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TITLE: Fundamental Studies for the Utilization of Carpet Waste

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RELEVANCE TO NTC GOALS

One of the NTC goals is to strengthen the nations textile and educational efforts by uniting diverse experts and resources in unique collaborative efforts. The union of a polymer chemist, a mechanical engineer (with a textile engineering undergraduate background), a polymer physicist, and a number of researchers in the textile and processing industries to work toward the solution to an important textile waste problem is indeed an example of such a collaborative effort.

Another NTC goal is the development of new materials and improved manufacturing processes. The fabrication of carpet waste fiber reinforced concrete is an excellent example of the development of new materials with potentially important properties. The extrusion of mixtures of compatibilized polypropylene/nylon involves not only the development of new materials but also the development of innovative processing techniques with the cooperation of process engineers from industry.

Our interactions with industrial companies will facilitate the utilization of the developed technologies. A number of invited [1,2] and contributed [3,4] presentations have been made on carpet recycling during the past year. The response to these presentations, to put it modestly, has been tremendous. Refereed journal research papers have been published [5,6], and a U.S. Patent application has been submitted [7]. One M.S. Thesis has been completed during this period [8], and work on other theses is currently underway (Shah and Chen). The carpet recycling (with particular emphasis on the concrete reinforcement) work has also featured in a Dalton, GA newspaper. Our work on the NTC funded carpet recycling has also helped Georgia Tech researchers to start a new initiative on the Recycling of Fibrous Solid Waste in Apparel, Carpet, and Textile Industries under Georgia's Consortium on Competitiveness funded by the state of Georgia. Under this initiative three Georgia industries will be targeted for reducing the solid fibrous waste, one in each of the following areas: apparel, carpet, and textile. Based on our work we also have had number of useful discussions with the industries; this list of industries include: BASF, Monsanto, Reichhold Chemicals, and Shaw industries. These discussions and in some instances the cooperative work between these industries and Georgia Tech has been of significant mutual benefit. The NTC support has helped in nurturing the polymer recycling expertise at Georgia Tech, and in the training of Graduate students in the recycling technologies. The discussions with the industries have clearly demonstrated the usefulness of this approach to the industries.

RESEARCH SIGNIFICANCE

The annual fiber consumption of the U.S. carpet industry is approximately 2.4 billion pounds. The most common fibers used by this industry are nylon, polypropylene, and, to a lesser extent, polyester. Current carpet manufacturing practice involves tufting the face fiber into a primary backing and then binding the primary backing to the secondary backing using styrene-butadiene-rubber latex (Figure 1). The latex binder holds the face fibers in place. Calcium carbonate is used as an economic filler in the latex, and does not contribute to the properties of the carpet. On curing, the SBR latex is crosslinked and hence becomes a thermoset. Nylon and polypropylene are the current common face fiber and the backing materials respectively. Carpet tiles and automotive carpets are generally made using thermoplastic binders; however, the different thermoplastic components even in these latter categories of carpets are not selected for their compatibility with each other. Therefore the recycling of even the current all-thermoplastic carpets is not straightforward. Over 60 million pounds of annual waste is generated in the carpet industry in Dalton, GA. In the current work, the attempt is being made to utilize this waste. The suggestion that over half the annual carpet production is used to replace used carpet means that as much as one billion pounds of fiber may currently be going into the US landfills by way of used carpets. Used carpet will in general be heavily contaminated, while most industrial carpet may possibly be retrieved in relatively uncontaminated form. However we anticipate that methods can be developed to de-contaminate the used carpets in such a way that the recycling technologies developed for the industrial waste can be used for recycling the used carpet as well.

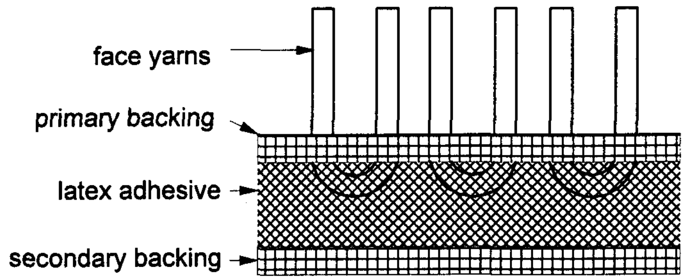

Figure 1. Illustration of Tufted Carpet Structure

The industrial waste generated before the application of the SBR latex adhesive is termed "soft waste" and the SBR containing waste is termed as "hard waste". The soft waste can be reused for filling purposes. Waste reduction and possible reutilization activities have been initiated over the past few years. Efforts are currently focused to develop cost effective technologies to recycle the carpet waste. A number of options are currently under evaluation, the principal ones being: (a) De-polymerization of the nylon; (b) manufacture of recyclable single material carpets, based on polyester; (c) alloying/ homogenization of the hard waste through reactive extrusion. Shredded carpet waste is also being evaluated for concrete reinforcement.

Other interesting alternatives would be the safe and efficient pyrolytic destruction of carpet wastes using plasma arc technology and effective separation of components using super critical solution techniques. In the current work, attempts are made to develop different technologies to convert carpet waste into useful products, including nylon depolymerization, reaction extrusion of the waste, and concrete reinforcement with the waste. Further descriptions of the current status of these technologies will be provided in the next section.

PROGRESS SUMMARY

Significant progress has been made in all the three areas: depolymerization of nylon, extrusion of waste by compatibilization, and use of waste for concrete reinforcement. Results to date are summarized below.

Depolymerization of Nylon:

Background: Our main objective is to develop a low temperature, atmospheric pressure process for the depolymerization of nylon 6 and nylon 6,6 in carpet waste utilizing phase transfer catalysis. In 1940, Hosino reported that nylon 6,6 is almost completely hydrolyzed with concentrated hydrochloric acid for 12 hours under reflux. The yields of adipic acid and hexamethylenediamine dihydrochloride recovered were 77% and 88%, respectively. In 1946, a du Pont patent reported a process for recovering adipic acid and hexamethylenedi-amine from nylon 6,6 waste fibers using 50% sulfuric acid for 5 hours at 120°C. Hexamethylenediamine was recovered by adding lime and purified by distillation. Adipic acid was purified by carbon treatment and recrystallization. Using an approach similar to that of the du Pont Company, Toyo Rayon recovered adipic acid and hexamethylenediamine in yields of 90% and 87%, respectively. In a 1961 patent, Monsanto reported the reclamation of waste nylon by re-action with molten adipic acid to dissolve the partially depolymerized polymer. After removal of stabilizers and other volatile materials by steam or inert gas sparging, the remaining liquid is hydrolyzed with water under pressure at 200 to 300° C. The resulting hot filtered aqueous solution is then reacted with hexamethylene-diamine to neutralize the excess acid groups. The filtered salt may be either subsequently polymerized or blended with freshly prepared salt for polymerization.

Results and Discussion: Acid hydrolysis of nylon 6,6 yarn was performed in both 46% aqueous formic acid and 7% aqueous hydrochloric acid solutions in the presence of phase transfer agent over a 24 hour period. The use of 46% formic acid at 95°C in the presence of phase transfer agent resulted in the recovery of a 56.4% yield of oligomers. In the absence of phase transfer agent 81.7% of unreacted nylon fibers were recovered. The use of 7% hydrochloric acid solution at 80°C for 24 hour in the presence of phase transfer agent resulted in an 85.3% yield of adipic acid and a 70.9% yield of hexamethylenediamine dihydrochloride.

For the formic acid process, 6g of nylon 6,6 yarn was reacted with 120 ml of 46% aqueous formic acid and 0.1g of phase transfer agent for 24 hours at 95°C. After completion of the reaction, water was evaporated and the residue was extracted with isopropanol in a Soxhlet apparatus for 24 hours. After evaporation of isopropanol, the products were dried in a vacuum oven at 30°C for 24 hours. The residue was washed with acetone to remove formic acid and water to remove the phase transfer catalyst. After drying in an oven at 100°C for 24 hours the weight of the dry product was 2.87g (56.4% yield). FTIR analysis indicated that nylon oligomers were formed.

For the hydrochloric acid process, 6g of nylon 6,6 yarn was reacted with 120 ml of 7% aqueous hydrochloric acid solution and 0.1g of phase transfer agent for 24 hours at 80°C. After completion of the reaction, the solution was cooled and extracted with three 50 ml portions of ether. The combined ether extracts were washed with water to remove traces of HCl and phase transfer catalyst and evaporated to produce a residue which was dried at 100°C in a vacuum oven for 24 hours. The weight of the recovered adipic acid was 2.76g (71.7% yield). The remaining aqueous solution was evaporated and the residue was dried in a vacuum oven at 100°C for 24 hours to give 4.24g (84.4% yield) of hexamethylenediamine dihydrochloride.

Reactive Extrusion of Carpet Waste:

Background: The use of compatibilizer to produce alloys of virgin polypropylene and nylon 6 has been reviewed in our last annual report [9]. The use of maleic anhydride grafted polypropylene (PP-g-MAH) as a compatibilizer for reactive extrusion of carpet face (nylon) and backing (polypropylene) has also been described in the report [9]. Our work[6,9] on the reactive extrusion and the work carried out at Monsanto [10] has clearly demonstrated that there is a significant potential for the utilization of carpet as a plastic resin. The initial experiments showed that by blending, compatibilizing, and extruding the carpet, the tensile strength as good or better than the tensile strength of polypropylene could be achieved. However, the tensile strain of the extruded carpet was rather low. Our work during the last year has focused on attempts to improve the tensile strain. The result of this study are presented below.

Experimental: Densified nylon 6 carpet in the form of pellets was received from BASF. This material was generated from their carpet separation plant operated jointly with Shred-Tech limited. The carpet face yarn is separated from the backing material and is sent through the condux plastocompactor (densifying mill). Based on the preliminary analysis done at BASF, the approximate sample composition was: Nylon 70-73%, calcium carbonate 6-14%, styrene butadiene rubber (SBR) 2-4%, polypropylene 10-20%, moisture 1-2% . These samples were used with the following two compatibilizers: Polybond™ (PP-g-MAH) from BP Chemicals and Kraton G1921x (SEBS-g-MAH) from Shell Co. The carpet waste and the compatibilizer were blended in the twin screw extruder (TW100). As based on the previous study the twin screw speed was chosen to be 150 - 170 RPM and the double pass technique was used. Two series of extrusion temperatures were used:

Series I—Temperatures of the four heating zones: 190, 210, 220, 235°C
Series II—Temperatures of the four heating zones: 150, 220, 235, 250°C

Extruded samples were compression molded at 235 and 260°C. Extrusion, compression molding, and mechanical test procedures were the same as reported before [6,9]. The mechanical properties of the various blends are given in Tables I -IV.

Table I. Mechanical properties of the blend using PB 3002 compatibilizer at Series I extrusion conditions

PB3002	Molding at 235°C			Molding at 260°C		
	Strength	Strain to failure	Work of rupture	Strength	Strain to failure	Work of rupture
wt %	*kpsi*	*%*	*lb-in*	*kpsi*	*%*	*lb-in*
0	6.65	5.99	1.10	6.71	6.96	1.05
5	6.43	5.44	0.92	6.94	5.14	0.54
10	5.91	4.44	0.61			
15	6.21	4.93	0.67	6.34	5.88	0.73

Table II. Mechanical properties of the blend using PB 3002 compatibilizer at Series II extrusion conditions

PB3002	Molding at 235°C			Molding at 260°C		
	Strength	Strain to failure	Work of rupture	Strength	Strain to failure	Work of rupture
wt %	*kpsi*	*%*	*lb-in*	*kpsi*	*%*	*lb-in*
0	5.36	4.06	0.58	6.48	10.27	
5	5.39	4.22	0.67	6.55	5.90	
10	6.41	5.95	1.30			
15	7.08	6.69	1.34	7.02	7.98	1.27

Table III. Mechanical properties of the blend using Kraton compatibilizer at Series I extrusion conditions

Kraton	Molding at 235°C			Molding at 260°C		
	Strength	Strain to failure	Work of rupture	Strength	Strain to failure	Work of rupture
wt %	*kpsi*	*%*	*lb-in*	*kpsi*	*%*	*lb-in*
0	6.65	5.99	1.10	6.71	6.96	1.05
5	5.95	6.08	1.05	6.34	9.19	1.51
10	5.61	6.54	1.27	5.57	15.27	2.32
15	5.71	17.50	4.30	5.26	30.93	4.30

Table IV. Mechanical properties of the blend using Kraton compatibilizer at Series II extrusion conditions

Kraton	Molding at 235°C			Molding at 260°C		
	Strength	Strain to failure	Work of rupture	Strength	Strain to failure	Work of rupture
wt %	*kpsi*	*%*	*lb-in*	*kpsi*	*%*	*lb-in*
0	5.36	4.06	0.58	6.48	10.27	
5				5.82	12.57	2.12
10	6.19	8.33	1.57	5.52	12.88	1.72
15	6.67	21.54	5.74	5.60	31.43	6.25

A comparison of the results in Table I -IV suggests that:

1. For the carpet system studied Kraton is a more effective compatibilizer than the polybond 3002. Work of rupture of the samples utilizing 15 % Kraton is excellent. Further work will be carried out to elucidate the compatibilization and the toughening mechanism using Kraton.

2. Variation in extrusion temperatures and in the molding temperatures result in small variation in properties.

Fiber Reinforced Concrete Using Carpet Waste Fibers:

Background: Concrete is durable, inexpensive, readily molded into complicated shapes, and has adequate compressive strength and stiffness, and it is the most heavily used man-made material in the world. However, concrete has low tensile strength, low ductility, and low energy absorption. Fiber reinforced concrete (FRC) is often made by adding a small fraction (usually below 2% by volume) of short fibers to the concrete mix during mixing. After extensive studies in the last three decades, it is now beyond doubt that such fiber reinforcement can significantly improve the tensile properties of concrete. Orders of magnitude increases in toughness (energy absorption) over plain concrete is commonly observed. Fiber reinforced concrete is currently being used in many applications including buildings, highway overlays, bridges, and airport runways. Fibers used in FRC include steel, alkali-resistant glass, and various synthetic fibers, most notably polypropylene. Recycled synthetic fibers such as those from textile industry waste offer low cost alternatives for concrete reinforcement. This work has focused on concrete reinforced with recycled fibers from hard carpet waste. It suggests that using recycled carpet fiber in construction could be a very cost-effective way to improve the durability and performance of concrete structures and at the same time to reduce the needs for landfill spaces.

Laboratory Evaluation of Carpet Waste Fiber Reinforced Concrete: The laboratory study carried out at Georgia Tech on concrete reinforcement with carpet waste fibers has been completed. The concrete matrix consisted of Type I Portland cement, river sand, crushed granite aggregate (maximum size=10 mm), and water. The weight ratios were 1.0 (cement)/0.35 (water)/0.85 (sand)/0.61 (aggregate). Recycled carpet waste fiber used was from hard carpet waste, disassembled mechanically by the Crown America, Inc. of Dalton, Ga. After disassembling, surface yarns (nylon) and some backing fibers (polypropylene) were collected. This collection is referred to as Type I waste fiber and had a typical length range between 12 to 25 mm. The disassembled waste after removal of Type I fiber is referred to as Type II, which contained backing fibers, SBR coated face yarns, and SBR particles. An analysis of Type II fiber indicated that the fiber length was about 3 to 25 mm and that about one third by weight was actually fiber, the other two thirds being $CaCO_3$ filled SBR. Fiber volume fractions for the waste fibers were 1% and 2%. Only the actual fiber portion was included for calculating fiber volume fractions for Type II waste fiber reinforced concrete. FiberMesh® virgin polypropylene fiber at 0.5% and 1% volume fractions was also included in this study for comparison purposes. The fiber length was 19 mm.

Four point flexural test and cylinder compressive test were conducted on a hydraulic testing machine. The age of specimens at testing was 28 days except the 1 day compressive test.

Results and Discussion: The results for compression and flexural tests of various mixes are given in Table V. Six or seven specimens were tested for each setup. In the one day compressive test, similar strength values were observed for plain concrete and various FRCs. It appeared that the 28 day compressive strengths of carpet waste FRCs were lower than that of plain concrete. The plain concrete specimens failed in a brittle manner and shattered into pieces. In contrast, all the FRC samples after reaching the peak load could still remain as an integral piece, with fibers holding the concrete matrices tightly together.

Table V. Compressive and Flexural Test Results

Fiber in FRC Mix	V_f	Compressive Strength		Flexural Strength	Flexural Toughness Indices	
		1 day	28 days		I_5	I_{20}
	%	*MPa*	*MPa*	*MPa*		
Concrete control	0	20.9	52.6	4.65	1.0	1.0
Fiber Mesh PP	0.5	24.2	52.2	4.58	2.6	6.9
Fiber Mesh PP	1.0	19.9	51.5	4.99	3.3	12.5
Type I waste fiber	1.0	22.4	59.4	5.24	1.8	4.4
Type II waste fiber	1.0	20.1	61.8	4.09	2.1	5.4
Type I waste fiber	2.0	20.9	39.7	4.69	3.3	7.5
Type II waste fiber	2.0	18.6	40.7	4.35	3.5	9.8

The flexural strengths of all mixes tested were essentially the same and the standard deviations were low. Figure 2 shows the typical load versus displacement curves for the flexural test. The plain concrete samples broke into two pieces once the peak load was reached, with very little energy absorption. The FRC specimens, on the other hand, exhibited a pseudo ductile behavior and fibers bridging the beam crack can be seen. Because of the fiber bridging mechanism, the energy absorption during flexural failure was significantly higher than that for plain concrete. One method of characterizing the energy absorbing ability, or toughness, of FRC is to calculate the toughness indices, as proposed in ASTM C1018. These indices indicate the energy absorption of FRC compared with a brittle material such as concrete (toughness index=1.0) for the deformation range specified.

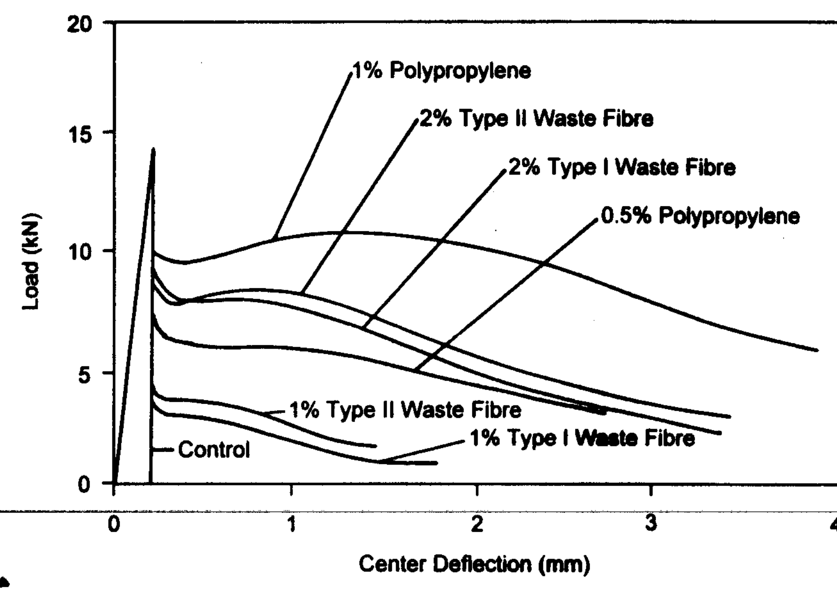

Figure 2. Typical Flexural Test Curves

Drying Shrinkage: The drying shrinkage of concrete was compared with that reinforced with carpet waste fiber and FiberMesh polypropylene. The test was carried out by measuring the length changes of 50.8×50.8×635 mm prism specimens at 19.5°C and 50%RH over a period up to 21 days. The reduction of drying shrinkage due to waste fiber reinforcement at 0.5% and 1% volume fractions was about 20%.

FRC for Construction Applications: Our laboratory study has indicated that the carpet waste fiber was very effective in improving the toughness and shrinkage properties of concrete. Shaw Industries Inc. has just completed its new 120,000 square-foot R&D Center in Dalton which used concrete reinforced with carpet waste fibers in the construction project. The presentation of the research findings of this NTC project to the Shaws new building team has contributed to Shaws' decision of using waste carpet reinforced concrete in this construction. About 40 thousand pounds of carpet production waste was consumed in the project, which would otherwise have gone to a landfill. Georgia Tech worked with Shaw Industries' building construction team on the specifications for waste shredding, fiber dosage selection, mixing, testing, and analysis. The amount of waste fiber added was 10 pounds per cubic yard and mixing was done by adding the fiber to the mixing truck directly. Such concrete containing waste fiber was used for the floor slabs, driveways, and walls of the building. The project demonstrated the feasibility of using large amount of carpet waste for concrete reinforcement in a full scale construction project. An estimation indicates that a one mile four-lane highway may use about 100,000 pounds of carpet waste for concrete reinforcement. Besides reducing the need for landfilling, the use of low-cost waste fiber for concrete reinforcement could lead to improved infrastructure with better durability and reliability.

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