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Recycling polymeric multi-material products through micronization

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ABSTRACT

The increasing usage of polymeric materials and the greater range of requirements in product design lead to the combined use of more than one material in the same product, component, the so-called multi-material products. These products represent a risk for the environment, as they make the conventional recycling process more complicated. The main problem in recycling polymeric multi-material products is related to the difficulty in separating their components. Thus, it is necessary to find solutions to allow multi-material recycling without the need to separate these materials. In this context, this study aimed to analyze the technical feasibility for the application of micronization in recycling polymeric multi-material products and to evaluate the potential of the resulting materials for use in new products. Therefore, a theoretical part, focusing on polymeric multi-material products and micronization, and a practical study, consisting of multi-material toothbrushes recycled via micronization, were presented. The experimental investigation involved the micronization of multi-material toothbrushes, followed by extrusion and injection molding. Subsequently, the resulting material was evaluated using scanning electron microscopy (SEM), tensile strength test and dynamic mechanical thermal analysis (DMTA). The results showed that micronization is a potential process to promote the recycling of multi-material products, and there was no degradation during the process. The recycled micronized material had a low level of interaction with the LLDPE matrix, which affected the elongation at break, causing loss of ductility and tenacity compared to virgin LLDPE. However, it did not affect the tensile strength, which presented an increase of 18.43% compared to the reference. Despite better performance in tensile strength, the recycled sample showed an intense decrease in the storage modulus in temperatures above 30 °C. This may limit the use of these materials in certain products. Based on the results obtained, it can be concluded that the resulting material has potential applications in new products.

1. Introduction

The use of polymers in diverse products has gained more importance in recent years (Allwood and Cullen, 2012; Gabrys et al., 2013; Julier, 2013). The evolution of polymeric materials and the increase in product requirements culminated in the combined use of more than one polymer in the same product component (Thomas and Yang, 2009; Wargnier et al., 2014), giving rise to so called multi-material products (Kromm et al., 2007).

Polymeric multi-material products are usually produced by co-injection molding (Kim and Isayev, 2015) and present several advantages related to Design for Manufacture and Assembly (DfMA), such as reduced manufacturing time and costs, final product quality, less need for manpower etc. (Advani and Hsiao, 2012; Wargnier et al., 2014). Despite the technical advantages, there are some disadvantages to the environment, due to problems with reprocessing, recycling and separation (Allwood and Cullen, 2012; Worrell and Reuter, 2014).

Difficulties in recycling polymeric multi-materials have a major impact on the environment, especially in the end of life, once it generates waste. Solid waste generation rates have increased and

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are responsible for stocking landfills, which opposes the idea of a fully closed material cycle (Bosmans et al., 2013). Furthermore, the difficulties in recycling these products impact on the extraction of raw materials for new products, since waste could be used to replace virgin raw materials, reducing the demand for primary production (Ichinose and Yamamoto, 2011; Kollikkathara et al., 2010; Koushal et al., 2014). In addition to the impact on environment, the recycling issues are also related to economic (Murakami et al., 2014; Ohnishi et al., 2016), social (Wilson et al., 2006) and regulatory (Campos, 2014) factors.

Therefore, it is necessary to find solutions to enable the recycling of multi-material products. Several studies have been conducted with the purpose of reprocessing difficult recycling materials using micronization (Casa and Castro, 2014; Hong et al., 2015; Prameetthaa and Bharatkumar, 2014; Stark et al., 2014). This research aimed to analyze the technical feasibility of applying micronization in the recycling of polymeric multi-material products and to investigate the potential of the resulting material for their application in new products. This was an interdisciplinary study, involving Product Design, Materials Sciences and Environmental Quality. It also aimed to achieve sustainable development.

2. Context overview and earlier studies

2.1. Polymeric multi-material products

Polymers have revolutionized the way that designers and consumers perceive the products (Julier, 2013). The demand for polymeric materials intensified after World War II and continued to grow rapidly (Allwood and Cullen, 2012).

The use of polymers is of increasing importance and has stimulated research on new types of polymers, new ways of processing and applications. These efforts resulted in a significant diversity of polymers available, being one of the factors leading to the use of more than one type of material in the same product component, the so-called multi-materials (Thomas and Yang, 2009). Other factors have also influenced the emergence of multi-materials. The need to reduce costs and to improve the performance of technical products has led designers and engineers to incorporate more functions in just one component (Wargnier et al., 2014). These requirements cannot be achieved when a product is composed of a single material. Therefore, the current trend is to develop multi-material products. The case of toothbrushes is an example. A few years ago, the material selection for hygiene products was restricted to health issues. Nowadays, however, ergonomics and aesthetics are also being considered (Yang et al., 2005). To fulfill these requirements, the use of different materials is necessary. The main advantages of multi-materials consist of improving component performance, integrating more functions to material, reducing costs, facilitating production and avoiding voluminous parts (Wargnier et al., 2014). The applications of multi-material products extend far beyond daily products. They range from personal care items, such as toothbrushes and razors, to vehicle panels, foods and beverage packages, tool handles, among others.

In industries, the most used process to manufacture these products since the early 70s is the co-injection molding (CIM), also known as sandwich injection molding. The possibility of combining two different polymers provides unique properties that could not be achieved through the traditional injection molding process, with one single injection (Kim and Isayev, 2015). CIM is a process in which two polymers are simultaneously or sequentially injected in the same mold cavity. The final result is a heterogeneous product, comprising the “core material” (the main material) and the “skin material” (the surface material applied in smaller quantities) that fuse (Zaverl et al., 2013). In the field of engineering (Advani and Hsiao, 2012; Boothroyd et al., 2011), this process presents several technical advantages, such as better quality, reduced manufacturing costs and shorter time, and consequently, the assembly work becomes unnecessary (Advani and Hsiao, 2012). Thus, the product parts can be produced economically, in a single step, by operating one machine and one mold, following the DFMA guidelines (Boothroyd et al., 2011). In addition, CIM is also one of the most promising methods from both the economic and ecological points of view (Kim and Isayev, 2015). However, the environmental quality issues are controversial. According to the ecodesign approach, co-injection molding of multi-materials could represent environmental risks. The melting of two different materials precludes their separation and recycling through the traditional process (Allwood and Cullen, 2012; Worrell and Reuter, 2014). Therefore, the main disadvantage of multi-material products, especially the difficulty in their reprocessing and recycling, is related to negative impacts on the environment.

Some authors (Cândido et al., 2011; Hopewell et al., 2009; Kreiger et al., 2014; Koushal et al., 2014; Nkwachukwu et al., 2013; Rajendran et al., 2012) consider recycling as the best choice in polymeric residue management. However, the amount of polymeric material destined to recycling is still small. From the 280 million tons of polymer produced globally in 2012, less than half was destined to recycling, landfilling or energy recovery through incineration. The remaining residues may be still in use or are being inappropriately discarded. In this sense, if the high demand for polymeric products continues, and the recycling rates remains the same, there will be an amount of 33 billion tons of plastics in the planet by 2050 (Rochman et al., 2013). In this context, the difficulty in recycling presented by polymeric multi-material products leads to many serious environmental damages, including high rates of solid waste generation that can result in an overload of landfill capacity (Kollikkathara et al., 2010), or waste in inappropriate places, presenting risks to the population (Ichinose and Yamamoto, 2011).

2.1.1. Polymeric multi-material recycling and characterization

Considering the need for expanding the practice of recycling, this research proposed the recycling of mixed polymers without the need of separating materials. This way, economical costs linked to the separation process could be reduced when providing recycling of mixed plastic wastes, making it more profitable (Bertin and Robin, 2002; Najafi et al., 2006). Many authors have discussed ways of reprocessing polymer-based composites by several methods (Perry et al., 2012; Palmer et al., 2009; Pickering, 2006; Yip et al., 2002). However, there are only a few specific studies regarding mixed polymers or polymeric multi-material recycling and end-of-life solutions. Among them, we may cite the study that applied a polymer milling process with liquid CO_2 to polymeric mixed waste, obtaining a powder material that was successfully utilized for a new composite material as a matrix (Cavalieri and Padella, 2002). The use of pyrolysis to recycle mixed plastics was also studied (Kaminsky, 1995; Kamisnky and Kim, 1999). Another study (Hopewell et al., 2009) showed that incineration with energy-recovery was indicated as the most suitable way for dealing with highly mixed plastics. Although, according to the UK Waste Resource Action Programme (WRAP, 2008), incineration has the second least favorable environmental performance, been only better than landfill. Despite that, landfill is still the most common destination for polymeric multi-material products and components. The research that analyzed the life cycle of multi-material car components reported that only steel is recycled in final disposal phase. Mixed polymers like polyamide and elastomers are destined to landfill (Ribeiro et al., 2007).

Regarding recycled polymeric materials, some studies were carried out concerning the final material characterization (Bertin and Robin, 2002; Ronkay, 2013; Lorenzo et al., 2014; Najafi et al., 2006). The final properties evaluation is important to select the best applications for a recycled material. Yang et al. (2012) suggested that the material from recycled polymer composites, in the near future, will be most likely used in lower levels of non-structural applications with less critical quality requirements. Usually, recycled polymers lose some mechanical performance due to its degradation when submitted to recycling cycles (Lorenzo et al., 2014; Moeller, 2008). The study made by Ronkay (2013), for example, evaluated some properties of recycled polycarbonate and observed a decrease of 75% in elongation at break, with recycled load concentration above 30% in the virgin polycarbonate.

Another concern when recycling mixed polymers is the interfacial adhesion, especially when mixed polymers are immiscible. In this sense, a study analyzed the mechanical recycling of mixed plastic wastes composed of low-density polyethylene (LDPE) matrix and polypropylene (PP) by compounding using single-screw or twin-screw extruders (Bertin and Robin, 2002). The results showed that adding compatibilizing agents have improved elongation at break and impact strength. Therefore, the recycled blends exhibit suitable properties for applications that require good mechanical properties. Another study has evaluated the mechanical properties of wood plastic composites made from sawdust and recycled mixed plastics (most commonly used HDPE and PP). Results showed that, generally, the mechanical properties of specimens containing recycled mixed plastics (HDPE and PP) were statistically comparable with those of composites made from virgin plastics (Najafi et al., 2006).

In summary, polymeric multi-material products cause environmental negative inputs. This statement counteracts the important technical advantages and competitiveness of this kind of product during the production and use by the end consumer. Therefore, it is necessary to develop processes to solve the end-of-life problems associated with polymeric multi-material products. In the present study, the use of micronization to recycle end-of-life polymeric multi-material products has been presented as an alternative to face this issue.

2.2. Micronization

Micronization is a process used to reduce a material particle size (Vandana et al., 2014). There are different micronization techniques and the most common and affordable are those involving grinding. In the present study, we analyzed micronization at room temperature. In this process, the material is triturated in a knife mill, operating at temperatures of up to 120 °C, therefore, it is not suitable for materials with low melting point (Shulman, 2004). This process is capable of generating particles of approximately 0.4 mm, with few finer particles (Ayyer et al., 2013). To obtain finer particles, micronization can be applied repeatedly until the desired particle size is achieved (Shulman, 2004).

Micronization is commonly used in the pharmaceutical industry to produce drugs in small particle sizes. This occurs because the powders provide a higher rate of dissolution of drug particles, enabling oral consumption (Joshi, 2011; Vandana et al., 2014). For pharmaceutical applications, the required particle sizes are about 10 microns or 0.01 mm (Vandana et al., 2014). In such cases, grinding micronization is not adequate for the product, therefore, more complex methods should be used, such as in-situ micronization (Vandana et al., 2014), spiral jet mill (Joshi, 2011), supercritical fluid micronization (Reverchon et al., 2015) etc. The aforementioned methods will not be covered in this study, since the required particle size for recycling are not so small.

Another interesting use of micronization is in natural fiber processing. Some researchers (Fernandes et al., 2008; Fornanri Jr. et al., 2008; Huang et al., 2014) have studied its application in natural fibers to develop composite materials. In such cases, this process is important for ensuring the homogeneity of the mixture, enhancing the properties of the final material (Fornanri Jr. et al., 2008).

The use of micronization to enable or facilitate recycling is already being studied in different fields and materials. In civil engineering, for example, micronization has been applied to recycle barite, a mineral used in the mortar manufacturing (Stark et al., 2014). In the same direction, Prameetthaa and Bharatkumar (2014) demonstrated the application of micronized silica (derived from rice husk) as a load for recycled concrete. The results showed that the recycled concrete with the micronized material had similar properties to the concrete blank (Prameetthaa and Bharatkumar, 2014). Another study (Casa and Castro, 2014) reported the potential use of recycled ash from biomass by micronization as raw material on the production of bricks for their application in construction. Micronization was also used for recycling the sludge drained from coal mines and subsequent development of pigment with the resulting material (Marcello et al., 2008).

However, the most common recycling applications of micronization are related to the reprocessing of post-consumer tire rubber (Ayyer et al. 2013; Colom et al., 2007; Zhang et al., 2009; Sonnier et al., 2008). This occurs because the material cannot be recycled by conventional methods, since it is a thermosetting polymer (Zhang et al., 2009). In this case, the conversion of the powder material and further mixing with thermoplastic polymer result in the creation of a thermoplastic elastomer amenable to injection molding, extrusion and other processes (Zhang et al., 2009).

With regard to micronization for recycling polymers, Mao et al. (2014) studied the effects of the particle size on the recycling of polyurethane by combining three different types of recycled polyurethane (PUR) material. Micronization was also used in the recycling of ethylene–propylene–diene monomer (EPDM) by Jeong et al. (2014) and Hong et al. (2015). Finally, micronization was used in polyamide (vehicle airbags) recycling (Basire, 2013).

Therefore, this research has considered the possibility of applying micronization in multi-material products as a recycling method.

3. Experimental procedures

3.1. Materials

For the practical study, the following materials were used:

- Five different models of multi-material toothbrushes;
- A commercial grade of linear low density polyethylene (LLDPE) with melt flow index of 10.5 g/10 min.

Toothbrushes were chosen as material in this study for being a typical example of multi-material product, which is expected to be made by at least 2 polymeric materials in the handle (some rigid thermoplastic and an elastomer), besides the bristles, which are commonly made of polyamide (Grover et al., 2012, Rosema et al., 2015), and the metal pieces that anchor the bristles. Moreover, the majority of dental practitioners recommend the replacement of toothbrushes every two or three months, since the prolonged use of toothbrushes may be a potential source of oral, blood and systemic infection (Bezirtzoglou et al., 2008). This need for frequent replacement generates a great amount of polymeric solid waste, according to the Chelsea Center for Recycling and Economic Development (2000), discarded toothbrushes account for an

estimated 50 million pounds per year of plastic waste in the United States, which is a relevant environmental problem.

For the preliminary experimental study, the toothbrushes used were from pre-consumption, and were acquired in the market in their original package. Different models were chosen to evaluate the range of materials used by diverse manufacturers, as well as the feasibility of reprocessing distinct products/materials without the need for separating them. The graphic representation of the used toothbrushes models is shown in Fig. 1. LLDPE was used as matrix due to its low cost and easy processability.

3.2. Procedures

3.2.1. Characterization of the toothbrushes materials and chemical composition

Before micronization, the polymeric materials of the toothbrushes were identified using Fourier transform infrared spectroscopy (FT-IR) in a PerkinElmer Spectrum 100, with a resolution of 4 cm^{-1} and 16 scans in the region between 4000 and 600 cm^{-1}. The polymeric materials were compared to the Sadtler digital library.

The FT-IR analysis of the toothbrushes was conducted in three parts of the product, which correspond to different materials. The parts were named as (a) body, (b) detail and (c) bristles, as shown in Fig. 2.

Additionally, the toothbrushes presented metallic clips to attach the bristles. This material was identified using an X-ray spectrometer Bruker S1.

3.2.2. Recycling: grinding and micronization

Previously to micronization, the toothbrushes were ground, using a Seibt MGHS 15/85, operating at 1125 rpm and 8 mm of sieve. The technique used to reduce the particles size was micronization at room temperature in a knife mill. So, after grinding, the material was submitted to two cycles of micronization in an AX Plastic Micronizer Machine, operating at 1600 rpm. Seives of 3 and 0.5 mm were used in the first and second cycles, respectively.

A Scanning Electron Microscopy (SEM) Hitachi® TM 3000 was used to identify the average diameter of the ground, micronized material. The magnifications used were 40× and 50×. Due to the particle irregularities, the largest dimension of each particle was considered for the measurement of its size.

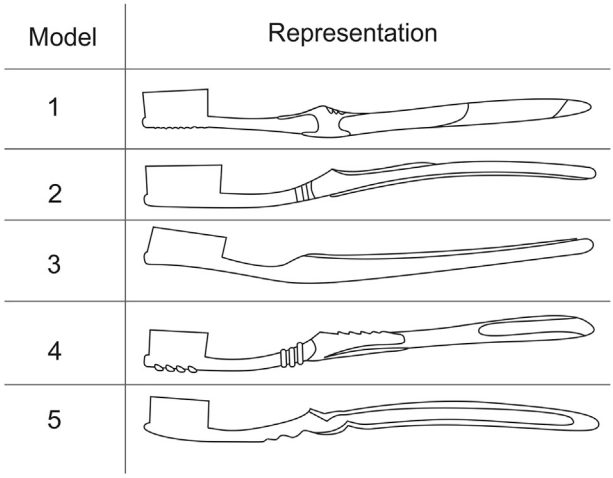


Fig. 1. Representation of toothbrush models used in the study.

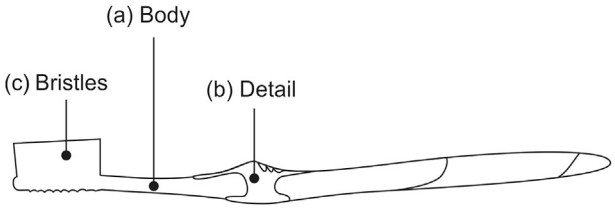


Fig. 2. Location of FT-IR analysis on the product.

Table 1
Injection molding parameters.

Melting temperature	200 °C
Mold temperature	30 °C
Injection pressure/post pressure	300/280 bar
Injection time	8 s

3.2.3. Mixture preparation and injection molding

The mixture was prepared by melt intercalation using a single-screw extruder Seibt MR6D59, operating at 35 rpm and temperature profile: 160–180–180–190–200 °C with 30 wt% of micronized material and 70wt% of the LLDPE (recycled sample) and neat LLDPE (virgin sample). Before extrusion, the polymers were dried in a hot-air-oven at 60 °C for 24 h. The residence[1] time calculated during extrusion was 40 s. After extrusion, the material was granulated using an Ax Plásticos AX GRAN pelletizer machine. The choice of 30 wt% of recycled material for 70 wt% of virgin matrix was made based on other similar studies that indicated that 30% of recycled load would present satisfactory mechanical properties without processing difficulties (Pedroso and Rosa, 2005; Jacob et al., 2001).

Samples for testing were prepared in a Haake Minijet II injection molding, according to ASTM D638, specimen type V. The parameters used are presented in Table 1. Before injection molding the materials were dried in a hot-air-oven at 80 °C for 3 h.

3.2.4. Testing: SEM, tensile testing and DMA

To analyze the resulting material obtained from extrusion and injection molding, the sample was observed using a Hitachi® TM3000 TableTop SEM. The images were used to verify interfacial adhesion.

Mechanical properties were evaluated by tensile test and dynamic mechanical analyses (DMA). Tensile properties were carried out in a Shimadzu® EZ-LX, with Trapezium X software at speed of 100 mm/min.

A PerkinElmer® Q8000 was used for the DMA of the materials at a fixed frequency of 1 Hz. DMA analyses were performed in single cantilever mode, using specimens with approximate dimensions of 30 × 3.2 × 12.75 mm. The samples were heated at temperatures ranging from 22 °C to 100 °C at a heating rate of 2 °C/min. No negative temperature was used due to equipment limitations. All results were compared with the neat material (virgin sample).

4. Results and discussion

4.1. Characterization of the toothbrushes materials and chemical composition

The FT-IR analysis and the comparison between the obtained spectra with the equipment library standard spectra and the bands characterized in previous studies, showed that all the studied

[1] The amount of time the material is in the extruder is the residence time.

toothbrushes models were basically made of polypropylene (PP), as the body material. It was also identified that ethylene–propylene–diene monomer (EPDM) was used in the details of models 1, 2, 4 and 5. The only exception (model 3) consisted of PP both in body and in the details. Although it seems that two different resins were used, it was found that model 3 was co-injected with two colors of the same material (PP). However, it is still considered a multi-material product, since the bristles and the metallic pieces are made of other materials, as follow.

For the bristles, it is recognized in the literature that polyamide (PA) is the most commonly used plastic material (Grover et al., 2012; Rosema et al., 2015). To confirm this statement, the FT-IR analyses were performed on the toothbrush bristles. As expected, the result confirmed that the bristles were made of PA. Figs. 3–5 show the comparative graphs of FT-IR transmittance spectra, where the *x* axis represents the wavenumber in cm^{-1}. Each analysis is compared to a standard material from the equipment library, which indicates the most likely material for that spectrum, after that, the bands were identified based on previous studies.

The spectra in Fig. 3 show a band at 2949.9 cm^{-1}, which is related to asymmetric stretching vibrations from CH_3. The bands between 2916.9 cm^{-1} and 2868.7 cm^{-1} are assigned to stretching vibrations of CH_2 (Nogueira et al., 2007). Bands around 1450 cm^{-1} and 1375 cm^{-1} are related to the CH_2 scissoring vibrations and to symmetric stretching vibrations of CH_3, respectively (Mitra et al., 2006). Finally, bands in 1167 and 890 cm^{-1} are assigned, by Carvalho et al. (2007) to the C–C bonding stretching and to the CH bending vibration, respectively. Thus, based on the analysis of the bands observed in Fig. 3 and the typical bands of polypropylene characterized by previous research (Carvalho et al., 2007; Nogueira et al., 2007), it is clear that the analyzed material is polypropylene (PP).

The FT-IR spectra from the products detail, presented in Fig. 4, show the bands around 2920 cm^{-1} and 2852 cm^{-1}, that are assigned to asymmetric and symmetric stretching vibrations of CH_2 (Mitra et al., 2006; Gunasekaran et al., 2007). The band at 1451 cm^{-1} is associated with CH_2 scissoring vibrations, and the band around 1376 cm^{-1} is related to symmetric stretching vibrations of CH_3 from the propylene unit present in EPDM (Mitra et al., 2006). Thus, comparing this spectra with previous studies (Mitra et al., 2006) it is possible to affirm that the detail material is EPDM.

Observing the spectra in Fig. 5 (relative to the bristles), we can see bands around 2923 cm^{-1} and 2853 cm^{-1}, already identified as asymmetric and symmetric stretching vibrations of CH_2, respectively. According to Evora et al. (2002), the band close to 3300 cm^{-1} is characteristic of polyamide and is related to the N–H stretching vibrations. Besides, to characterize polyamide, there may be bands related to the C=O stretching vibrations and bending vibrations from N–H (Evora et al., 2002), these bands appear in Fig. 5 around 1680 cm^{-1} and 1515 cm^{-1}, respectively. This proves that the bristles material is polyamide. During bristle extraction, a metallic material was found in all analyzed models. The toothbrushes contain metallic pieces, smaller than 3 mm, whose function is to anchor the bristles in the product. This metallic material was identified as a copper-zinc alloy (65% Cu/35% Zn).

4.2. Grinding and micronization

In grinding and in the first micronization, the PA bristles and the metals were not crushed. The materials were passed through the sieve maintaining their original format. The concentration of long bristles did not harm the process. The material was pushed towards the blade and passed without clogging the equipment.

In the second micronization, the largest part of the bristles was ground, getting the consistency of a powder; a few longer polyamide strands were visible when handling the micronized material. It was difficult to visually identify the metal pieces among the micronized toothbrushes; however, a few pieces could be identified, meaning that they have passed through the sieve. Fig. 6 shows the resulting material from each process.

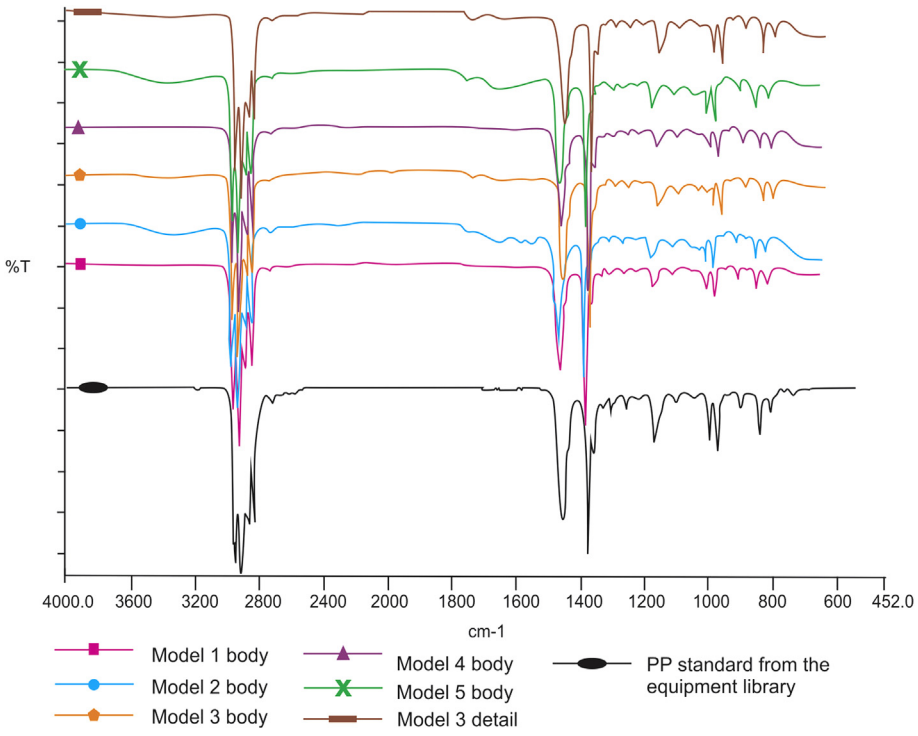


Fig. 3. Toothbrush FT-IR spectra characterizing polypropylene (PP) as the body material.

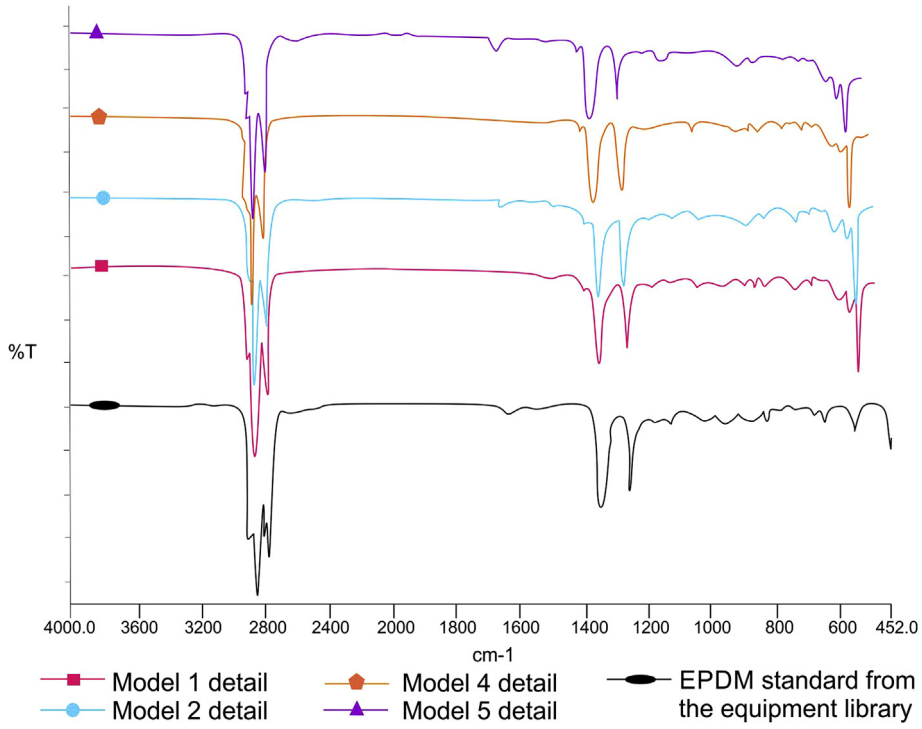


Fig. 4. Toothbrush FT-IR spectra characterizing ethylene–propylene–diene (EPDM) as the detail material.

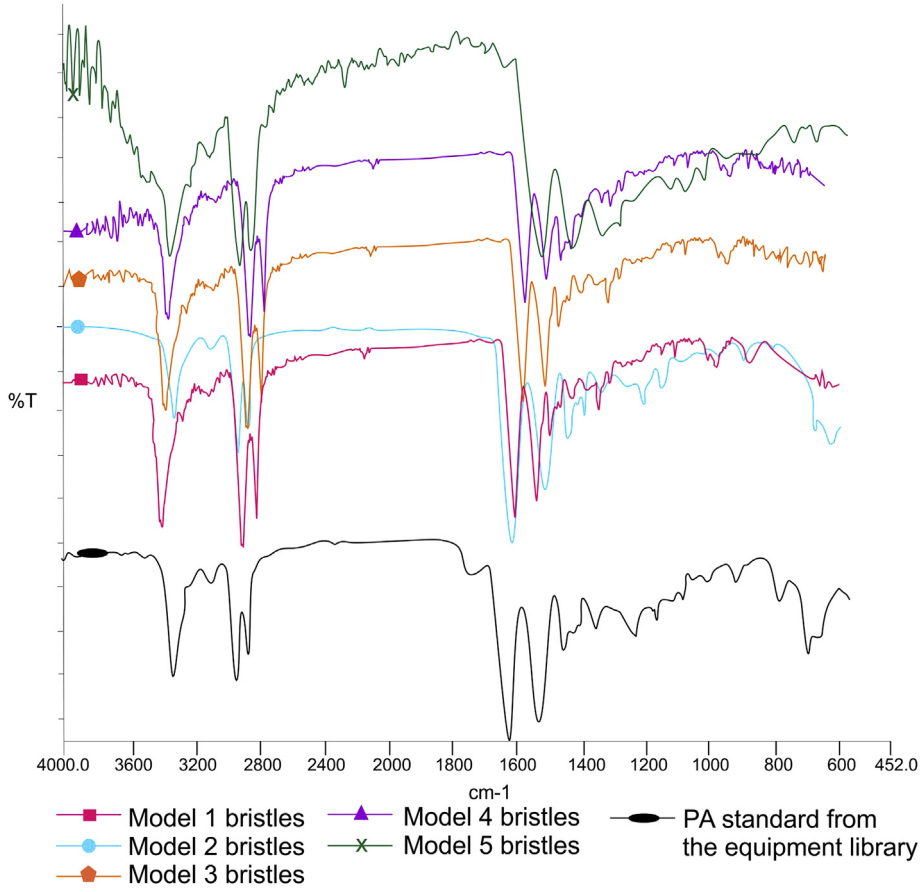


Fig. 5. FT-IR spectra characterizing polyamide (PA) as the bristle material.

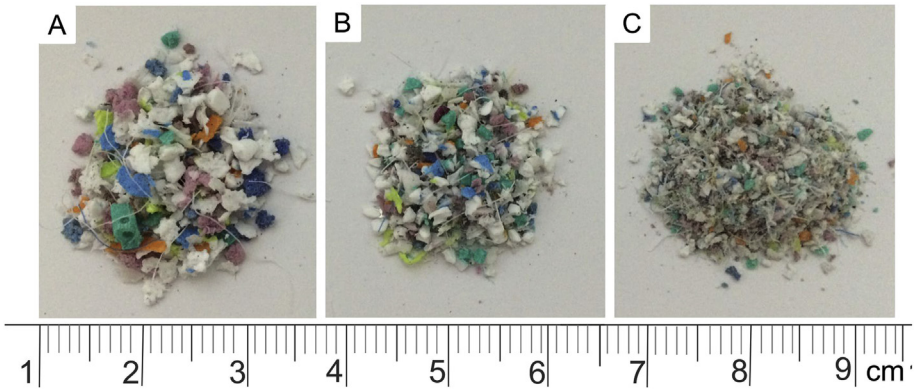


Fig. 6. Ground material (A), from the first micronization (B) and from the second micronization (C).

It was found, through measurements made in SEM images, that the average size of the particles generated by grinding ranged from 3.2 mm to 3.7 mm. In the first micronization there was more irregularity among the particle size, which ranged from 1.7 mm to 3.2 mm. Finally, the measurements of the sample generated after the second micronization revealed great irregularities in the size of the particles. There were many fine particles (ranging from 0.008 mm to 0.3 mm) among some larger particles (up to 1.4 mm). For better visualization, Fig. 7 shows the variation of the mean particle size in each process and the SEM images.

It was observed that micronization has significantly reduced the particle size; however, the particle-size distribution (PSD) of the resulting material was not uniform, especially after the second processing. Table 2 shows that the particle size generated in the first micronization did not reach the size suggested by the literature for room temperature micronization, whose value is determined by approximately 0.4 mm (Ayyer et al., 2013). After the second micronization, the particle size determined by the literature as the standard size for room temperature micronization was partially achieved.

4.3. Testing: SEM, tensile testing and DMA

The material obtained from extrusion showed no apparent degradation and had homogeneous appearance, except for the small metal pieces that became apparent in the extruded material. Many of these metal pieces fell during pelletizing. The equipment was not damaged by the metallic materials. During the injection molding, there was also no apparent degradation. The specimens showed homogeneous appearance. The few metallic pieces that remained in the material neither harmed the injection process nor damaged the equipment.

The analysis of the morphology was carried out in two stages: after extrusion and after injection molding, both using SEM images. The SEM image obtained after extrusion presents two distinct phases (Fig. 8A), however, the micronized material is well distributed in the matrix. The injected material can be seen in Fig. 8B.

As observed in the extruded material, the injected sample also has two phases, which indicates a low level of interfacial adhesion between the micronized particles and the matrix. This may be due to the poor compatibility between the LDPE, EPDM and PA (Jose et al., 2011).

The evaluation of the mechanical behavior of the obtained recycled material was conducted using a tensile strength test and DMA. The recycled material (recycled sample) was compared to the virgin linear low density polyethylene (LLDPE), the reference material (virgin sample). For the tensile test, ten specimens of each sample were prepared and tested, in order to ensure a reliable average in the results. However, only nine results of each sample were considered. As shown in Fig. 9, the specimen 6 from the virgin sample and the specimen 5 from the recycled sample had a non-standard behavior and had to be discarded. In relation to the valid tests, the mean and standard deviation (Table 2) were considered. Fig. 10 shows the stress (δ) −strain (ε) behavior of both samples based on the mean values.

Table 2 shows a significant reduction in the elongation at break of the recycled sample (reaching only 51.9% of the reference performance). In Fig. 10, the tenacity is represented by the area under the curves. It can be observed that the area under the curve of the virgin sample is much larger. Thus, the incorporated recycled

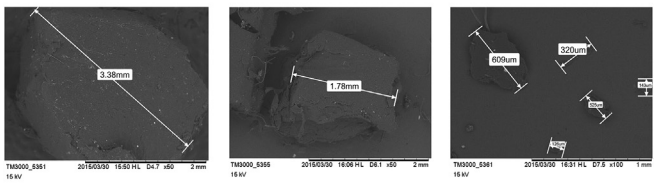
Process	Grinding (Fig. 6A)	First Micronization (Fig. 6B)	Second Micronization (Fig. 6C)
SEM image			
Size variation (mm)	3.2 to 3.7	1.7 to 3.2	0.008 to 1.4

Fig. 7. Variation of the mean particle size in each process + SEM measurements.

Table 2
–Means obtained in the tensile strength test for virgin and recycled samples.

Samples	Yield strength (Mpa)	Ultimate tensile strength (Mpa)	Elongation at break (mm)	Young modulus (MPa)
Virgin (reference)	17.67 ± 0.73	18.99 ± 1.18	88.28 ± 18.59	808.75 ± 134.17
Recycled	20.92 ± 0.51	21.33 ± 0.82	45.81 ± 24.91	728.93 ± 27.1

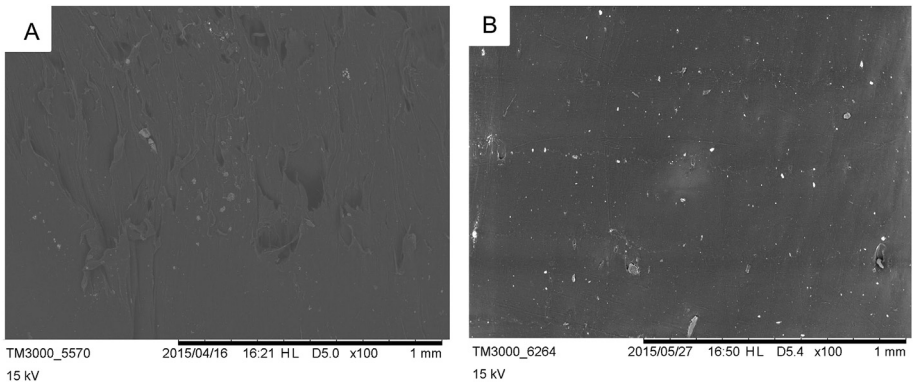


Fig. 8. SEM images.

material led to a decrease in ductility and tenacity of the resulting material. This may be related to the low level of interaction at the interface between the LLDPE matrix and the micronized material particles, as seen in the SEM images (Fig. 8). In ductile matrices, particulate fillers increase the brittleness of the material because of the low interfacial adhesion (Fu et al., 2008), causing an anticipated rupture when the material is under tensile strength.

Young's modulus presented a slight decrease (9.9%), however, which was considered insignificant if considering the standard deviation. This result showed no sensitivity to the low interfacial adhesion. The tensile strength test showed that the addition of a recycled load has led to a slight increase in material resistance, which supported 18.43% more stress (relative to reference) without undergoing plastic deformation. The increased resistance in the recycled sample was also perceived in DMTA, according to Fig. 11.

However, DMTA demonstrated that, although the resistance has increased compared to the virgin sample, the recycled sample was more susceptible to higher temperatures. This can be noted in the intense decrease of the storage modulus curve of the recycled sample in temperatures above 30 °C. As seen in SEM images and observed in the elongation at break, this behavior may be related to the poor interfacial adhesion between LLDPE and the micronized material (Santos et al., 2009). Despite being more susceptible to temperatures, the recycled sample has maintained a higher storage modulus during the test, even in higher temperatures.

5. Conclusion

This study showed the technical feasibility of reprocessing different co-injected materials by means of micronization. Based on the results obtained, it can be concluded that micronization is a potential process to promote the recycling of multi-material products. Furthermore, the process proved to be efficient in reducing the particle size of the analyzed material, without degradation or damage to the equipment. It is noteworthy, however, that such effectiveness in reducing the material particle size

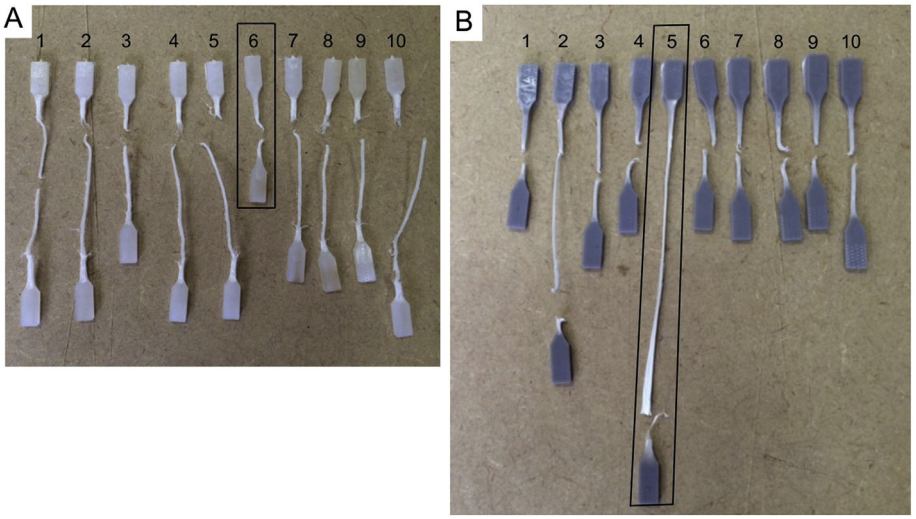


Fig. 9. –Virgin (A) and recycled (B) specimens after the tensile strength test.

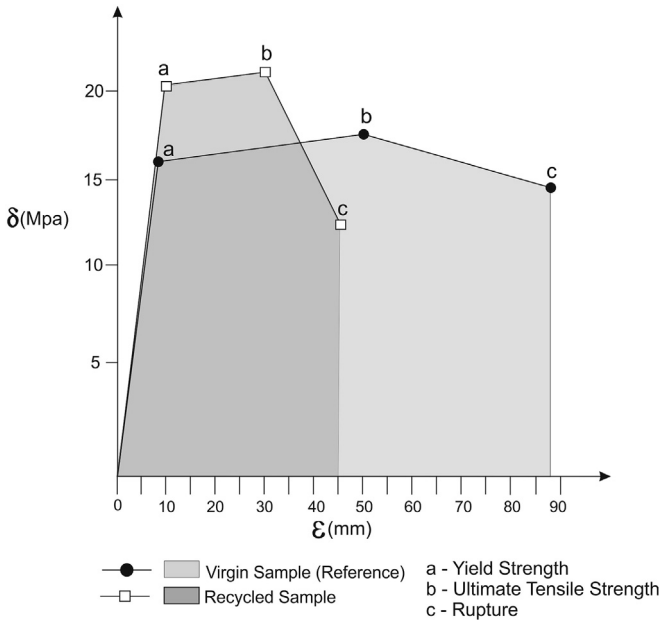


Fig. 10. Stress (δ) - strain (ε) graphic.

may be maximized by separating smaller grains with a fine sieve. Larger particles could be submitted to a third micronization, which could reduce the disparity between the obtained grain sizes. Although the literature has indicated a particle size average slightly smaller for room temperature micronized materials, it was noticed that the particle size obtained in this study was sufficient to promote the recycling of the product.

Regarding the technical feasibility of using extrusion and injection techniques, the results obtained were satisfactory. Using materials of different grain sizes did not hinder the extrusion process. Considering the technical factors of extrusion and injection molding techniques, there was no degradation of the material, even when subsequent techniques were applied to the recycled material. In addition, the metallic parts (copper–zinc alloy) mixed in the recycled material did not damage the equipment.

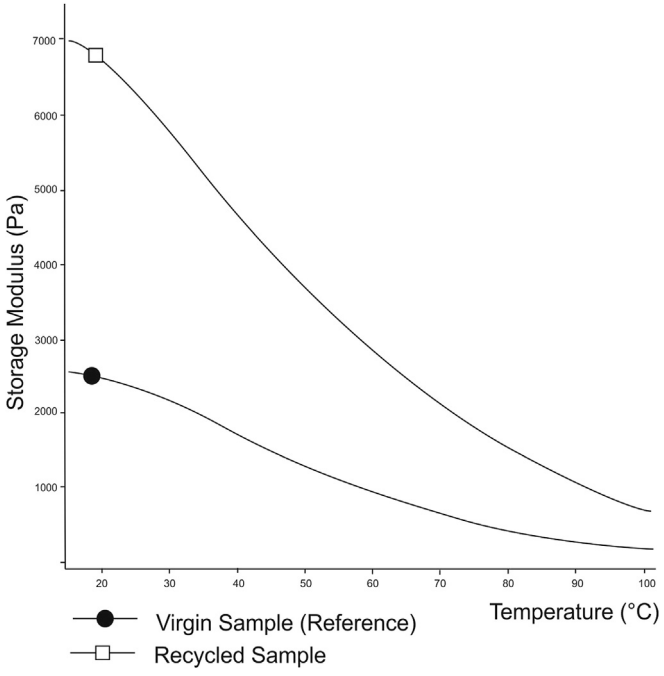


Fig. 11. Storage Modulus curves for both virgin and recycled samples.

The study showed the possibility of reprocessing multi-material toothbrushes, which were obtained by co-injection without the need for prior separation of materials. Moreover, reprocessing can be done in a few steps and with low material loss rate. The loss rate perceived was only that inherent of each process, since there were no losses from sorting and separation.

For the mechanical properties of the resulting material, this study has examined the tensile strength and the dynamic-mechanical thermal behavior. The results indicated, as expected, that the recycled micronized material presented inferior strength properties when compared to the virgin material, since recycling cycles in polymers usually worsens mechanical properties (Cândido et al., 2011). For this reason, currently, recycled materials are usually used as load in applications with no aggregated value, such as concrete (Isamail and Al-Hashmi, 2008; Siddique et al., 2008; Rao et al., 2007; Katz, 2003) or asphalt (Rezende and Carvalho, 2003; Akbulut and Gürer, 2007; Huang et al., 2007; Chiu et al., 2008).

In summary, the material resulting from the reprocessing of co-injected multi-material toothbrushes has potential applications in new products, however, the use of experimental material in this research should be considered, being necessary further studies on post consume multi-material products. Additionally, further studies on material characterization are necessary, in order to define the best applications for this specific material, considering its reduced mechanical properties compared to virgin materials.

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