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Different rotations in the acrylonitrile-butadiene-styrene extrusion process and their influence on fracture and mechanical properties

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ABSTRACT

ABS resins have wide application in the market, mainly in the manufacture of household appliances, telephones, automotive industry and others, and need to be processed by extrusion for the manufacture of products. The variables used in the process of extrusion of polymers have a direct influence on their mechanical properties. The objective of this study was to evaluate the different speeds of the extruder screw (15, 30, 45 and 60 rpm) on the mechanical properties of raw ABS after the extrusion process. Izod impact resistance and tensile tests, were also performed. The fractures of the samples tested by Izod impact were observed via optical microscopy, scanning electronic microscopy (SEM), and roughness was measured. Calorimetric tests (DSC) and thermogravimetry (TGA), as well as melt flow index (MFI), were also performed. The most significant results were observed for the extruder screw speed of 60 rpm. For the impact test, a reduction in impact energy of approximately 3% was observed, with the same result for tensile resistance. However, an increase in the value of the elastic modulus and the MFI was observed due to the increase in the degree of crystallinity, which was caused by the higher shear of the polymer mass in the screw of the extrusion equipment. Additionally, an atypical case occurred for the ABS polymer in the highest rotation, since it was the polymer that suffered the greatest shear in the polymer chains due to the degradation of the butadiene phase.

Keywords: Polymer; ABS; Processing; Extrusion; Mechanical properties.

1. INTRODUCTION

Polymers are one of the most versatile classes of materials, and are rapidly replacing materials such as metals and ceramics in a wide variety of technological applications [1–8]. Among the polymers, acrylonitrile-butadiene-styrene (ABS) stands out. The copolymer, formed by styrene-acrylonitrile (SAN), has characteristics such as thermal and chemical resistance to acrylonitrile, though the gloss, moldability and rigidity are conferred by styrene, the incorporation of butadiene rubber confers a greater mechanical resistance against impacts to the material [9].

The first parts molded using ABS polymer were sheets, profiles and tubes and, from the 1950s, their use expanded to industrial, automobile, aeronautical areas and nanotechnology [10–12]. As such, its applications are conditioned according to the concentrations of each monomer that constitute the ABS; however, the processing conditions can also interfere with the results [13].

The literature is very extensive in regards to studies on ABS, and includes studies related to degradation [14], composition in recycling [13], behavior in the extrusion process [15], and alternative polymeric mixtures [16] that aim to improve its properties. Using a mechanical analysis, the work carried out by BOLDIZAR and MÖLLER [14] sought to evaluate the degradation of ABS during repeated processing cycles, and it was observed that from the second to the sixth cycle the elongation of the rupture calculated by the tensile test presented a drastic drop, due to the repetitive extrusion processing. Through thermal and chemical tests, the alterations of the thermo-oxidative degradation of butadiene and the physical aging of the SAN phase were confirmed.

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In the study by MANISH *et al*. [13], through mechanical testing of new and recycled ABS with different chemical compositions of SAN and butadiene, it was observed that new ABS achieved the best tensile test results when compared with recycled ABS. In order to improve the tensile performance of recycled ABS, it was necessary to increase the additives of SAN and butadiene in new processes, for which their compositions varied from 5 to 60% physical (SAN) and elastic (butadiene) phases, in order to obtain an improvement similar to new ABS.

The research carried out by VARGHESE and MURUNGAN [15] investigated the influence of the deformation rate in ABS, high density polyethylene (HDPE) and isotactic polypropylene (PP–I) as a function of extrusion on the mechanical behavior. Uniaxial stress tests were performed in samples cut in three different orientations (0^o, 45^o and 90^o). It was found that the three polymers achieved an increase in the rate of deformation as a result of an increase in flow, irrespective of the orientations used in the tests. The researchers observed that the deformation rate that was applied exerts strong influence on the mechanical behavior, while the orientation of the extrusion direction exerts no significant influence.

In the work performed by LOPES and D'ALMEIDA [16], the researchers evaluated the addition of carbon fiber in different proportions in ABS. The thermal analyses showed that thermal stability is influenced by the weight percentage of carbon fiber in ABS. When comparing pure ABS and the carbon fiber reinforced material, an increase of up to 38% in tensile strength was noted in the carbon fiber reinforced ABS, with a 90% increase in Young's modulus. The results showed the great potential that carbon fiber has for improving the mechanical properties of ABS.

Thus, this work aimed to evaluate the influence of different revolutions (rpm) of the screw on the mechanical properties of ABS during the extrusion process. For this, tensile, flexion, and impact tests were performed. In addition, other analyses were performed, such as the melt flow index (MFI), thermogravimetric analysis (TGA), roughness and scanning electron microscopy (SEM).

2. MATERIALS AND METHODS

For the present study, natural ABS polymer LG ABS HF380 (high impact and high flow) was used in granule form, without pigmentation, with a specific weight of 1.04 N/m^3 with the contraction of this material varying between 0.4 and 0.7% and a melt flow rate of 43 g/min.

A single-screw extruder (TECKTRIL, EMT 35), with an L/D ratio of 24, capacity of 25 kg/35 h, was used. In this work, the following parameters of revolutions of the screw were employed: 15, 30, 45 and 60 rpm, and the processing temperatures adopted for all the rotations were 215 ºC in the feeding zone, compression zone at 225 ºC and dosing zone at 235 ºC.

After the extrusion process, the ABS was injected for the production of specimens for the tensile and impact tests. The injection parameters were the same for the extruded ABS at all revolutions. The injection process used the following parameters: injection speed of 75 mm/s, injection pressure 900 bar, booster pressure 800 bar, booster time 15 s, injection time 5 s, cooling time 30 s.

For this test, an electromechanical universal testing machine was used (Emic DL2000), with a 2 kN load cell, and equipped with the Tesc data capture program (version 3.04). The test was performed using the ASTM standard D638-010 (type-I sample) and with a traction speed of 50 mm/min at a temperature of 23 °C. A total of 10 specimens was used for this test.

The Izod impact test was carried out on an impact testing machine, (Tinius Olsen, 892). For this test, the ASTM-D 256-02 standard was used at a temperature of 23 °C. A total of 10 specimens was used for this test.

The surface roughness of the fracture from the impact test, was analyzed using a roughness meter (Mitutoyo, Surftest SV-400); the measurements were made in Ra with $\lambda = 0.8$ mm. A total of 5 measurements were performed in each specimen with different rotations. The measurements were performed along the length of the surface of the impact fracture and then the averages were calculated.

To obtain the melt flow index (MFI), using the viscometer, (DSM, MI-3P), the analyses were performed according to the ASTM-D 1238 standard. A total of 10 specimens was used for this test.

For this analysis, a simultaneous thermogravimetric analyzer and differential scanning calorimeter was used (TA Instruments, SDTQ600). In this characterization, an alumina crucible, purge gas with synthetic air at a flow rate of 50 mL/min, heating ratio 20 °C/min, final temperature of 700 °C and sample mass of 4 mg were used.

For the analysis of the fracture surface after the Izod impact test, a scanning electron microscope (Hitachi, TM 3000) was used. To obtain SEM images of fracture surfaces, SEM was used (JEOL IT 500HR).

3. RESULTS AND DISCUSSIONS

3.1. Tensile test

Figures 1 and 2 show the results of the tests of tensile strength, elongation, elastic modulus, and Izod impact as a result of the different ABS processing parameters adopted in the study. Figure 1 shows that the tensile strength and elongation remained constant, despite the deviation, at revolutions of 15 and 45 rpm. For the 60-rpm rotation, there was a reduction in tensile strength and elongation. The tensile strength and elongation behavior of the polymeric materials showed a decrease and this may be associated with increased screw rotation speed. Higher screw revolutions in the extrusion process caused an increase in the degree of molecular orientation and degradation of the butadiene phase (B) of the ABS molecule [13, 17]. The double bonds in the butadiene phase (B) were broken and produced oxygen-containing groups such as carbonyl and hydroxyl. These groups can cause phase separation, reduction of molecular weight and loss of mechanical properties [14, 18–20].

In the analysis of the elastic modulus, an increase was observed when using the parameter of 60 rpm (Figure 2). This behavior occurred due to the increase in the degree of crystallinity caused by the greater shear of the polymer mass in the screw of the extrusion equipment [20]. However, the ABS extruded at this same rotational speed showed a lower Izod impact resistance when compared with the ABS extruded at the other speeds (15, 30 and 45 rpm). A higher processing speed implied an increase in the process temperature of the polymer, which causes the reduction of the tangle of the chains in the elastic phase of the acrylonitrile styrene. Thus, this caused an increase in shear stresses in the ABS molecules, which may have led to their degradation [20, 21].

For the melt flow index, it is observed that there was an increase in viscosity (increase in MFI values) with the increase in screw rotation up to 45 rpm (Figure 2). The heating caused by the shear between the molecules and caused a thermo-oxidative degradation of the ABS molecules, thus causing the increase in the MFI value [16, 21].

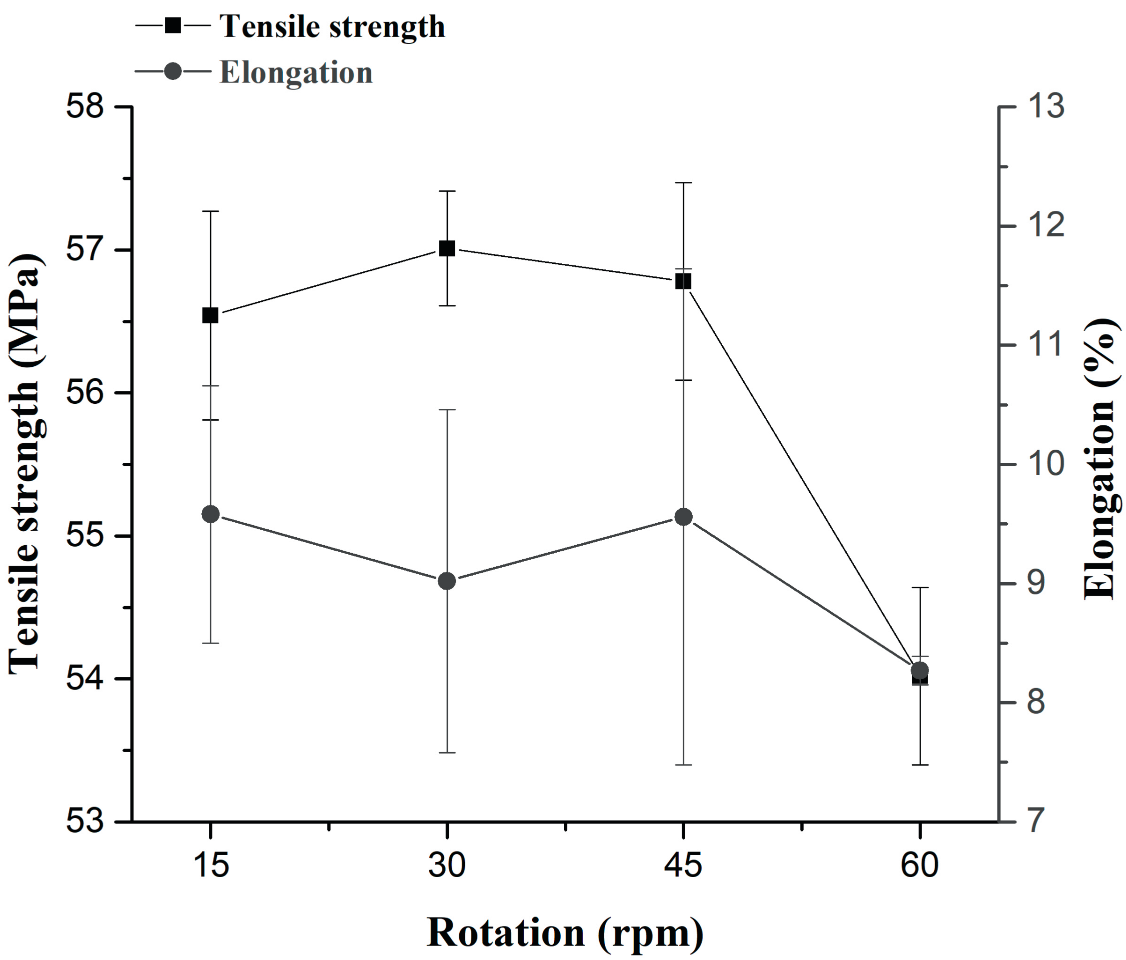

Figure 1: Tensile strength and elongation and different rotations of the screw during the extrusion process.

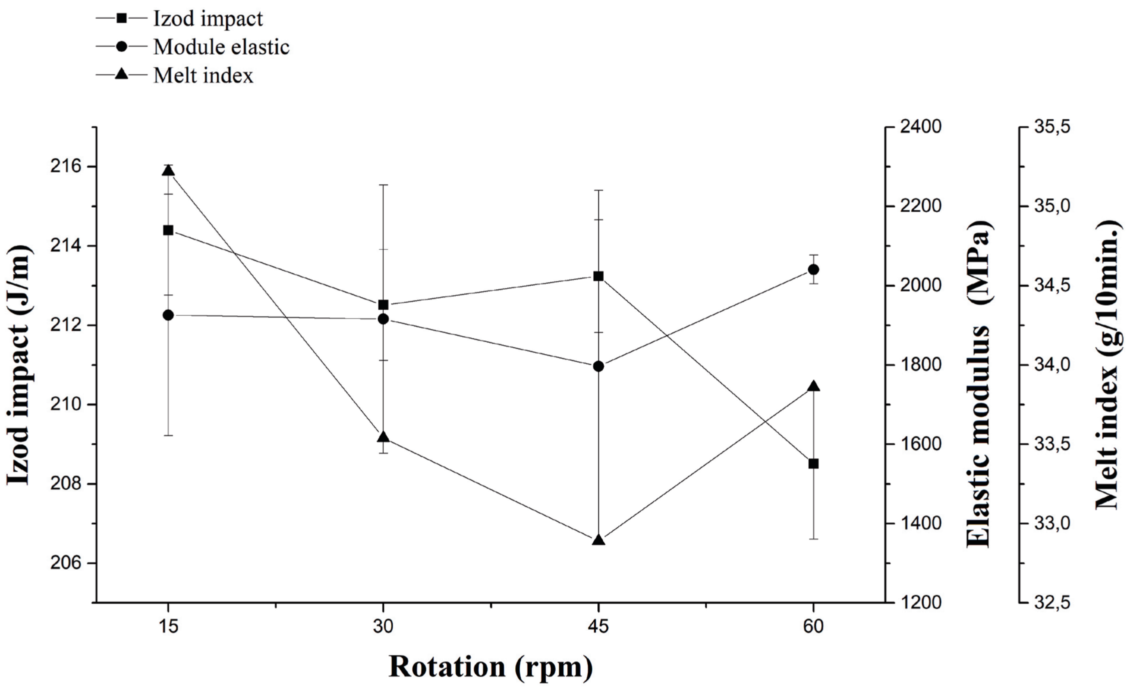

Figure 2: Influence of rotation on impact resistance, tensile strength, elastic modulus and the melt flow rate of the ABS.

3.2. Fracture

In order to complement the mechanical analysis, micrographs were obtained using optical microscopy (OM) and scanning electron microscopy (SEM). Roughness was also observed in order to observe the fracture surfaces of the ABS polymers processed at different screw rotation speeds. Thus, it is possible to correlate the nature of the fracture with the Izod impact assay.

According to CORREA *et al.* [22], ductile fractures in polymers present shear regions, whereas brittle fractures are represented by mirror or smooth surfaces resulting from the possible cleavages. This behavior exposes possible energy paths of crack propagation, stress concentrations, and defects in the processed material.

In tenacious thermoplastic polymers, such as ABS, which has a fragile matrix, when fractured at room temperature, it typically has ductile fracture characteristics with the main energy release through the formation and growth of multiple micro-fibrillation (crazing). This fracture mechanism in ABS is a consequence of the local flow around the largest elastomeric particles due to the high stress concentration, which causes the formation of voids that propagate in the normal direction at the maximum stress. However, the voids are stabilized by the polymer fibrils that support the opening and prevent the crack from growing. In practice, the smaller polybutadiene particles do not normally initiate this mechanism, but are responsible for the formation of voids (cavitation of the particles due to cohesive or adhesive failure). This contributes to the advancement of crazing, and triggers other mechanisms and also the phenomenon of bleaching (stress whitening) in the material [23-27]. According to NAGARAJAN *et al.* [28], this phenomenon is related to the high plastic deformation of the polymer, which modifies the refractive index of the material and causes the light to spread and reflects all wavelengths of visible light. In addition, as an additional fracture mechanism, it can present flow under shear (shear yielding).

In this context, Figure 3a-d illustrates the fracture surface images obtained by scanning electron microscopy (SEM) after the impact test of the ABS samples produced at revolutions of 15, 30, 45 and 60 rpm, respectively. It is also worth mentioning the optical microscopy images of the samples (Figure 3). In Figure 3, it can be observed that in the sample extruded at 15 rpm, there was a higher incidence of bleaching, while, in the revolutions of 30, 45 and 60 rpm, the occurrence of this bleaching behavior was lower.

The chemical explanation for this phenomenon is related to the SAN phase (matrix phase), which is responsible for the adhesion between the phases in the ABS polymer [21, 22, 29]. As the speed increases in the process, this phase decreases and, thus, the transition phase appears, which is represented by black circles. BAI *et al.* [21] describe that with an increase in revolutions in the polymer process, the SAN phase is degraded, thus reducing the molecular interaction with butadiene, which leads to morphological changes as well as thermal and mechanical properties. In addition, according to XU and XU *et al*. [25], the difference in the behavior of the different types of the ABS polymer is associated with the quantity, composition, morphology and the distance between the particles of the tenacious phase.

Additionally, within the transition phase, in the magnification of Figure 3, it is visually perceived that the fracture surface in the images obtained by SEM of the fractured ABS is rough at the speed of 15 rpm and this becomes apparently rougher and has more micro-voids at the speeds of 30, 45 and, mainly, at 60 rpm. As a result, roughness tests were performed to elucidate the reason for this phenomenon. The verification of the irregularities of the fracture surface can be performed by measuring the average surface roughness (Ra). The

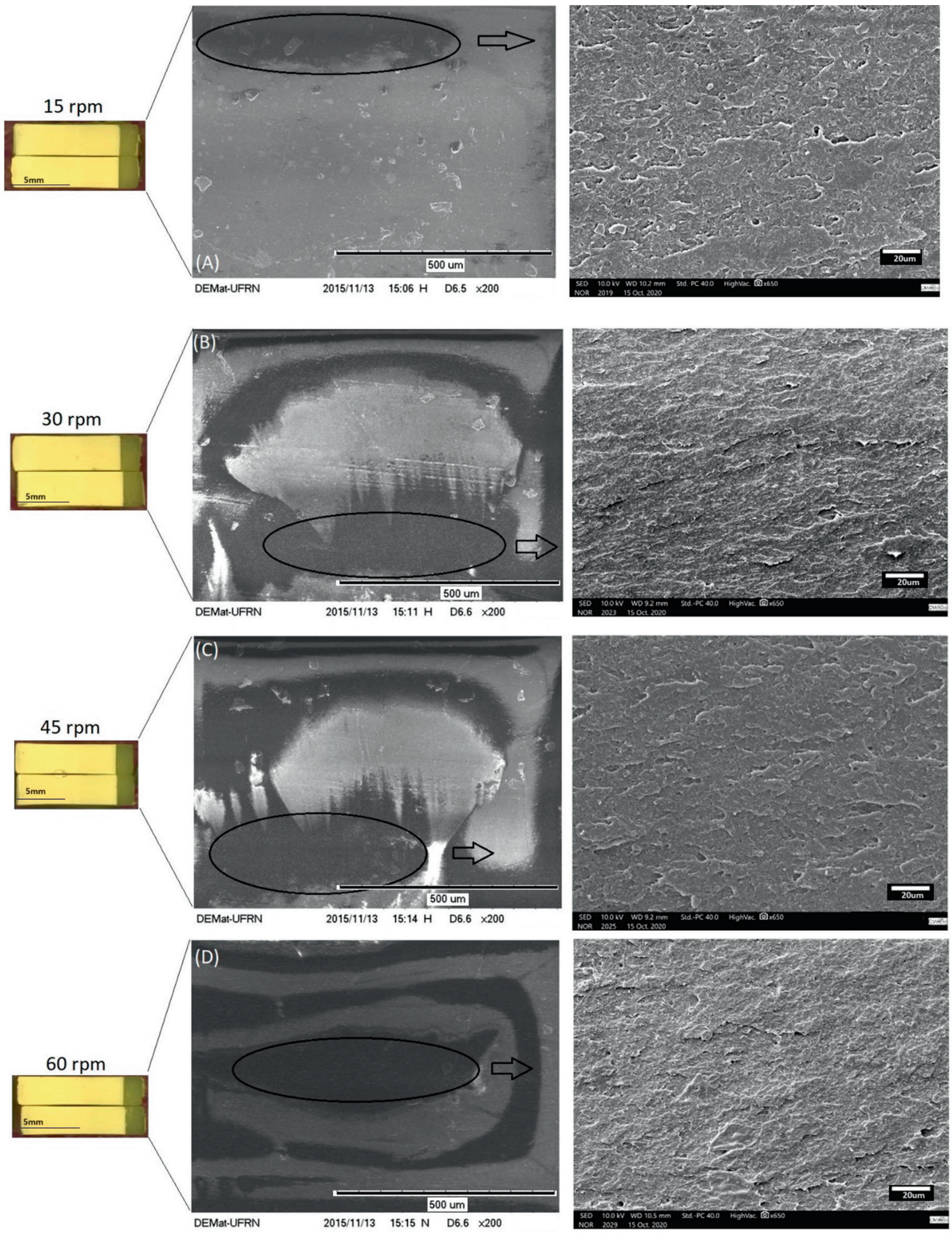

Figure 3: Optical microscopy (OM) images illustrating the fracture surface after the Izod impact test via SEM in two magnifications of the samples extruded at different speeds, (a) 15 rpm and (b) 30 rpm (c) 45 rpm and (d) 60 rpm.

roughness on the fracture surface was measured at 5 points along the sample. Figure 4 shows the results obtained for Ra after the fracture surface of the samples was tested with the roughness meter.

Initially, the fracture of the pure ABS polymer at room temperature is considered ductile, as illustrated by SEM images (Figure 3) as well as the Izod impact energy results illustrated in Figure 2. According to Figure 4, the samples made using a rotation speed of 15 rpm have the lowest surface roughness value Ra (0.60 µm), while at 30 and 45 rpm, they have a similar behavior, considering the margin of error of the measurements. In other words, the values varied by approximately 0.70 µm. However, for the rotation of 60 rpm, the value of Ra was approximately 0.75 µm.

The roughness values are directly related to the amount of energy dissipated during the fracture. Higher Ra values result in greater deformation or irregularity on the fracture surface and are consequently related to higher impact energy values. As previously described, in the fracture images, the cavitation and multiple microfibrillation processes are the main fracture mechanisms observed in this work. An atypical case occurred for the ABS polymer made at the highest rotation since it was the polymer that underwent greater shear in the polymer chains, due to the degradation of the butadiene phase (unsaturated bonds C=C). This is the most susceptible to degradation initiated by a thermo-oxidative and self-catalyzed mechanism [21, 30, 31], and obtained the highest value for average surface roughness Ra.

According to the data obtained for Izod impact energy, at the highest rotation speed of the screw (60 rpm), there was a reduction of approximately 3% of the impact energy value (J/m), i.e., a reduction in impact resistance and toughness. However, even with the reduction of energy release, through the reduction of the transition phase (whitening), the polymer remained ductile after undergoing thermo-oxidative and self-catalyzed degradation. The superior results of mean roughness (Ra) obtained in the fracture region for this polymer corroborate the evidence obtained from the images obtained using electron microscopy (SEM) presented in Figure 3.

3.3. Thermogravimetric and calorimetric analysis (TGA/DSC)

In order to observe the effects of rotations (rpm) on the ABS (terpolymer) obtained at 15, 30, 45 and 60 rpm, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed (Figure 5). According to Figure 5a, the materials begin to degrade and this process occurs in two stages; the first stage starts near 200 ºC ending at ~ 475 ºC and the second stage occurs from 475 to 600 ºC. In the first stage of degradation, it is possible to observe that several chemical reactions occur simultaneously such as functional groups of esters and benzenes [32–35].

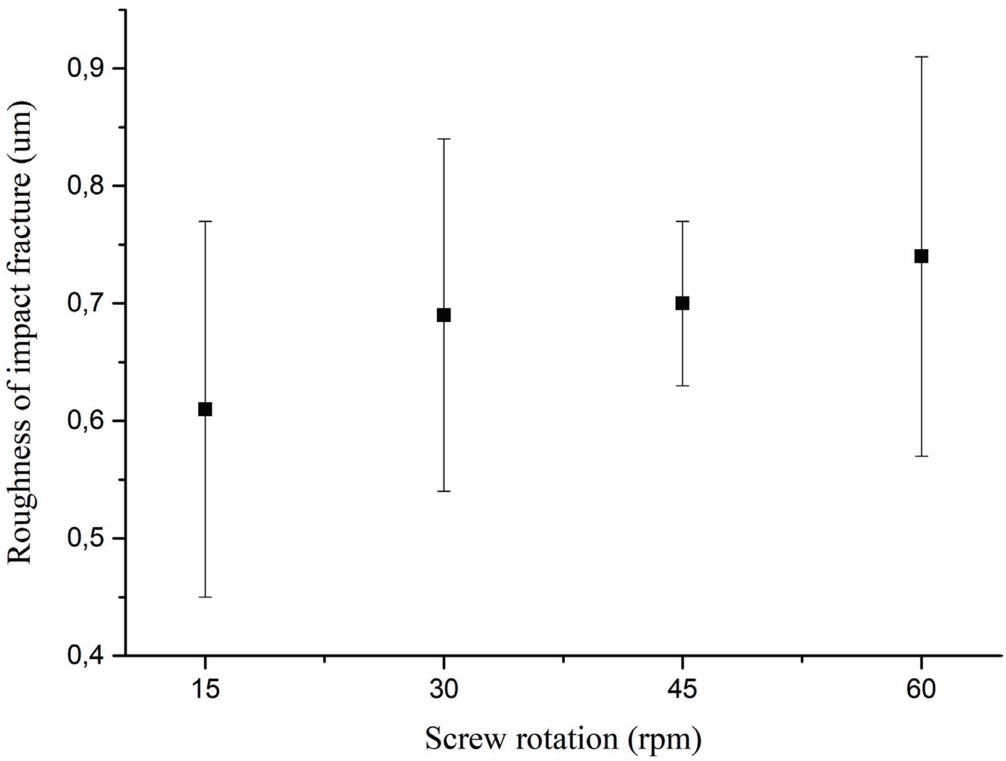

Figure 4: O Average roughness (Ra) of the surface of the fracture of the ABS polymer at different screw rotational speeds.

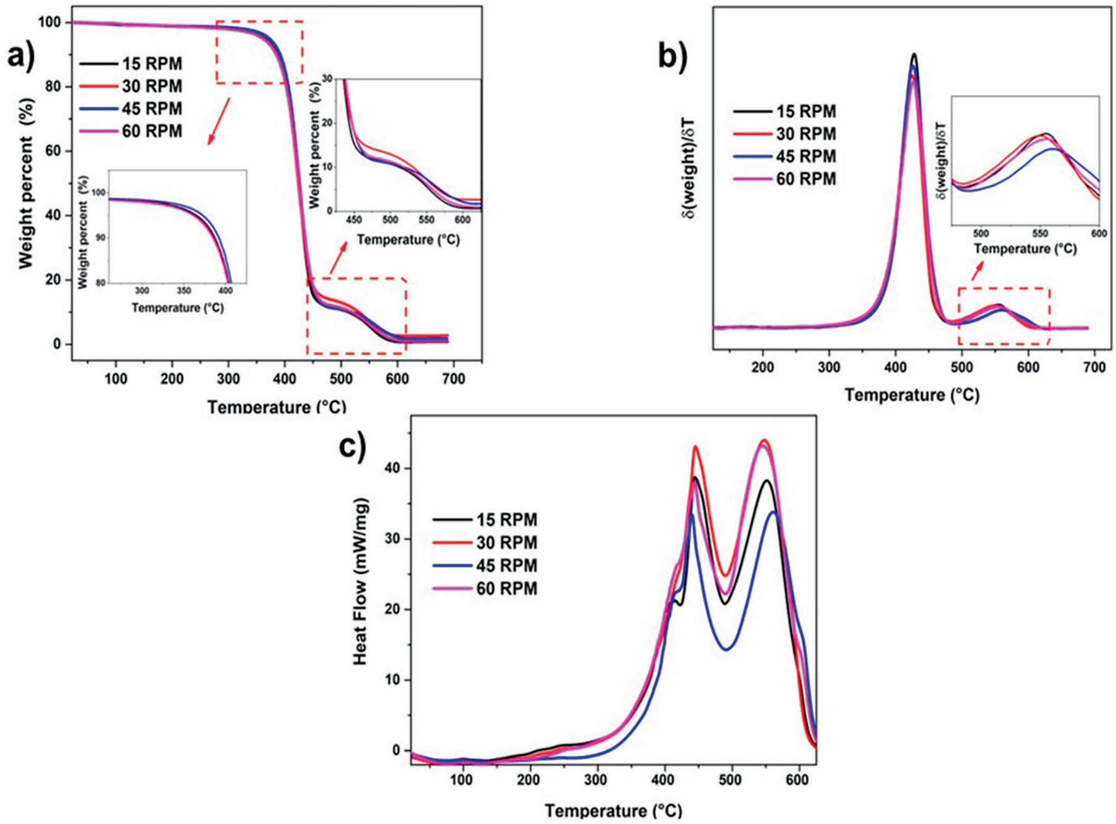

Figure 5: Thermal behavior (a) TGA (b) DTG and (c) DSC of extruded ABS samples at different processing speeds.

In the first stage of degradation of the ABS samples that were studied, between temperatures of 325 to 405 ºC, this is associated with decompositions of butadiene ~ at 345 ºC and 365 ºC for styrene; while, at 412 ºC, the degradation of acrylonitrile begins, such reports are also described in the works of YANG *et al*. [26] and FENG *et al*. [35]. In the second deposition step (475 to 600 ºC), this is associated with the end of the degradation of acrylonitrile. It was observed that between ~ 200 ºC and 325 ºC other decompositions occur; however, the reactions involved are volatiles that exist in the ABS [36-38], similar observations are also described in the literature [39–42].

In this study, all the thermograms obtained showed similar behavior in their degradations, so much so that, via the DTG (Figure 5b), it is possible to observe such behavior. With regard to the percentages of the masses of decay from the ABS degradation process obtained in the rotations used in the work, their decompositions are 2.5 and 3.5% for butadiene and styrene, respectively. For acrylonitrile, for which the beginning of its decomposition already occurs in the first step, there is an initial loss of ~ 7.25% mass and, at the end of the second step, the degradation. The resulting residues of the materials are 2.3% (30 rpm), 1.9% (45 rpm), 1.1% (60 rpm) and 0.90% (15 rpm).

4. CONCLUSIONS

In this study, it was possible to evaluate the effects of different extruder screw speeds (15, 30, 45 and 60 rpm) in the extrusion process on the thermal, mechanical and morphological properties of ABS380. The different extrusion speeds of the polymer affected the mechanical properties studied to a greater degree, mainly the toughness, elastic modulus and melt flow index. For the highest speed (60 rpm), through the thermal analyses, it was observed that there are no chemical changes arising from the thermal stability when compared with the other rotational speeds used in the ABS, even when there is greater friction in the polymer chains during processing. The effects of this degradation were ob-served in the reduction of tensile strength, Izod impact energy and elevation of melt flow index. Additionally, even with the highest thermal degradation, the average roughness (Ra) results and the SEM images of the fracture surfaces showed that the polymer processed at this speed (60 rpm) remained ductile compared to the lower screw speed of 15 rpm. In this context, this work showed how important it is to evaluate the alterations in the parameters of polymeric processing on the final properties of polymers; however, is often neglected in various industries. Finally, depending on the desired properties, the same polymer can meet several specifications, though it is necessary to carry out processing in the correct manner in order to obtain the best properties.

5. ACKNOWLEDGMENTS

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