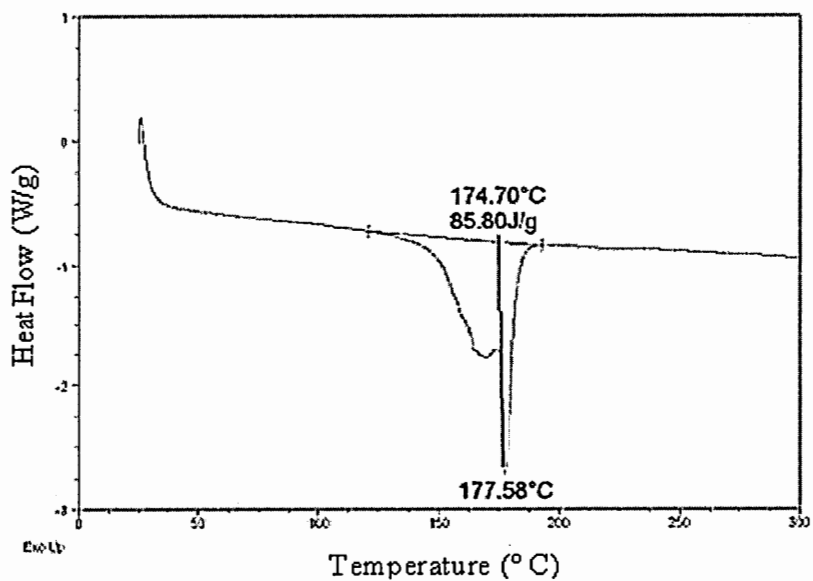


Figure 3.5 First heat cycle DSC curve of single PP backing fabric from shredded PP PCC

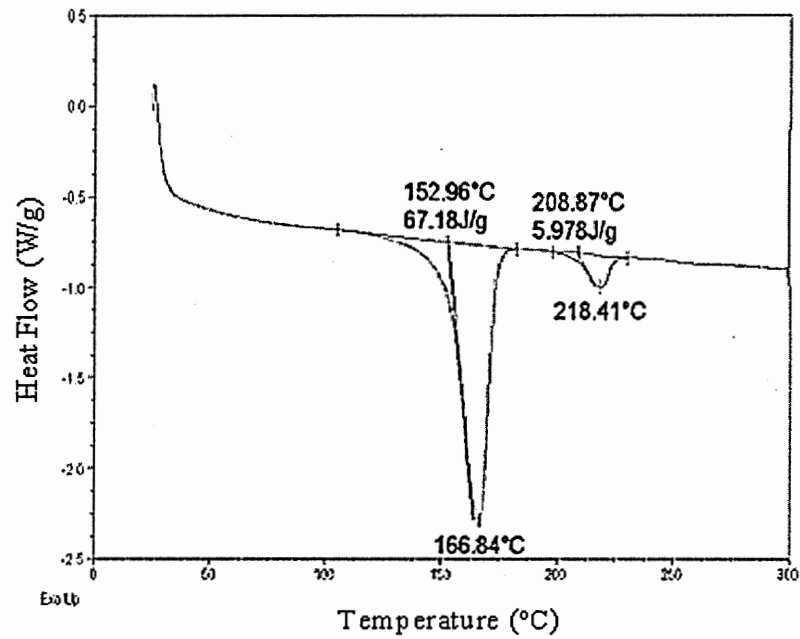


Figure 3.6 First heat cycle DSC curve of shredded PP PCC

Table 3.2 Estimated crystallinity fraction of composition of PP PCC

Component in PP PCC	H_m (J/g)	H_m^* (J/g)	Crystallinity fraction (%)
PP face fiber	74.64	188[17]	40
Backing material	85.80	188[17]	46
PP materials (general)	80.22		43
Nylon 6 fiber	69.50	189[17]	37

Table 3.3 Estimated weight fraction composition of PP PCC

Component in PP PCC	H_m (J/g)	H'_m (J/g)	Weight fraction (%)
PP face fiber & Backing material	67.18	80.22	84
Nylon 6 fiber	5.978	69.50	9

3.2 Thermal Stability of Shredded Post Consumer Carpet

Thermal analysis is important in understanding the structure-property relationship and mastering technology for industrial production. Both DSC and thermogravimetric (TG) techniques have been used for thermal analysis for fiber-reinforced composite[18]. In the previous section, DSC tests have already provided information on crystallinity of post consumer carpet. Data from TG tests indicate the stages of thermal breakdown and it is possible to reveal thermal decomposition mechanisms of the materials, which in turn can be used to set the temperature range in processing.

3.2.1 Experimental

Materials used for tests were prepared in the same manner as described in section 3.1.1. All the samples were tested on Hi-Res TGA 2950 and heated from 23.8°C to 1000°C at a heating rate of 10°C /min with continuously purging air to simulate the processing condition in real world.

3.2.2 Results and Discussion

Figure 3.7 demonstrates the thermal degradation behavior of nylon 6 face fiber and PP backing material from shredded N6 PCC. The polypropylene backing material began to decompose below 250 °C and lost most of its weight around 400°C. The majority weight loss of nylon face fiber started around 300°C and almost completed around

500°C. The about 3% loss shown in the curve of nylon face fiber is possible due to the loss of moisture absorbed by face fiber. The thermal degradation process of shredded carpet is consistent with that of its components. For the purpose of comparison, the SBR containing filler material was also tested by TGA and shown in Figure 3.8. The sieved material may still include a small amount of fibers and other impurities. It can be seen that all the polymer materials in the shredded carpet completed decomposition when the temperature reached around 700°C. Figure 3.9 shows that shredded PP PCC started to degrade as the polypropylene fiber did. The contaminated nylon 6 and SBR content accounted for the decomposition curve after 400°C.

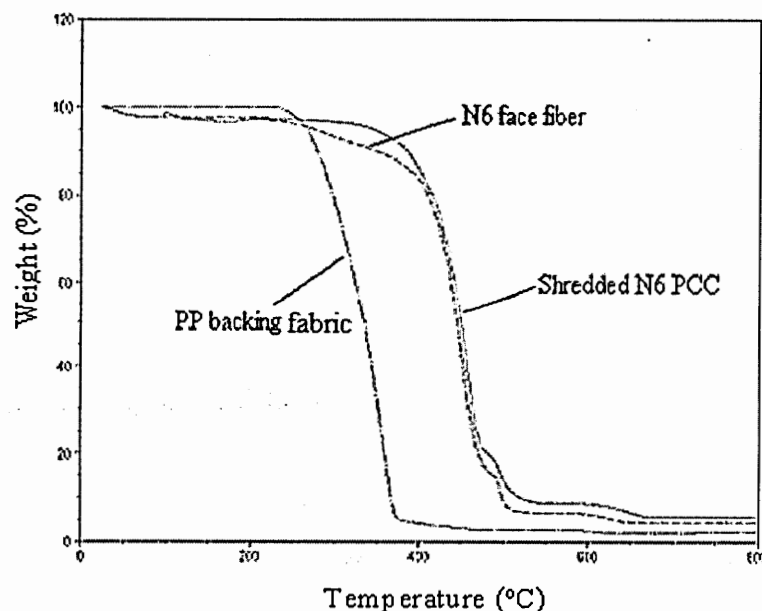


Figure 3.7 TGA diagrams of single nylon 6 face fiber, polypropylene backing fabric and shredded N6 PCC

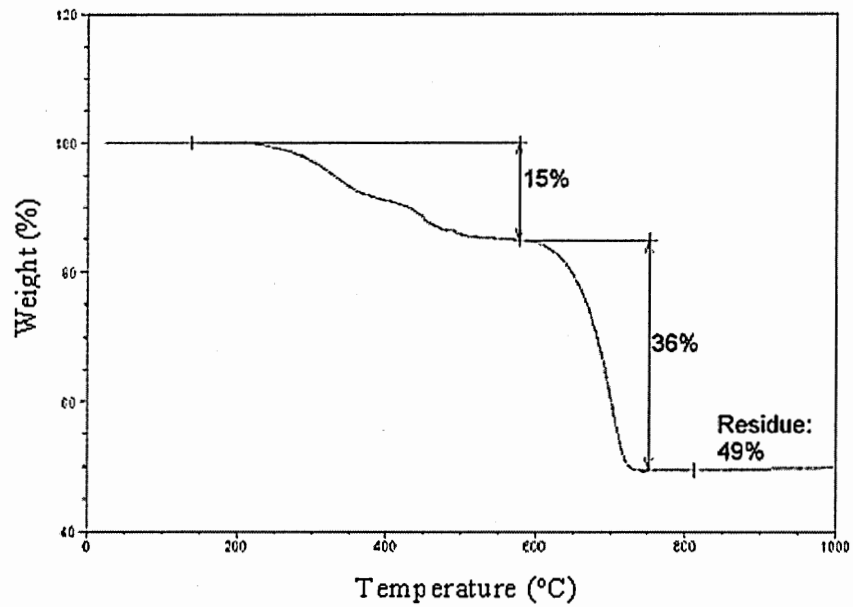


Figure 3.8 TGA diagram of SBR containing CaCO_3 filler

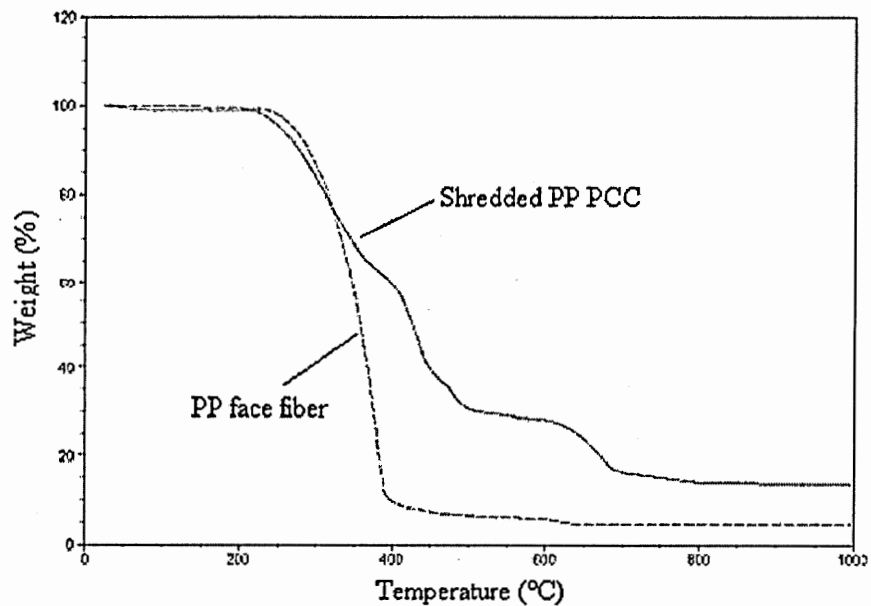


Figure 3.9 TGA diagrams of shredded PP PCC and single PP face fiber from shredded PP PCC

CHAPTER 4

Material Preparation and Compression Molding

4.1 Material Preparation

The shredded carpet has low density and this bulky nature makes it difficult to be interleaved with glass fiber mat for compression molding or other practical processing such as injection molding and extrusion. Therefore it is essential to convert shredded carpet into a more convenient form for further processing. In this study, two procedures were applied to convert the shredded carpets into the useful forms for compression molding.

4.1.1 Pelletizing

The shredded N6 PCC and PP PCC carpet were converted into pellets, separately, by using a NGR A-Class Type 55 VSP Repelletizing system from Next Generation Recycling Maschinen GmbH. The NGR unit is a single screw extruder with a shredder incorporated in the feed section of the extruder. The shredded carpet was forced into the in-line shredder by crammers and was blown into the extruder barrel where the shredded carpet was melted and vented. Then the melt was filtered with a 20-mesh screen and extruded as strands into a cool water bath. The cool strands were converted into pellets using a Reiter pelletizer. The pellets were ground into smaller particles by a Wiley mill.

The temperature profile for processing PP PCC is: intake 163°C (325°F), meter & vent 232°C (450°F), vent meter 246°C (475°F), adaptor through die 260°C (500°F). In the case of processing N6 PCC, the meter & vent was set at 260°C (500°F) in order to melt both polypropylene and nylon 6.

4.1.2 Debulking

Since shredded carpet has a low bulky density, it is necessary to consolidate the shredded carpet before it is stacked with glass fiber mat in compression molding. Debulking is a process without melting the shredded carpet. The shredded carpet was weighed first according to the designed weight ratio of glass fiber mat. The weighed shredded carpet was spread evenly on a Teflon sheet 30 x 30 cm (12 in x 12 in) and covered by another Teflon sheet of the same size. A bigger aluminum foil sheet was used to wrap the two Teflon sheets together in case of sliding to each other when pressure was applied on them. The wrapped sheets were preheated to 215°C (420°F) for around 3 minutes and then cooled in a 90 kN Wabash press with a pressure of 95 kPa until the temperature was lowered to 26°C (80°F).

4.1.3 Glass Fiber Mat

Glass fiber mat could be either swirled mat or short fiber mat. The former is made of undispersed glass roving and it has a continuously looped structure. The short fiber mat comprises of well-dispersed short fibers. Short fiber in GMT typically is about 5 ~ 20 mm long, which is much longer than those in injection molded products.

In this study, the chopped strand glass mat from Eleison, Inc. was used. The glass fibers were bonded with polyester resin. The mat contains 10 polyester and 90% E-glass. The glass mat has a basis weight of 110 g/m². The cut length of chopped strand was 50 mm.

4.2 Pellet Characterizations

4.2.1 Melt Flow Rate (MFR) Test

The MFR is defined as the weight of polymer extruded through a specified die in 10 minutes under standard condition of temperature and pressure. MFR is not a fundamental polymer property and may not correlate directly with processing

behavior, but it has been used as one important measure by the processors. MFR characterizes the behavior of the molten plastics including uniformity of the flow, and can be used for differentiating different grades of polymer and for quality control.

In this research, the tests are performed on an extrusion plastometer CEAST 6841 and method ASTM D1238-01 procedure A was followed. Procedure A is a manual cutoff operation based on time used for materials having flow rates within the range between 0.15 and 50 g/10 min. Since PP PCC contains more than 80% of polypropylene and less than 10% of nylon 6, the condition of its test is referenced to standard conditions for polypropylene, namely, 230°C with 2.16 kg total load including piston. In the case of N6 PPC, the standard conditions for nylon were applied, 235°C with 2.16 kg total load.

The MFR results for the pellets from shredded carpets are listed in Table 4.1. The pellets made from N6 PCC have a higher MFR than the pellets from PP PCC. Since nylon 6 accounted for up to 77% of the N6 PCC and nylon 6 has a lower viscosity than that of polypropylene, the result is reasonable and expected.

Table 4.1 Results of MFR measurements for the pellets extruded from PCC

Sample	Condition		MFR
	Temp (°C)	Load (kg)	(g/10min)
PP PCC pellets	230	2.16	11.0 ± 0.761
N6 PCC pellets	235	2.16	14.4 ± 0.540

4.2.2 DSC and TGA Tests

During palletized processing shredded carpet experienced re-melt and re-crystallizing and therefore it is necessary to investigate possible thermal degradation brought by processing. Figure 4.1 and Figure 4.2 show the results of TGA and DSC tests on shredded carpet fibers of N6 PCC and pellets of N6 PCC from the NGR extruder. The DSC diagram demonstrates the melting point temperature of two major components in the pellets set at almost exact same points as they did in the received shredded carpets. It indicates that the melting and cooling process during the course of extrusion didn't change the crystallinity of the polymer components. No significant shifts between melting peaks of components in two types of material sources are observed. In Figure 4.1 the major thermal decomposition part of the shredded N6 PCC and pellet N6 PCC overlapped very well. It is reasonable to conclude no obvious significant thermal degradation occurred due to the extrusion and pelletizing processes. In addition, the start of decomposition occurred at a higher temperature than shredded carpet, which suggests that the pellets were more stable than the shredded carpet. In a similar way, Figure 4.3 show the PP PCC pellet and shredded carpet started from almost same temperature to degrade, and the shredded carpet continued to decompose until around 700°C while PP PCC pellet completed its degradation around 400°C. The difference of weight loss between two shredded PP PCC and PP PCC pellet implies that the components are distributed more uniformly in pellets than in shredded mixture. The shredded PP PCC in the Figure 4.3 may contain more SBR and filler. No noticeable shifts between melt peaks of shredded PP PCC and PP PCC pellet are observed in Figure 4.4 and therefore, no significant thermal degradation occurred during palletizing process for PP PCC materials.

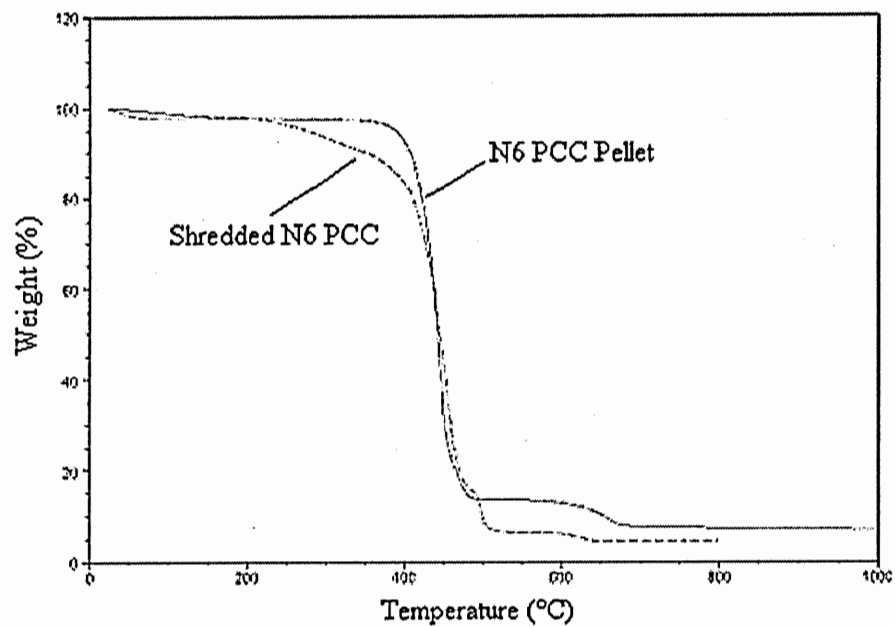


Figure 4.1 TGA diagrams of shredded N6 PCC and N6 PCC pellets

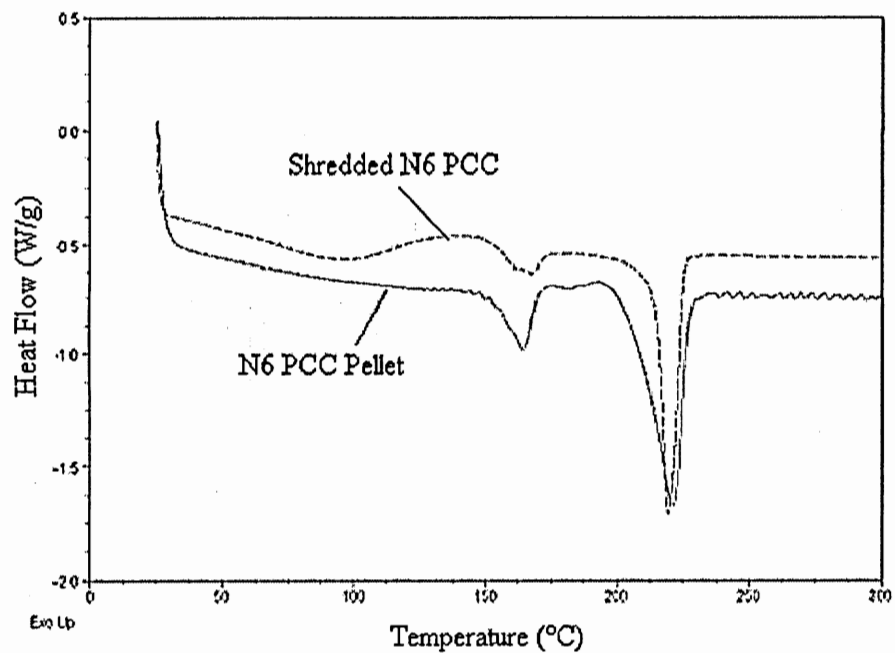


Figure 4.2 DSC diagrams of shredded N6 PCC and N6 PCC pellet

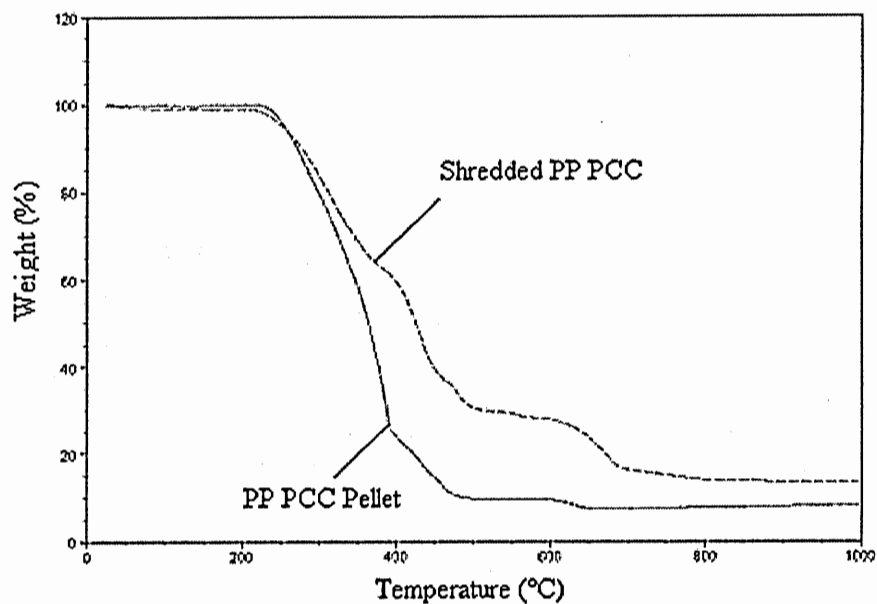


Figure 4.3 TGA diagrams of shredded PP PCC and PP PCC pellet

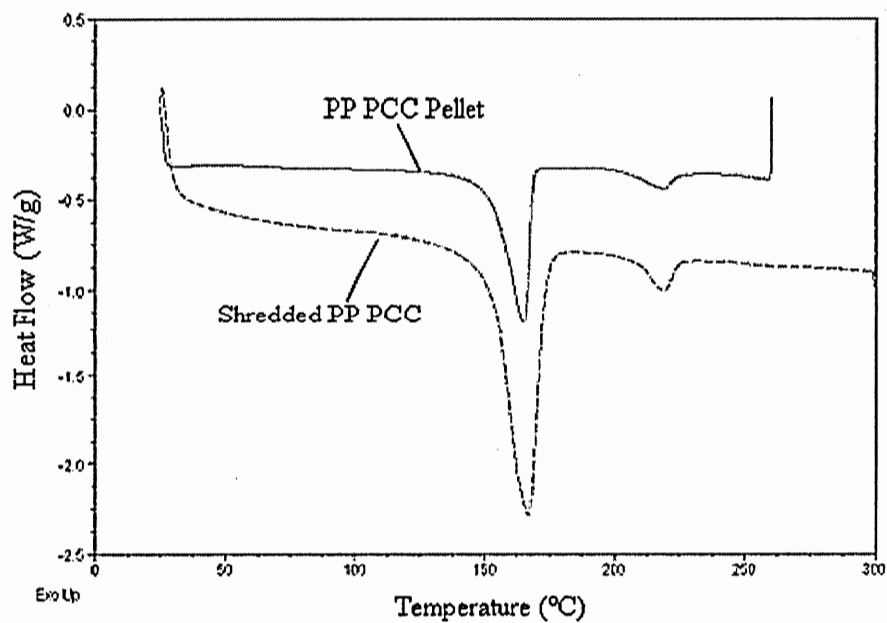


Figure 4.4 DSC diagrams of shredded PP PCC and PP PCC pellet

4.3 Sample Fabrication by Compression Molding

Glass fiber mat reinforced thermoplastic (GMT) is a sheet composite that can be fabricated via many methods: melt-impregnation method, slurry deposition method and preforming method. In this research, analogous GMT panels were made of glass fiber mat and post consumer carpet using compression molding that is based on the principle of melt-impregnation process. Glass fiber mat / PPC was heated above the temperature of PPC melting point and the melt permeated into layers of glass fiber mat. Then the mold was quenched and pressure was applied to facilitate the impregnation and consolidation of the panel.

A 30 x 30 cm mold and a Wabash 445 kN vacuum compression press with 46 x 46 cm platens were used. The total input weight of the material was controlled to be about 400 grams. All the glass fiber mats were cut into slightly less than 30 x 30 cm and the average weight of glass fiber mat is around 10 ~ 11 g/sheet. Two series of glass fiber mat reinforced panels were prepared in this research.

Series A was prepared using pre-weighed pellets with glass fiber mat concentration 0%, 20%, 30%, 40%. The pre-weighed pellets was spread on the bottom of the mold and one sheet of pre-cut glass fiber mat was laid on it, the interleaving process was repeated until all the materials were stacked up together. The detailed composition assigned is listed in Tables 4.2, and 4.3. The stacked mold was first heated in the Wabash Press with vacuum on and then it was quenched to cool down the laminate while it was being consolidated by the hydraulic press. The applied conditions to fabricating PCC composites were shown in Table 4.4. A1 is series made from PP PCC pellets and A2 is made from N6 PCC pellets. Series B was prepared using debulked PPC sheets interleaved with glass fiber mat of weight fraction 0% and 30%.

Table 4.2 Composition designed for sample made by pellet method

Desired sample using pellet method	PCC (g/layer)	Glass fiber mat (sheets)
0% GMT	400	0
10% GMT	72	4
20% GMT	35.6	8
30% GMT	21.5	12
40% GMT	14.1	16

Table 4.3 Composition designed for sample made by debulking method

Desired sample using debulking method	Debulked PPC (g/layer)	Glass fiber mat (sheets)
0% GMT	80	0
30% GMT	21.5	12

Table 4.4 Conditions applied to sample fabrication

Sample series	Material	Heating	Cooling
		Time(min)/Temp(°C)	Time(min)/Pressure(kPa)
A1 (0% ~ 40%)	PP PCC	20/216	2/100 → 5/150 → 15/200
A2 (0% ~ 40%)	N6 PCC	20/294	5/47 → 5/100 → 15/150
B1 (0%, 30%)	PP PCC	30/216	2/47 → 5/14.8 → 15/100
B2 (0%)	N6 PCC	25/260	5/47 → 5/14.8 → 15/150
B2 (30%)	N6 PCC	20/260	5/47 → 20/10.4

CHAPTER 5

MECHANICAL AND PHYSICAL PROPERTIES

5.1 Mechanical Tests

5.1.1 Flexural Properties Test

Flexure testing is a method for measuring behavior of materials subjected to simple bend loading. Flexural properties in this research were determined following ASTM D 790-02 using an Instron Series IX tester at a crosshead speed of 1.3 mm/min (0.05 inch/min). Six specimens for each sample with dimensions 127 x 25 x 3.4 mm (5 x 1 x 0.135 inch) were cut for test and a supporting span of 50.8 mm (2 inch) was used. Support span-to-depth ratio of 16 (tolerance ± 1) is generally recommended for molding materials. The tests were conducted at 23°C and 50% relative humidity.

The width and depth of the specimen were measured to the nearest 0.03 mm (0.001 inch) at the center of the support span. The rate of crosshead motion was set as calculated by the following equation:

$$R = ZL^2/6d \quad (5.1.1)$$

Where

R = rate of crosshead motion, mm (inch)/min

L = support span, mm (inch)

d = depth of beam, mm (inch)

Z = rate of straining of the outer fiber, mm/mm/min (inch/inch/min). Z shall be equal to 0.01.

The flexural modulus was the ratio of maximum stress to maximum strain, within elastic limit of stress-strain diagram obtained in the flexure test.

The maximum stress occurs at the mid-span for a specimen loaded as a simple beam at its midpoint. It is calculated by the formula:

$$S = \frac{3PL}{2bd^2} \quad (5.1.2)$$

Where

S = maximum fiber stress, MPa (psi)

P = load, N (lb)

L = span, mm (inch)

b = width of the beam, mm (inch)

d = depth of the beam, mm (inch)

5.1.2 Drop Impact Test

Drop impact test is useful to provide load versus deformation response of specimen at impact velocity. A Dynatup[®] 8250 drop weight impact test machine equipped with Dynatup 830-I data acquisition system was used. ASTM D 3763-92 was followed for the tests. Four specimens for each sample were prepared in dimension 10.2 cm x 10.2 cm (4 inch x 4 inch). The thickness of the samples is measured. Energy to maximum load, total energy absorbed by specimen and maximum load are recorded.

The impact energy absorbed by the specimen is calculated as

$$U = \frac{W}{g} (u_1^2 - u_2^2) \quad (5.1.3)$$

Where

W = weight of the striking weight load, kg

u_1 = velocity of the striking head just before impact ($\sqrt{2gH}$), m/s

u_2 = measured velocity of the striking head just after impact, m/s

g = acceleration of gravity, m²/s

H = drop height, m

5.2 Physical Properties Measurements

5.2.1 Density Determination

The density determination of samples was carried on a set up for the density determination of solids. The procedures were basically the same as described by ASTM D 792 – 00. The fundamentals of density determination of solids are governed by the following equation derived from Archimedes' principle:

$$\rho = \frac{A}{A - B} \times \rho_0 \quad (5.2.1)$$

where

ρ = density of the solid, g/cm³

A = weight of the solid in air, g

B = weight of the solid in the auxiliary liquid, g

ρ_0 = density of the auxiliary liquid at the given temperature, referred from a density table, g/cm³

Two specimens for each sample with dimension about 2.5 cm x 2.5 cm (1 inch x 1 inch) were cut from the specimens for the flexural tests of each sample. The specimen was weighed in air and weight A was noted. Then it was placed in the holder that was suspended into an air-free liquid. In this research, distilled water was used as the air-free liquid. After the balance reached stability the weight B was noted. The temperature of the water was also recorded before each specimen was tested.

5.2.2 Glass Fiber Concentration

ASTM D 2584-02 was followed to determine the weight fraction of glass fiber in the samples. The specimens used for density determination were dried in the oven at 70°C for 1 hour. After removing moisture, the specimens were placed into the crucibles and weighed to the nearest 1.0 mg. The crucible and specimens were heated in the oven at 600°C for about 3 hours until all carbonaceous material has disappeared.

However, the glass fiber reinforced composites made in this research also contain a little amount of inorganic component, calcium carbonate, which does not decompose around 600°C. Therefore, in this research, the glass fibers were picked out from the crucible and weighed after the crucible was cooled down to room temperature. The solid particles at the bottom of crucibles were observed. The following equation was used to calculate the ignition loss of the specimen in weight percent:

$$\text{Ignition loss, \%weight} = (W_2/W_1) \times 100 \quad (5.2.2)$$

where

W_1 = weight of specimen, g

W_2 = weight of residue, g

5.2.3 Void Content Determination

ASTM D 2734-94 - method A, which is a standard test method for void content in reinforced plastics, was used to determine the void content of the samples. The following equation was used for calculation:

$$V = 100 (T_d - M_d)/T_d \quad (5.2.3)$$

where

V = void content, volume%

T_d = theoretical composite density, g/cm³

M_d = measured composite density, g/cm³

The theoretical density of a composite was calculated as following:

$$T_d = 100/(w_f/\rho_f + w_m/\rho_m) \quad (5.2.4)$$

where

w_f = weight fraction of reinforcement in composite, weight%

ρ_f = density of reinforcement, g/cm³

w_m = weight fraction of matrix in composite, weight%

ρ_m = density of matrix, g/cm³

After rearrangement of the equation (5.2.3) and (5.2.4), the void content was obtained:

$$\text{Void vol \%} = 100 \times \left[1 - \rho_c \left(\frac{w_r}{\rho_r} + \frac{1 - w_r}{\rho_m} \right) \right] \% \quad (5.2.5)$$

the weight fraction of reinforcement w_r and the weight fraction of resin were determined by the ignition loss method described in Section 5.2.2; the composite density ρ_c and resin density ρ_m were measured using the reinforced and un-reinforced samples as described in Section 5.2.1.

5.3 Results and Discussion

(1) Void Content

On calculating void contents using 2.54 g/cm³ for the density of E-glass negative results were obtained. Equation (5.2.5) suggests that using too high a value for the composite density ρ_c , or reinforcement weight fraction w_r , or too low a value for the glass fiber density, or the matrix density. Thomason[19] suggested the relative importance of the factors is dependent on the fiber fraction with the exception of the composite density. Moreover, it was suggested that the two most likely sources of error could be the use of nominal value for the glass fiber density and the potential difference in matrix density between the composite matrix and the un-reinforced sample used to determine ρ_m .

The glass fiber mat used in this research was also the composite consisting of 10% amorphous polyester and 90% E-glass determined by ignition loss test. Weight ratio of polyester and glass fiber is 0.1111. On consideration of polyester in the glass fiber mat, the ignition loss determined above should include 10% of polyester, the density of which is 1.32 g/cm³ quoted from the manufacturer. Since the matrix density of the

composite was measured using composite without glass fiber mat, the theoretical density of composite was modified as following:

$$D_t = 1/(w_f/\rho_f + w_{PET}/\rho_{PET} + w_m/\rho_m) \quad (5.3.1)$$

w_f was determined by ignition loss test, ρ_f is 2.54 g/cm³; w_{PET} was 0.1111 w_f and ρ_{PET} is 1.32 g/cm³, ρ_m is determined from un-reinforced samples. Equation (5.2.5) is modified as following:

$$Void\ vol\% = 100 \times \left[1 - \rho_c \left(\frac{w_f}{\rho_f} + \frac{0.111w_f}{\rho_{PET}} + \frac{1 - w_f - 0.111w_f}{\rho_m} \right) \right] \% \quad (5.3.2)$$

After obtaining the void content of the composites using equation (5.3.2), the volume of glass fiber in samples is calculated by following equation:

$$GF\ vol\% = \frac{w_g/\rho_g}{w_g/\rho_g + w_{PET}/\rho_{PET} + w_{PCC}/\rho_{PCC} + void\%} \quad (5.3.3)$$

The results of determination of apparent density, glass fiber concentration and void content corresponding to the designed sample are listed in the appendix A.

Figure 5.1 shows a plot of composite void content as a function of glass fiber concentration for two series of composites made from N6 PCC and PP PCC. It is assumed that the void content in composites with 0% glass fiber mat is zero. It is not true in reality but it simplifies the case. It is clear that the void content goes up generally with increasing glass fiber concentration. The most likely reason for this trend is that the degree to which melting impregnation is completed decreases with the number of layers of glass fiber mat increasing. For debulking method, the apparent density of composites is less than that of corresponding composite made of pellets (Appendix A) and the Figure 5.1 indicates that the composites made from the debulking process has introduced more void content during compression molding than those made from pellets. It is possible that the processes of extruding, pelletizing and

grinding improved impregnation in compression molding by increasing the shredded PCC into more uniform and dense materials. On the other hand, Figure 5.1 also shows the composites made from N6 PCC introduced more void content than those made from PP PCC. The possible reason is that less pressure was applied during the quenching process of composites with N6 PCC in order to avoid flashing out. During the sample fabrication, it was found that more flashing out occurred in molding N6 PCC than in PP PCC case. This could be explained by the difference of MFR index between two kinds of materials. In addition, nylon 6 is much more susceptible to absorbing moisture in the air than PP even though the materials have been dried in oven for pre-test conditioning, and the voids are possibly attributed to the release of more moisture absorbed by N6 PCC pellets.

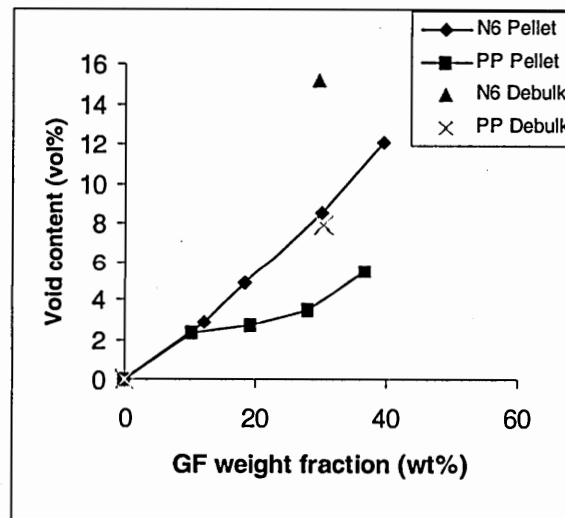


Figure 5.1 Void content of PCC composite vs. glass fiber weight fraction

(2) Flexural Properties

Figure 5.2 represents the flexural modulus of the composites with the variation of glass fiber content. It indicates generally the stiffness of these composites increases with increasing glass fiber concentration and it is very consistent with the results from previous studies about the influence of fiber concentration on flexural properties[20]. But when the glass fiber concentration was initially raised from 0% to around 10%, the value of flexural modulus fell. The possible reason is that glass fiber mat introduced a big increase in void content while the reinforcement was not strong enough to improve the modulus of the composites against the crack propagation brought by the voids. After the glass fiber content increase enough to overcome the void defects due to processing, the reinforcement started to play an important role to show an improvement of mechanical property. But this important role is limited by the introduction of voids and other possible defects caused by processing condition. In N6 PCC case, a significant decrease is observed after glass fiber weight fraction reach around 40%, it may also explain the influence of voids on the flexural modulus.

The composites made from pellets generally have higher modulus than those made from debulking PCC with the same reinforcement. The relations observed between flexural strength and glass fiber volume content are shown in Figure 5.3. It can be seen that the flexural strength basically depends on the glass fiber concentration and the matrix material used. The composites using N6 PCC have higher flexural strength than those using PP PCC. It also clearly shows that the flexural strength of composites made from debulking process is lower than those made from pellets with corresponding glass fiber concentration.

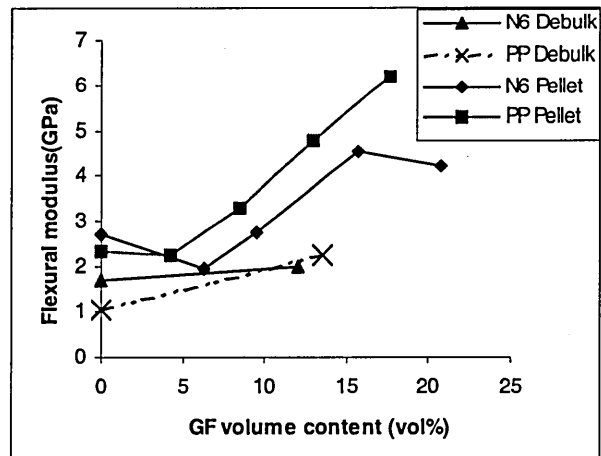


Figure 5.2 Flexural modulus of PCC composites vs. glass fiber volume fraction

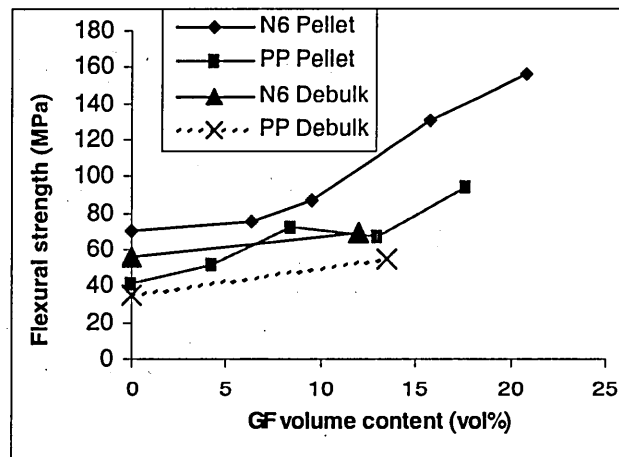


Figure 5.3 Flexural strength of PCC composite vs. glass fiber volume fraction

Table 5.1 shows the properties of GMT composites made by AZDEL, Inc.[21]. The composites are commercially available glass mat thermoplastic laminates based upon a random oriented chopped fiber mat contained within a polypropylene resin matrix. The flexural modulus and flexural strength for the composites of AZDEL[®] products are compared with those of PP PCC in this research in Figure 5.4 and Figure 5.5. The flexural modulus for both materials showed no significant difference between each other. The flexural modulus of composite of PP PCC with 40% and 30% glass fiber mat are greater than that of AZDEL[®]. It could be explained that the impurity particles in the post consumer carpet, particularly calcium carbonate, possibly served as additional reinforcement. The results indicate that the difference between matrix material such as post consumer carpet and virgin polymer does not have a major impact on their flexural modulus. However, AZDEL[®] laminate using virgin PP as matrix material showed better performance level in flexural strength.

Table 5.1 Properties of AZDEL[®] chopped fiber laminate[21]

Properties	AZDEL [®] C220-B01	AZDEL [®] C321-B01	AZDEL [®] C401-B01
Glass fiber content (wt%)	22	32	40
Glass mat basis weight (kg/m ²)	3.8	4.0	4.4
Specific gravity (ASTM D-792)	1.04	1.12	1.17
Flexural modulus (GPa) (ISO 178)	3.50	4.57	5.52
Flexural strength (MPa) (ISO 178)	95	104	146
Multi-axial Impact (4.0 mm thickness) Energy to max load (J)	12	9	10

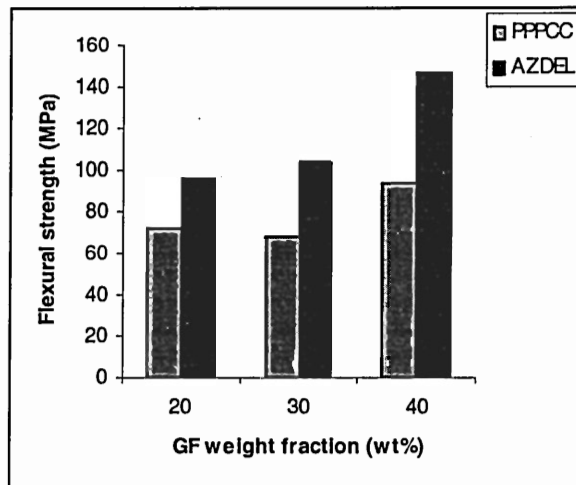


Figure 5.4 Comparisons of flexural strength between two types of composites

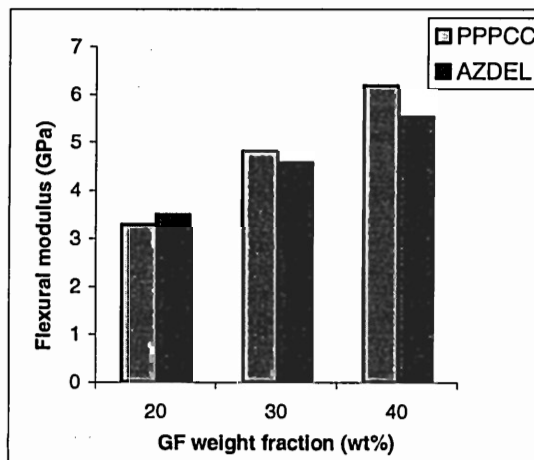


Figure 5.5 Comparisons of flexural modulus between two types of composites

(3) Impact Properties

The results of total energy absorbed from multi-axial drop impact tests are summarized in Figure 5.6 as a function of fiber concentration. The impact behavior depends on the thickness of the sample tested. The total energy absorbed in Figure 5.6 is normalized by total energy measured from tests divided by the sample's thickness and times 4.0 mm for the purpose of comparison with AZDEL[®] products. Overall the data show similar trends as previously observed for flexural modulus and strength. First, the increasing glass fiber concentration dominated the increase of total energy consumption during the impact test. Second, the matrix material, N6 PCC and PP PCC did not show their difference in their contribution either to total energy absorbed or to the value of maximum load that happens around fracture propagation. Despite the deviation occurred around 20% weight fraction of glass fiber the trends of four series are similar. It indicates that the matrix materials used in the research have little influence on the impact properties. It is interesting that the panels made from debulked post consumer carpet without reinforcement showed more than two-fold higher energy absorbed than the other corresponding samples. It was mostly likely attributed to carpet fibers remaining in the laminates due to incomplete melting.

In terms of energy to maximum load absorbed by the composites, Figure 5.7 shows that PP PCC composites have higher value of normalized energy to maximum load absorbed than AZDEL[®] products do after glass fiber weight fraction reaches above 30%.

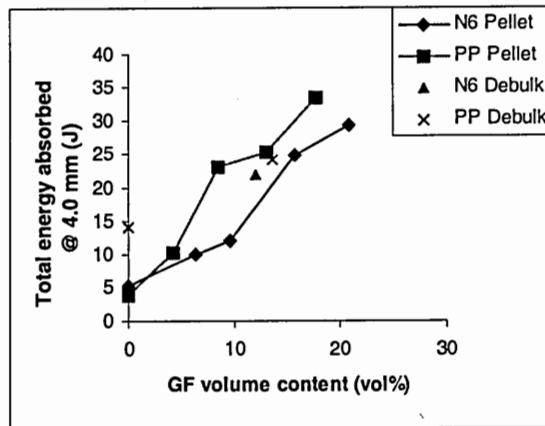


Figure 5.6 Normalized total energy absorbed by composites vs. glass fiber volume fraction

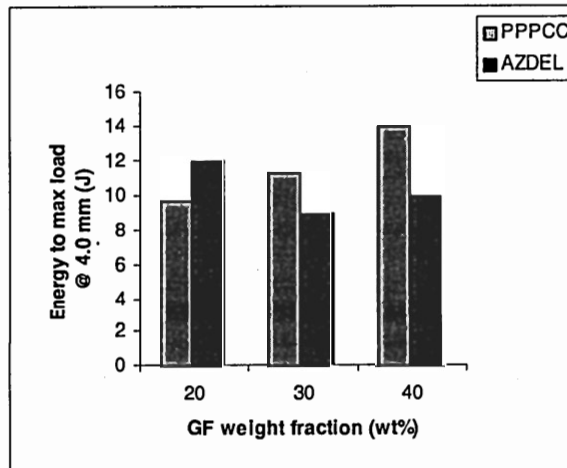


Figure 5.7 Comparisons of energy to maximum load between two types of composites

(4) Comparison with Composites Made from Glass Fiber Filled Extruded Board

The composites shown in Figure 5.8 and Figure 5.9 were all made from PP PCC pellets as above described and virgin PP. But composites with NFM labels were different in material preparation. The PCC pellets made from NGR extruder were sent to NFM Welding Engineers and reprocessed into continuous boards filled with 25.4 mm (1 inch) and 12.7 mm (0.5 inch) long fibers at various glass fiber concentrations, and the boards were cut into 280 x 51 mm (11 x 2 inch) boards. The process uses a counter rotating non-intermeshing twin-screw extruder. About 4 boards were spread into a 30 x 30 cm (12 x 12 inch) compression mold and made into a composite laminate under similar condition as that described. It is difficult to make extruded boards or rods filled with glass fiber more than 25.4 mm (1 inch) in length. These fiber lengths, 12.7 mm and 25.4 mm, are quite long in extrusion-molded products. The relevant properties of panels made from these extruded boards are listed in Table 5.2. Observing Figure 5.8 and Figure 5.9, the laminate made with glass fiber mat has the best performance in both impact test and flexural tests. The properties of the laminates made of glass fiber filled extruded boards did not show the dependence on glass fiber length. One reason for lack of clear trend might be caused by mechanical degradation of the glass fibers during their filling and mixing process in the extruder. The screws break the glass fibers and the frictions incurred between fibers and melting mixtures could also either break the fibers or damage the fibers. In order to investigate the fiber lengths in the composites the average fiber lengths were calculated from a minimum 200 fibers recovered from incineration of extruded boards. The fiber lengths were manually measured on a LEICA Qwin image analysis system. Figures 5.10 and 5.11 show the distribution of lengths of fibers in the final composites. Although the glass fiber samples investigated were only randomly selected from a small part of the whole

extruded boards, it evidently demonstrated that the fibers had been broken during the filling and extruding process and the majority of the fibers were reduced to half of the original length.

Secondly, the fibers filled into extruder might not be dispersed well and it led to poor wetting and fiber bundles, which could lower the performance level due to low effective aspect ratio of the fiber bundles. The variation in distribution of fiber length and their orientation in the composite contributed to the difficulty in controlling the mechanical properties of the products. In contrast, the glass fiber mats do not have the problems with reduction of fiber length and rearrangement of fiber distribution.

Table 5.2 Properties of composites made from glass fiber filled extruded broad[22]

Extruded board	PP PCC	PP(virgin)	PP PCC	PP(virgin)
Designed glass fiber wt%	30%	30%	32%	32%
Glass fiber cut length (mm)	12.7	12.7	25.4	25.4
Density (g/cm ³)	1.11	1.14	1.09	1.12
Real GF (wt%)	32	33	31	33
Void content (vol%)	4.5	2.0	7.5	5.1
Flexural modulus (GPa)	4.3	2.6	3.7	3.7
Flexural strength (MPa)	64.5	56.1	58.2	71.4
Drop impact test	4.8	9.7	6.8	2.0
Energy to max load (J)				
Drop impact test	6.8	13.6	6.8	3.5
Total energy @ 4.0 mm (J)				

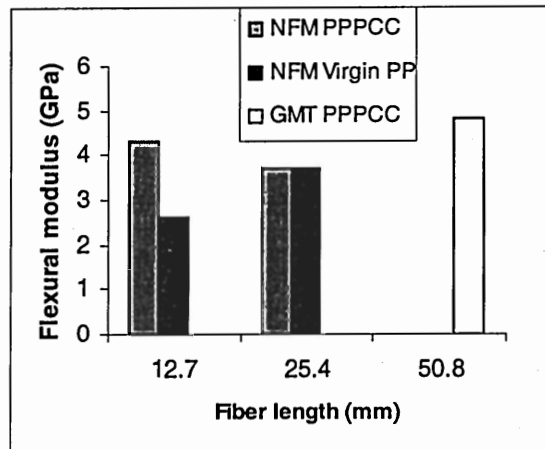


Figure 5.8 Comparisons of flexural modulus of composites (30wt% Glass Fiber) with various glass fiber lengths

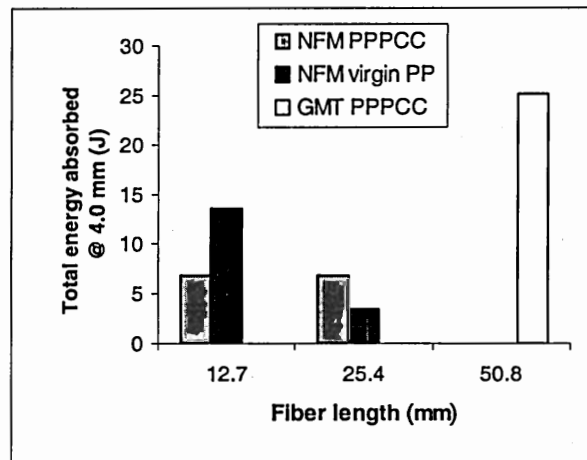


Figure 5.9 Comparisons of normalized energy absorbed of composites (30wt% of Glass Fibers) with various glass fiber lengths

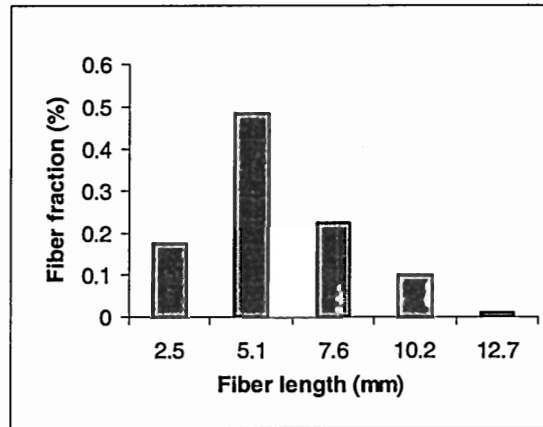


Figure 5.10 Fiber fraction with certain length in sample NFM PP (virgin) with 30wt% 12.7 mm long fibers

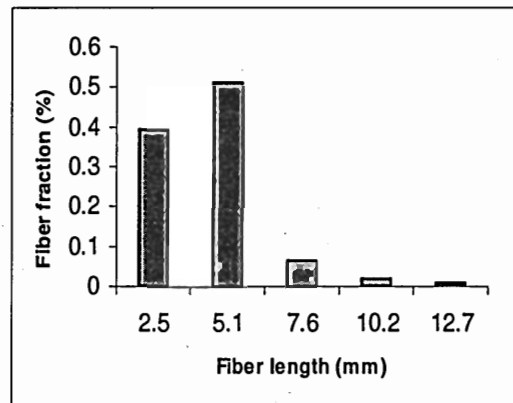


Figure 5.11 Fiber fraction with certain length in sample NFM PP PCC with 30wt% 12.7mm long fibers

CHAPTER 6

CONCLUSIONS

In this research the glass fiber mat reinforced thermoplastic composites made from two types of PCC have been investigated. Two material preparation methods, pellet method and debulking method, have been carried out. Shredded post consumer carpets with majority of polypropylene content used as GMT matrix demonstrated higher flexural modulus, total energy absorbed in the drop impact test than those with majority of nylon 6 face fibers. However, in terms of flexural strength, N6 PCC are greater by about 50% than those made from PP PCC pellets.

The pellet method took advantage of extruding and pelletizing process to make material more uniform and less porous. The flexural strength of the glass fiber reinforced PCC fabricated by the pellet method is generally two-fold higher than those produced by the debulking method.

The concentration of glass fiber volume played a critical role in improving the flexural properties and drop impact resistance. Generally, the experimental results proved that increasing concentration of glass fiber increased the flexural strength and impact damage resistance of the composites made by both methods. However, the contribution of glass fiber concentration to the improvement of mechanical properties is compromised by the void content in the composites. Within the range of 20% to 35% glass fiber concentration, the properties tested in this study were dominated by the glass fiber concentration.

Compared with glass fiber feeding extruded boards with 30% glass fiber concentration, GMT reinforced PCC composites with the same glass fiber content were around 25% higher in flexural modulus and 70% greater in energy absorbed in

drop impact test than boards made of either virgin PP or PP PCC. The flexural strength of the three types of composites is very close to each other. It proved that glass fiber mat is more effective to improve flexural modulus and impact resistance due to its controllable fiber length and orientation. PP PCC as a matrix material is comparable with virgin PP in flexural strength.

Comparison with commercially available GMT reinforced virgin PP laminates, AZDEL[®] laminates, also showed that GMT reinforced PP PCC was comparable with them in flexural modulus and energy to maximum load absorbed. But composites with virgin PP have higher flexural strength than those with PP PCC.

It is a feasible and interesting approach to use post consumer carpet without separation as a matrix material for glass fiber reinforced composites. Their properties in flexural bending and drop impact are comparable with virgin materials, and the composites have great potential to be applied in practice as load carrier for suitable duty.

CHAPTER 7

RECOMMENDATIONS

The processing conditions such as cooling rates and pressure applied for fabrication of glass fiber mat reinforced composites using post consumer carpet through compression molding is worthwhile to be explored. The processing condition has great impact on melt impregnation, void introduction and in turn affects the improvement of mechanical performance. Various types of glass fiber mat should be used to investigate the potential improvement of the properties of composites. Additional mechanical tests on glass mat reinforced PCC composites should be conducted to investigate the effect of processing condition and glass fiber concentration on the other fiber-dominated properties such as longitudinal tensile strength and strain.

The cost-effective goal could be achieved by expanding the application of the reinforced composites from PCC to fill the gap between moderate end-use performance and relatively limited end-use performance. It is worth investigating the optimized processing conditions to produce the composites with stable and characteristic properties such as good fatigue resistance. Therefore, additional mechanical tests such as tensile test, shear and compression tests could be investigated for possible application identification. On the other hand, investigation of influence of various temperature conditions for mechanical testing could be conducted. Maleic-anhydride-modified polypropylene has been demonstrated by other researchers that it can improve the mechanical properties such as strength and fatigue behaviors of glass fiber reinforced polypropylene composites[2, 23, 24], it would be interesting to apply m-PP to glass fiber mat reinforced composites made from PCC

and investigate the possible property improvement by m-PP. Also, further characterization of transverse, shear and compressive strength of the composites should be conducted on the m-PP modified PCC composites.

The process using post consumer carpet as matrix material could also be applied to other post consumer carpet with synthetic face fibers rather than nylon 6 or polypropylene.

APPENDIX A

Data for Physical Properties of Composite Samples Fabricated

A tabulation of the data for the apparent density and glass fiber content of the samples made from pelletized post consumer carpets with polypropylene face fiber and nylon 6 face fiber is given in Tables A.1 and A.2 respectively. The apparent density of the samples made from debulked post consumer carpets is given in Table A.3. The standard density of water is 0.99707 @ 25°C and 0.99681 @ 26°C respectively referenced from density tables for distilled water included in the density determination set-up. Two specimens for each sample have been tested. The method for apparent density determination is described in Section 5.2.1 and the method for determining glass fiber content of the samples is described in Section 5.2.2. Tables A.4 and A.5 summarize the values of void content, glass fiber volume and theoretical density of composite samples calculated from the data listed in Tables A.1, A.2 and A.3.

Table A.1 Apparent density of samples made from pelletized PCC with polypropylene face fiber and designed glass fiber mat content tested in water @ 25°C

Sample	Apparent density (g/cm ³)	Glass fiber content (wt%)
PP0%	1.0036, 1.0045 mean = 1.004	----
PP10%	1.0464, 1.0538 mean = 1.0501	9.63, 10.19 mean = 9.91
PP20%	1.1143, 1.1125 mean = 1.1116	18.03, 18.10 mean = 18.06
PP30%(1)	1.1828, 1.1833 mean = 1.1831	35.71, 36.01 mean = 35.86
PP30%(2)	1.1815, 1.1756 mean = 1.1786	28.95, 28.53 mean = 28.74
PP40%	1.2358, 1.2374 mean = 1.2366	36.16, 37.54 mean = 36.85

Table A.2 Apparent density of samples made from pelletized PCC with nylon 6 face fiber and designed glass fiber mat content tested in water @ 26°C

Sample	Apparent density (g/cm ³)	Real glass fiber content (wt%)
N60%	1.2798, 1.2769 mean = 1.2784	----
N610%(1)	1.3378, 1.3431 mean = 1.3404	18.90, 18.32 mean = 18.61
N610%(2)	1.1983, 1.1978 mean = 1.1980	13.82, 14.50 mean = 14.16
N610%(3)	1.3238, 1.3330 mean = 1.3284	13.12, 11.40 mean = 12.26
N620%(1)	1.3041, 1.2740 mean = 1.2890	25.05, 21.13 mean = 23.09
N620%(2)	1.0426, 1.0364 mean = 1.0395	22.56, 22.74 mean = 22.65
N630%	1.3837, 1.3681 mean = 1.3759	29.36, 30.64 mean = 30.00
N640%(1)	1.2880, 1.2648 mean = 1.2764	33.33, 32.01 mean = 32.67
N640%(2)	1.3974, 1.4160 mean = 1.4067	38.69, 40.31 mean = 39.50

Table A.3 Apparent density of samples made from debulked PCC with designed glass fiber mat content tested in water @ 26°C

Sample	Apparent density (g/cm ³)	Glass fiber content (wt%)
PP0%	0.9837, 0.9982 mean = 0.9910	----
PP30%	1.1467, 1.1330 mean = 1.1398	30.14, 30.76 mean = 30.45
N60%	1.1407, 1.1083 mean = 1.1245	----
N630%	1.1247, 1.1727 mean = 1.1487	28.97, 30.13 mean = 29.55

Table A.4 Calculated void content, glass fiber volume and theoretical density of composite samples made from pellet PCC

Sample (Pellet method)	Theoretical density (g/cm ³)	Void content (vol%)	Glass fiber volume fraction (vol%)
PP0%	1.004	0 (assume)	----
PP10%	1.075	2.30	4.30
PP20%	1.144	2.73	8.45
PP30%(1)	1.297	8.81	16.36
PP30%(2)	1.221	3.50	12.96
PP40%	1.307	5.41	17.62
N60%	1.278	0 (assume)	-----
N610%(1)	1.408	4.80	9.55
N610%(2)	1.380	13.0	6.77
N610%(3)	1.362	2.84	6.33
N620%(1)	1.467	11.1	12.8
N620%(2)	1.437	27.4	8.93
N630%	1.504	8.53	15.7
N640%(1)	1.528	16.5	15.8
N640%(2)	1.593	12.1	20.8

Table A.5 Calculated void content, glass fiber volume and theoretical density of composite samples made from debulked PCC

Sample	Theoretical density (g/cm ³)	Void content (vol%)	Glass fiber volume fraction (vol%)
PP0%	0.991	----	0
PP30%	1.140	7.91	13.5
N60%	1.125	----	0
N630%	1.149	15.2	12.1

APPENDIX B

Data for Mechanical Properties of Composite Samples Fabricated

Table B.1 Data of Flexural Properties of samples made from pelletized PCC with polypropylene face fiber

Sample	Width (mm)	Depth (mm)	Flexural Strength (MPa)	Flexural Modulus (GPa)
PP0%	24.1(6.45%)*	4.04(2.76%)	41.6(6.20%)	2.34(16.9%)
PP10%	24.2(4.17%)	4.03(1.67%)	51.2(7.53%)	2.56(12.0%)
PP20%	24.4(9.21%)	3.86(1.92%)	72.1(12.0%)	3.29(10.4%)
PP30%(1)	23.3(8.00%)	3.75(3.33%)	5.14(12.0%)	3.19(12.4%)
PP30%(2)	24.1(6.44%)	3.91(2.97%)	67.6(6.11%)	4.81(5.77%)
PP40%	23.8(3.33%)	3.88(1.47%)	94.0(9.85%)	6.20(10.2%)

* Coefficient of variation based on 6 specimens for each sample

Table B.2 Data of flexural properties of samples made from pelletized PCC with nylon 6 face fiber

Sample	Width (mm)	Depth (mm)	Flexural Strength (MPa)	Flexural Modulus (GPa)
N60%	24.2(2.74%)*	3.67(2.26%)	70.2(16.2%)	2.71(3.71%)
N610%(1)	23.9(3.50%)	3.82(3.18%)	87.3(3.85%)	2.74(24.8%)
N610%(2)	24.1(45.0%)	3.40(2.69%)	78.4(6.65%)	2.13(1.14%)
N610%(3)	23.7(2.76%)	3.82(0.91%)	76.5(3.84%)	1.95(1.31%)
N620%(1)	24.2(2.73%)	2.98(3.22%)	103(11.7%)	4.11(18.9%)
N620%(2)	24.8(3.31%)	4.37(1.60%)	60.6(8.34%)	1.86(10.9%)
N630%	23.3(6.45%)	3.08(1.53%)	135(18.1%)	4.55(26.1)
N640%(1)	23.3(3.93%)	3.24(0.95%)	157(7.10%)	4.19(12.3%)
N640%(2)	24.0(5.6%)	3.06(0.176%)	128(10.8%)	3.89(15.4%)

* Coefficient of variation based on 6 specimens for each sample

TableB.3 Data of flexural properties of samples made from debulked PCC

Sample	Width (mm)	Depth (mm)	Flexural Strength (MPa)	Flexural Modulus (GPa)
PP0%	24.1(4.32%)*	4.36(3.96%)	34.8(6.45%)	1.02(41.0%)
PP30%	23.6(8.31%)	3.67(3.66%)	54.4(31.8%)	2.23(33.8%)
N60%	23.8(6.48%)	4.00(1.38%)	55.8(11.6%)	1.72(17.2%)
N630%	23.3(4.05%)	3.56(2.61%)	69.8(12.7%)	1.99(32.3%)

* Coefficient of variation based on 6 specimens for each sample

Table B.4 Data of drop impact properties of samples made from pelletized PCC

Sample	Thickness (mm)	Maximum load (N)	Energy to maximum load (J)	Total energy absorbed (J)
PP0%	4.08(1.19%)*	768(15.5%)	3.48(36.9%)	3.84(29.6%)
PP10%	3.89(2.09%)	1124(17.2%)	5.41(3.33%)	9.90(20.0%)
PP20%	3.61(4.07%)	2167(9.80%)	9.65(11.1%)	20.7(9.18%)
PP30%(1)	3.38(1.50%)	2112(4.15%)	7.46(9.15%)	17.4(7.10%)
PP30%(2)	3.18(3.97%)	2247(9.16%)	9.02(8.37%)	20.0(2.05%)
PP40%	3.77(1.59%)	3573(6.07%)	13.2(15.7%)	31.4(12.0%)
N60%	3.67(5.34%)*	652(11.6%)	4.43(39.5%)	4.73(39.7%)
N610%(1)	3.23(5.93%)	1231(28.0%)	4.88(25.0%)	8.80(33.2%)
N610%(2)	3.37(8.05%)	1107(12.9%)	5.20(28.1%)	8.25(22.3%)
N610%(3)	3.91(6.01%)	1339(2.39%)	5.97(13.7%)	9.65(63.6%)
N620%(1)	3.11(3.16%)	1705(20.8%)	6.82(23.0%)	13.4(15.2%)
N620%(2)	4.31(4.96%)	2203(13.9%)	10.1(12.3%)	19.5(24.3%)
N630%	3.02(4.80%)	2322(16.6%)	9.63(16.2%)	18.8(20.2%)
N640%(1)	2.93((6.62%)	2615(16.3%)	10.2(23.7%)	19.7(18.5%)
N640%(2)	3.28(3.52%)	3003(10.6%)	13.3(14.1%)	24.0(9.21%)

* Coefficient of variation based on 4 specimens for each sample

Table B.5 Data of drop impact properties of samples made from debulked PCC

Sample	Thickness (mm)	Maximum load (N)	Energy to maximum load (J)	Total energy absorbed (J)
PP0%	4.31(4.62%)	1075(19.9%)	7.65(14.7%)	15.0(29.5%)
PP30%	3.75(2.72%)	2436(12.5%)	9.84(22.0%)	22.7(16.7%)
N60%	4.06(1.62%)	658(9.37%)	2.91(18.2%)	3.59(10.4%)
N630%	3.70(3.70%)	2187(4.91%)	8.12(9.32%)	20.2(7.60%)

* Coefficient of variation based on 4 specimens for each sample

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