#### **Recycling of Carpet and Textile Fibers**

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A large amount of fibrous waste is generated each year in the United States. For economic and environmental reasons, industries and research organizations have been looking for various technologies to recycle fibrous waste. This chapter reviews the waste statistics, waste characteristics, recycling rates, and recycling technologies for fibrous waste.

#### 1. Introduction

According to the US Environmental Protection Agency (EPA) [1,2] the municipal solid waste generated in the US is about 210 million tons per year, among them about 40% being paper products, 9% plastics, and 4% carpet and textiles.

Most of the fibrous waste is composed of natural and synthetic polymeric materials such as cotton, polyester, nylon, polypropylene, among others. Table 1 presents the amount of fibrous waste including textiles and carpet. In 1996, approximately 6.8 million tons of textile waste was discarded, and 2.3 million tons of carpets and rugs (carpet contains fiber, rubber, and inorganic filler) were generated in the municipal solid waste. About 1% of carpet waste was recovered for recycling (Table 1). In comparison, 20 million tons of plastics were generated and 6% was recovered, and 4 million tons of rubber tire waste was generated and 19% was recovered in the United States.

There are several disadvantages associated with the current landfilling of fibrous waste. First, a tipping fee is required. Second, due to environmental concerns, there is increasing demand to ban polymers from landfills. Third, landfilling polymers is a waste of energy and materials. A variety of technologies have been developed in response to customer demands for recycled products and as alternatives to landfilling [3,4,5,6,7,8]. Except for the case of direct reuse, which is a common form of utilization for discarded textiles, some processing is involved to convert the waste into a product. Typically recycling technologies are divided into primary, secondary, tertiary, and quaternary approaches. Primary approaches involve recycling a product into its original form; secondary recycling involves melt processing a plastic product into a new product that has a lower level of physical, mechanical and/or chemical properties. Tertiary recycling involves processes such as pyrolysis and hydrolysis, which convert the plastic wastes into basic chemicals or fuels. Quaternary recycling refers to burning the fibrous solid waste and utilizing the heat generated. All these four approaches exist for fiber recycling.

#### 2. Textile Waste and Recycling

According to the Secondary Materials and Recycled Textiles Association (SMART) and the Council for Textile Recycling [9,10], more than one thousand businesses and organizations employing many tens of thousands of workers divert some 2 million tons of textile waste from the solid waste stream. Textile waste can be classified as either pre-consumer or post-consumer. Pre-consumer textile waste consists of

by-product materials from the textile, fiber and cotton industries. Each year 750,000 tons of this waste is recycled into raw materials for the automotive, furniture, mattress, coarse yarn, home furnishings, paper and other industries. Approximately 75 percent of the pre-consumer textile waste is recycled.

Post-consumer textile waste consists of any type of garments or household article, made of some manufactured textile, that the owner no longer needs and decides to discard. These articles are discarded either because they are worn out, damaged, outgrown, or have gone out of fashion. They are sometimes given to charities but more typically are disposed of into the trash and end up in municipal landfills. Approximately 1,250,000 tons of post-consumer textile waste (4.5 kg per capita) is recycled annually. However, the recycled amount represents less than 25 percent of the total post-consumer textile waste that is generated. Almost half (48 percent) of the recovered post-consumer textile waste is recycled as secondhand clothing, which is typically sold to third-world nations. Approximately 20 percent of the material processed becomes wiping and polishing cloths. Finally, 26 percent of this post-consumer waste is converted into fiber to be used in products similar in nature to those manufactured from pre-consumer textile waste.

## 3. Carpet Waste and Composition

During carpet manufacturing, the edges of a tufted carpet need to be trimmed and the face yarns sheared. This waste is approximately 60% edge trim and 40% shear lint. In the Dalton, Georgia, region where there is a high concentration of carpet manufacturing activities, over 20 thousand tons of carpet manufacturing waste is generated every year. The carpet waste from the fitting process is concentrated in the automotive and prefabricated housing industries [11,12]. During the fitting process, the carpet is formed and cut into various irregular shapes, and waste is generated as a result. The largest amount of carpet waste, however, is from the discarded post-consumer carpet. It is estimated that about 2.3 million tons of carpet and rugs were placed in the municipal solid waste stream in 1996, of which only 1% was recovered for recycling.

Carpet is a complex, multi-component system. The tufted carpet, the most common type (90%) as shown in Figure 1, typically consists of two layers of backing (mostly polypropylene fabrics), joined by CaCO<sub>3</sub> filled styrene-butadiene latex rubber (SBR), and face fibers (majority being nylon 6 and nylon 6,6 textured yarns) tufted into the primary backing. The SBR adhesive is a thermoset material, which cannot be remelted or reshaped. The waste containing the SBR (post consumer and some industrial waste) has not found suitable uses and it forms the major part of the carpet waste going into the landfills. Figure 2 shows the typical masses for the various components [13].

Because about 70% of the carpet produced is for replacing old carpet, it is important to understand the amount and types of carpet produced. Using the typical life of a carpet of 5-10 years, one can estimate the amount of carpet being disposed of currently and to be disposed of in the next few years. According to carpet industry statistics [14], the total fiber consumption in 1999 was about 1.7 million tons: Nylon 57%, Olefin 36%, Polyester 7%, and Wool 0.4%. Among the nylon face fiber, about 40% is nylon 6 and 60% is nylon 6,6. In recent years, the use of polypropylene (labeled as polyolefin or olefin) in carpet is increasing, as tabulated in Table 2 [15]. Based on the aforementioned data, one expects the current rate of carpet disposal to be about 2 million tons per year; or about 70% of the shipment in 1996. The amount of the various materials used in carpet manufacturing in the U.S. for the 1996 is given in Table 3.

Nylon generally performs the best among all synthetic fibers as carpet face yarn, but it is also the most expensive. Typical price per kg for the plastic resins are: Nylon \$2.50, Polyester \$1.20, and Polypropylene \$0.75. This price list provides a perspective on the economics of recycling as well. For

example, if it takes the same processing effort to convert the fiber into resin, an operation on nylon would be most profitable. This also explains why most of the recycling effort is on nylon recovery.

## 4. Fiber Recycling Technologies

Many carpet manufacturers, fiber and chemical suppliers, recycling companies, and academic institutions are actively pursuing various methods to recycle fibrous waste. The approaches include chemical processes to depolymerize nylon and other polymers, recovery of plastic resins from carpet fibers, direct extrusion of mixed carpet waste, composites as wood substitutes, fibers for concrete and soil reinforcement, waste-to-energy conversion, and carpet as feedstock for cement kilns.

#### 4.1. Depolymerization of Nylons

Because of the higher value of nylon resin in comparison with other polymers used in carpet, nylon carpet has been looked at as a resource for making virgin nylon via depolymerization. The majority of polyamides used commercially are nylon 6,6 or nylon 6, and the largest supply of waste for recycling of nylons is obtained from used carpets. The waste carpets are collected, sorted and then subjected to a mechanical shredding process before depolymerization.

### Hydrolysis of Nylon 6

A process for depolymerizing nylon 6 scrap using high pressure steam was patented by Allied Chemical Corporation in 1965 [16], and subsequent patents by AlliedSignal, Inc. were obtained [17,18]. Ground scrap was dissolved in high-pressure steam at 125-130 psig (963-997 kPa) and 175-180<sup>0</sup>C for 0.5 hour in a batch process and then continuously hydrolyzed with super-heated steam at 350<sup>0</sup>C and 100 psig (790 kPa) to form ε-caprolactam at an overall recovery efficiency of 98%. The recovered monomer could be repolymerized without additional purification. Braun et al. [13] reported the depolymerization of nylon 6 carpet in a small laboratory apparatus with steam at 340<sup>0</sup>C and 1500 kPa (200 psig) for 3 hours to obtain a 95% yield of crude ε-caprolactam of purity 94.4%. Recently, patents were issued to AlliedSignal for the depolymerization of polyamide-containing carpet [19,20].

Acid hydrolysis of nylon 6 wastes [21,22] in the presence of superheated steam has been used to produce aminocaproic acid which under acid conditions is converted to  $\varepsilon$ -caprolactam, and several patents have been obtained by BASF [23,24]. Acids used for the depolymerization of nylon 6 include inorganic or organic acids such as nitric acid, formic acid, benzoic acid, and hydrochloric acid [25,23]. Orthophosphoric acid [24] and boric acid are typically used as catalysts at temperatures of 250-350 $^{\circ}$ C. In a typical process, superheated steam is passed through the molten nylon 6 waste at 250-300 $^{\circ}$ C in the presence of phosphoric acid. The resulting solution underwent a multistage chemical purification before concentration to 70% liquor, which was fractionally distilled in the presence of base to recover pure  $\varepsilon$ -caprolactam. Boric acid (1%) may be used to depolymerize nylon 6 at 400 $^{\circ}$ C under ambient pressure. A recovery of 93-95%  $\varepsilon$ -caprolactam was obtained by passing superheated steam through molten nylon 6 at 250-350 $^{\circ}$ C [23].

Sodium hydroxide has been used successfully as a catalyst for the base-catalyzed depolymerization of nylon 6. At  $250^{\circ}$ C, a pressure of 400 Pa, and a sodium hydroxide content of 1%, the yield of  $\varepsilon$ -caprolactam was 90.5% [26].

## Catalytic pyrolysis

Catalytic pyrolysis has been studied as a hybrid process for recovering caprolactam from nylon 6 followed by high temperature pyrolysis of the polypropylene into a synthetic natural gas. Czernik *et al* [27] investigated the catalysis of the thermal degradation of nylon 6 with an  $\alpha$ -alumina supported KOH catalyst in a fluidized bed reactor. In the temperature range of 330°C to 360°C the yield of caprolactam exceeded 85%.

Bockhorn *et al* [28] use a liquid catalyst composed of a eutectic mixture of 60 mol% NaOH and 40 mol% KOH which melts at 185°C. At 290°C the caprolactam yield exceeded 95%. At this temperature the polypropylene is not degraded significantly. Based on a preliminary feasibility study, this process could be economically viable [29].

## Recovery of Caprolactam

Approximately 10-12% by weight of oligomers is formed in the synthesis of polycaprolactam (nylon 6). These oligomers are removed by extraction with water or by distillation under vacuum. In the process, two types of liquid wastes are formed: (1) a 4-5% aqueous solution of low-molecular weight compounds, consisting of ca. 75% by weight of caprolactam and ca. 25% by weight of a mixture of cyclic and linear caprolactam oligomers and (2) a caprolactam-oligomer melt, containing up to 98% caprolactam and small amounts of dimer, water, and organic contaminants. The recycle of caprolactam involves two different stages: depolymerization of polymeric waste and purification of the caprolactam and oligomers obtained.

A general recovery of caprolactam from liquid waste generates 20-25% oligomers along with organic and inorganic compounds as impurities. The distillation of caprolactam under reduced pressure produces a residue which consists of inorganic substances such as permanganates, potassium hydrogen sulfate, potassium sulfate, sodium hydrogen phosphate, and sodium phosphate. The larger portion of the residue contains cyclic and linear chain oligomers plus 8-10% of caprolactam. The types and exact amounts of impurities depends on the method used for the purification and distillation of caprolactam.

The cyclic oligomers are only slightly soluble in water and dilute solutions of caprolactam. They tend to separate out from the extracted waste during the process of concentration and chemical purification of the caprolactam. The cyclic oligomers tend to form on the walls of the equipment used in the process equipment. 6-Aminocaproic acid or sodium 6-aminocaproate may also be found in the oligomeric waste especially if sodium hydroxide is used to initiate the caprolactam polymerization.

Many impurities are present in commercial caprolactam which pass into the liquid wastes from polycaprolactam (PCA) manufacture from which caprolactam monomer may be recovered. Also, the products of the thermal degradation of PCA, dyes, lubricants, and other PCA fillers may be contained in the regenerated caprolactam. Identification of the contaminants by IR spectroscopy has led to the detection of lower carboxylic acids, secondary amines, ketones, and esters. Aldehydes and hydroperoxides have been identified by polarography and thin-layer chromatography.

Caprolactam is a thermally unstable compound which on distillation may form methyl-, ethyl-, propyland n-amylamines. Also, at high temperatures, caprolactam reacts with oxygen to form hydroperoxides which in the presence of iron or cobalt ions are converted into adipimide. N-alkoxy compounds are also formed by the reaction of caprolactam with aldehydes during storage.

Therefore, caprolactam and the depolymerized product from which caprolactam is regenerated contain various impurities, which are present in widely fluctuating amounts depending on the processes involved.

In particular, the presence of cyclohexanone, cyclohexanone oxime, octahydrophenazine, aniline, and other easily oxidized compounds effects the permanganate number. Also volatile bases such as aniline, cyclohexylamine, cyclohexanol, cyclohexanone, nitrocyclohexanone, and aliphatic amines may be present in the caprolactam.

Caprolactam must be very carefully purified to exclude small concentrations of (1) ferric ions, which would catalyze the thermal oxidative degradation of polycaprolactam and (2) aldehydes and ketones, which would markedly increase the oxidizability of caprolactam. The impurities in caprolactam may retard the rate of caprolactam polymerization as well as having a harmful effect on the properties of the polymer and fiber.

In the vacuum depolymerization of nylon 6, a catalyst must be used because in the absence of a catalyst, by-products such as cyclic olefins and nitrides may form which affects the quality of the caprolactam obtained [3].

The caprolactam obtained must meet the specifications of permanganate number, volatile bases, hazen color, UV transmittance, solidification point, and turbidity, in order to be used alone or in combination with virgin caprolactam [25]. Reported caprolactam purification methods include recrystallization, solvent extraction, and fractional distillation. One solvent extraction technique involves membrane solvent extraction. Ion-exchange resins have been shown to be effective in the purification of aqueous caprolactam solutions. In one such process, the oily impurities are removed by extraction with organic solvents, followed after treatment with carbon at 60-80°C. Cationic and anionic exchange resins are then used to complete the purification process. Ion-exchange resins remove all ionic impurities as well as colloidal and floating particles i.e. alkali metal salts formed in permanganate treatment are removed during ion-exchange treatment. Also the treatment of aqueous solutions of caprolactam with ion-exchange resins helps to remove the distillation residue. Treatment of caprolactam with activated carbon helps to remove anionic and cationic impurities.

Impurities in caprolactam have also been destroyed by oxidation with ozone followed by distillation. Ozonation treatment of waste caprolactam leaves no ionic impurities. However, the most commonly used oxidizing agents are potassium permanganate, perboric acid, perborate and potassium bromate. Treatment of caprolactam with these oxidizing agents is carried out in a neutral medium at 40-60°C. Strongly alkaline or acidic conditions accelerate the oxidation of caprolactam to form isocyanates. The undesirable oxidation reaction is fast above pH 7 because of the reaction with isocyanate to form carbamic acid salts which shifts the equilibrium to form additional isocyanate.

In a typical process, potassium permanganate is used to treat the cracked liquor exiting the depolymerization plant without any pH adjustment. The liquor is usually acidic because it contains some of the phosphoric acid depolymerization catalyst. The KMnO<sub>4</sub> treatment is followed by treatment of the caprolactam aqueous solution with carbon followed by filtration. Next the filtered 20-30% caprolactam aqueous solution is concentrated to 70% and the pH is adjusted to 9-10 by addition of sodium hydroxide. The caprolactam alkaline concentrate is treated with KMnO<sub>4</sub> followed by distillation under reduced pressure to remove water and low boiling impurities.

Also the caprolactam aqueous solution may be hydrogenated at 60°C in the presence of 20% sodium hydroxide and 50% palladium absorbed on carbon to provide caprolactam of very high purity after distillation. Treatment with an ion-exchange resin before or after the oxidation or hydrogenation process also improves the quality of the caprolactam obtained after distillation.

Caprolactam has also been purified by treatment with alkali and formaldehyde followed by fractional distillation to remove aromatic amines and other products.

Also, nylon 6 waste may be hydrolyzed in the presence of an aqueous alkali metal hydroxide or acid to produce an alkali metal or acid salt of 6-aminocaproic acid (ACA). The reaction of nylon 6 waste with dilute hydrochloric acid is very fast at 90-100°C. The reaction mixture is poured into water to form a dilute aqueous solution of the ACA salt. Filtration is used to remove undissolved impurities such as pigments, additives, and fillers followed by treatment of the acid solution with a strong cation-exchange resin. A sulfonic acid cationic-exchanger absorbs ACA and pure ACA is eluted with ammonium hydroxide to form a dilute aqueous solution. Pure ACA is obtained by crystallization of the solution.

Alternatively, nylon 6 waste may be hydrolyzed with aqueous sodium hydroxide and the sodium salt of ACA converted into pure ACA by passing the aqueous solution through an anion-exchange resin.

## Applications of Depolymerized Nylon 6

Chemical recycling of nylon 6 carpet face fibers has been developed into a closed-loop recycling process for waste nylon carpet [25,30,31,32]. The recovered nylon 6 face fibers are sent to a depolymerization reactor and treated with superheated steam in the presence of a catalyst to produce a distillate containing caprolactam. The crude caprolactam is distilled and repolymerized to form nylon 6. The caprolactam obtained is comparable to virgin caprolactam in purity. The repolymerized nylon 6 is converted into yarn and tufted into carpet. The carpets obtained from this process are very similar in physical properties to those obtained from virgin caprolactam.

The "6ix Again" program of the BASF Corp. has been in operation since 1994. Its process involves collection of used nylon 6 carpet, shredding and separation of face fibers, pelletizing face fiber for depolymerization and chemical distillation to obtain a purified caprolactam monomer, and repolymerization of caprolactam into nylon polymer [32].

Evergreen Nylon Recycling LLC, a joint venture between Honeywell International and DSM Chemicals, was in operation from 1999 to 2001. It used a two-stage selective pyrolysis process. The ground nylon scrap is dissolved with high-pressure steam and then continuously hydrolyzed with super-heated steam to form caprolactam. The program has diverted over one hundred thousand tons of post consumer carpet from the landfill to produced virgin-quality caprolactam [30,31].

#### Hydrolysis of Nylon 6,6 and Nylon 4,6

The depolymerization of nylon 6,6 and nylon 4,6 involves hydrolysis of the amide linkages which are vulnerable to both acid- and base-catalyzed hydrolysis. In a patent granted to the DuPont Company in 1946, Myers [33] described the hydrolysis of nylon 6,6 with concentrated sulfuric acid which led to the crystallization of adipic acid from the solution. Hexamethylene diamine (HMDA) was recovered from the neutralized solution by distillation. In a later patent assigned to the DuPont Company by Miller [34], a process was described for hydrolyzing nylon 6,6 waste with aqueous sodium hydroxide in isopropanol at 180°C and 2.2 MPa pressure. After distillation of the residue, HMDA was isolated and on acidification of the aqueous phase, adipic acid was obtained in 92% yield. Thorburn [35] depolymerized nylon 6,6 fibers in an inert atmosphere at what was reported to be a superatmospheric pressure of up to 1.5 MPa and at a temperature in the range of 160-220°C in an aqueous solution containing at least 20% excess equivalents of sodium hydroxide.

Polk et al [36] reported the depolymerization of nylon 6,6 and nylon 4,6 in aqueous sodium hydroxide solutions containing a phase transfer catalyst. Benzyltrimethylammonium bromide was discovered to be an effective phase-transfer catalyst in 50% sodium hydroxide solution for the conversion of nylon 4,6 to oligomers. The depolymerization efficiency (% weight loss) and the molecular weight of the reclaimed oligomers were dependent on the amount and concentration of the aqueous sodium hydroxide and the reaction time. Table 4 exhibits the effects of experimental conditions on the depolymerization efficiency and the average molecular weight of the oligomers. The viscosity-average molecular weight was calculated from the Mark-Houwink equation:  $[\eta] = KM_v^a$ , where  $M_v$  is the viscosity-average molecular weight,  $K = 4.64 \times 10^{-2} \text{ dL/g}$  and  $a = 0.76 \text{ at } 25^{\circ}\text{C}$  in 88% formic acid. Nylon 4,6 fibers ( $M_v = 41,400 \text{ g/mole}$ ) did not undergo depolymerization on exposure to 100 mL of 25 wt% sodium hydroxide solution at  $165^{\circ}\text{C}$ . Out of 6.0 g of nylon fibers fed for depolymerization, 5.95 g were unaffected. When the concentration of sodium hydroxide was increased to 50 wt%, the depolymerization process resulted in the formation of low molecular weight oligomers. Hence, even in the presence of a phase transfer agent, a critical sodium hydroxide concentration exists between 25 and 50 wt% which is required to initiate depolymerization under the conditions used. Soluble amine salts, were also obtained.

In order to establish the feasibility of alkaline hydrolysis in respect to recycling of nylon 4,6, it was necessary to determine whether the recovered oligomers could be repolymerized to form nylon 4,6. For this purpose, solid state polymerization was performed on nylon 4,6 oligomers formed via alkaline hydrolysis with 50 wt% NaOH at 165°C for 24 hours. The solid state polymerization process was carried out in a round bottom flask at 210°C for 16 hours under vacuum. Solid state polymerization of the nylon 4,6 oligomers resulted in an increase in intrinsic viscosity from 0.141 to 0.740 dl/g. That corresponds to an increase in viscosity average molecular weight from 1846 g/mole to 16,343 g/mole. In theory, higher molecular weights would be obtained by heating for a longer time interval.

The product of the depolymerization of nylon 6,6 with 50% aqueous sodium hydroxide solution was relatively low molecular weight oligomers. A series of experiments were run in order to examine the applicability and efficiency of benzyltrimethylammonium bromide [BTEMB]as a phase transfer catalyst in the depolymerization of nylon 6,6. Table 4 shows the effect of the feed ratio of the nylon 6,6 to BTEMB on the viscosity-average molecular weight of the depolymerized nylon. The product of the run with no phase transfer agent showed a 15.9% increase in weight compared to the weight of the original nylon 6,6. The calculated percent increase in weight for a 19-fold decrease in molecular weight (due to the addition of water) would be ca. 1%. Therefore, a large part of the increase must be due to leaching of silicates of the glass container (resin reaction kettle) by the strong alkali (50 wt%) at the temperature of the reaction (130°C) over 24 hours. The oligomer obtained had a viscosity average molecular weight of 1644 g/mole (the original nylon 6,6 had a molecular weight of 30,944 g/mole). The runs with phase transfer agent produced oligomers with decreases in weight of 40-50%. Although the occurrence of leaching of silicates from the glass container made quantitative assessment difficult, these results suggested that in the absence of phase transfer agent only oligomers are formed; however, soluble low molecular weight products are formed in the presence of phase transfer agent. The oligomers obtained were repolymerized in the solid state by heating at 200°C in a vacuum. The viscosity-average molecular weight of the solid state polymerized nylon 6,6 obtained was ca. 23,000 g/mole (the molecular weight of the oligomeric mixture was 1434 g/mole).

In order to isolate adipic acid, nylon 6,6 fibers were depolymerized under reflux with a 50% NaOH solution in the presence of catalytic amounts of benzyltrimethylammonium bromide. The oligomers formed in successive steps were depolymerized under similar conditions. The yields in steps 1, 2, and 3 were 57.8%, 38.7%, and 100% theoretical. However, hexamethylene diamine was not isolated. The overall yield of adipic acid was 59.6%.

## Ammonolysis of Nylon 6,6

Ammonolysis currently is the preferred route currently in use at the DuPont Company for the depolymerization of nylon 6,6 carpet waste [7,37]. McKinney [38] has described the reaction of nylon 6,6 and nylon 6,6/nylon 6 mixtures with ammonia at temperatures between 300 and 350°C and a pressure of about 68 atmospheres in the presence of an ammonium phosphate catalyst to yield a mixture of the following monomeric products: HMDA, adiponitrile, and 5-cyanovaleramide from nylon 6,6 and ε-caprolactam, 6-aminocapronitrile, and 6-aminocaproamide from nylon 6. The equilibrium is shifted toward products by continuous removal of water formed. Most of the monomers may be transformed into HMDA by hydrogenation. Kalfas [39] has developed a mechanism for the depolymerization of nylon-6,6 and nylon-6 mixtures by the ammonolysis process. The mechanism includes the amide bond breakage and amide end dehydration (nitrilation) reactions, plus the ring addition and ring opening reactions for cyclic lactams present in nylon 6. On the basis of the proposed mechanism, a kinetic model was developed for the ammonolysis of nylon mixtures.

Bordrero, *et al.* [40] utilized a two step ami/ammonolysis process to depolymerize nylon 6,6. The first step is based on an amminolysis treatment of nylon 6.6 by n-butylamine at a temperature of 300°C and a pressure of 45 atm. Free HMDA and NN'-dibutyladipamide are generated. The second step is ammonolysis of NN'-dibutyladipamide at a temperature of 285°C and a pressure of 50 atm. The end product is adiponitrile (ADN). It is estimated that the yields could be about 48% for ADN and about 100% for HMDA at optimized reaction conditions.

#### Recovery of Nylon 6,6 Monomers

Adipic acid and hexamethylene diamine (HMDA) are obtained from nylon 6,6 by the hydrolysis of the polymer in concentrated sulfuric acid (Figure 3). The adipic acid is purified by recrystallization and the HMDA is recovered by distillation after neutralizing the acid. This process is inefficient for treating large amounts of waste because of the required recrystallization of adipic acid after repeated batch hydrolyses of nylon 6,6 waste. In a continuous process [25], nylon 6,6 waste is hydrolyzed with an aqueous mineral acid of 30-70% concentration and the resulting hydrolysate is fed to a crystallization zone. The adipic acid crystallizes and the crystals are continuously removed from the hydrolysate. Calcium hydroxide is added to neutralize the mother liquor and liberate the HMDA for subsequent distillation.

Continuous recovery requires adipic acid crystals having an average diameter of ca. 40-50 nm. Such crystals are obtained by continuously introducing the hot hydrolysate containing 10-20% adipic acid into an agitated crystallization vessel while maintaining an average temperature of 20-30°C. The slurry obtained from the crystallization vessel is filtered to collect the adipic acid crystals and the filtrate which contains the HMDA acid salt is continuously neutralized with calcium hydroxide. The calcium salt formed is removed by filtration and the HMDA in the filtrate is isolated by distillation.

In the case of nylon 6,6 waste recycled by ammonolysis, nylon is treated with ammonia in the presence of a phosphate catalyst. Reaction occurs at 330°C and 7 MPa. Distillation of the reaction mixture produces ammonia which is recycled and three fractions containing (a) caprolactam, (b) HMDA and aminocapronitrile and (c) adiponitrile. Aminocapronitrile and adiponitrile are hydrogenated to yield pure HMDA and the caprolactam is either converted to aminocapronitrile by further ammonolysis or distilled to produce pure caprolactam. The HMDA produced by this process is extremely pure (>99.8) [3]. The main impurities are aminomethylcyclopentylamine and tetrahydroazepine which are expected to be removed more effectively in the larger distillation columns employed in larger plants.

#### 4.2. Fiber Identification and Sorting

For many recycling processes such as nylon depolymerization and polymer resin recovery, it is desirable or required to sort the feedstock according to the fiber type. For carpet, the sorting is according to the type of the face fiber. Melt point indicator is an inexpensive instrument that can identify most fiber types, but it is generally slow and cannot distinguish between nylon 6,6 and polyester. Infrared spectroscopy is a must fast and accurate technology. A typical instrument consists of an A/C powered base unit for data acquisition, analysis and display, and a probe connected to the based unit via a fiber-optical cable. Such units are suitable for carpet sorting in a central warehouse. A portable infrared spectrometer has been developed by Kip et al [41], which is a lightweight, battery operated unit. It is designed to identify the common carpet face fibers: nylon 6, nylon 6,6, polypropylene, polyester, and wool. Unidentifiable fibers, either due to operating conditions or fiber types other than those in the above list, would be shown as "unknown".

### 4.3. Mechanical Separation of Polymers from Carpet

Mechanical methods have been utilized to separate carpet components. One or more segregated components then are recycled into products that generally compete with products produced from virgin polymers.

In a process developed by DuPont [7,42], nylon 6,6 carpet first is passed through dry processes consisting of a series of size reduction and separation steps. This provides a dry mix of 50-70% nylon, 15-25% polypropylene and 15-20% latex, fillers and dirt. Water is added in the second step where the shredded fiber is washed and separated using the density differences between the fillers, nylon and polypropylene. Two product streams are obtained: one 98% pure nylon and the other is 98% pure polypropylene. The recycled nylon is compounded with the virgin nylon at a ratio of 1:3 for making automotive parts.

The United Recycling process [43,44] starts with clipping the face fibers on loop carpet to open the loops. The next step is debonding, in which the carpet is bombarded with a combination of air and steam to loosen the calcium carbonate-filled latex backing. The secondary backing then is peeled off mechanically, exposing the primary polypropylene backing. Next mechanical picks pluck the face fibers. It is claimed that the cost of this process is low and that it yields a product stream with 93-95% pure face fibers. Other devices employing water jet [45] or mechanical actions [46,47,48,49,50,51,52,53] for size reduction and separation of carpet have also been reported. Many types of equipment are commercially available for processing textile and carpet waste [54].

A process has been developed to separate carpet waste using a cold, dry abrasive step [55]. Dry ice pellets are shot into an abrasive zone as a segment of discarded carpet on a conveyor system is stripped apart and disassembled. The dry ice pellets freeze the binder material (usually latex), lowering it to a temperature that makes the binder brittle and easy to break apart. The dry ice pellets sublimate directly into gas without any liquid residues. This process eliminates the need for a drying operation, which saves energy and avoids potential chemical pollution.

#### 4.4. Physical/Chemical Separation of Polymers from Carpet

Solvent extraction has also been used to separate the high value nylon from carpet waste. The solvents used are aliphatic alcohol [56], alkyl phenols [57] and hydrochloric acid [58]. In the process developed by Booij, *et al.* [56], the carpet waste is shredded into 0.5-20 cm<sup>2</sup> pieces. Then the carpet pieces are mixed with the extraction agent, such as methanol. The weight ratio of solvent to carpet waste is generally 5 to 20. An extraction time of 60 minutes is found to be sufficient to dissolve nylon 6 at a

temperature of 135-140°C and a pressure of 0.2-2 MPa. Solids are filtered out, the solution cooled down and the nylon 6 precipitated. The nylon obtained has at least 90% of the relative viscosity of the nylon present in the carpet waste, indicating that no serious degradation takes place in the extraction process. In addition, the yield of nylon is high (above 90%). The drawbacks of solvent extraction are the chemicals involved, modest temperature and pressures required, and time required. In comparison, the use of hydrochloric acid [58] as a solvent requires lower temperature (20 to 100°C) and shorter dissolution time (2 to 30 minutes.). Based on relative viscosity data, no degradation of the recycled nylon is observed. However, hydrochloric acid solvent is not recyclable due to its reaction with the calcium carbonate filler in the carpet waste.

Another approach to separate carpet components is to use a supercritical fluid (SCF) method [59,60]. The solubility of the polymer changes with the variation in pressure and temperature of the SCF. Sikorski [59] disclosed that the individual polymers in carpet could be extracted sequentially using a SCF such as CO<sub>2</sub> by increasing temperature and pressure. However, high temperatures (170-210°C) and pressures (500 to 1000 atm) are required to dissolve the various polymers in the SCF solvent. A recent development enables the separation of carpet waste at close to room temperature and moderate pressure [60]. Up to 2.3 w% nylon was dissolved in an 88 wt% formic acid solution. Then supercritical CO<sub>2</sub> as an anti-solvent was added to precipitate the nylon out of solution at a temperature of 40°C and a pressure between 84 and 125 atm. Both the solvent and the anti-solvent can be recycled. The whole process is very controllable and the resultant nylon is of high quality.

#### 4.5. Plastic Resin Compounds From Waste

Most carpet waste contains two immiscible plastics, nylon and polypropylene. The immiscibility of these two components leads to poor mechanical properties. When carpet is recycled using melt blending, compatbilizers are used to improve the properties of the blends [61,62,63,64,65,66,67].

United Recycling Inc. (URI), which was in operation from the early 1990s to 1999, introduced two extruded blends (URI 20-001 and URI 10-001) from post consumer carpet waste for injection molding in 1993 [61]. These were the first commercial recycled carpet compounds. The process used both polypropylene and nylon carpet. Their products were described as proprietary blends containing polypropylene. The composition and properties of the two compounds are listed in Table 5. The molding compounds developed by URI were used to extrude carpet tack strip.

In 1994, Monsanto patented a process to recycle all the components of post consumer nylon 6,6 carpet, without separation, into a filled thermoplastic product suitable for injection molding [62, 63]. They used a twin screw extruder to accomplish high intensity mixing of the thermoplastic from carpet samples. The recycled material contained 35-67 wt% nylon, 8-21 wt% polypropylene, 5-29 wt% SBR and 10-40 wt% inorganic filler. In one study, no compatibilizer was used [62]. The carpet samples were fed directly into a twin screw extruder operating at about 250-260°C and at a shear rate of 200 to 400 s<sup>-1</sup>. The tensile and Izod strengths of the extruded carpet were compared with those of the control sample, virgin polystyrene (Table 6). The properties of the directly extruded thermoplastic were comparable to those of the virgin polystyrene. However, the properties of the extruded carpet were lower than virgin nylon 6,6 [68]. In their subsequent work [63], a maleic anhydride grafted polypropylene, PolyBond 3150, was added to compatibilize the nylon and polypropylene in the carpet waste. Addition of 3 wt% PolyBond 3150 resulted in a tensile strength of about 40 MPa, tensile modulus of 3.1 GPa and Izod impact strength of 35 J/m. The elongation at break was low, about 2.3%. These properties appeared promising enough for the product to compete with some virgin polymers.

Studies have been carried out on the compatibilization of polypropylene and nylon using maleated polypropylene, PolyBond 3002 [64]. An addition of 3 wt% of PolyBond 3002 to the nylon/polypropylene carpet increased the blend tensile strength to the level of virgin nylon 6,6. In aother study [65], a compatibilizer, Kraton (Kraton is a trademark of Shell Company), and a toughening agent, styrene-ethylene/butylene-styrene block copolymer (SEBS) were also used in the extrusion process. It was found that the addition of compatibilizer or toughening agent improved the strain to failure. The addition of Kraton increased the strain and the work of rupture significantly while the tensile strength was decreased slightly (Table 7). This decrease in tensile strength was attributed to the elastomeric character of Kraton.

Young, *et al.* [66] patented a recycling method of preparing a polymeric blend formed from carpet scrap through the use of selected compatiblizing agents - PolyBond, Kraton or poly (ethylene-co-vinyl acetate) (EVA). The carpet scrap was from automotive carpet and the composition of the waste was significantly different from that of the residential carpet waste. The automotive carpet scrap contained 14 wt% nylon 6,6, 4 wt% PP, 11 wt% EVA, and 71 wt% BaSO<sub>4</sub> filled EVA. The addition of the compatiblizing agents significantly improved the mechanical properties. For example, PolyBond 1001, even at 2 wt%, increased the tensile strength from 4.7 to 9.1 MPa, elongation at break from 7.6 to 17.5% and Izod impact strength (un-notched) from 254 to 471 J/m.

Zegler and Weinle [69] developed a process to make the secondary backing for new carpet from the shredded carpet waste. The carpet waste could contain nylon 6, nylon 6,6, polyvinyl chloride, vinyl copolymer, polypropylene and polyethylene. The content of nylon was about 15 to 50 wt%. The chopped carpet waste was extruded at a temperature of 215 °C, then calendared and bonded to a primary backing of the new carpet through an adhesive coating.

## 4.6. Use of Recycled Polymers in Glass Fiber Reinforced Composites

Glass fiber reinforcement can be used to enhance the properties of melt processed carpet. In the study by Hagberg, *et al.* [63], carpet waste was first compatibilized with 10% maleic anhydride grafted polypropylene (PP-g-MA), and then compounded with 15 or 30% glass in an extrusion process as shown in Figure 4. The average composition of the carpet waste is 50/30/15/5:nyon/CaCO<sub>3</sub>/PP/SBR. Mechanical properties of the glass filled and unfilled carpet waste are compared in Table 8. The addition of 30% glass fibers improved the tensile strength by about 180%, tensile modulus by about 190% and Izod impact strength by about 130%. The properties of the glass filled carpet waste are competitive with several commercial resins.

Mantia, *et al*, [70] used short glass fibers to reinforce a PET/HDPE mixture (Table 9), which is one of the typical compositions in packaging waste. The mechanical properties of the blends, except for the elongation at break, are enhanced with the increase of glass fiber content. The addition of 20 wt% glass in PET/HDPE blends increased the tensile modulus by 50%, tensile strength by 110%, and impact strength by 70%. In addition, the heat distortion temperature of the blend was remarkably improved.

One of the important applications of polypropylene is to make glass mat reinforced thermoplastic (GMT) composites. Such composites are extensively used in the automotive industry. Both carpet edge trim and polypropylene separated from carpet waste are effective substitutes for virgin polypropylene in GMT (Table 10) [71]. The Azdel PM 10400 is a commercial GMT based on virgin polypropylene. Compared to short fiber reinforced thermoplastics these GMT materials are both rigid and tough. Their primary application is in automotive bumper beams. This approach to recycling carpet waste has been patented [72]. Also, the material is produced commercially by Georgia Composites, Inc. [73].

#### 4.7. Polymer Composites Utilizing Waste Fibers as Reinforcement

Kotliar et al [74,75,76] have explored the use of carpet face yarn and textiles as a fibrous filler for a composite or laminate. Because of the fine diameter of the fibers involved, a low viscosity prepolymer in a water base was used to insure complete coverage of the fibers. Adhesives were selected to result in a high modulus and creep resistant material with good weathering characteristics.

The work emphasized shredded carpet selvage to which various amounts of cut waste fibers such as nylon 6, nylon 6,6, polyester and cotton were added. Fabric bits of waste denims and cotton-polyester fabrics were also used. The waste carpet blend was then coated with phenolic or urea formaldehyde resins that were dispersed in a water base. The composites contained various amounts of different fibers or fabrics and 7.5 to 20 wt% adhesive solids with respect to the fiber content. The fibers were spray coated and molded in a heated press at 150 to 200°C and 3.4 MPa. Test results show that one can achieve high flexural moduli of 2.4-2.8 GPa with face yarn, i.e., fibers that bind to the matrix such as nylon, polyester and cotton. These values together with flexural strengths of 34-48 MPa make the products suitable for many outdoor and transportation applications.

Laminates directly from waste carpet pieces were also made by coating the face yarn with a phenol formaldehyde resin and molding the carpet pieces back to back with the face yarn on the outside to achieve a high flexural modulus [75]. Holes were punched into the carpet prior to spray coating the face yarn so that protrusions of the matrix material could flow into the backing during the molding process to avoid shear delanination. Additional work has been done to make honeycomb sandwich structures for high flexural stiffness and light weight.

Gowayed, *et al.* [77] explored the use of edge trim of polypropylene (PP) fabric waste (from the carpet backing) to reinforce a polyethylene (PE) matrix. Four layers of 0.1 mm thick PE film were laid with a single layer of washed PP fabric waste. Then they were molded at a temperature of 150°C and a pressure of 290 kPa. It was found that the resulting PE/PP composite, with 25% PP volume fraction, exhibited a three-time increase in tensile strength and a 60% increase in flexural modulus when compared to the properties of pure PE. The composite developed can be used as a lining material for corrosive mixtures.

#### 4.8. Waste Fibers as Reinforcement in Concrete and Soil

#### Fiber Reinforced Concrete

Concrete is the most heavily used construction material in the world. Adding a small fraction (usually 0.5-2% by volume) of short fibers to the concrete mix can increase the toughness (energy absorption) of concrete by orders of magnitude. Reduced shrinkage cracking has been observed even with fiber volume fractions as low as 0.1% of polypropylene fibers. Besides reducing the need for landfilling, the use of waste fiber for concrete reinforcement could lead to improved infrastructure with better durability and reliability. Potential applications could include pavements, columns, bridge decks and barriers, and for airport construction as runways and taxiways.

In a study on concrete reinforcement with carpet waste fibers [78,79], recycled carpet waste fibers about 12 to 25 mm in length and fiber volume fractions of 1% and 2% were used. FiberMesh, a virgin polypropylene (PP) fiber (19 mm long), at 0.5% and 1% volume fractions was included for comparison.

Four point flexural test and cylinder compressive test were conducted on a hydraulic testing machine and the results are given in Table 11. 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 FRCs with 2% carpet waste fibers 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.

The flexural strengths of all mixes tested were essentially the same and the standard deviations were low. 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 (Figure 5), 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. The flexural toughness indices ( $I_5$  and  $I_{20}$ ) were calculated according to ASTM C1018.

Shaw Industries, Inc., the largest carpet manufacturer in the world, constructed a 11,000 m<sup>2</sup> R&D Center in Dalton, Georgia, which used concrete reinforced with carpet waste fibers in the construction project [86]. The amount of waste fiber included was 5.95 kg/m<sup>3</sup>, and about 20 tons of carpet production waste was consumed in the project. Mixing was done by adding fibers to the mixing truck directly, after which the fibers were found to be uniformly dispersed in the concrete without balling or clumping. Mixing, pouring, and finishing followed standard procedures, used conventional equipment, and went smoothly. The compressive and flexural strengths exceeded specifications, and reduced shrinkage cracking was observed. Such concrete containing waste fibers was used for 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.

Other studies on the use of polypropylene fibers from carpet waste in concrete [80], used tire cords in concrete [81,82], and using recycled nylon fibers to reduce plastic shrinkage cracking in concrete [83] have also been reported and reviewed [84]. Gordon, *et al.* [85] used the waste nylon fibers and ground carpet to stabilize asphalt concrete. Increase of asphalt content in asphalt concrete is favorable because it leads to more durable roads. But it is limited by the resultant flushing and bleeding of pavements and possible permanent deformation of the pavement. Addition of 0.3 wt% waste fibers increased the allowed asphalt content by 0.3 to 0.4 wt%.

## Fiber Reinforced Soil

Studies reported in the literature have shown that fiber reinforcement can improve the properties of soil, including the shear strength, compressive strength, bearing capacity, post-peak load strength retention, and the elastic modulus. Recycled fiber reinforced soil was studied by laboratory evaluation, field trials, and ranking of potential applications [86,87,88]. Carpet waste, apparel waste, and virgin fibers were used for this study at different dosage rates. Additionally, the effect confining pressure and saturation were investigated. Compaction tests were performed to determine the moisture density relationships of the fiber-reinforced soil.

The tests were conducted on a silty to clayey sand. The soil fiber mixture was allowed to hydrate for 24 hours prior to compaction and triaxial testing. The triaxial test specimens were compressed hydraulically with a static-loading machine in a metal split mold. To determine the effect of reinforcing fibers on the moisture density relationship, standard compaction tests (ASTM D 698) were conducted for soil reinforced with carpet fibers and polypropylene fibers at fiber contents of 1, 2, and 3%. It was observed that the reinforcing fibers impeded the compaction process, and thus increasing the fiber content had the same effect as reducing the compactive effort (energy). With the addition of fibers, more water was required to lubricate the soil grains during compaction, resulting in a higher optimal moisture content.

The triaxial compression tests were performed on as-compacted and soaked samples. Fiber type, fiber content and confining pressure were varied and for each specimen the dry density and moisture content was maintained at 1597 kg/m<sup>3</sup> (100 lb/ft<sup>3</sup>) and 19.0%, respectively. In the unconfined compression tests performed on soil reinforced with carpet fiber, polypropylene fiber, and apparel fibers, the peak compressive stress increases with increasing fiber content for all three fiber types with the exception of the 0.3% polypropylene reinforced specimen which showed a decrease in peak compressive stress. Fiber reinforcement also resulted in a reduction of post peak strength loss with increasing fiber content for all fiber types. The control, 0% fiber content specimen exhibits strain-softening behavior whereas strainhardening behavior is exhibited at higher fiber contents. Triaxial compression tests on soil reinforced with carpet fibers under confining pressures of 34.5 kPa and 69.0 kPa were also performed (Figure 6, Table 12). The fiber reinforced specimens showed significant increases in peak stress ranging from over 121% to 303%. More importantly, with increasing fiber content, the soil behavior changed from strain softening to strain hardening. The compressive stress at an axial strain of 10% is given in Table 12. From these tests, it was generally observed that the enhancement of soil properties generally occurs at large deformation levels. At very small strains, the stiffness of the soil is actually decreased due to a reduction in soil compaction.

The specimens in the soaked triaxial compression tests were allowed to absorb water for 48 hours prior to testing. These tests were used to simulate in service saturation that can occur during periods of heavy rainfall or due to other natural or man-made events. The soaked tests showed reduced strength at all strain levels as compared to the as-compacted condition. However, the 1 and 2% fiber content soaked specimens confined at 34.5 kPa and 69.0 kPa, exhibited increases in strength over the unreinforced soaked specimens. Thus, the use of fiber reinforcement can greatly reduce the strength losses associated with in service saturation.

A field study was carried out to demonstrate the feasibility of incorporating carpet waste fibers in road construction [86]. The field trial sites for unpaved county roads were selected to represent typical types of soils found in the State of Georgia. Trial sections with carpet waste fibers and virgin fibers were installed in several Georgia counties. The installation involves ripping the soil to a 150 mm depth, spreading the shredded carpet fibers, blend the fibers into the soil, and smooth and compact the soil. The fibers used were shredded into a length up to 70 mm long, and about 0.33% by weight of fibers were added. It was observed that the fibers could be mixed into soil with reasonable consistency in the field. Assessment of the unpaved roads by visual inspections confirmed that fibers in soil could improve the durability for certain types of soils, and thus reducing the need for frequent regrading. Promising applications for fiber reinforced soil include: levees, landfills, retaining structures backfill, roadway slopes, and sports surfaces.

#### 4.9. Waste to Energy Conversion

The energy content of the waste materials may be recovered, at least in part, by burning the waste materials in air (incineration) [3,89,90,91,92]. Together, about 100 municipal solid waste (MSW) combustion facilities incinerated about 17% (35 million tons) of the municipal solid waste in the United States in 1996 [1,2]. Most of these facilitates have a waste to energy conversion process. Waste containing used paper/wood products, contaminated packaging, and discarded tires has been combusted. The volume of these MSW is reduced by about 75% after incineration. The post-combustion ash still needs to be treated separately and then landfilled. Public concerns exist for the incineration of polymer waste. However, with advanced technologies and proper management, waste-to-energy conversion can be a viable alternative to landfilling. It is estimated that, if all the MSW that is currently generated in the United States were incinerated, the resultant carbon dioxide would be only 2% of that produced from the combustion of all other fossil fuels [90]. The current challenges for the incineration of polymer waste include further improving the incineration efficiency and reducing the harmful end products in the form of

ash and noxious gases. The high combustion energy of polymer waste leads to decreased incineration capacity if the incinerator is heat limited [3].

Carpet waste has a simple composition compared to some other plastic waste streams. The major component in current carpet design is nylon, which requires up to 155 MJ/kg to manufacture, but gives off 29 MJ/kg when burned (see Table 13 [89]). The criterion for energy efficiency for incineration is that if it takes more than twice as much energy to make a plastic than what is recovered by burning, it is better to recycle the plastic than to burn it [61,66]. This criterion clearly favors recycling nylon carpet waste over incineration. In general, the following factors have made incineration a viable option for carpet waste:

- a. Incineration is becoming more acceptable for handling the waste due to more and more restrictions and regulations being put on landfilling waste [93].
- b. The combustion energy values of typical components in carpet are listed in Table 13. These values are comparable to, or higher than, coal (29 MJ/kg). Thus carpet waste is a good candidate for heat generation.
- c. The use of polypropylene in carpet manufacturing is increasing continuously. Polypropylene in carpet manufacturing is about 40% of total fiber used [14,15]. Incineration may be a better option for carpet waste with polypropylene as the major component. Polypropylene requires 73 MJ/kg to make and releases 44 MJ/kg when burned.
- d. The incineration option also can fit with other recycling routes. For example, after the valuable face yarns, such as nylon, have been separated, the remaining components may be gainfully incinerated.

Solid waste such as tires has been use in cement kilns as fuel supplement for making Portland cement. In an Atlanta, Georgia, plant, the use of tires has decrease the plant's air emissions by up to 30%, and allow the company to meet tighter nitrogen oxides (NOx) guidelines [94]. The use of carpet waste in cement kilns is also quite attractive and an effort is being made in this direction [95]. The relatively high fuel value of carpet polymers can reduce the need for fuels, and the calcium carbonate in carpet becomes raw material for cement.

#### 4.10. Carpet Redesign and Reuse

As the search of technology for carpet recycling continues, carpet also is being redesigned for better recyclability. Reuse of reconditioned carpet is being considered for extending its life.

One approach is to make a carpet from one type of material to eliminate the need for separation in recycling. Hoechst-Celanese Corporation manufactures a one-component recyclable carpet from polyester fibers [96,97]. The carpet used un-dyed polyester as the face yarns in the carpet. These face yarns were tufted on a primary polyester nonwoven backing fabric. A secondary backing fabric, also a polyester nonwoven, was sewn to the back of the tufted primary backing to lock the tufted face yarns in place. At least one of the two backings contained low melt polyester fibers having a melt temperature of approximately 105-110°C. When the carpet was heated in the subsequent dyeing and drying steps, the two backings melted together thereby keeping the face yarns from being removed easily. The Hoechst process is suitable for polyester fiber, which is not pre-dyed in the fiber forming stage.

A recyclable carpet based on polypropylene fibers also has been developed [98,99]. Unlike polyester fiber, polypropylene fiber is pigmented during the fiber forming stage. The tufted polypropylene fiber is manufactured by the use of a needle-bonding method to interlock the primary and secondary backings. The polypropylene face yarns were tufted into the primary backing first. Then the secondary backing was

needle-punched into the tufted primary backing. The secondary backing then was heated using infrared radiation to strengthen the bond between the two backings.

Carpet is removed from homes or businesses due to a variety of reasons, including being dirty, stained, worn out, or requiring a style change. In many cases, used carpets can still be reused. The "Earth Square" program by Milliken Company is to promote re-use through re-conditioning old carpet tiles [100]. Milliken's process includes four steps: collecting the old carpet, cleaning, restoring the texture, and adding new color/design. This process helps to extend the service life of the carpet.

## 5. Summary

A large amount of textile waste is disposed of in landfills each year. This not only poses economical and environmental problems to the society, it also represents a severe waste of resources. Waste statistics, waste characteristics, recycling rates, and recycling technologies for fibrous waste have been reviewed in this chapter. There have been several commercial carpet recycling operations in the U.S., and the products range from virgin-equivalent nylon 6 resin (or fiber), resin for automotive parts, fibrous mats, and vinyl carpet backing, among others. While many of commercial operations are still active, many of the carpet recycling facilities have been discontinued due to economical reasons. Although a recycling operation handling one type of carpet waste can be feasible, the overall economical viability is compromised if only part of the carpet collected can be recycled. This signifies the need for further research to develop diversified approaches that can recycled all types of fibrous waste collected.

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Table 1 Selected products in municipal solid waste in the United States in 1996 (in thousands of tons) [1].

Products	MSW generation	Recovered for Recycling
Carpet and Rugs	2,310	30 (1.3%)
Clothing and footwear	5,340	700 (13.1%)
Towels, sheets, and pillowcases	750	130 (17.3%)

Table 2 Relative market share of the top three synthetic fibers used by the U.S. carpet industry [15].

Year	1992	1996	2000
Nylon	66%	62%	55%
Polyester	10%	6%	7%
Polypropylene	24%	32%	37%
Total (million tons)	1.4	1.6	1.7

Table 3 Estimate of amount of materials in carpet produced in 1996

	Mass (million tons)	Percent
Nylon 6	0.4	14%
Nylon 6,6	0.6	21%
Polyester	0.1	3%
Polypropylene	0.5	18%
Adhesive/filler	1.3	44%
Total	2.9	100%

Table 4 Effects of experimental conditions on depolymerization efficiency and average molecular weight of oligomers [36]

Experiment	1	2	3	4	5	6
Feed Ratio of	5	10.3	20.6	29.4	59.8	no PTA
Nylon 6,6/BTEMB						
(wt/wt)						
Decrease in weight	40.3	49.5	55.5	42.5	40.8	-15.9
of Oligomers (%)						
M <sub>v</sub> of Oligomers	1556		1912	1697	2396	1644

Table 5 Composition and properties of molding compounds produced by United Recycling, Inc. from carpet [61]

	URI 20-001	URI 10-001
Composition		
Nylon 6, min.%	60	75
PP, max.%	15	10
Other polymers, max.%	10	10
Inorganic filler, max.%	15	15
Moisture, max.%	2	2
Properties		
Tensile strength, MPa	38	23
Elongation at break, %	10	10
Flexural modulus, MPa	790	760
Melt Flow Index, g/10 min	6.0	8.6
Extrusion/injection temp., °C	260-288	232-288

Table 6 Comparison between Extruded Carpets and Virgin Plastics [62]

Samples		Impact Properties		
	Tensile strength, MPa	Tensile modulus, GPa	Elongation at break, %	Izod impact strength, J/m
Extruded Carpets	26-31	2.1-2.2	4.5-6.7	41-57
Control sample, Polystyrene	34	2.8	2-3	22
Nylon 6,6*	77	1.7	>200	267

<sup>\*</sup>The data for nylon 6,6 are from "Nylon Plastics Handbook" [68]. All the data are collected at room temperature and 50% RH.

Table 7 Effect of Weight Fractions of Kraton on the Mechanical Properties of Extruded Carpets. The Carpet was composed of approximately 70% nylon, 20% PP and 10% SBR and Calcium Carbonate [65]

Kraton content,	Tensile strength,	Strain at failure,	Work of rupture,
wt%	MPa	%	J/cm <sup>3</sup>
0	46	7	1.6
5	43	9	2.3
10	38	15	3.5
15	37	31	8.0

Table 8 Effect of the Addition of Glass Fibers on the Carpet Waste Compatibilized with 10 % Maleic Anhydride Grafted PP (PP-g-MA) (estimated based on the charts presented in reference [63])

Samples	Tensile strength,	Tensile	Izod impact
	MPa	modulus, GPa	strength, J/m
Unfilled carpet waste	42	3.0	37
Carpet waste			
with 15% glass	77	5.2	69
Carpet waste			
with 30 % glass	117	8.8	87

Table 9 Mechanical Properties of Glass Fiber (GF) Filled PET/HDPE blends [70]

Blend	Tensile Modulus, GPa	Tensile Strength, MPa	Elongation at break,	Impact Strength, J/m	Heat Distortion Temperature, °C
PET/HDPE	1.6	12	1.3	19	120
PET/HDPE					
+ 10% GF	2.0	21	1.4	28	158
PET/HDPE					
+ 20% GF	2.4	26	1.5	33	233
PET/HDPE					
+ 40% GF	3.1	30	1.4	39	239

Table 10 Density and mechanical properties of compression molded GMT samples containing 40 wt % glass [71]

Materials	Edge trim	Separated PP	Azdel PM 10400
Density (g/cm <sup>3</sup> )	1.52	1.18	1.18
Tensile strength (MPa)	109	95.8	95.2
Tensile modulus (GPa)	5.72 *	6.83	6.27
Elongation at break (%)	7.9	8.8	3.0
Total energy absorbed (J) **	21.4	28.3	22.4
Sample thickness (mm) **	3.0	4.0	3.6

<sup>\*</sup> estimated without the use of an extensometer.

<sup>\*\*</sup> ASTM D3793 Drop weight impact test at 3.4 m/s.

Table 11 Compressive and Flexural Tests for FRC [86]

Fiber	$V_f$	Comp. Strength	Flex. Strength	$I_5$	$I_{20}$
	%	MPa	MPa		
None	0	52.6	4.65	1.0	1.0
FiberMesh PP	0.5	52.2	4.58	2.6	6.9
FiberMesh PP	1.0	51.5	4.99	3.3	12.0
Waste fiber	1.0	61.8	4.09	2.1	5.4
Waste fiber	2.0	40.7	4.35	3.5	9.8

Table 12 Compressive stress (kPa) at 10% axial strain for soil with carpet, polypropylene, and apparel fibers [87,88]

Fiber type	Fiber	Comp. Stress at	Comp. Stress at	Comp. Stress at
	%	0 kPa confinement	34.5 kPa	69.0 kPa
			confinement	confinement
Carpet	0	0	253	337
	1	213	355	342
	2	318	489	577
	3	501	736	859
Polypropylene	0	0	253	337
	0.3	111	474	335
	0.5	192	553	494
	1.0	335	635	722
Apparel	0	0		
	1	163		
	2	341		
	3	480		

Table 13 Energy Content and Combustion Energy of Various Polymers used in Carpets [89]

Plastic	Combustion energy,	Energy Content
	MJ/kg	MJ/kg
Nylon	29	155
Polypropylene	44	73
Polyester	31	84

# **Figure Captions**

- Figure 1 Typical carpet construction
- Figure 2 Component mass/area for a typical carpet (g/m²). Total is 2223 g/m².
- Figure 3 Depolymerization of nylon 6,6 by hydrolysis
- Figure 4 Extrusion process developed by Monsanto for recycling carpet waste [63]
- Figure 5 Typical flexural test curves of fiber reinforced concrete [78]
- Figure 6 Stress-strain relationships for triaxial compression test of carpet fiber reinforced soil confined at 34.5 kPa [87]

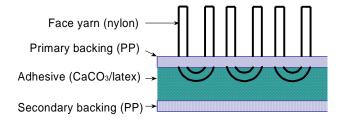


Figure 1 Typical carpet construction

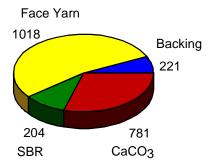


Figure 2 Component mass/area for a typical carpet (g/m2). Total is 2223 g/m2.

Figure 3 Depolymerization of nylon 6,6 by hydrolysis

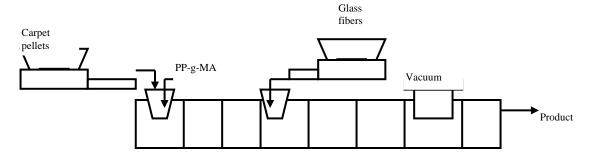


Figure 4 Extrusion process developed by Monsanto for recycling carpet waste [63]

### Load (kN) 20 1% Polypropylene 2% Waste Fiber 15 0.5% Polypropylene 10 5 1% Waste Fiber Control 0 3 2 Center Deflection (mm) 0 1 4

Figure 5 Typical flexural test curves of fiber reinforced concrete [78]

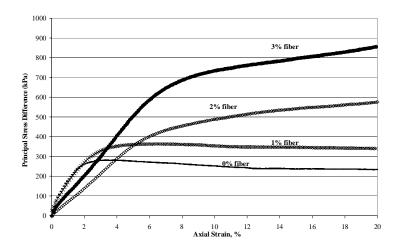


Figure 6 Stress-strain relationships for triaxial compression test of carpet fiber reinforced soil confined at 34.5 kPa