R E V I E W A R T I C L E

Recent advances in the use of Polyamide-based materials for the automotive industry

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Abstract

Polyamide (PA) is a well-known and researched thermoplastic due to its excellent mechanical and physical properties, making it developed in the automotive sector, suitable for lighter vehicles, and, consequently, lower fuel consumption. This review manuscript presents the applications of PA-based materials in the manufacture of vehicle parts, with a description of their processing, a discussion about their thermal properties and the crystallization of polymer structure, the challenges of machining PA-based composite materials, and the feasibility of recyclability. This work aims to revise literature about the use of polyamide 6 (PA6), polyamide 66 (PA66), and polyamide 12 (PA12) and their composites reinforced with fiberglass (FG) and carbon fiber (CF) focused on the potential that these materials have as alternative materials for the automotive industry.

Keywords: *automotive industry, carbon fiber, composites, polyamide.*

1. Introduction

Polyamide (PA) is a semi-crystalline thermoplastic widely used in the automotive and aerospace sectors due to its excellent thermal and mechanical properties. However, PA is a highly hygroscopic polymer due to the presence of oxygenated groups (carbonyl and hydroxyl) in its polymer chain, that favors the permeation of water molecules between the chains, favoring good moisture absorption[1]. In addition, PA-based composites can be degraded by hydrolysis, and therefore PA must be subjected to the drying process before any processing[2]. However, even with the inclusion of this step before processing, PA has been standing out from other high-performance polymers, such as poly(phenylene sulfide) (PPS), poly(ether-ether-ketone) (PEEK), poly(aryl ether ketone) (PAEK) and poly(ether imide) (PEI), in use in the automotive and aerospace industries. This is because PA requires lower processing temperatures, making it possible to use the same equipment as conventional polymers, such as polypropylene (PP) and polyethylene (PE), lower raw material cost, good abrasion resistance, good chemical temperature (40-50 ºC) compatible with a different application.

In addition to being able to be recycled by mechanical, thermal, and chemical processes.

The wide use of PA in the automotive industry is justified by its numerous characteristics, such as its extreme durability, mechanical strength, good stability at high temperatures (< 130 ºC), good abrasion resistance, good chemical stability, and resistance to fuel, grease, and oils. In addition, it has an excellent oxygen barrier, provides excellent cost/benefit, and can be recycled[3,4].

However, the mechanical properties of PAs vary a lot with the level of absorbed water and the working temperature. Dry PAs are fragile at room temperature and have a glass transition temperature (T_g) above 50 °C; if applied at temperatures below 50 °C, the PAs have a high elastic modulus of maximum stress and low deformation at the break. Stress and elastic modulus values decrease with increasing temperature while straining increases[5]. Furthermore, another attractive mechanical property for the automotive industry is the high friction coefficient of PA, leading to high abrasion of the parts. This characteristic

makes PAs an excellent choice for replacing other materials in components such as gears, shafts, bushings, bearings, screws, and nuts[6]. For the application of these parts, manufacturers often use further processing to force water absorption, preventing the region from becoming brittle or from water absorption during the product's life cycle leading to part swelling[7]. The presence of moisture can have some advantages, as the absorption of water in finished parts can be the desired effect by manufacturers because the water can act as a plasticizing agent in the polymer; that is, the absorption of water tends to leave the toughest end product[8].

In the automotive industry, PA has already been widely used in airbags, car engine protections, car bodies and internal components, fastening elements (bolts, bushings, threads), and also in the manufacture of high-performance components, and in this way, it contributes with the reduction in the weight of vehicles and, consequently, in the decrease in fuel consumption[9]. Figure 1 shows the main applications of PA in the automotive sector.

PA-based materials can be processed using injection molding, extrusion, compression, and additive manufacturing. The choice of the most suitable PA-based materials processing method depends mainly on the final shape of the product and the type of reinforcing agent (particles, short fibers, or continuous fibers). However, due to the hygroscopic nature of PA, a key step in PA processing is the drying step before processing, as parts may form with various defects caused by the presence of water in the material[10].
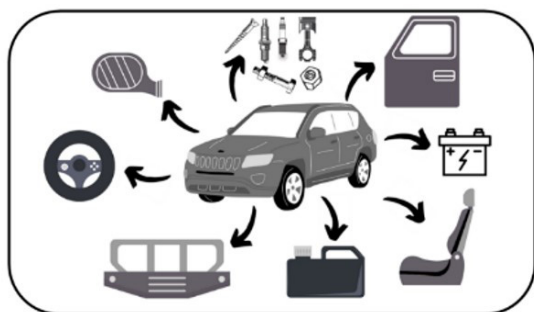

Figure 1. Main PA applications in the automotive sector.

The family of PAs is quite broad, as they have different structures that enable them to have other characteristics and properties, according to the number of carbons between the amide groups present in the macromolecules (Table 1). PA6 and PA66 are the most used due to their durability and strength. PA6 and PA66 have excellent mechanical properties, good chemical and oil resistance, but low chemical resistance to strong acids and bases, low gasoline permeability and excellent gas barrier properties, wear and abrasion resistance, good resistance to long-term heat, flame-resistant, feature the highest moisture absorption rate when reinforced with fiberglass (FG) or other materials can provide superior modulus of elasticity and strength, and PA6 and PA66 parts feature excellent surface finish. The main difference between them is the monomers (Table 1). PA 6 is more flexible while the PA66 is highly rigid, in addition to the PA6 being resistant to a temperature of 180°C and the PA6 notwithstanding working temperatures above 130°C[3].

However, by modifying chemical structures, such as chain length and chemical organization, it is possible to obtain other PA family members, such as the PA12. Among all commercially available PAs, PA12 has the lowest concentration of amide group, promoting its characteristics, such as lower moisture absorption, excellent impact resistance (even at temperatures below freezing point), excellent resistance to chemicals, good abrasion resistance, and high processability. Still, it has a high cost[4].

Understanding the kinetics of crystallization, degree of crystallinity (X_c), and thermal properties, such as melting temperature (T_m) and glass transition (T_g), are essential in choosing the processing parameters of polymers and their composites. Different degree of crystallization affects their chemical and mechanical properties. Higher degrees result in higher stiffness and tension strengths, while lower crystallization results in higher impact energy absorption[1].

Although there are several attractive properties to the industry, such as excellent strength and low specific weight properties, machining presents a current challenge. One of the main attention points related to the manufacture and processing of carbon fiber reinforced polymers (CFRP) and fiberglass reinforced polymers (FGRP) composite materials

Table 1. Chemical structure, monomers, melting temperature (T_m), glass transition temperature (T_g), and tensile strength of PA6, PA66, and PA12.

PA type	Chemical structure	Monomers Used to make	Tm (°)	Tg (°C)	Tensile strength (MPa)
PA6	$\begin{pmatrix} H & & O \\ \| & & \| \\ -N & -(CH_2)_5 & -C- \end{pmatrix}_n$	Caprolactam	220	40	78
PA66	$\begin{pmatrix} H & & N & O & & O \\ \| & & \| & \| & & \| \\ -N & -(CH_2)_6 & -N & -C- & (CH_2)_4 & -C- \end{pmatrix}_n$	1,6-Hexamethylene diamine and adipic acid	260	50	77
PA12	$\begin{pmatrix} H & & O \\ \| & & \| \\ -N & -(CH_2)_{11} & -C- \end{pmatrix}_n$	Aminolauric acid	180	40	53

are the difficulty of performing the cutting process during the machining[11,12].

Most due to the anisotropic and heterogeneous structure of thermoplastic composite laminates and the highly abrasive nature of the FG and CF, several critical damages are observed during the machining, along with high tool wear rates[13]. According to Meinhard *et al.*[14], CFRP machining can lead to different types of damage patterns that can be divided into two main types: permanent damages (i.e., delamination, chipping, fiber pullouts) and defects passible of reworking (i.e., irregular shape of holes and burr formation). The majority of machining focused research that studied FG and CF reinforced PAs highlighted the highly abrasive impact of fibers during machining, leading to poor surface finish, rapid progression of tool wear, and damages such as delamination in the machined surface[11,15-17].

This review study aims to present the latest research (2018-2022) related to the PA family, in particular PA6, PA66, and PA12, from the numerous applications in the automotive industry, thermal and mechanical properties, the most suitable means of processing and feasible, facts that influence machining, in addition to the possibility of recycling.

2. PA6, PA66, and PA12: application in the automotive industry

The European Commission regulation EU 2019/631[18] motivated the search for lighter materials in automotive vehicles. By the regulation, between 2020 and 2024, all manufactured passenger cars must have emissions of less than 95 g/km of CO_2. This emission is strongly related to vehicle mass, and the weight reduction strategy is cited by Nguyen-Tran *et al.*[19] to control the emission of CO_2.

However, regardless of the energy source, powered by a combustion engine, electric motor, or any alternative, the reduction in the vehicle's total weight impacts its energy performance in transport in general, showing the importance of the search for low-weight materials by the automotive industry. Grätzl *et al.*[20] showed that 37% of the total vehicle weight is related to body weight. Continuous fiber-reinforced plastics are a good alternative due to their high mechanical resistance/weight rate. Besides that, the European Union[21] has the legal requirement that 95% of the vehicle's total weight must be recyclable by 2025, which leads to a preference for thermoplastics over thermosetting polymer matrices.

According to Amasawa *et al.*[22], high-performance polymer materials can potentially change cars production. Nowadays, an average vehicle is made of 1163 kg of iron and steel and 150 kg of plastic. The use of polymers can affect car designs, enhance design flexibility and reduce vehicle weight. The authors cited the Japanese government initiative, with Japan ImPACT program, launched in 2014, that developed a prototype car composed of 47% of polymers and polymer composites that weighed 850 kg versus 1333 kg of a comparable vehicle using steel and glass. The main goal of the research was to analyze the life cycle greenhouse gas emission of the prototyped vehicle. The authors found that polymers and composites have

significant potential in reducing the life cycle greenhouse gas emissions of a battery-powered electric vehicle. Still, some innovations in fabrication, design, and powertrain resizing must be developed.

One of the alternatives used as a lightweight material is the PA and fiber-reinforced PA-based composites, such as FG and CF. Among the engineering thermoplastic alternatives, PAs are easy to process, are among the cheapest, and have the necessary mechanical properties, having strong fracture toughness if compared with thermoset like epoxy resin. PA-based composites are used in the automotive industry, where they are submitted to mechanical stress and high temperatures. The Polyamides are used in tires, seat belts, fan blades, car upholstery, air-intake manifold, fuel system, cooling systems, switch housing, and oil pans. The automotive sector is responsible for 35% of PA consumption, the largest PA consumer[23]. Polyamide 6 and PA66 are also prominent in the manufacture of gears for automotive use[24-28], while the use of nylon 66 can also be seen in the manufacture of leaf springs[29].

PA-based composites can be used in plain bearing, body coils, guide and coupling parts, nuts, and slides[8]. According to Singh *et al.*[30], PA6 is used in automotive industries to fabric gear, bearing, and fitting via extrusion, compression, or injection molding and machining. By its good thermo-mechanical properties, Chen *et al.*[31] reported that PA6-based composites could be applied where are required creep and fatigues resistance, substituting traditional metal materials in manufacturing structural parts and components in the engine compartments, bearing, and gears for the automotive industry. Spronk *et al.*[32] tested CF/epoxy and FG/PA6 composites used in the automotive industry with the concern of understanding the damage caused to the material in an eventual crash, demonstrating how energy is absorbed during an impact. Murray *et al.*[33] have also studied the impact strength of the FG reinforced PA6 composite for structural automotive parts.

Ishikawa *et al.*[34] mention the use of CF-reinforced PA6 matrix composite to manufacture a complete chassis of "Lotus Elise", originally made of aluminum alloy, to reduce the vehicle's weight. It is essential to ensure that a material used in the automotive industry can pass through most of the automotive process chain. For these reasons, Grätzl *et al.*[20] tested the effect of the painting process of PA6 or PA66 based long CF composites on the mechanical properties. These materials can be used in the vehicle body to reduce the total weight. The authors described a painting process that exposed the automotive piece to a temperature above 190 °C for 20 min. They concluded that the painting process does not deteriorate the mechanical properties of the composites. The study of Caltagirone *et al.*[35] showed no difference in strength and stiffness in using recycled or virgin CF in manufacturing PA66 composite-based material for automotive uses.

Caputo *et al.*[36] investigated the use of PA6/FG composite in manufacturing an innovative engine encapsulation system for the automobile, aiming at acoustic and thermal insulation. Volpe *et al.*[37] studied the microcellular injection molding of FG-reinforced PA6. PA66 has a higher cost than PA6, and their fiber-reinforced composites can be

used to produce under-the-hood automotive components. FG-reinforced PA66 is used in manufacturing automotive engine bay components and its production by injection molding was simulated by Moisey *et al.*[38]. Wei *et al.*[39] researched the aging properties of PA12 exposed to biodiesel at high temperatures, citing that PA6 and PA12 can be used in vehicle fuel systems because of their excellent balance of mechanical properties and barriers properties against hydrocarbons. Hıdıroğlu *et al.*[40] studied the reduction of daily rate hydrocarbons evaporation from PA12 automobile fuel transfer pipes prepared by radiation crosslinking. In Table 2 is presented a summary of the important uses of polyamides in automotive industry.

2.1 Processing

PA are polymers with a high tendency to absorb water, and for this reason, special care is needed to ensure the quality of processing. As with all thermoplastics, one must pay attention to processing parameters such as viscosity, T_m, the possibility of oxidation at high temperatures or in the presence of oxygen, and the behavior of the polymer when undergoing cooling[1].

Due to the tendency to absorb water, PA pellets must be very well dried before use in any processing. This drying step is crucial for the rest of the polymer cycle, as the use of wrong temperatures and times can result in a much shorter useful life of the parts that will be manufactured. Most literature recommends drying between 70 and 90 ºC, not exceeding 12 h in an oven. A more extended temperature or exposure time can lead to pellet surface oxidation, resulting in a defective final product. PA12 has a much lower water absorption than the rest of PAs and can be processed as a common thermoplastic; the pellets can be dried much shorter[31]. In addition to water absorption, there is a widespread phenomenon of PAs after processing, called post-processing shrinkage. This phenomenon must be considered, especially when using injection molding, since the mold must be produced considering the material shrinkage rate. This shrinkage leads to higher process costs, as a mold is needed just to process the PA[41-43].

PA-based materials can be processed by extrusion, injection molding, compression molding, and selective laser sintering (SLS). Determining the proper processing technique will depend on the shape of the product. Parts with structural applications are usually processed by injection molding, replacing metallic elements. Teixeira *et al.*[44]

prepared PA66/FG (60/40) composites by injection molding using a mold with the ability to simulate controlled flow restriction. From mathematical models, the authors verified that controlled flow restriction affects the fiber length and fiber orientation, influencing the final mechanical properties of the composite. However, the most considerable difficulty encountered in processing is the addition of fillers in the PA matrix, which increases the melt viscosity, generating greater shear in the equipment[45]. Specific equipment must be used to degrade the parts and avoid loss of properties[46].

Another type of processing widely used by the automotive industry for PA is compression molding, where polymer plates, usually with long fibers as filler, are pressed in positive molds, generating the desired parts[47-49]. In the study carried out by Murray *et al.*[33], resin transfer molding was used to manufacture PA6/FG composites. The impact strength results proved promising for manufacturing parts to be applied in the automotive sector. When processed by extrusion, PAs must be dried, or an extruder with a degassing system must be used. Furthermore, an extruder with a slight compression zone is more suitable for processing due to PAs' relatively low molar mass. These factors again lead to an increase in the production cost, as a specific extruder must be used or more time must be spent drying the material[50,51].

Hıdıroğlu *et al.*[40] prepared oil and fuel transfer ducts through direct extrusion in a layer extruder, where there are different dosing zones to insert different phases into the final product. According to the authors, it was proven that the degree of crosslink is directly linked to the final properties of the product, such as mechanical properties and permeability to gases. On the other hand, SLS is a relatively new process that is very promising for manufacturing high-performance PA parts, but it has not been widely investigated. However, some studies indicate good results[31,41,42].

Borrelli *et al.*[52] studied the performance of automotive PA components with basalt as load printed in 3D in collision tests performed by the finite element method (FEM). According to the authors, it was verified that the 3D printed parts do not present any type of deficit in properties compared to the features currently used, which consist of polypropylene (PP) and talc plates welded by vibration. This result is significant because printed pieces are relatively lighter than contemporary pieces. Lupone *et al.*[53] researched a new methodology to process composites and PA/CF by joining FDM and laser writing. This new additive manufacturing methodology proved ideal for achieving an alignment of

Table 2. Summary of the important information and uses of polyamides in the automotive industry.

Highlight information	References
Regulations about CO_2 automobile emission and recyclability of the used materials in automobile fabrication.	[18],[21]
Use of polymers and reinforced polymers composites as a strategy to reduce weight and improve cars energy efficiency.	[19], [20], [22], [34]
Use of polyamide in tires, seat belts, fan blades, car upholstery, air-intake manifold, fuel system, cooling systems, switch housing, and oil pans.	[23]
Use of polyamide for plain bearing, body coils, guide and coupling parts, nuts and slides.	[8]
Use of PA6 for gears and bearing.	[24], [25], [28], [30], [31]
Use of Carbon Fiber Reinforced PA6 for structural car parts.	[33], [34]
Use of PA66 for gears or leaf springs.	[24], [25], [29]
Use of PA6 and PA66 for under-the hood components, engine encapsulations system and engine bay components.	[36], [37], [38]
Use of PA6 and PA12 in vehicle fuel system parts and fuel transfer parts.	[39], [40]

short CF, which guarantees good electrical and mechanical properties when the effort is towards the fibers.

2.2 Thermal analyses of PA and the influence of humidity

Thermal analyzes of a polymer and its composites provide essential information for choosing processing parameters, such as temperature, time, heating, and cooling rates. The crystallization rate of semicrystalline polymers, such as PA, is strongly influenced by temperature and occurs between the T_g and the T_m of the material, making knowledge of these temperatures essential. The crystallinity degree (X_c) for high-performance thermoplastics directly impacts the chemical and mechanical properties of the polymer. The crystalline phase increases the stiffness and tensile strength, while the amorphous phase affects energy absorption[54].

Searching for the best understanding of the parameters that influence the properties of PA6, Liu et al.[55] evaluated the non-isothermal crystallization of PA6. The samples were heated in the temperature range of 25 ºC to 250 ºC, with a heating rate of 50 ºC/min to remove the previous heating history. It was noted that a lower crystallization temperature and a more excellent temperature range are obtained with an increasing cooling rate. As the cooling rate increases, the initial crystallization temperature decreases, indicating that the molecular chain does not have enough time to form nucleation at high temperatures. Through Avrami crystallization kinetics, it was possible to conclude that the non-isothermal crystallization of PA6 comprises a nucleation mode with one-dimensional and two-dimensional growth. Furthermore, Liu et al.[55] also apply the Liu and Mo kinetic equation[56] to provide a more accurate description of the non-isothermal crystallization process. The analysis carried out by POM shows the formation of spherulites in PA6 under the experimental conditions.

Zaldua et al.[57] evaluated thermoplastics produced by thermoplastic matrix transfer molding (T-RTM) with PA6/CF (53/47) and PA6/FG (40/60). Since the residual monomers from the polymerization effect have a plasticizing effect on the mechanical properties, TGA (thermogravimetry) analysis was conducted to determine the residual amount and the degree of conversion of the monomers into longer chains. The incorporation of fibers makes the improvement in thermal stability evident compared to neat PA6. Composites reinforced with CF had better mechanical properties, such as modulus and tensile strength, compared to composites with FG reinforcement. The influence of X_c on the mechanical properties is also evaluated by Taketa et al.[58] using different cooling temperatures in PA6/CF, PPS/CF, and PP/CF composites with unidirectional reinforcement. The results obtained attest to the decrease in crystallization temperature with the increase in cooling rates. Tensile strength tests showed increased strength due to the X_c. Interfacial shear was also evaluated, showing a reduction with decreasing cooling rates.

PA6/CF composites manufactured via vacuum-assisted resin transfer molding (VARTM) were analyzed by Li et al.[59] with different molding temperatures. As preliminary results, it is observed that the satisfactory temperature for impregnation of the material is 140ºC. At this temperature,

less water absorption by the material is observed. The authors concluded that the ideal temperature range for polymerization is 140 to 160ºC, and the molding process must be conducted within 20 seconds, regardless of the size and shape of the sample.

Yaghini and Peters[60] addressed the primary and secondary isothermal crystallization kinetics of PA6 with a more robust and complete model, considering structural characteristics, such as lamellar thickness, and a rigid amorphous fraction (RAF), and a mobile amorphous fraction (MAF). The results are satisfactory for predicting properties based on the processing conditions, converging with the experimental data. Uematsu et al.[61] determined the relationship between the crystalline structure of PA6 and CF with the mechanical properties of unidirectional PA6/CF composites using Raman spectroscopy. The analysis of crystallinity and interfacial properties shows that the increase and decrease in the proportion of crystalline phases of the material influence the final strength of the composite. Handwerker et al.[62] used a new way of producing reinforced materials, both with chopped CF and continuous FG, using fused filament fabrication (FFF) or also called fused deposition melting (FDM). The heat application increases strength and stiffness, especially when annealed at a temperature of 200ºC for 6h. Although heat treatment increases Young's modulus, microscopic analysis shows a concentration of air voids.

One of the biggest concerns when working with PA matrix composites is the influence of water absorption influence on the properties of the material. Hagihara et al.[63] used chemical and structural analysis to study the degradation mechanisms of thermoplastic composites with a PA6 matrix reinforced with CF exposed to hot steam. With crystallization, there is a decrease in the density of the amorphous phase, increasing the free volume and allowing water molecules to occupy this space, thus causing the hydrolysis of PA6 in the composite. Resistance analyzes showed that, after 28 days of aging under hot steam at 140 ºC, there was a decrease in the mechanical properties. Ma et al.[64] studied the water absorption behavior and its influence on the mechanical properties of PA6 boards and PA6 laminates reinforced with CF with different fiber orientations. Half of the absorbed water remained in the spacings between the polymeric chains, and the other half permeated in the fibers gaps and between the matrix/fiber interface. The decrease in water absorption upon reaching equilibrium results from the rejection of water from the crystalline phase and hydrolytic degradation of the matrix. Decreased mechanical properties are observed in both materials due to matrix plasticization and deterioration of the matrix/fiber interface. Chabaud et al.[65] evaluated the hydromechanical and mechanical behavior in relation to the processing of PA6/CF and PA6/FG composites by 3D printing. In addition, although the 3D printing process allows for the fabrication of more complex geometries, an increase in the porosity of the material and fiber twist was evidenced, which may result in a greater concentration of stresses. A summary of thermal properties, methods, parameters used and values, found in literature, can be seen in Table S1, for PA6 and PA6 based materials, and in Table S2, for PA66 and PA66 based materials, of the Supplementary Material.

The effects of post-condensation on PA12 crystallization kinetics have been studied by Paolucci et al.[66]. The samples were subjected to thermal annealing at different temperatures for different time intervals. It was evident that the molecular weight increases with annealing time and temperature. Although the crystallization rate is similar for samples annealed at different temperatures, it was observed that the lower the crystallization kinetics for longer annealing time. Ma et al.[67] researched the thermal properties and crystal structure of neat PA12. The analysis showed that the different crystal shapes influenced the tensile properties. Thus, it is possible to obtain PA12 with an advanced mechanical performance from the adaptation of crystalline forms with the correct preparation. A summary of the thermal properties for PA12, found in the literature, can be seen in Table S3 of the Supplementary Material.

2.3 Machining of PA

Most components made from CF and GF reinforced polymers can be manufactured to dimensions close to the final shape. However, the excess material is removed using machining to meet assembly requirements[16,68]. Furthermore, the machining process can present more economic advantages than molding processes when it demands lower production volumes[69]. Moreover, most automotive PA reinforced composites are generally manufactured by extrusion process requiring additional machining operations[70].

Nonetheless, with the increase of additive manufacturing techniques applied in the industry, the need for further machining processes is increasing. Since the lack of geometrical accuracy is one of the critical disadvantages of melted filament fabrication of reinforced thermoplastic polymers (PA12) parts, a strong need for developing hybrid manufacturing technologies was generated by combining additive manufacturing techniques and machining to improve surface roughness[71]. However, even with the accuracy provided by the machining of printed parts, the necessity of specific hardware and pre-machining routines still demands the in-deep study of the influence factors of the process.

Conventional machining methods are used to machine CFRP and FGRP composite material[11]. The drilling, milling, and grinding processes represent a significant part of machining these materials in the automotive industry[72,73]. The drilling process has an essential role in the final assembly of large and structural parts, as well as for lightweight and automotive components[13], since considering the higher loads applied to structural applications and due to safety reasons, there is still the necessity of classical mechanical joining involving machining of correlated screws holes[14].

Literature analysis revealed that the machining investigation of fiber-reinforced polymer machinability was usually realized through comparative studies to the non-reinforced polymer machinability. The machining processes of CFRP are related to abrasive solid tool wear and represent a significant process challenge due to their anisotropic structure and brittle fracture behavior. According to Meinhard et al.[14], burr formation is the dominant type of damage in the machining of CF-reinforced PA12 laminates, in contrast to delamination in the case of machining CFRP. Recent studies showed that surface roughness is highly regarded when machining PA. Most studies try to avoid delamination and, at the same time to, achieve good surface finish results. There are several difficulties to the drilling machining process due to the vast number of influencing factors that can change the quality of drill holes and alter the damage dynamics.

Several factors can impact the machining of fiber-reinforced thermoplastic polymers for automotive applications[74]. Among others, the temperature during cutting[75], the cutting tool material and geometry[76], material composition, preparation of the machined material, and their properties also play an essential role[11]. Regarding the influence of temperature in machining, Yan et al.[73] concluded that the excellent machinability of fiber-reinforced polymer composites is due to the viscoelastic behavior of the thermoplastic matrix as temperature increases. Results show that the viscoelastic behavior also leads to a better surface finish. Bertolini, Ghiotti, and Bruschi[15] observed that the size and surface defects density increased when machining PA6 at lower temperatures.

Since the thermoplastic matrix consists of mostly linear and high molar mass entangled chains, the temperature of the process is essential for machining. Its increase can lead to changes in mechanical properties. According to Meinhard et al.[14], the machining process must be executed due to the particular thermoplastic polymeric chemical bonds and the risk of matrix melting or depolymerization at low temperatures.

Meinhard et al.[14] stated that the cutting tool geometry is critical in drilling reinforced composites since the drilling process evaluates the drill-hole quality. The diamond-coated tool, deposed by the chemical vapor deposition (CVD) method, is indicated for thermoplastic machining[77]. However, Gaugel et al.[13] alert that the advantages of using diamond coating as cutting tools to machine CF reinforced polymers are only valid for high-quality laminates.

According to Kataras et al.[78], the tribological properties, corrosion resistance, and good mechanical and fatigue strength of PA6 components can favor minimum roughness results. An excellent surface finish can also lead to uniform distribution of stresses on the workpiece.

According to Kießling et al.[79], the extensive mechanical properties of PA would be the basis for investigating its machinability and manufacturing behavior. Even though thermoplastic composites laminates exhibit very high in-plane strength, delamination is a common challenge during machining due to poor transverse strength and low delamination fracture toughness. The heterogeneity and anisotropic properties of the fiber-reinforced polymer composites allow these materials not to exhibit plastic deformation, allowing several applications in the automotive industry[11]. However, in the automotive industry, PA reinforced joints are also necessary to consider the interfacial strength on the polymer moisture content and the impact of interfacial crystallinity on the temperature of machining[79]. Another important consideration is the final porosity of the material. According to Gauge et al.[13], the residual porosity has a high impact on machining CFRP composites laminates.

It is also essential to consider the failure mechanisms and fibers orientation to assess the machinability of CFRP

and FGRP materials to both conventional and modern manufacturing and machining methods[11]. Ferreira *et al.*[71] observed that the addition of short CF in the PA12 matrix decreased the cutting load's levels and tool wear, thus indicating improvement in machinability. However, parameters such as building orientation did not influence the final surface roughness. As considering the micromachining of reinforced PA, Kuram[16] investigated the micro-machinability of PA6 and FG-reinforced PA6 manufactured by injection molding, concluding that forces increased with feed rate and spindle speed. The principal observed damages were protrusion of fibers, fiber failure/fracture, and fiber/matrix debonding.

About the drilling machining process of CFRP, Gaugel *et al.* [13] observed that diamond-coated tools presented minor coating rounding of the coating until chipping occurrence and being the coating fracture and excessive substrate wear the most predominant mechanisms wear. Meinhard *et al.*[14] investigated the drilling of quasi-isotropic CFRTP laminates made of 24 unidirectional carbon/PA12-plies, at 100 m/min and a feed rate of 0.6 mm/rev using a carbide cutting tool. The authors observed that the decrease in cutting speed and feed rate caused damage to fraying, burring, plasticization, and fiber deflection, which was also associated with increased tool wear.

FG and CF reinforced thermoplastic polymers' tuning process was also studied. Davim *et al.*[70] investigated the precision turning process of PA66 with 30 wt% of FG applying four different cutting tools. The authors observed the formation of continuous coiled microchips in all testing conditions. Bertolini *et al.*[15] investigated the turning of PA6 using cutting speeds from 50 to 200 m/min and deep-of-cut from 0.25 to 1 mm in cryogenic and abundant lubrication. The authors observed the change of chip morphology during turning of PA6 with the decrease of temperature, from continuous to serrated until the T_g

region and after that from serrated to dust form. The dust form was correlated with poor surface finish.

Considering the association between additive manufacturing and machining processes of CFRP and FGRP, the contribution of machining is usually related to surface quality improvement, boosting the development of hybrid equipment to allow the fast transition of processing to machining components. Ferreira *et al.*[71] investigated the milling of short CF reinforced PA12 printed parts. The authors also highlight that with hybrid equipment and techniques that associate machining and additive manufacturing, a reduction of 50% in building time was observed, proving its potential application in several industrial processes.

2.4 PA recycling processes

Disposing of polymeric waste in landfills or incineration is becoming increasingly disadvantageous for industries. The development of new guidelines and environmental legislation that regulate the recyclability of these materials, which have high costs due to the high technology involved in the production process, are being led to new projects in the sector[80]. In Figure 2, it could be the possible PA recycling processes.

The automotive industry is committed to developing projects that solve environmental problems resulting from the waste generated during manufacturing components. The recycling of thermoplastics is a setback within the automotive sector and the feasibility of recycling end-of-life automotive parts. According to European Union guidelines[81], this regulation establishes that as of 2020, new car fleets must release an average of about 95 g of CO_2/km. Directive 200/53/EC of the European Parliament & Council of the European Union[82] aims to end the incorrect disposal of end-of-life vehicles (EVL) and their waste. The long-term
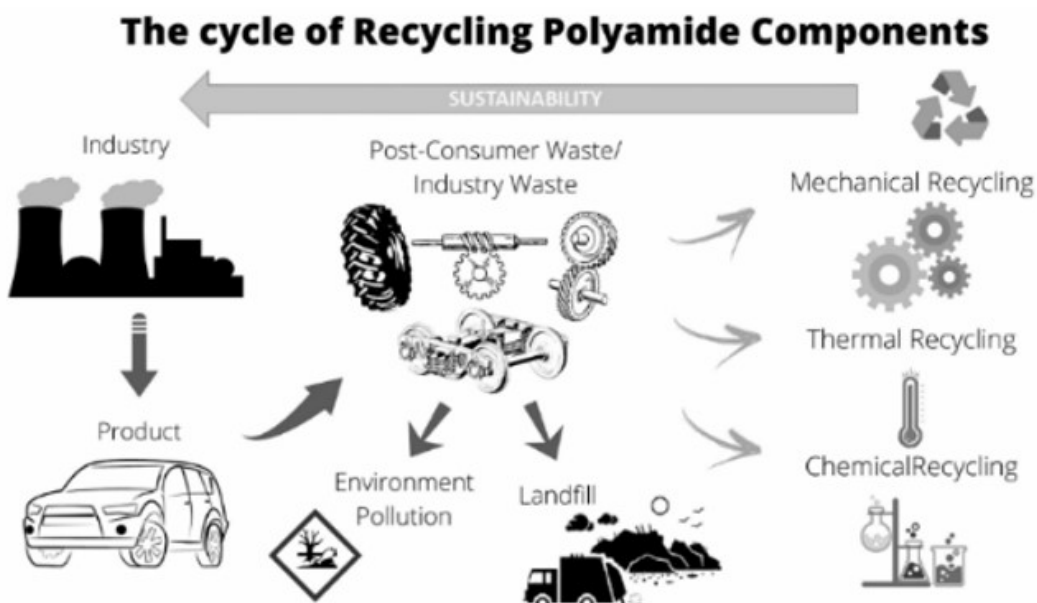

Figure 2. Feasibility of recycling PA components.

perspective is to change the way cars are designed and produced, so the directive targets the recovery and recycling of EVL and waste. To reduce the waste generated, vehicle manufacturers must develop and manufacture vehicles that have easy disassembly, reuse, and recycling of EVLs, increase the use of recycled materials in vehicle manufacturing and reduce the use of hazardous substances in the design of new vehicles[82].

The recycling of thermoplastics can be subdivided into four processes: mechanical, thermal, chemical, and energy[83]. However, it is clear that there is a need to develop sustainable production, and mechanical recycling has been gaining prominence among methodologies as it combines technical feasibility, moderate costs, and environmental benefits[84]. This approach is more suitable for short fiber reinforced polymers, as milling induces fiber breakage. Reprocessing has decreased the material's mechanical properties compared to chemical and thermal recycling that allowed the recovery of fibers and matrix in organic substances; however, the high energy consumption of these methods makes them less sustainable for composites with high commercial value fibers[84].

PA-based materials are feasible to be recycled, and in this way, it has been drawing the attention of PA consumer industries due to the high recycling potential resulting in suitable quality materials that can be reinserted into the production process of the generating industry or even incorporated as a material raw in another sector. It can be used in the automotive, electrical, and electronics sectors for packaging; consequently, there was a significant evolution in increasing the volume of PA residues. To remedy this problem, Chanda[85] discusses PA recycling methods; mechanical recycling is considered the easiest way to recycle PA, but as it is difficult to separate and clean the waste and be careful with repetitive melt processing, it can cause degradation of the PA properties.

Different processes can perform chemical recycling of PA. Lee et al.[86] address the chemical recycling route via catalytic pathways to recover caprolactam from PA6 with a high conversion rate. Chanda[85] discusses studies carried out with the types of decomposition of PA through hydrolysis, glycolysis, alcoholysis, ammonolysis, and mediation of ionic liquids, explaining the particularities of each technique and the parameters used in the studies. The studies by Nemade and Zope[87] carry out dissolution processes with different solvents such as dimethyl sulfoxide, dimethylformamide, formic acid, and p-cresol to degrade PA residues, with formic acid being excellent for the degradation of PA.

Alberti et al.[88] performed the depolymerization of PA6 through ring closure reactions to produce N-acetyl caprolactam, using 4-(dimethylamino)pyridine (DMAP) as an organocatalyst and acetic anhydride as a cheap and abundant depolymerization reagent. Kamimura et al.[89] studied the depolymerization of PA6 in hydrophilic ionic liquids under microwave irradiation at 300 ºC, separating caprolactam under simple extraction, and the ionic liquid, in turn, can be reused several times. Furthermore, the authors verified an improved procedure that avoids direct distillation (DD), contributing to an energetically more economical technique.

Kumar et al.[90] used a green and sustainable approach to hydrogenative PA depolymerization. The monomers obtained by this process can be dehydrogenated to return in the form of poly(oligo)amide, establishing closed-loop recycling of PA. Thus, the development of these strategies should allow better recognition of the PA residual value for the industry. The study performed by Tapper et al.[80] also presented the closed-loop recycling methodology to recover the high-quality constituents, the PA6 matrix, and the CF. The matrix was recovered by dissolving it completely in benzyl alcohol followed by precipitation, and the fibers were filtered from the PA6 solution. The authors found that even with the decrease in the mechanical properties of the recycled composite, these values were higher for any recycled thermoplastic composite and any discontinuous CF composite with thermoplastic or thermoset matrices. Mondragon et al.[91] carried out an interesting study on the recycling of PA6 fishing nets recovered from the ocean by fishermen. According to the authors, the thermomechanical processing did not affect the final properties of PA6. The PA6 residues could be reused in other applications since the analyzed properties are similar to the commercialized material.

Kunchimon et al.[92] prepared polymer blends from PA6 and polyurethane (PU) from textile waste, using the double screw melt spinning technique. According to the authors, it was possible to verify the feasibility of recycling end-of-life multi-material polymeric products to replace neat polymers such as PU. The study by Souza et al.[93] used vacuum bags of PA6 discarded after the curing process in an autoclave in the aerospace sector to produce PA6/LLDPE (linear low-density polyethylene) blends. According to the authors, this process demonstrated a new strategy for PA6 recycling, contributing to the reduction of waste from the aerospace industry.

The work of Moreno and Saron[94] was also based on recycling PA6 and low-density polyethylene (LDPE) components from multilayer polymeric packaging films. The presence of PE-g-MA proved to be economically viable due to its relatively low value compared to other additives used in polymers and its use boosting the potential application of the recycled blend. Pietroluongo et al.[95] investigated the mechanical recycling of parts of vehicle end-of-life radiators composed of PA66 reinforced with 35.7 wt% short FG to be applied in new structural or semi-structural components. The authors verified a reduction in mechanical properties is also related to different lengths of medium fibers and increasingly shorter fibers, making them less effective in resisting matrix movement. According to the study, recycled material can be used in the automotive sector to replace components made from unreinforced PA66 or with a low percentage of FG reinforcement. It may contribute to enforcing strict EU regulations on reuse and recyclability of at least 85% by weight per vehicle and reuse and recovery of at least 95% by weight per vehicle[21].

Češarek et al.[96] studied the degradation of PA66 through hydrolysis at different temperatures (170°C and 200°C) and molar ratios of 0.5, 1.0, and 1.25 HCl/amide in times ranging from 3.5 to 40 min under microwave irradiation. This methodology proved profitable for the recovery of PA precursor monomers and allowed the recovery of

the reinforcement present in the composite, making it promising. Another method of chemical recycling is the chemical decomposition of PA66 carried out by glycolysis processes with an excess mass of ethylene glycol (EG) as a decomposition agent in the presence of a catalyst, resulting in liquid samples and amino glucose with a mixture of EG with triethylenetetramine (TETA) as a decomposition agent that resulted in solid samples, proposed by Datta *et al.* [97]. The products obtained had the presence of molecules with hydroxyl groups and amines. The attractiveness of this technique is that it was possible to apply the chemical decomposition products to synthesize new PU with glycolysis intermediates originating from recycled PA66, replacing the commercially used polyol in the amount of 10 or 15 wt% in a satisfactory application of the process.

After an in-depth discussion of the most recent PA recycling techniques currently used, it is possible to see the extent of the viability of this process within industries. Each type of processing focuses on producing different products from recycling and demonstrates potential application in other materials and sectors. From the analyzed studies, the range of recycling techniques used for PA6, PA66, and PA12 is noticeable. Furthermore, the increased use of this material is indicative of its excellent properties and application versatility. It has great potential to close the production cycle and fit into the circular economy, benefiting the automotive sector to comply with EU laws and guidelines.

3. Conclusions

Concerns about environmental issues have led governments to establish increasingly strict rules for the automotive industry. PAs are among the most widely used thermoplastics in engineering polymers due to their excellent balance between performance and cost. In the automotive sector, they are widely used, resulting in lighter vehicles and lower fuel consumption. The keys finding regards to PA (Nylon) 6, 66 and 12 in this review research was:

- PA6-based materials are an alternative for automotive production, used inclusively in structural parts such as chassis where good mechanical performance is required;

- PA6-based materials and PA66-based are used in automotive industry in the fabrication of gears, bearing, leaf springs, under-the hood components, such as engine encapsulation and engine bay parts;

- PA12 is most used in the fuel transfer system, the fuel systems pipes;

- Due to the tendency to absorb moisture, PA must be dried before processing, which can be by injection molding, extrusion, compression molding, and additive manufacturing;

- Higher cooling rates result in lower crystallization temperatures and a more comprehensive temperature range. The incorporation of fibers increased the thermal stability of neat PA6. Higher mechanical strength was achieved with a higher degree of crystallinity. A decrease in interfacial shear resistance was obtained with a reduction of cooling rates;

- The mainly machining problems when processing PA-based composites are delamination and burr occurrence. The described relevant factors that affect machining were cutting tool temperature, cutting tool material and geometry, machined material composition, and thermal properties;

- PA-based materials can be recycled mechanically, thermally, and chemically and have been drawing the attention of companies generated from PA-based waste due to the excellent quality of the resulting recycled material.

According to the information gathered, the use of PA 6,66 and 12, and PA-based materials has the potential to assist the automotive industry in complying with increasingly stringent regulations for emission of gases and use of recyclable materials, also helping the energy efficiency of the vehicle by reducing its total weight.

4. Author's Contribution

- Conceptualization – Marcel Yuzo Kondo; Larissa Stieven Montagna ; Mirabel Cerqueira Rezende.

- Data curation – Marcel Yuzo Kondo; Larissa Stieven Montagna; Guilherme Ferreira de Melo Morgado; André Luiz Guimarães de Castilho ; Larissa Anne Pereira dos Santos Batista.

- Formal analysis – Marcel Yuzo Kondo.

- Investigation – Marcel Yuzo Kondo ; Larissa Stieven Montagna.

- Methodology – Marcel Yuzo Kondo ; Larissa Stieven Montagna.

- Project administration – Edson Cocchieri Botelho.

- Resources – Edson Cocchieri Botelho; Michelle Leali Costa; Fabio Roberto Passador; Mirabel Cerqueira Rezende; Marcos Valério Ribeiro.

- Software – Larissa Anne Pereira dos Santos Batista.

- Supervision – Mirabel Cerqueira Rezende; Marcos Valério Ribeiro.

- Validation – Marcel Yuzo Kondo ; Larissa Stieven Montagna.

- Visualization – Marcel Yuzo Kondo ; Larissa Stieven Montagna.

- Writing – original draft Marcel Yuzo Kondo; Larissa Stieven Montagna; Guilherme Ferreira de Melo Morgado; André Luiz Guimarães de Castilho; Larissa Anne Pereira dos Santos Batista.

- Writing – review & editing – Marcel Yuzo Kondo; Larissa Stieven Montagna; Edson Cocchieri Botelho; Michelle Leali Costa; Fabio Roberto Passador; Mirabel Cerqueira Rezende; Marcos Valério Ribeiro.

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Supplementary Material

Supplementary material accompanies this paper.

Table S1 – Summary of the investigation of thermal properties of PA6.
Table S2 – Summary of the investigation of thermal properties of PA66.
Table S3 - Summary of the investigation of thermal properties of PA12.

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