

Nylon Recycling Processes: a Brief Overview

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Plastic pollution is a significant environmental issue, and recycling has been identified as one of the ways to reduce its impact. This review provides therefore a brief overview of the various processes involved in recycling of polyamide plastic waste. Nylon is a material extensively utilized in the creation of clothes, textiles, and other products thanks to its high mechanical and thermal properties, as well as its strength and durability. This latter feature turns in a very slow decomposition if polyamide-based objects are abandoned in the environment, making it a significant environmental pollutant. The review covers all the possible recycling processes for this material, that are mechanical recycling, which involves shredding and melting plastic waste to make new products, solvent dissolution, where a solvent selectively dissolved only the desired polymer leaving behind the unwanted impurities, and chemical recycling, which uses chemical reactions to break down plastic waste into its chemical constituent parts, i.e. Monomers. The advantages and limitations of these processes are discussed.

1. Introduction

The textile and fashion industry is one of the largest in the world, with a very sustained growth in terms of consumption and production volumes and a linked economy that is predominantly linear, with huge exploitation of raw materials and production of waste destined to be sent to landfill, incineration or, at worst, to uncontrolled dumping in the environment (The Fiber Year Consulting, 2018). It is therefore urgent to transform the production model linked to the world of textiles from linear to circular and a great effort must therefore be made to recover both post-industrial and post-consumer waste, bringing them back into the production cycle (Boston Consulting Group and Global Fashion Agenda, 2017). Synthetic fibres make up about 62% of the textile material produced (Textile Exchange, 2021), with polyamide accounting for about 5% of this fraction. This percentage, smaller than the one of other polymers, generates another problem in terms of volume, since nylon constitutes only a small fraction of total municipal plastic waste that, in the case of nylon, is constituted also by a small fraction of flexible food packaging. Therefore, the recycling of this material is applicable on a large scale mainly on post-industrial waste and it is therefore very limited. In addition, other very common nylon applications are carpet and parts in the automotive industry: these latter applications do not enter in the municipal plastic waste stream and the pre-treatment costs for obtaining an exploitable nylon waste stream are sometimes still too high. Therefore, of the total amount of polyamide produced (5.58 million tons in 2019), the 98% is synthesized by the exploitation of new resources, while only 2% comes from recycling processes (compared to 15% of polyester, the most recycled synthetic fibre). Nylon-based waste poses some environmental problems, such as the microplastic pollution in aquatic environments (from fishing nets and synthetic textile fibres from laundering) (Le, 2018). To overcome all these issues, factors like the increasing demand for recycled polymers as well as European investments, that can advance R&D and consolidate technologies that are not yet applied on an industrial scale, allow to foresee important prospects for the spread of recycling processes (Ballabio et al., 2022). The European Commission is also working on a common roadmap for industrial technologies applicable to circularity, which aims to rationalize industrial research and innovation, including in the field of textile recycling. (European Commission, 2022)

The following review is therefore focused on the methods of recycling post-industrial and post-consumer Nylon-based wastes.

2. Types of recycling processes

The recycling of polymeric materials can be divided in three main families, i.e. thermomechanical, by solvent dissolution and chemical, according to the process and the characteristics of the final product.

2.1 Thermomechanical recycling

The thermomechanical recycling is a process that involves grinding and subsequent re-extrusion of plastic wastes to reuse them. The plastic waste is first shredded and grinded into smaller pieces, which are then melted into chips/pellets that can be moulded or spun into new products: grinding is necessary because only a homogeneous material of defined size can be introduced into extrusion or injection molding plants. The quality of the recycled material may be lower than that of the original polymer, so it is often used for less demanding applications (such as plastic application). This type of process can be performed both with post-industrial, such as scraps or processing waste, and post-consumer nylon waste. In the first case the process is simpler, because materials are generally homogeneous, relatively uncontaminated and can be recycled in the same process as the original polymer. (Goodship, 2007)

A fundamental procedure required before the thermomechanical recycling of polyamide-based materials is drying, because it's important to ensure that the water content is as low as possible to limit, during processing, the thermal hydrolysis reaction, which results in a decrease of the molecular weight of the polymer and therefore of mechanical properties of the resulting material. This issue can also be partially overcome by adding a stabilizer. In this situation, the recovered material can exhibit good qualities even after multiple reprocessing cycles and without drying (La Mantia et al., 2002). Although condensation and trans-reactions can provide molecular weight growth, the repetitive melting process can cause severe degradation of nylon, resulting in deterioration of all its characteristics. Studies show how virgin Nylon 6 may be treated up to seven times without affecting its physical-mechanical qualities or morphology; the only difference observed is the color. Pieces of lower quality are obtained after the seventh cycle; nevertheless, by mixing with virgin material or adding a chain-extender, the quality can be improved. (Lozano-Gonzalez et al., 1999) The case of post-consumer nylon waste is different: in this case polyamide is often combined with other materials, such as polyurethane, polyester and glass fibers, in order to provide different mechanical properties, but this in turn becomes a problem for the following thermomechanical recycling. In fact, since the main material is polyamide, a polymer with a high melting point, the re-extrusion must be carried out above 260 °C, an operation that could cause the degradation of polymers with lower thermal and chemical stability than nylon (such as polyurethane in textile fibers), leading to the formation of impurities that can jeopardize the quality of the recycled product, limiting its applications (Lv et al., 2015). Always in the field of mechanical recycling, a possible solution for highly contaminated wastes may come from the use of compatibilizing agents (Czarnecka-Komorowska, et al., 2021), where specific chemicals are able to keep together polymers of different nature still maintaining the mechanical properties of the main component: also in this case, anyway, it is difficult to find "universal" compatibilizers being able to maintain the mechanical properties regardless the nature and quantity of the contaminants.

Till now, the main nylon-based products recycled by a thermomechanical approach (figure 1) are the following:

- Carpets, that are made up of three main parts: the face fiber, which is usually nylon 66, nylon 6 or polypropylene (PP), at least one backing, made of PP, and an adhesive material made of styrene-butadiene rubber (SBR) applied as a latex and typically filled with an inorganic filler like calcium carbonate. Because of the different properties of their separate components, these products pose many difficulties when mechanically recycled. There are recycling processes for these types of products that involve separating the individual materials, through numerous processing steps, and recovering the material for re-use, but these procedures are extremely expensive, and the energy required reduces the environmental benefit of the process itself. Possible solutions of this problem are reported by Monsanto, which patented a process involving the development of a melt blend of the unseparated carpet material under temperature, pressure, and intensive mixing conditions sufficient to generate a composition with thermoplastic qualities suitable in a range of applications (David et al., 1994).
- Fishing nets, in which the main component is nylon. A recent study shows that the ageing in the marine environment does not affect the thermal, mechanical and chemical properties of nylon and that the thermomechanical recycling process on samples composed of the same type of fishing nets produces a material with properties not inferior to that present on the market and therefore suitable for similar applications. (Mondragon et al., 2020)
- Post-industrial Nylon 6 fibers waste: because of PA6 significantly oxidizes during long-term storage and at high melting temperatures, PA6 fibers and yarn waste cannot be simply recycled to make other fibers. In the thermomechanical recycling process the fibrous waste is ground and sent to the forced feeding system with simultaneous drying through a pneumatic conveyor and then to the extruder. The melt is then filtered (to remove physical impurities), degassed and discharged to produce standard-sized chips that can be used as it is or combined with virgin granulates without risk of phase-separation into the melt. To obtain

injection molding-grade product, additives can be added during the extrusion process. Alternatively, for highly contaminated waste, the melt can be also supplied to the depolymerization plant to obtain the monomers. (Keshav V. Datye, 1991)

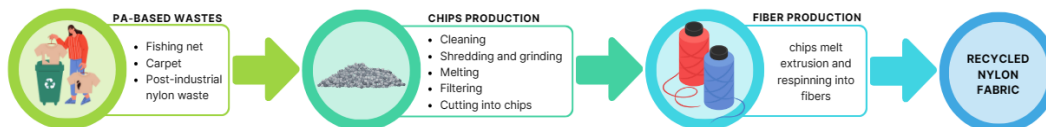


Figure 1: thermomechanical recycling process scheme

2.2 Dissolution recycling

The main problem associated to the exploitation of mechanical recycling of nylon-based materials, which is the most attractive technique due to its low energy demanding and costs, is the fact that very often PA is combined with other polymers (such as polyesters, polypropylene or polyurethanes) or inorganic/organic fillers (such as TiO_2 , glass fibers, carbon black, dyes, etc.) in order to give the desired properties or aspects to the final products. All this contaminants not only limit the applicability of thermomechanical recycling, since it is impossible for the recycled PA to maintain its original properties, but also of the chemical recycling, since the depolymerization reaction conditions, whatever technology used, also degrade polymers/organic fillers other than nylon, producing mixtures of heavily polluted monomers that need to be purified to obtain products suitable for use as raw materials in a new polymerization cycle; in addition, the presence of materials other than nylon can poison the catalysts, also reducing the efficiency of the depolymerization process itself. For these reasons nylon recycling is very limited to the post-industrial scraps and few PA-based materials such as fishing nets and pre-treated carpets, as previously reported.

A possible solution could be the dissolution/precipitation approach (Zhao et al., 2018), represented in figure 2, in which one or more polymeric materials, or other impurities, are selectively dissolved using a solvent or a mixture of solvents. After filtering the residual solid (which can be nylon or the undesired impurities), two different scenarios are possible: precipitation using an anti-solvent and precipitation by temperature lowering/concentration increasing. The polymer obtained by this process, purer than the starting material, becomes therefore suitable for further recycling by mechanical or chemical approach, the second one only if the quality of the recovered polymer is not suitable for every application required by the market (e.g., textile sector).



Figure 2: dissolution recycling process scheme

Regarding nylon scraps recycling by selective dissolution-precipitation method, different solvents have been proposed in literature to selectively dissolve PA from different matrix (carpets, molded plastics, textile wastes, above all post-industrial scraps): carboxylic acids (such as formic and acetic acids), glycols (Pallatheri M. Subramanian, 1995), glycerol (Stefandl, 2000), aliphatic alcohols (such as methanol at high temperatures and pressure) (Martin Booij et al., 1996) and aromatic alcohols (phenol and benzylic alcohol), inorganic acids (HCl (Sarian et al., 1996) and H_3PO_4 , (Davis & Dellinger, 1992)), fluorinated alcohols working at low temperatures, such as hexafluoroisopropanol (Teti, 1972), cyclic organic carbonates (ethylene and propylene carbonates, (Mauldin & Cook, 2005)), other polar compounds like dimethylsulfoxide and caprolactam, and salt mixture such as ionic liquids and alcoholic solution of CaCl_2 (Rietzler et al., 2021).

Some textile scraps, where nylon has been combined with polyurethane (PU, elastane) to provide specific properties, are recycled by selective dissolution of PU leaving the polyamide unaffected. This can be achieved using dimethylsulfoxide, dimethylformamide (Katsuragi & Ono, 2006) or mixtures of water-lactams (such as N-methylpyrrolidone, (Murata R., 2009)). Aquafil technology (Robello et al., 2011) avoids the use of solvents by heating the scraps near the melting point of nylon, causing the selective degradation of polyurethane and successively washing away the degraded chains with a polar solvent like ethanol.

Another very common material combination is made up of PA66 and silicone resins in the production of airbag. Silicone is spread on polyamide surface in order to provide gas impermeability to the PA yarn. The recycling of these scraps is generally accomplished by selective degradation/dissolution of silicon resin, causing its detachment from nylon which is then filtered off. To achieve this, mixtures of alkali/alcohol/surfactants can be employed (Rachel Andrews, 2007) as well as mixtures of Lewis acid/glycols (Ho & Yang., 2010) or mixtures alkali/amines/surfactants (Takayuki Mihashi, 2016). Another problem that must be faced during nylon recycling is the one related to the color of the waste. Commonly used colorants for polyamides are acid dyes, which strongly interact with amine terminal groups. The removal of dyes is for these reasons not easy and, after dissolution of PA, colors are mostly retained in the recycled nylon. Specific technologies using solvents have been developed by BASF (Ozer et al., 2008), which use a mixture of lactams/amines and alcohols as a swelling

agent plus ammonium salts as stripping agent, and by (Mu et al., 2022), where a mixture of dimethyl sulfone and water (95:5) is able to remove most of the colorants from PA66, hence producing a high-quality fiber.

2.3 Chemical recycling

The process of transforming polymeric waste into substances that can be reused as raw materials to produce plastics or other products is known as chemical or tertiary recycling (figure 3). Technologies used for nylon tertiary recycling are often based on depolymerization (Al-Salem et al., 2009). To perform a depolymerization, which is cost-competitive with the traditional process from fossil, the purity of the raw material stream must be high enough to not complicate the final purification process. If the purity is not high enough, chemical recycling is not suitable and a pre-treatment is required to improve the competitiveness of the technology.

Regarding PA, the depolymerization processes in the state of the art are:

- Hydrolysis: the reaction, that involves the attack on carboxyl amide by water molecules, can be applied both for nylon 6 and 66. This process can be promoted by high temperatures and pressures (thermal hydrolysis) (Sifniades et al., 1999), using acid catalysts (acid hydrolysis) (Born et al., 2007), or alkaline reagents (alkaline hydrolysis) (Moran, 1994).

Acid Hydrolysis: the acid-base catalytic process exploits acidic substances that lower the activation energy hence favoring the breaking of the polymeric chains. This technology is predominantly applied to nylon 6 and it is the most studied process in both scientific and patent literature: different reactor configurations, catalysts and purification processes are described to obtain the monomer with the chemical quality required by the market. Acid hydrolysis is, to date, the only technique used on a large scale for the depolymerization of post-industrial and post-consumer nylon 6. The main acidic catalyst is orthophosphoric acid (H_3PO_4), which is a cheap chemical and also a strong dehydrating agent; other catalysts, that respond well to the process such as sulfonic acid and boric acid (H_3BO_3), are less used due to their higher cost. The yields of this process are about 85-97% according to the kind of inlet raw material.

Thermal hydrolysis: this technique exploits the attack of water molecules having higher kinetic energy to the amide group of nylon, leading to the fragmentation of the chain and the subsequent release of the monomer. Examples of technologies that exploit this reaction are reported in the patent literature, with different reactor configurations, also including the operations necessary to obtain a purified caprolactam. The absence of acid catalysts avoids problems of plant corrosion and formation of phosphorous byproducts, leading to a yield of 90%.

Alkaline Hydrolysis: this technology exploits the much more nucleophilic reagent -OH to break the amide bonds and recovering monomer such as caprolactam and 1,6-hexanediamine. It is mostly exploited for nylon 66 depolymerization, since in some configurations there is the possibility to remove 1,6-hexanediamine from the reaction environment, shifting the equilibrium towards the monomers. Chemical plants exploiting this technology were used in the past in order to recover polymer scraps coming from PA66 polymerization plant, but now these wastes undergo mechanical recycling for economic reasons.

- Pyrolysis: this reaction involves only PA6, in absence of water, in inert atmosphere and using heat. The commonly accepted mechanism is an intramolecular cyclization, which occurs at temperatures higher than 300°C and lead to the formation of caprolactam; the reaction is generally accelerated by a basic catalyst that promotes the deprotonation of the amide group thus favoring the internal cyclization. Commonly used catalyst are alkaline and alkaline-earth hydroxides and oxides, sodium and potassium carboxylates, potassium carbonates, and the heterogeneous system KOH/Al₂O₃ (Bockhorn et al., 2001). The pyrolysis catalyzed by strong bases potentially has some advantages compared to traditional hydrolysis, deriving from the absence of water or solvents. In addition, another great advantage offered by pyrolysis is the possibility of working under vacuum, a condition that facilitates the removal of caprolactam from the reaction environment allowing the shift of the equilibrium reaction towards the desired product and leading to a yield of about 90%. Even though this technology appears attractive with respect to hydrolysis, at the best of our knowledges, there are no commercial plants, limiting to some pilot configurations (Kopietz et al., 1996).
- Enzymatic depolymerization: several studies into the enzymatic hydrolysis of polyamide materials have been performed. A thermostable variant of an enzyme named nylon hydrolase has been shown to catalyze the hydrolysis of a nylon-6 powder. The mechanism of enzymatic depolymerization of nylon involves the enzymatic breakdown of the amide bonds in the polymer chain, which results in the formation of shorter oligomers. These oligomers can then be further broken down into monomers, which can be recycled or used to create new nylon. Nylon-66 is also degraded by the enzyme, although less efficiently than PA6. Enzymatic depolymerization has the potential to be a more sustainable alternative to traditional methods of nylon disposal, with a conversion degree of 75% for nylon 6. However, more research is needed to optimize the process and make it economically viable on a large scale. (Thiyagarajan et al., 2022)
- Ammonolysis: it is a process where the polymer chains of nylon (6 or 66) are broken with a transamidation reaction with ammonia at high temperatures (280-330°C). This process can give a significantly high yield of almost 100% if the non-converted oligomers are recirculated. Although ammonolysis is less documented in the literature than the other techniques previously illustrated, a process that exploits this reaction has been patented by DSM (Buijs et al, 2000) to recycle PA6.

- Glycolysis: applicable both to PA6 and PA66, this process takes advantage of the nucleophilicity of hydroxyl moieties and the high boiling point of glycols in breaking the amide bonds of nylon and forming monomers (sometimes in their ester forms). In order to boost the reaction, a catalyst can also be used, either acidic or alkaline (Booij et al, 2000). Since the yields are still low (37%) and some byproducts are formed, there is no industrial applications of this technology.
- Solvent-assisted depolymerization: it is a chemical process that allows PA6 to be depolymerized using a supercritical, subcritical or high boiling point solvent. The solvent has the purpose of disrupting the structure of the polymer bringing it into high temperature mixture, facilitating the intramolecular cyclization of the amino groups and therefore the formation of caprolactam. Regardless the solvent used, all these technologies are, by the best of our knowledge, at laboratory scale.

With hydrocarbons: the process consists of heating polyamide 6 in a hydrocarbon solvent plus a small part of water under supercritical or subcritical conditions. Hydrocarbons are chemically inert towards polyamide: their function is only to bring the polymer to high temperature in the liquid phase, thus favouring the intramolecular cyclization described above. The solvents suitable for this type of process are both aliphatic, such as n-heptane, n-dodecane, and aromatic, where the best choice is toluene. The yields are higher than 90% (Kaiso et al., 2006).

With alcohols: PA6 is dissolved in an alcohol under supercritical or subcritical conditions. In particular, the solvents that give the best results are secondary and tertiary alcohols, which are less nucleophilic and promote the desired reaction, leading to a yield of about 90%. This process does not involve the use of water or a catalyst, hence minimizing operating and fixed costs and the problem of corrosion of steels. (Kamimura et al., 2006)

With ionic liquids: ionic liquids are becoming solvents of interest in chemistry, as they have unique properties of solubility, non-volatility, thermal and chemical stability, low flammability, etc. By exploiting these properties, it is possible to conduct a solvolysis at high temperature and at the same time at reduced pressure, a condition that facilitates the removal of the monomer once it forms in the reaction environment, leading to good yields of about 85%. (Kamimura et al., 2007)

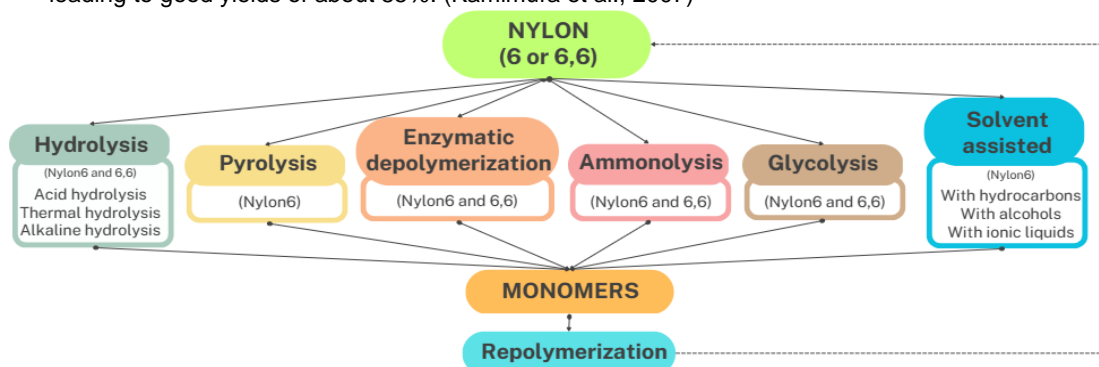


Figure 3: Chemical recycling processes scheme

3. Consideration and conclusion

The process of recycling nylon can help to reduce the amount of waste that ends up in landfills or incineration plants. Furthermore, recycled nylon can be used to create new products, thus reducing the demand for virgin materials, derived from fossil sources, and the associated environmental impacts of their extraction and production. Among the technologies reported, the mechanical recycling offers the best performance in terms of environmental impact and costs, but it is generally applied for post-industrial waste and only in the plastic sector, whereas the chemical recycling is the most suitable for textile applications, but it's applied on industrial scale only for PA6, it's the most expensive and requires a pure inlet raw material. Between these two, solvent dissolution may offer an alternative for nylon recycling, especially for high polluted waste. New technologies (e.g., enzymatic depolymerization) show very high potential, but they are still far from an industrial application.

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