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Research Article

Synthesis and Thermomechanical Characterization of Nylon 6/Cu Nanocomposites Produced by an Ultrasound-Assisted Extrusion Method

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A nylon 6 nanocomposite with copper nanoparticles processed by ultrasound-assisted extrusion was prepared at concentrations between 0.01 and 0.50 wt.%, and its thermal and mechanical properties were determined. The presence of the crystalline phase α (α_1 and α_2) in the polymer matrix was confirmed by X-ray diffraction, and the presence of the α_2 phase showed a greater increase than the α_1 phase as a function of the copper nanoparticle concentration. This process was attributed to secondary crystallization. Furthermore, it was determined that the chemical composition of the nanoparticles is a blend of metallic copper and cupric oxide. The formation of copper nanowires was observed by scanning electron microscopy, and the concentration of 0.10% exhibited the best dispersion in comparison with the other concentrations. The melting temperature of the nanocomposites underwent a slight decrease in comparison with the nylon 6, while thermal stability, crystallization temperature, and crystallinity were increased in relation to the pure polymer. This behavior is attributed to an efficient dispersion of the nanoparticles and to their functionality as crystal nucleation sites. For the 0.10% concentration nanocomposite, higher mechanical properties were obtained; tensile strength increased by 8.9%, and the tensile modulus increased by 25.4%; as a consequence, elongation at break was 62% less than that of the polymer matrix.

1. Introduction

Currently, one of the ways to improve the properties of polymers is to disperse inorganic nanoparticles to form polymer nanocomposites; to mention some of the properties that can be improved and that are of interest for technological development, we have mechanical properties, thermal and dimensional stability, antifriction characteristics, catalytic capability, electrical and thermal conductivity, protection against UV radiation, flame retardancy, and gas barrier properties, among others [1, 2]. The improvement depends not only on the chemical nature of the nanoparticles, but on concentration, particle size, morphology, dispersion, and spatial distribution, as well as the crystallinity of the matrix, usually influenced by the presence of the nanoparticles [3, 4]. The improvement of the properties has been so successful that nanocomposites have been used in different industrial applications in many fields such as automotive, electrical, electronic, food packaging, and textiles [5, 6].

In particular, nanocomposites based on nylon 6 formulated with copper nanoparticles have aroused the interest of the academic and industrial scientific community since they exhibit excellent properties such as high tensile strength and tensile strain modulus [7–9], high electrical conductivity [10] and thermal stability [11], attractive optical properties [12], improved tribological characteristics [13, 14], and many others. For this reason, these nanocomposites are attractive in applications where high-performance materials are required, such as electrical and electronic markets, because they perform better than the alternative materials [11, 15].

However, only a few papers regarding this composite have been published. In 2015, Xu et al. obtained nylon 6 nanocomposites with nanoparticles of metallic copper [14]. The polymer was synthesized by ring-opening polymerization of ε-caprolactam and copper oxide as a metallic source at the concentrations of 0.1, 0.3, 0.5, 0.7, and 0.9 wt.%. After polymerization, the nanoparticles with an initial size of around 4-5 nm formed clusters with a size of 30 to 50 nm. The authors claimed the presence of copper metallic nanoparticles by X-ray diffraction (XRD), but small peaks at around $2\theta = 40^{\circ}$ may be associated with copper oxide and a change of the crystalline phase α to the γ phase was observed as the concentration of copper oxide increased. The tensile strength of the 0.3 wt.% sample increased by 47.5% compared to that of the nylon 6.

In 2013, Komeily-Nia et al. reported the preparation of copper nanowires in polyamide 6 composites at 0.5 wt.%, by the chemical reduction of copper sulfate; in this case, the authors claimed the presence of metallic copper and the presence of the crystalline phase α of the nylon 6, as confirmed by the X-ray diffractograms and the energy disperse spectrometers. Scanning electron microscopy showed that the nanoparticles had an average size of 85 nm, and the tensile strength of the nylon 6 increased by 9% with the addition of the nanoparticles [7].

Copper nanowires in nylon 6 were also observed by Xu et al. in 2013; the polymerization in situ was kept in a reducing atmosphere in order to prevent the oxidation of the nanowires found at the concentration of 0.5 wt.%. In this case, the presence of the nanowires did not affect thermal stability or melting temperature, but crystallinity increased by 1.38%, crystallization temperature decreased by 1.79°C, and tensile strength increased by 77.4% compared to the pure polymer [8].

Martelli et al. synthesized the nylon 6 nanocomposite with metallic copper nanoparticles, with a diameter of around 8 nm at 5 wt.%, through a ring-opening polymerization of the ε-caprolactam monomer. The reaction was carried out under high vacuum conditions in order to avoid the oxidation of the copper and the polymer. In spite of the presence of the nanoparticles, no significant changes in thermal conductivity or polymer crystallinity were observed [12].

In 2009, Rusu and Rusu synthesized nylon 6-copper microcomposites at concentrations from 0 to 25 wt.% through anionic ring-opening polymerization of ε-caprolactam. The X-ray diffractograms confirmed the formation of metallic copper, and the transition from phase α to phase γ of the nylon 6 was associated with the increase in copper concentration; as long as melting temperature decreased, crystallinity temperature increased and the degree of crystallinity decreased as well. The microcomposites at concentrations of 6 and 15% were analyzed by X-ray diffraction and only the metallic copper peak at $2\theta = 43.4^{\circ}$ was found. For the specific concentration of 25%, the decomposition temperature of the composite was 450.5°C, higher than the 428.0°C reported for the nylon 6 [11].

As previously mentioned, most authors have used in situ polymerization to obtain the nylon 6 composite. Despite the improvement in the mechanical properties reported in these works, the synthesis involves expensive reactants, specialized equipment, very specific reaction conditions, long reaction time, and low production capacity.

As an alternative, this work presents synthesis by ultrasound-assisted reactive extrusion. The procedure has the following advantages: (i) the reaction is held in the melting state without the use of solvents, (ii) it is possible to handle many polymers or copolymers branched or grafted with distribution of molecular weight, even to recycle polymers, (iii) the reaction time is in the order of minutes and more product can be processed simultaneously and continuously, and (iv) the required equipment has standard characteristics and flexibility in the control of the process variables [4, 16]. It is important to note that the production of the nanocomposite without the formation of copper oxide nanoparticles is quite complicated due to the strong tendency of the metallic copper to form the oxide metal under environmental conditions [17].

The aggregation of the nanoparticles inside the polymer matrix is related to the high superficial energy of the particles [14]. Usually the presence of the aggregates implies a decrease in the mechanical and thermal properties of the nanocomposite. Furthermore, it is well known that aggregation can be avoided by ultrasonic waves at a specific power and frequency without degradation of the polymer matrix [18, 19]; for this reason, ultrasound is implemented during the reactive extrusion in this work.

This document reports the synthesis of nylon 6 nanocomposites formulated with different contents of metallic copper nanoparticles (nylon 6/nano-Cu), using the melt mixing route in a single-screw extruder. The preparation was carried out using an ultrasound system to disperse the nanoparticles inside the nylon 6 matrix. The physicochemical characterization of the raw and obtained products was done by different experimental techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and the tensile strength test. It should be mentioned that currently, in the scientific literature, there is no report of a similar work and that only a few studies of nylon 6 nanocomposites with metallic copper particles (described above) are available.

2. Experimental Methods and Materials

2.1. Chemical Products. Granulated nylon 6 commercialized as Zytel®7301 NC010 by Dupont™ with a density of 1.13 g/cm^3 and a melting point of 230°C [20] was used. Likewise, metallic copper nanoparticles with an average

diameter of 21 nm obtained by a previously reported procedure were used [21, 22].

2.2. Preparation of the Nylon 6 and Synthesis of the Nanocomposites. First, the nylon 6 was dried in a vacuum oven at 80°C for four hours according to the data sheet provided by the supplier [20] Then, the pure polymer and the samples with a concentration of metallic copper nanoparticles of 0.01, 0.05, 0.10, and 0.50 wt.% were processed under the same conditions in the single spindle Moreli extruder with a length/diameter ratio of 13.0, set to 230°C at 23 rpm, a feed of 400 g/h, and a residence time of 15 minutes. An ultrasonic device with a power of 450 W and a frequency of 35 kHz was coupled with the extruder. In every sample, 100 g was taken and used for the physicochemical characterization.

2.3. Characterization Techniques

2.3.1. X-Ray Diffraction (XRD). To determine the crystalline structure of the nylon 6 and the nanocomposites, X-ray diffraction was used. The samples were evaluated in a diffractometer model D-5000 manufactured by Siemens, operated at a current intensity of 25 mA and a voltage of 35 kV to obtain the radiation corresponding to the Cu Kα at a longitude wave of 1.54056 Å. The scanning in the 2θ scale was from 15° to 80° with a sample velocity of 0.02°/s.

2.3.2. Scanning Electron Microscopy (SEM). The morphology of the polymer and its nanocomposites were determined by means of a field-emission scanning electron microscope model JSM-7401F manufactured by Jeol. A secondary electron detector, LEI, was fixed with a voltage of 3.0 kV and a working distance of 3 mm. The samples were frozen in liquid nitrogen and fractured. In order to make their surface conductive, the samples were coated with a gold-palladium alloy using a sputtering technique. All micrographs were taken at a magnification of 30,000x.

2.3.3. Differential Scanning Calorimetry (DSC). The polymorphic structure, melting temperature, crystallization temperature, and degree of crystallinity of all the samples were determined by means of a calorimeter model DSC-2920 Modulated DSC provided by TA Instruments. The heating and cooling rate were set at 10°C/min, and a high purity nitrogen (UAP, 99.999%) with a flow rate of 50 mL/min was used to avoid oxidation of the samples, and between 10 and 15 mg of each sample was tested from 0 to 260°C. The degree of crystallinity was calculated using the following equation [23]:

$$X_{\mathrm{C}}(\%) = \frac{\Delta H_{\mathrm{f}}}{(1-\varnothing)\Delta H^{*}} \times 100, \quad (1)$$

where ΔH_{f} is the heat of fusion of every sample and ΔH^{*} is the heat of fusion of the pure nylon 6 with 100% crystallinity, equal to 188 J/g for the specific case of our polymer, and $\varnothing$ is the weight fraction of the copper nanoparticles.

2.3.4. Mechanical Properties. Mechanical properties were determined according to the *Standard Test Method for Tensile Properties of Plastics* ASTM D638-10 [24] in a universal materials testing machine model 4301 supplied by INSTRON. The operating conditions were as follows: the length between jaws (L) was 25.4 mm, a load cell of 100 kg, and an elongation velocity of 5 mm/min. All tests were carried out at room temperature, and 5 specimens were tested for every polymer or nanocomposite. To obtain the testing probes, the processed polymer and the nanocomposites were injected in a proper mold with an injection machine manufactured by Dynisco. Before the probes were tested, they were vacuum dried at 80°C for four hours in order to eliminate the residual humidity in the samples. The statistical analysis indicated that the standard deviation was below 2.6% for tensile strength stress, 2.2% for tensile strength modulus, and 3.0% for tensile elongation at break.

3. Results and Discussion

3.1. X-Ray Diffraction of the Polymer and Composites. The diffractograms of the pure nylon 6 and the nanocomposites for the concentrations of 0.01, 0.05, 0.10, and 0.50 wt.% are shown in Figure 1. It can be seen that the pure nylon 6 shows peaks at 20 and 23.7° with crystalline planes (200) and (002, 202), attributed to the crystalline phases α_1 and α_2, respectively [25]; both signals have nearly the same intensity. In the case of the nanocomposites in Figures 1(b)–1(e), the same peaks remain after the incorporation of the copper nanoparticles whose presence is confirmed for the new peaks observed. The intensity of the crystalline plane (002, 202) is higher than the intensity of the reflection (200) which indicates that the nanoparticles favor the formation of the α_2 phase in the polymer matrix. This behavior has been previously observed by other authors in spherical nanoparticles dispersed in nylon 6; these authors concluded that this phenomenon is closely related to a secondary crystallization process, where crystal growth is held onto the crystalline plane with the higher energy level [26–29].

The new peaks located at 37.1° and 43.3° were associated with the nanoparticles. The first corresponds to the crystalline plane (111) assigned to the copper II oxide (Cu_2O) with a body-centered cubic structure (BCC) [29], while the second is related to a crystalline plane (111) assigned to the metallic copper (Cu^0) with a face-centered cubic (FCC) [7, 8]. As the concentration of nanoparticles increases those peaks increase as well, the peak corresponding to the oxide increases in a higher proportion which means that the increase in concentration increases the oxidation of the metallic copper nanoparticles. The transition from metallic copper to the oxidized state is attributed to the conditions during the preparation of the nanoparticles, for example, the presence of air, the residence time, and the process temperature.

3.2. Morphology and Nanoparticle Distribution. The surface of the pure nylon 6 after the fracture at low temperatures is presented in Figure 2. It can be seen that the fracture was

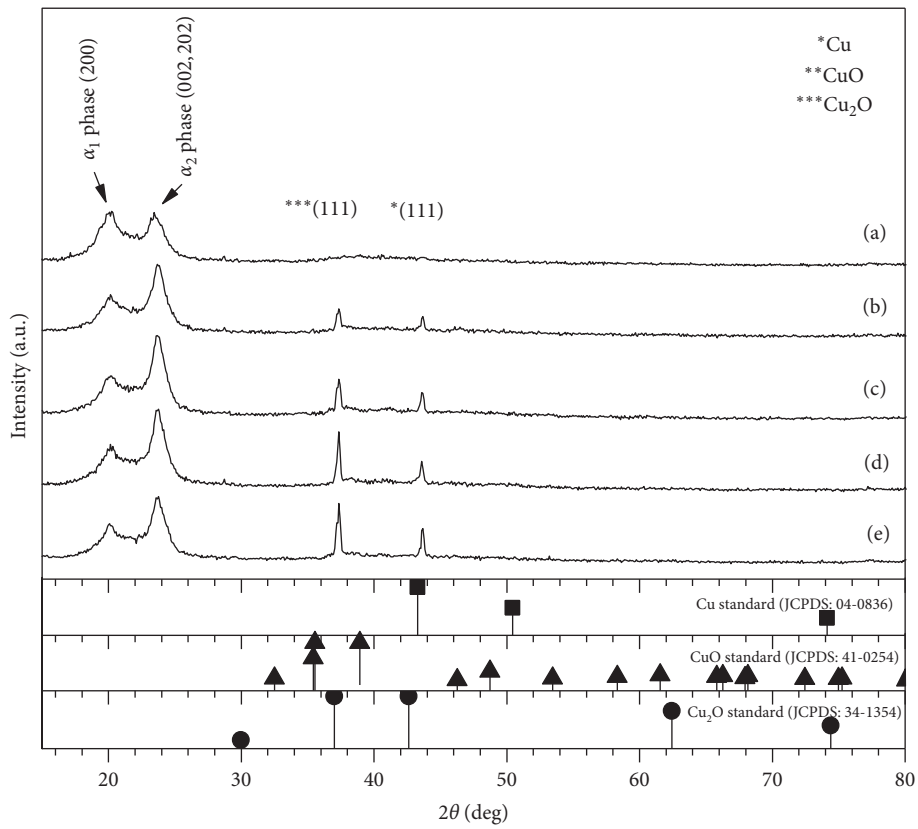

Figure 1: XRD diffractograms for all the samples: (a) pure nylon 6 and nanocomposites at the evaluated concentrations: (b) 0.01, (c) 0.05, (d) 0.10, and (e) 0.50 wt.% of copper nanoparticles.

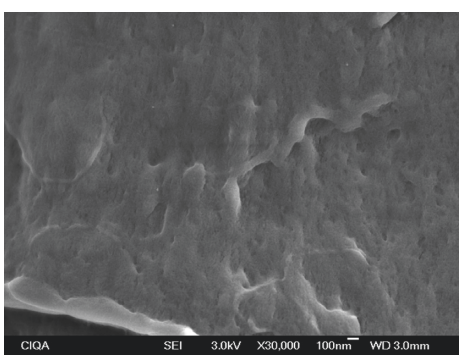

Figure 2: Fractured surface of the nylon 6.

ductile, due to the fact that it undergoes a plastic deformation and there is no evidence of tear and the surface remains smooth and uniform.

Figure 3 shows the micrographs corresponding to the obtained nanocomposites. At the low concentration of nanoparticles (0.01 and 0.05%), the matrix of the polymer has a smooth appearance without the presence of tears or hollows, and some spherical nanoparticles smaller than 100 nm can be observed. In the case of 0.05%, some aggregates have the same morphology as that exhibited by the monodispersed nanoparticles.

In particular, Figure 3(c) shows the surface of nanocomposite with a weight percent equal to 0.10 of copper nanoparticles. The polymer matrix of this nanocomposite exhibits zones with smooth features and others with the presence of cracks. As can be seen, scattered spherical particles with sizes less than 100 nm and aggregates in form of wires are observed. The particles and nanowires of the nanocomposite exhibited are brighter than those of the matrix, perhaps due to the good electrical conductivity of the copper nanoparticles. Chen et al. [30] prepared nylon 6 nanocomposites with different amounts of multiwalled nanocarbon tubes (MWNTs) using the fusion blend method. By SEM analysis of a fractured sample with liquid nitrogen, they saw high brightness in the MWNTs, attributed to their high electrical conductivity.

Figure 3(d) illustrates the micrograph of the nanocomposite with 0.50 wt.% of copper nanoparticles. It shows that the polymer matrix has smooth characteristics and also some cracks. Additionally, monodisperse spherical particles and aggregates of subparticles of smaller size were observed. The distribution of both isolated particles and agglomerates is relatively uniform. In addition, a higher content of nanoparticles is visualized compared to the other nanocomposites, and this agrees with the XRD results shown in Section 3.1.

3.3. Thermal Characterization. The formation of crystals of different sizes of the nylon 6 polymer matrix on the bulk of the nanocomposites during the heating-cooling process can

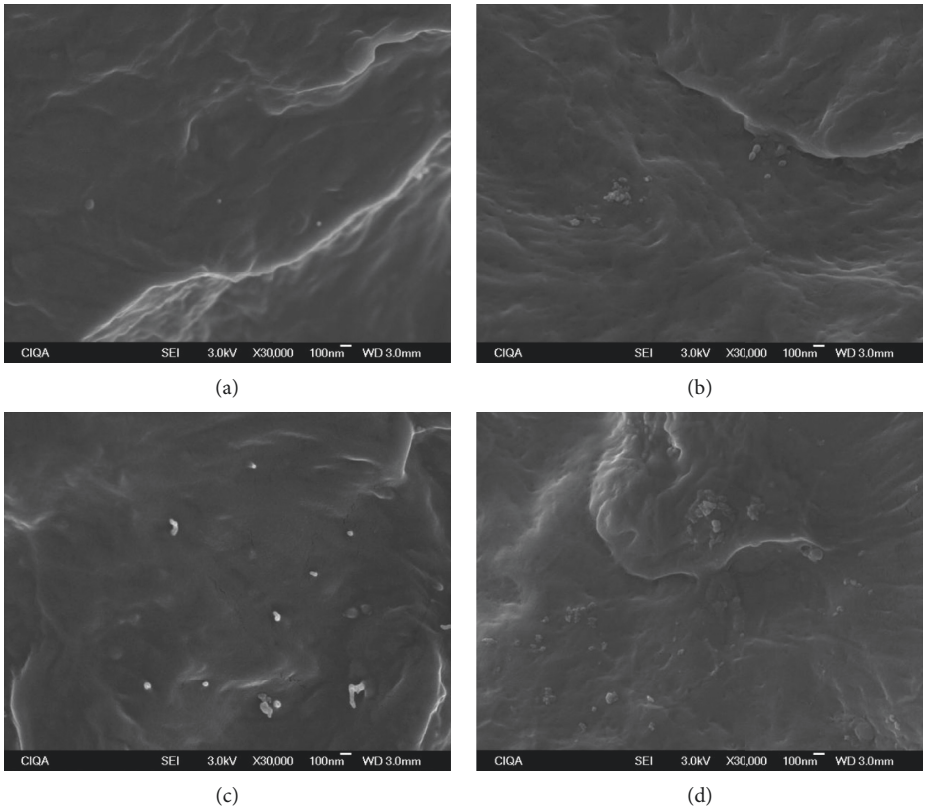

Figure 3: Fractured surface of the nanocomposites: (a) 0.01, (b) 0.05, (c) 0.10, and (d) 0.50 wt.% of copper nanoparticles.

show significant dependence on the content of copper nanoparticles. In this case, it is essential to study the processes of melting and crystallization of the polymer matrix in nanocomposites using DSC thermal analysis.

3.3.1. Effect of the Nanoparticles in the Fusion Process. The endothermic thermograms of the nylon 6 and the nanocomposites at the concentrations of 0.01, 0.05, 0.10, and 0.50 wt.% of nanoparticles are presented in Figure 4.

In Figure 4(a), for the nylon 6, the fusion endotherm exhibits a single peak with a melting temperature (T_m) of 222.3°C attributed to the crystalline phase α, in agreement with the scientific literature [28]. On the contrary, for the nanocomposites, two well-defined peaks were found. For the nanocomposites at 0.01, 0.05, and 0.1 wt. %, in Figures 4(b)–4(d), the melting temperature of peak 1 decreases compared to the pure polymer with values of 222.1, 221.5, and 220.8°C, respectively. The decrease in the melting point may be related to the presence of the copper nanoparticles which acts as a nucleation promoter [31]. Figure 4(e) shows the endotherm of nanocomposite at 0.50 wt.% with a melting temperature of 221.8°C, quite similar to that of the 0.05 wt.% concentration. This behavior is evidence of the possible rearrangement of the polymer crystals which undergo a slight growth in size [32]. Thus, such variations in the melting temperature support the idea that the nanoparticles participate in the nucleation process of the polymer matrix [11].

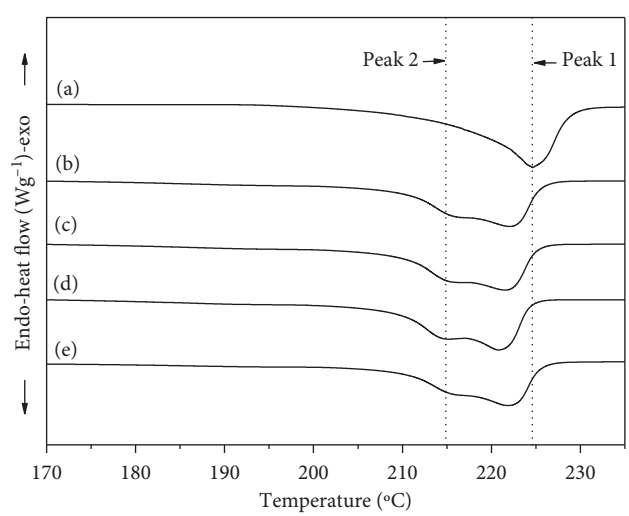

Figure 4: Endothermic thermograms of the nylon 6 (a) and the nanocomposites at the concentrations of 0.01 (b), 0.05 (c), 0.10 (d), and 0.50 (e) wt.%.

The second peak observed for the nanocomposites shown in Figure 4 and found at approximately 215°C has a lower intensity than that of peak 1. In general, the presence of a second peak can reveal that the crystals have imperfections or defects in the crystalline structure and in their spatial distribution [33]. This peak has also been observed in other nanocomposites prepared with different nanoparticles in the development of engineering plastics such as

polyethylene terephthalate and polycarbonate. The authors of those works attributed the presence of the second peak to a secondary crystallization process in the polymer matrix that occurs after the formation of the primary crystals which are more thermodynamically stable [34, 35]. Other authors have associated this behavior with the formation of the crystalline phase γ [11, 36].

3.3.2. Effect of the Nanoparticles on the Crystallization Process. The exothermic thermograms of the nylon 6 and the nanocomposites at the concentrations of 0.01, 0.05, 0.10, and 0.50 wt.% of nanoparticles are shown in Figure 5.

The crystallization exotherm of the nylon 6 is shown in Figure 5(a); the wide crystallization peaks have a crystallization temperature (T_c) of 188.7°C and a degree of crystallinity of 42.7%. On the contrary, for the nanocomposites with concentrations of 0.01, 0.05, and 0.1 wt.% of copper nanoparticles, a single peak slightly more narrower than that of the nylon 6 with an increase in the crystallization temperature at values of 189.9, 190.5, and 190.8°C for the respective concentrations was observed. Additionally, the degree of crystallinity slightly increases, producing the corresponding values of 43.1, 45.9, and 46.9, respectively. In this case, it is difficult to draw conclusions about the origin of this behavior because opposite behaviors have been reported in the literature: an increase in the degree of crystallinity [8], a decrease in this variable [11], and an insignificant effect of the nanoparticles on this variable [12]. However, our findings do support the idea that the copper nanoparticles change the nucleation process in the polymer matrix, as has been reported by the other authors [27, 32, 37].

The exotherm of the sample with a concentration of 0.5 wt.%, presented in Figure 5(e), exhibits a completely different behavior with a crystallization temperature of 189.3°C and a degree of crystallinity of 40.1%. In these cases, the crystallization temperature decreases to a value closer to that corresponding to the concentration of 0.01%, the lowest crystallinity degree of all of the nanocomposites, even considering the pure polymer. It can also be seen that the peak is wider and less intense than the others. This behavior can be associated with the formation of aggregates of particles that are not homogeneously dispersed in the continuous phase as can be seen in Figure 3(d); the nucleation process is less favorable, and crystalline growth is limited. Furthermore, the aggregates restrict the movement of the polymer chain which hinders the orientation and packing of the chains; this mechanism is supported by the reduced degree of crystallinity of the polymer matrix [23]. Table 1 gives a summary of the results observed in Figures 4 and 5.

The thermal stability of the nanocomposites determined by thermogravimetric analysis indicates that the presence of the nanoparticles does not produce a considerable effect; for example, the maximum increase in this variable ranges from 452.4°C for nylon 6 to 453.4°C for the nanocomposite at 0.5 wt.% of copper nanoparticles. This result is similar to the cases reported previously [8, 11].

As previously described, there are only a few reports regarding the synthesis of nylon 6/copper nanoparticles

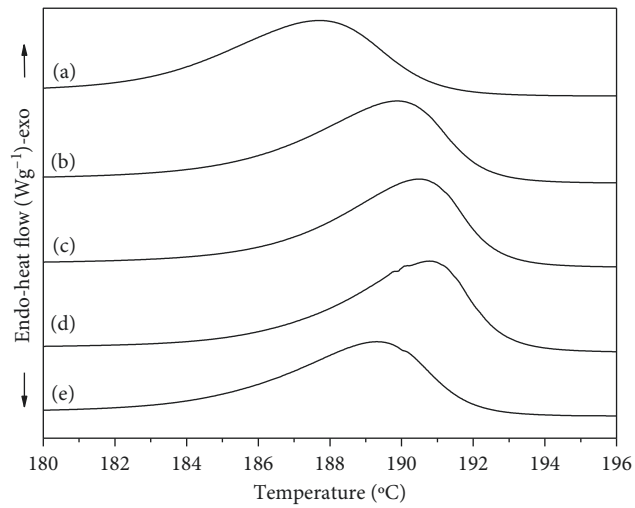

Figure 5: Exothermic thermograms of the nylon 6 (a) and the nanocomposites at concentrations of 0.01 (b), 0.05 (c), 0.10 (d), and 0.50 (e) wt.%.

[8, 11, 12] in addition to the information presented in this work. It is difficult to find good agreement among all the works with regard to the mechanical or thermal behavior of the nylon 6/Cu nanocomposites; this may be attributed to the fact that the characteristics of the nanoparticles depend on the conditions of synthesis and processing. Unfortunately, in the works mentioned, not all the parameters are equal, and a detailed comparison cannot be made. In the present work, it can be said that the physicochemical and mechanical properties of nylon 6 are improved by the presence of the copper nanoparticles for concentrations below 0.5 wt.%.

3.4. Mechanical Behavior of Nylon 6 and Composites. Nylon 6 is an engineered polymer that exhibits excellent mechanical properties [15]. In nanocomposites, these characteristics can be improved or diminished depending on the chemical composition, content, shape, and dispersion of the particles embedded in the polymer matrix [38]. In this section, the tensile strain properties determined experimentally are presented.

3.4.1. Tensile Strength. Figure 6 shows the results of the tensile stress test performed with pure nylon 6 and nylon 6 nanocomposites with a copper nanoparticle weight content of 0.01, 0.05, 0.10, and 0.50 wt.%.

Tensile strength increased with the concentration of the copper nanoparticles by 8.9% compared to the pure nylon 6, which corresponds to a magnitude of 71 MPa instead of 65.13 corresponding to the polymer. Again, this behavior correlates with the good dispersion of the nanoparticles or their aggregates in the polymer matrix, which increases the interfacial adhesion and the mechanical performance of the material [39, 40]. It is important to note that the tensile strength values determined for the nanocomposites via extrusion are superior to those of similar compounds prepared by the in situ polymerization method [8, 14].

Table 1: Thermal properties of nylon 6 and its nanocomposites with copper nanoparticles.

Nylon 6/nano-Cu	Melting temperature, T_m (°C)	Crystallization temperature, T_c (°C)	Degree of crystallinity, X_c (%)
Pure nylon 6	222.3	187.7	42.7
Nylon 6/0.01	222.1	189.9	43.1
Nylon 6/0.05	221.5	190.5	45.9
Nylon 6/0.10	220.8	190.8	46.9
Nylon 6/0.50	221.8	189.3	40.1

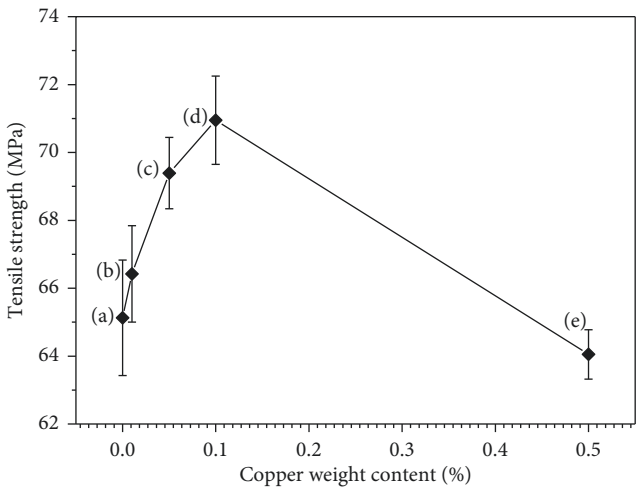

Figure 6: Tensile strength: (a) nylon 6 and nanocomposites with (b) 0.01, (c) 0.05, (d) 0.10, and (e) 0.50 wt.% of copper nanoparticles.

At the 0.50 wt.% concentration of the nanoparticles, a decrease of 1.7% in tensile strength was observed (Figure 6). This behavior has been attributed to the formation of particle aggregates [41]. In Figure 3(d), such aggregates can be observed, and in Figure 7, the composition of the particles was confirmed by elemental mapping determination using energy dispersive spectroscopy (EDS); groups of particles concentrate in small spaces, and the formation of some elongated aggregates quite similar to nanowires were observed as well.

These aggregates can act as stress concentration centers, causing a drastic reduction in the mechanical properties due to the fact that there is no homogenous distribution of the nanoparticles within the nylon 6 matrix [42]. Another possible cause of this behavior is the low crystallinity of the polymer matrix in the compound (Table 1), which favors a decrease in the rigidity of the polymer, and as a consequence, a reduction in its resistance to the tension [43].

In polymers such as polypropylene, it is known that ultrasound-assisted extrusion may have a slightly degradative effect on the polymer matrix [44], and this effect can be greater at higher nanoparticle concentrations [45]. The use of ultrasound and nanoparticles with polyamides is quite controversial [46–48], and further studies are necessary in order to clarify these points.

3.4.2. Tensile Modulus. Figure 8 shows the tensile modulus for the nylon 6 and the nanocomposites prepared with the copper nanoparticles.

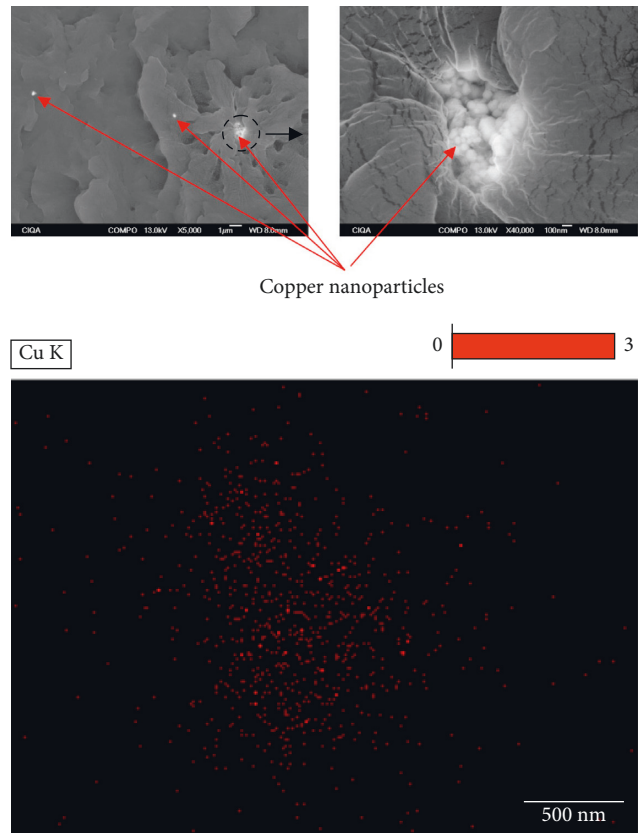

Figure 7: Copper elemental mapping by energy dispersive spectroscopy (EDS).

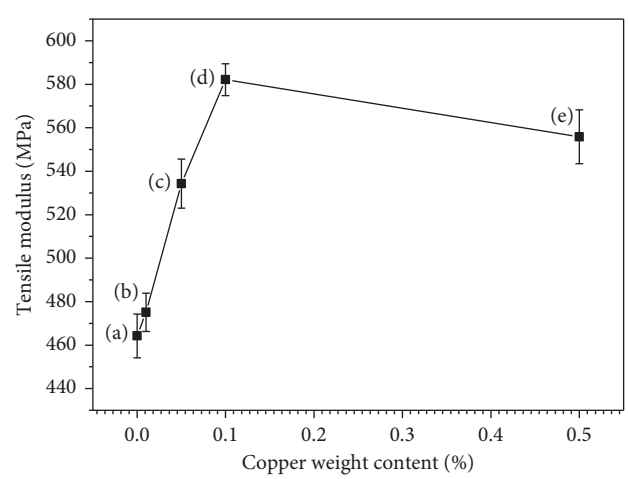

Figure 8: Tensile modulus: (a) nylon 6 and nanocomposites with (b) 0.01, (c) 0.05, (d) 0.10, and (e) 0.50 wt.% of copper nanoparticles.

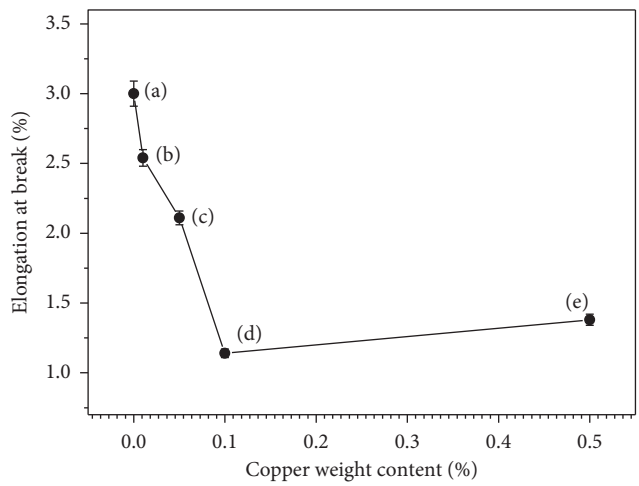

Figure 9: Tensile elongation at break: (a) nylon 6 and nanocomposites with (b) 0.01, (c) 0.05, (d) 0.10, and (e) 0.50 wt.% of copper nanoparticles.

Table 2: Mechanical properties and standard deviation of Nylon 6 and the nanocomposites.

Nylon 6/nano-Cu*	Tensile strength (MPa)	Tensile modulus (MPa)	Tensile elongation at break (%)
Nylon 6	65.13 ± 1.70	464.22 ± 10.12	3.00 ± 0.09
99.09/0.01	66.42 ± 1.42	475.08 ± 8.85	2.54 ± 0.06
99.05/0.05	69.39 ± 1.05	534.27 ± 11.26	2.11 ± 0.05
99.90/0.10	70.95 ± 1.30	582.13 ± 7.34	1.14 ± 0.03
99.95/0.50	64.05 ± 0.73	555.82 ± 12.41	1.38 ± 0.04

*The total weight of each sample is of 100 g. Values shown with a ± symbol belong to the standard deviation.

The tensile modulus of the nylon 6 had a magnitude of 464.22 MPa. For the nanocomposites (Figures 8(b)–8(d)), the modulus of the concentrations of 0.01, 0.05, and 0.10 wt.% increased by 2.3, 15.1, and 25.5%, respectively. As noted above, the incorporation of nanoparticles in the compound favors increased rigidity and hardness of the polymer matrix [49, 50].

Regarding the compound with 0.50 wt.% of nanoparticles whose tensile modulus is 19.7% higher than of the nylon 6 but lower that of the nanocomposite with 0.10 wt. % of nanoparticles, this mechanical behavior can be explained by the fact that the nanoparticles in the compound are not homogeneously distributed and dispersed properly throughout the continuous phase, which causes zones where the polymer matrix exclusively remains, in which stresses causing discontinuities are concentrated which weakens the structure [42].

3.4.3. Tensile Elongation at Break. Figure 9 shows the elongation at break of the nylon 6 and the nanocomposites with concentrations of 0.01, 0.05, 0.10, and 0.50 wt.% content of nanoparticles.

In contrast to the other mechanical properties, the tensile elongation at break decreases with the presence of nanoparticles. For the concentrations of 0.01, 0.05, and 0.10 wt.% of nanoparticles, decrease in elongation at break by 15.3, 29.7, and 62%, respectively, was observed. This is in agreement with the information shown in Figure 6 on tensile strength for these nanocomposites; since the content of nanoparticles within the nanocomposites increases, their continuous phase or polymer matrix acquires more rigid characteristics, due to a synergistic effect between the particles and the polymer. The theoretical explanation of this observation is based on the fact that frequently polymer compounds that exhibit high tensile strength usually acquire fewer elongations at rupture because their hardness tends to increase [51].

The sample at 0.50 wt.% of nanoparticles showed reduced elongation at break by 54% compared to the nylon 6, as shown in Figure 9(e), but it is slightly higher than the elongation at break of the 0.10% sample. This can be explained by the fact that the formation of aggregates leaves some regions free of nanoparticles in the polymer matrix which are susceptible to more deformation before breakage [41].

These aggregates of particles can act as centers of stress concentration that can be considered possible points of failure, which contributes to the deformation of the material. Additionally, they may change the spatial configuration of the hydrogen bonds between the polymer chains, preventing the reinforcing action that these bonds provide to the nylon 6 matrix [52]. Table 2 shows the results of the mechanical tests shown in the previous graphs accompanied by their standard deviation (error bars shown in the graphs).

4. Conclusions

Through the ultrasound-assisted reactive extrusion method, it was possible to synthesize nylon 6 nanocomposites with

copper nanoparticles. The XRD studies showed the presence of Cu and Cu_2O nanoparticles, and this result confirms the oxidation of nanoparticles at the reaction conditions used. It was also demonstrated that the presence of nanoparticles favors the formation of the α_2 crystalline phase in the nylon 6 matrix.

The results of the scanning electron micrographs showed that the compound with 0.10% had the highest degree of dispersed particles with spherical and elongated shaped nanowires. This nanocomposite showed the greatest tensile strength and tensile modulus, as well as a lower elongation at break. In general, the incorporation of copper nanoparticles into nylon 6 improves its physical and mechanical properties as the concentration increases; 0.10 wt. % produces the best results while higher percentages cause agglomeration and decreased mechanical properties.

The nylon 6 nanocomposites with copper nanoparticles are prominent candidates for applications in which high-performance materials are required, mainly those in which the materials must withstand high mechanical stresses and maintain their dimensional stability such as automotive, aviation, and aerospace parts.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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