

Article

Thermal and Thermomechanical Characterization of Polypropylene-Seed Shell Particles Composites

José Luis Rivera-Armenta ^{1,*} , Beatriz Adriana Salazar-Cruz ¹, Ana Cecilia Espindola-Flores ², Diana Samantha Villarreal-Lucio ³, Claudia María De León-Almazán ⁴ and Jorge Estrada-Martínez ⁵

¹ Centro de Investigación en Petroquímica, Instituto Tecnológico de Ciudad Madero/Tecnológico Nacional de México, Altamira 89600, Mexico

² Centro de Investigación en Ciencia Aplicada y Tecnología Avanzada, Unidad Altamira, Instituto Politécnico Nacional, Altamira 89600, Mexico

³ Coordinación para la Innovación y Aplicación de la Ciencia y Tecnología, Universidad Autónoma de San Luis Potosí, San Luis Potosí 78210, Mexico

⁴ Independent Researcher, San Nicolás de los Garza 66495, Mexico

⁵ Instituto Tecnológico de Altamira/Tecnológico Nacional de México, Altamira 89600, Mexico

* Correspondence: jlrivieraarmenta@itcm.edu.mx; Tel.: +52-833-5342961

Abstract: The use of plant waste, such as husks, fibers, and bagasse, as additives in polymeric matrices generated great interest recently, since they became an option to be used as additives, reinforcers, or only as fillers. The pistachio is a seed for human consumption, while the *Jatropha curcas* (JC) is a seed from which oils are extracted, and are used to obtain biofuels; however, their shells are not used when discarded because no useful application is known. An interesting application for this waste material is to use it as a bioadditive in polymeric matrix. The objective of the present work is to prepare polymer composites with polypropylene (PP) as a matrix and as a bio-additive particle in seed shells, in this case *Jatropha curcas* shell particles (JA) and pistachio shell particles (PI), in different content from 2 to 10 phr. The composites were characterized by techniques such as differential scanning calorimetry (DSC) and X-ray diffraction (XRD) to evaluate crystallinity; by using thermogravimetric analysis (TGA), the thermal stability was studied, and by using dynamic mechanical analysis (DMA) the viscoelastic behavior of composites was evaluated. According to DSC, results suggest that a nucleating effect occurs with the addition of the JA and PI, which was reflected in an increase in the percent of crystallinity of the composites. The TGA results show that thermal stability of the composites was modified with the presence of JA and PI, compared with that PP pristine. In addition, dynamic mechanical analysis (DMA) reported that the viscoelastic behavior was modified, finding a higher capacity to dissipate energy for composites, PI being the one that presented a greater effect on them.

Keywords: *Jatropha curcas* shell particles (JA); pistachio shell particles (PI); thermal stability; crystallinity



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1. Introduction

The composites polymers using renewable or waste materials attracted interest in recent years, providing an option to take advantage of these kinds of materials, which have no industrial application, but can generate improvements in polymer matrix properties.

Lignocellulosic wastes are materials which mainly have cellulose, hemicellulose, lignin, and some other polysaccharides in its composition, and that are studied as reinforcements, fillers, or as additives in polymeric matrices [1–3]. Some of these waste materials, such as fibers, agroindustrial wastes, and seed shells, demonstrated that they can generate improvement in mechanical properties, such as hardness, abrasion, textural stress, and impact stress, among others [4–7]. One of the reasons why composite materials are prepared using lignocellulosic waste materials as reinforcements is because benefits are generated in their production. Lignocellulosic waste reduces energy consumption, it is a cheap raw

material, in addition to being biodegradable, and can produce improvement in mechanical and thermal properties, among others. Some of the applications of these materials have a wide range in industrial areas.

Polypropylene (PP) is a polymer that, according to its properties, has a wide variety of applications, such as in toys, vessels, construction materials, auto parts, carpets, etc. Melt flow is an important property related to the molecular weight of a polymer, and depending on this property, its areas of application are too wide.

Pistachio is one of the world's top three favorite nuts, and is widely cultivated in hot, arid, saline areas of the Middle East, Mediterranean countries, and the United States. The use of shells was studied to obtain energy sources and/or raw material for the synthesis of organic materials, mainly through the pyrolysis method; for this reason, this material captured interest, however, a pyrolysis process represents high energy consumption [8].

On the other hand, the *Jatropha curcas* (JA) is a seed from which biofuels are obtained with great success, however, it is a material which is not fully studied, since its mainly applications are in gasification and pyrolysis processes [9]. Although some research groups report that JC wastes have good antioxidant properties, meaning they could expand their areas of application [10–12].

A good option for these lignocellulosic waste materials is their use as bioadditives in polymeric matrices, since it is reported that they can produce a beneficial effect on some properties. Several polymer matrices were modified with addition of lignocellulosic waste materials, and Table 1 reports some of the studied polymers and main findings. Table 1 reports different types of waste material, polymer matrix, and findings.

Table 1. Different types of waste materials used in polymer matrix as additives.

Waste Material	Matrix	Findings	Reference
Lignocellulosic fibers	PP	Improvement in tensile and impact strength, transcrystallinity was achieved, and bulk crystallinity was affected by processing conditions.	[3]
Pistachio shell and sunflower husk	High density polyethylene (HDPE)	Improvement in impact strength, stiffness, and tensile strength.	[4]
Pistachio shell particles	Polyester matrix	Improvement in mechanical properties such as tensile and flexural strength, and impact strength with particles.	[7]
Pistachio shell	Natural rubber/styrene-butadiene rubber	Abrasion resistance was significantly improved, and a moderate change in thermal stability, tensile, and aging properties were achieved.	[13]
Pistachio shell	Polymethyl methacrylate (PMMA)	Decreasing tensile strength and elongation and increasing one Young's modulus.	[14]
Almond shell	PP	To chemical treatments improve the mechanical and viscoelastic properties, and thermal stability increases.	[15]
Groundnut shell	Recycled PP	Thermal stability and crystallinity were improved, and composites were biodegradable.	[16]
<i>Jatropha curcas</i> shell	Vinyl ester	Improvement in tensile strength, decreasing in flexural strength, thermal stability was improved.	[17]
<i>Jatropha curcas</i> seed	Epoxy	Tribological study	[18]
Peach and cherry stones	PP	Incompatibility between polymer matrix and particles, cherry and peach stones retard degradation, and increase in stiffness of matrix	[19]
Waste walnut shell	Ethylene-propylene-diene monomer rubber (EPDM)	Increase in tensile strength, no considerable changes in thermal properties, results show that these particles can be considered an effective filler.	[20]
Wood	PP	Chemical treatment increases the nucleating ability.	[21]
Rapeseed straw particles	PP	Decrease in the crystallinity index value and increase in beta phase is attributed to processing conditions.	[22]
Coconut, wood flour, and walnut shell	PP	Strength and stiffness were improved, and flammability and burning time decreased.	[23]

One of the main characteristic of PP is its crystallinity, this is related to mechanical properties, and it is reported that it is related to an improvement in the interfacial adhesion. Additionally, it is reported that the decrease in α crystals in PP is composed with an increase in β -crystals, which provides good mechanical performance. An option for increasing the PP crystallinity is chemical modification with acid, basic mediums, or with esterification, causing an improvement in adhesion between the polymer matrix and natural materials [21]. Another way to improve the crystallinity is the use of inorganic fillers combined with lignocellulosic materials, this is an option for cost reduction and to get antimicrobial properties and biodegradable materials [24]. In a previous work by our research group, it was reported that chemical modification of Pistachio shell particles is a good option for increasing the crystallinity of PP acting as a nucleating agent; this was attributed to better interfacial adhesion between particles and the polymer matrix [25].

In the present work, the preparation of polymer composite using a medium melt flow index (MFI) PP as a polymer matrix is reported, adding *Jatropha curcas* shell particles (JA) and pistachio shell particles (PI) in concentrations of 2 to 10 phr (parts per hundred of resin), evaluating the effect of the particles on the crystallinity of PP by XRD, DSC, its thermal stability by TGA, and its viscoelastic behavior by DMA.

2. Materials and Methods

2.1. Materials

The PP used was donated by the company Indelpro S.A. of C.V., Altamira, México, with a melt flow rate of 12 g/10 min, and in accordance with ASTM D-1238, the shells of JA and PI were collected domestically; later, the shells were crushed in an IKA model A11 basic mill and sieved with a 35 mesh to have a 500 μm particle size, the appearance of JA and PI particles is depicted in Figure 1. The shell particles were used as bioadditives in concentrations of 2, 4, 6, 8, and 10 phr, using the following codes for their identification: composites PP-*curcas* *Jatropha* shell particles (JA): PP-2-JA, PP-4JA, PP-6JA, PP-8JA, and PP-10-JA, respectively, and PP-2PI, PP-4PI, PP-6PI, PP-8PI, and PP-10PI for the PP-pistachio shell particles (PI), respectively. For this purpose, a Brabender mixing chamber was used, a model Intelli torque with a 60 cm^3 chamber using roller type blades. The mixing conditions were 180 $^{\circ}\text{C}$, 50 rpm, and 20 min, and later the materials were pressed in order to obtain the specimens for the characterizations.

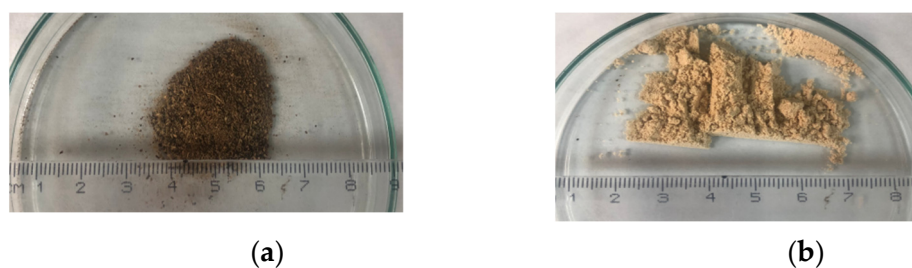


Figure 1. Appearance of JA (a) and PI (b) particles.

2.2. Composites Characterization

The composites were characterized by differential scanning calorimetry (DSC) using a Perkin Elmer model DSC8000, using a 10 mg samples that were encapsulated in aluminum sample holders that were hermetically sealed, performing a double scan in order to delete the thermal history of the composites, the first from -40 to 300 $^{\circ}\text{C}$ with a heating rate of 20 $^{\circ}\text{C}/\text{min}$; later, the material was left in isothermal mode at a temperature of 200 $^{\circ}\text{C}$ for 5 min, during which it was reported the crystallization temperature (T_c) and the ΔH_c of crystallization; samples were subsequently cooled at a rate of 20 $^{\circ}\text{C}/\text{min}$ to -40 $^{\circ}\text{C}$, and again the material was left isothermally for 5 min to subsequently carry out a second scan up to 200 $^{\circ}\text{C}$, with a heating rate of 10 $^{\circ}\text{C}/\text{min}$, taking the data of the second scan for the

analysis, and reporting the melting temperature (T_m) and the melting ΔH_m to calculate the % of crystallinity (%C) using the following formula:

$$X_c = (\Delta H_f / \Delta H_f^\circ) \times 100 \quad (1)$$

For the above calculation, 207 J/g was used as the reference value for ΔH_f° ; this is the melting enthalpy per mass unit in a 100% crystalline PP [23].

The thermal stability of the composites was evaluated by means of thermogravimetric analysis (TGA), for which a TA Instruments model Q600 was used, 10 mg of sample was used in a platinum crucible. The temperature range was from 30 to 700 °C with a heating rate of 10 °C/min in an atmosphere of N₂ with a flow of 100 mL/min.

The dynamic mechanical properties of the composites were determined using a TA Instruments model Q800, with a dual cantilever clamp, the sample dimensions were 20 × 10 × 3.2 mm³, in a temperature range of −40 to 150 °C, with a heating rate of 5 °C/min and a fixed frequency of 1 Hz. The storage modulus (E') and the tan δ were reported. These conditions were established based on the previous work of our group [25].

The composites structural properties were evaluated with an X-ray diffraction analysis, it was carried out in a Bruker model D8 Advance, with a range of 5 to 40° on the 2 θ scale, with a step size of 0.02°, a voltage of 40 kV, a current of 40 mA, and a radiation of Ka from Cu (0.15405 nm).

3. Results

3.1. DSC Results

The DSC thermogram for pristine PP is presented in Figure 2, all the PP-JA and PP-PI composites showed the same behavior, and from them were obtained the crystallization temperature (T_c), the crystallization enthalpy (ΔH_c), the melting temperature (T_m), the melting enthalpy (ΔH_m), and the % crystallinity of the PP-JA and PP-PI composites are reported in Table 2. An important variable that affects the crystallization of PP is the melt index [22], in our case the PP melt flow is medium. From the data obtained, PP's T_c is 111 °C with minimum variation, and when JA is added, there is no appreciable variation in this value. In general, the addition of JA slightly decreases ΔH_c however, with the increase in the particle content, this value also increases, PP-6-JA being the composite with the highest ΔH_c . The addition of lignocellulosic causes an increase in the crystallization temperature of pristine PP [21]. Other reports indicate that the addition of lignocellulosic fibers does not have a significant effect on the melting temperature of pristine PP [26–28].

Table 2. Thermal data obtained by DSC for PP, PP-JCSP and PP-PSP composites.

Composite	T_c , °C	ΔH_c , J/g	T_m , °C	ΔH_m , J/g	% X_c
PP	111	92.11	171	62.48	30.18
PP-2-JA	113	84.6	165	63.14	30.5
PP-4-JA	111	81.36	164.7	65.52	31.65
PP-6-JA	111	89.74	164.8	73.23	35.37
PP-8-JA	112	83.24	163.7	64.63	31.22
PP-10-JA	112	87.88	166	71.26	34.4
PP-2-PI	111	92.45	167	68.78	33.22
PP-4-PI	112	98.47	165	78.36	37.85
PP-6-PI	112	86.8	165	65.87	31.82
PP-8-PI	112	85.85	167	65.67	31.72
PP-10-PI	113	78.25	166	62.68	30.28

On the other hand, the addition of PI presented a similar behavior in regard to T_c , which remains unchanged. However, with respect to ΔH_c , there is a slight increase at low concentrations (PP-2-PI, PP-4-PI), higher than that of PP; however, at a higher content of particles, the enthalpy decreases to values lower than pure PP. Borysiak et al. [24] reported a T_c value of 113°C, a value similar to that reported in our work, despite the fact that a PP

with a lower melt index (12 g/10 min) was used in their study. These results are similar to those reported by Krupa and Luyt [29] who evaluated that PP irradiated with UV light did not undergo changes in its crystallization behavior. Furthermore, Quillin et al., [30] reported that the crystallization process of PP with lignocellulosic materials can be modified with a chemical treatment or with the presence of coupling agents. Regarding the T_m value, it showed a decrease of up to 6 °C compared to the T_m of pure PP (171 °C), which is indicative of an affection in the crystalline structure of PP. This T_m value differs from some previous works, which report a T_m of 166 °C and 164 °C, but with a melt flow PP of 17 g/10 min and 12 g/10 min, respectively [21,22]. The crystallinity of the PP was calculated from the ΔH_m of 62.48 J/g, and a % of crystallinity of 30.18% was determined, a value close to that reported in previous work with a similar melt flow value of PP [27]. On the other hand, it was reported that the melt flow value has a significant effect on the crystallinity of the PP [22]. Regarding the ΔH_m , the addition of JA produces an increase in this value, which is reflected in the content of % crystallinity with respect to pure PP, being higher for a JA content of 6 phr, while in the case of PI, the increase was higher for a content of 4 phr, decreasing with an increase in the concentration of the particles. According to these results, the presence of particles in low contents produces an effect of nucleation that is reflected with an increasing in the crystallinity of the PP matrix. This behavior was previously reported, attributing this increase in crystallinity in a PP matrix to a better interfacial interaction between the particles and the PP, generating a nucleating effect on the polymer matrix [16,26]. Other works report that the presence of lignocellulosic particles in a PP matrix does not produce significant changes in the crystalline structure [19].

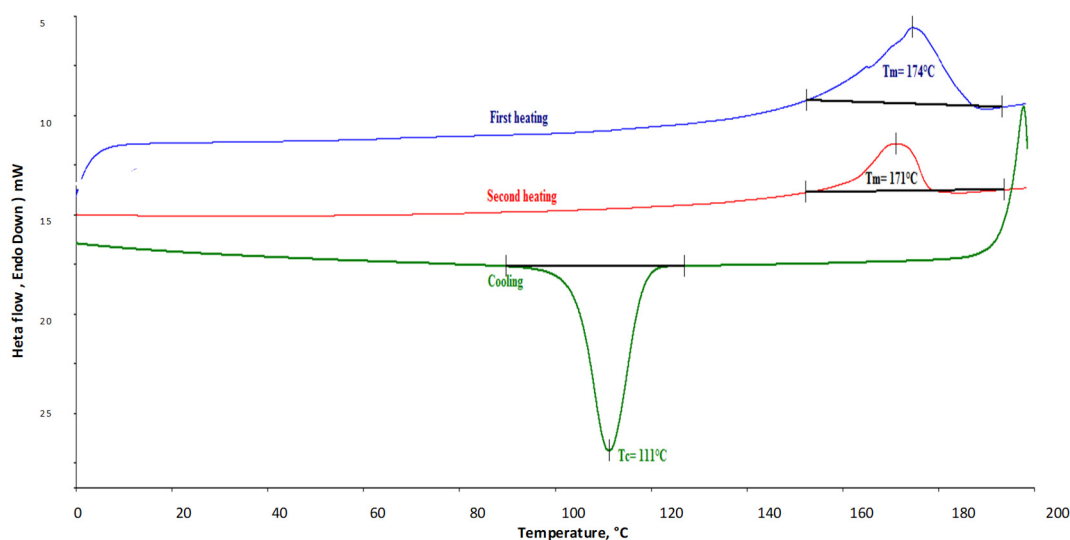


Figure 2. DSC thermogram of PP.

3.2. TGA Results

Figure 3a illustrates the TGA thermograms of pristine PP, JA, and PP-JA composites, it can be seen that the decomposition of the PP is carried out in a single stage, presenting the decomposition at an approximate temperature of 390 °C, presenting at the end of the analysis a minimum ash content. On the other hand, the JA shows two main stages of decomposition, the first attributed to the decomposition of hemicellulose, while for the second, approximately 350 °C is attributed to the deposition of lignin and cellulose. These results are similar to other works that report the thermal decomposition of wastes of lignocellulosic origin [31,32]. One way to verify if the decomposition of a material takes place in different stages is by using the derivative of the weight loss with respect to temperature, which can also help identify the decomposition susceptibility of a material with average temperature [33]. Figure 3b shows the DTG curve for PP, JA, and PP-JA composites, and it can be seen that the average decomposition temperature does not show

a significant difference, however, the ratio of the curve does show changes, which, as mentioned above, can be associated with the decomposition susceptibility of the material. It is observed that the PP-4-JA and PP-6-JA composites show a lower ratio, which indicates that the speed with which they lose weight is slower compared to the rest of the composites, as well as the pure PP.

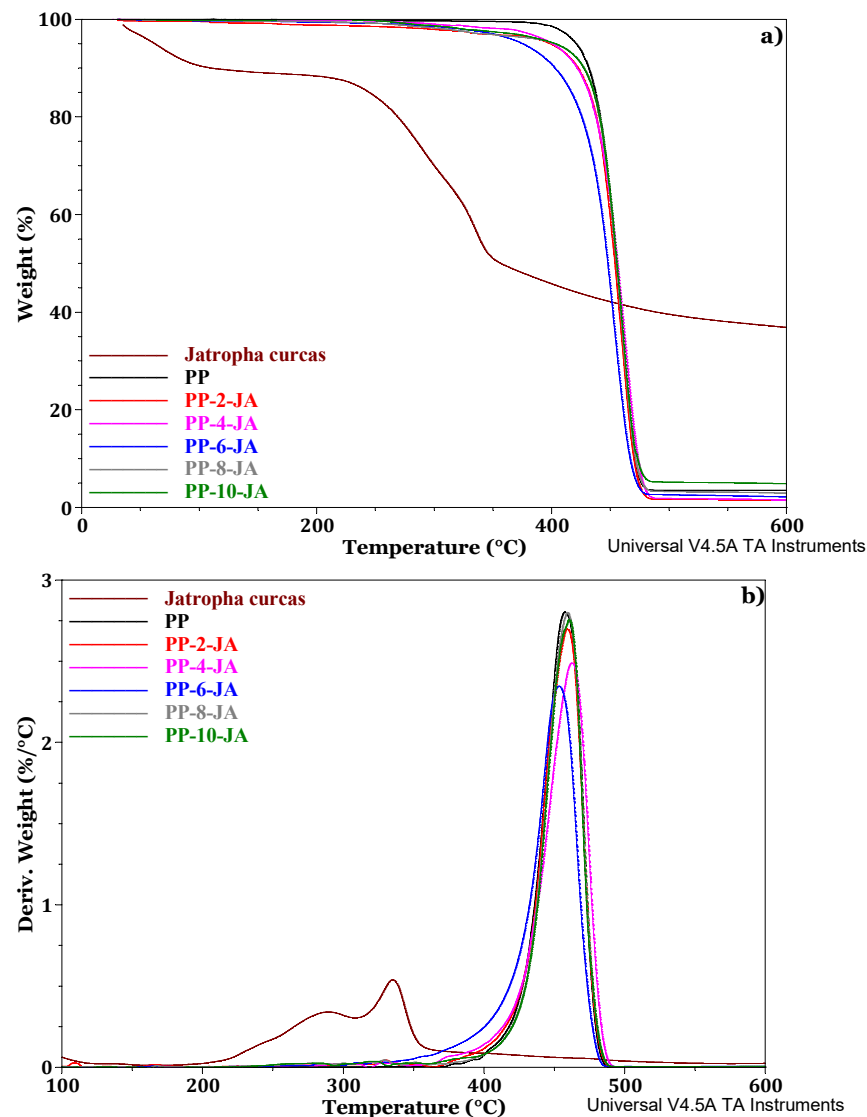


Figure 3. TGA thermogram of % weight loss (a) and derivative of %wt/°C (b) for PP, JA, and PP-JA composites.

On the other hand, Figure 4a,b shows the TGA thermograms of weight loss and derivative of % weight loss against temperature for pristine PP, PI, and PP-PI composites. The PI shows two main decomposition stages, first attributed to removal of hemicellulose, and the second one to degradation of lignin cellulose [34]. It can be observed that the behavior is similar to that observed for the composites with JA, in the case of the PI, it also presents a decomposition prior to the decomposition of the pure PP, and as the PI content increases, the composites begin to decompose at a lower temperature. Moreover, the DTG curves allow us to identify that both composites and pure PP have a very similar average decomposition temperature, close to 457 °C. However, the ratio for the PP-8-PI composite is the one with the lowest ratio, compared to the rest of the composites and the PP. As discussed above, the low value in the ratio indicates that the material is less susceptible to weight loss, thus showing greater stability compared to the rest of the composites.

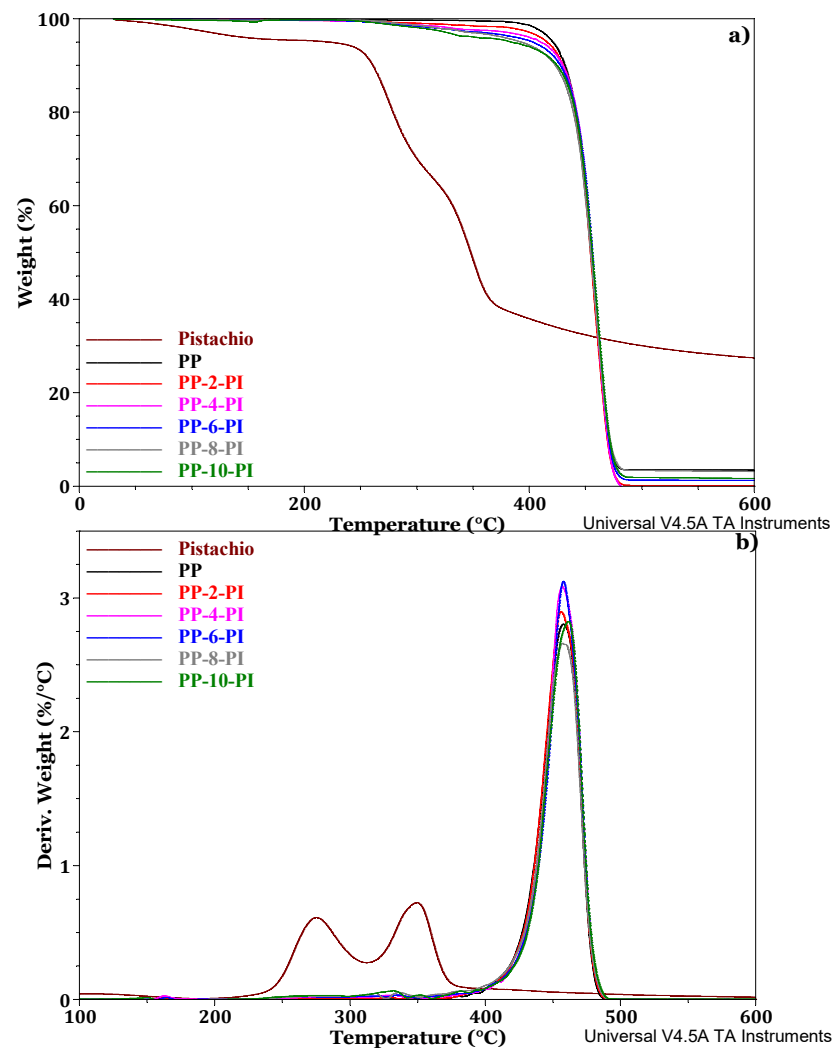


Figure 4. TGA thermogram of % weight loss (a) and derivative of %wt(°C), (b) for PP, PI, and PP-PI composites.

3.3. DMA Results

Figure 5 shows the DMA thermograms of the storage modulus and $\tan \delta$ with respect to the temperature of the PP and PP-JA composites. In Figure 5a, it can be observed that the addition of JA causes a decrease in the value of the storage modulus when the JA content increases, being that PP-8-JA is the composite that presented a lower value of storage modulus. This behavior is due to the fact that the presence of JA generates an agglomeration effect in the vitreous region, as was reported previously with high particle concentration (50%wt) [26]. The results obtained are contrary to other works that report an increase in the storage modulus with the addition of pistachio particles in polyolefins [4], attributing this behavior to a better adhesion between the PI and the polymeric matrix.

Figure 5b shows the behavior of $\tan \delta$ with respect to temperature. This signal allows for the identifying of the relaxations experienced by a polymer with respect to temperature, and the magnitude of the $\tan \delta$ peak establishes the behavior of the polymer in terms of temperature, its ability to flow or deform elastically, so that the width and height of the peak can be associated with the damping ratio of the material. It can be seen that the ratio of $\tan \delta$ for the composites is higher compared to that of the pristine PP; in addition to the fact that its width increases, this behavior is typical when the materials have a greater capacity to dissipate energy, that is, they have a greater tendency to flow than to deform, which is also associated with more complex structures. This behavior was previously reported [34], and attributed to the presence of rigid lignocellulosic fillers that can replace

the amorphous regions of the matrix, which is reflected in the variation in the $\tan \delta$ ratio. On the other hand, two relaxations can be observed both in the pristine PP and in the PP-JA composites; the first at an approximate temperature of 10 °C, which is associated with the glass transition of the PP, which is observed not to present a change significant in its value as the concentration of JA increases, and the second relaxation at a temperature close to 80 °C, which is attributed to a laminar and rotational slip mechanism in the crystalline phase of the polymer. These results differ from previous works, which found that the addition of particles of lignocellulosic materials produces an increase in Tg, since the presence of the particles prevents the movement of the polymer chains [4,35].

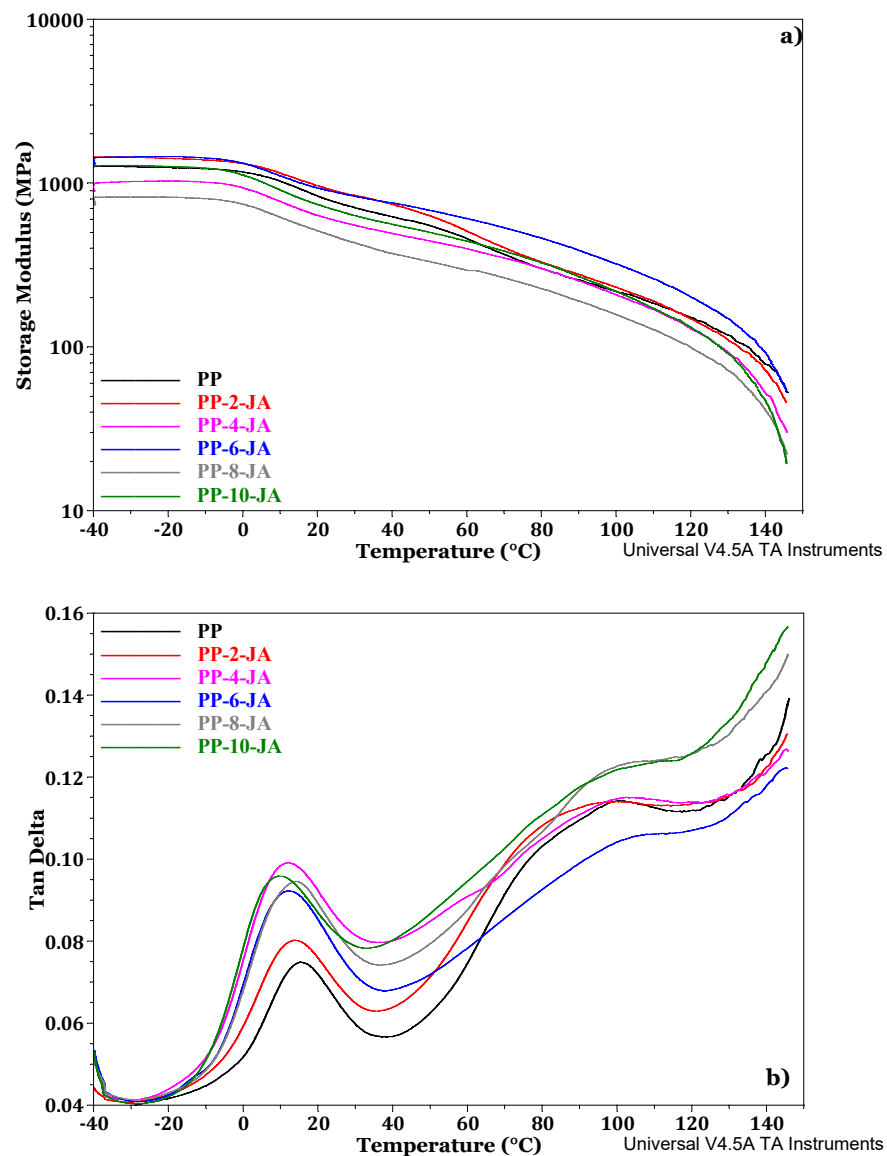


Figure 5. DMA thermogram for PP and PP-JA composites. (a) Storage modulus versus temperature, and (b) $\tan \delta$ versus temperature.

On the other hand, Figure 6a shows the DMA thermograms of storage modulus against temperature for PP and PP-PI composites, where it can be seen that the PP-8-PI composite was the one that presented a lower storage modulus, compared to the rest of the composites, which had modulus values very close to the pristine PP. This behavior is opposite to that previously reported by our working group, with the difference of the properties of PP, which has a higher melt flow value, in addition to the fact that the pistachio particles underwent a chemical treatment with NaOH solution [25].

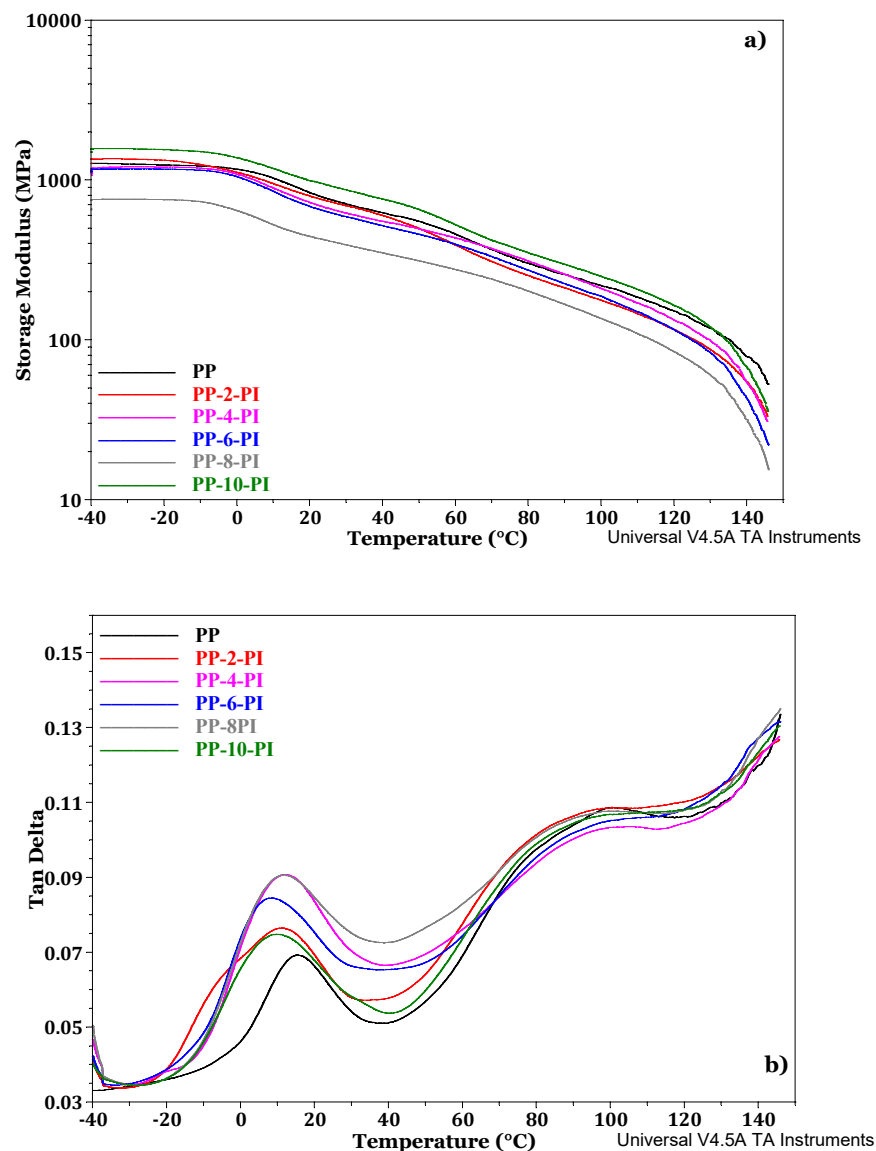


Figure 6. DMA thermograms for PP and PP-PI composites. (a) Storage modulus versus temperature, and (b) $\tan \delta$ versus temperature.

Figure 6 shows the DMA thermograms of the storage modulus and $\tan \delta$, with respect to temperature for pristine PP and PP-PI composites; a similar behavior is observed for PP-PI composites, which show a similar modulus value to pristine PP, except for the PP-8-PI composite, which shows a significant decrease in the temperature range of the analysis. On the other hand, in Figure 6b it is observed that the $\tan \delta$ curve shows a behavior similar to that shown by the PP-JA composites; however, in this case, the peak is much wider and a dissimulation in the Tg of the compounds is identified. Composites presenting a difference between 2 and 4 °C include PP-4-PI and PP-8-PI, being the ones that report a greater difference with respect to the pristine PP.

3.4. XRD Results

Figure 7 shows the X-ray diffraction patterns of the pristine PP and the PP-JA composites. It can be seen that PP shows characteristic peaks at 14, 17, 18, and 21° that correspond to the monoclinic crystalline phase alpha with planes 110, 040, 130, and 111, respectively, in addition to two small peaks at 25 and 28°, that were reported with plans 060 and 220, and attributed to the same monoclinic structure of the PP [36,37]. In the PP-JA composites at 4, 6, 8, and 10, differences with respect to the pristine PP are identified, with the appearance

of small peaks at 16° , which are associated with β -type crystals with 300 planes [21,24,36]. Previous works report that the β phase of PP is identified by the appearance of a peak at 16° [22,26,38]. The presence of the β phase in the PP-JA composites can be related to the presence of lignocellulosic materials that promote its appearance, due to a nucleating effect; however, other works indicate that the lignocellulosic materials do not influence the presence of the β phase. PP has β crystals, but the presence of these are due to the high shear stresses that are reached during processing [22]. Li et al. [39] report that the β phase in PP provides a better performance and that using nucleating agents from wastes, such as eggshell, would be a good option.

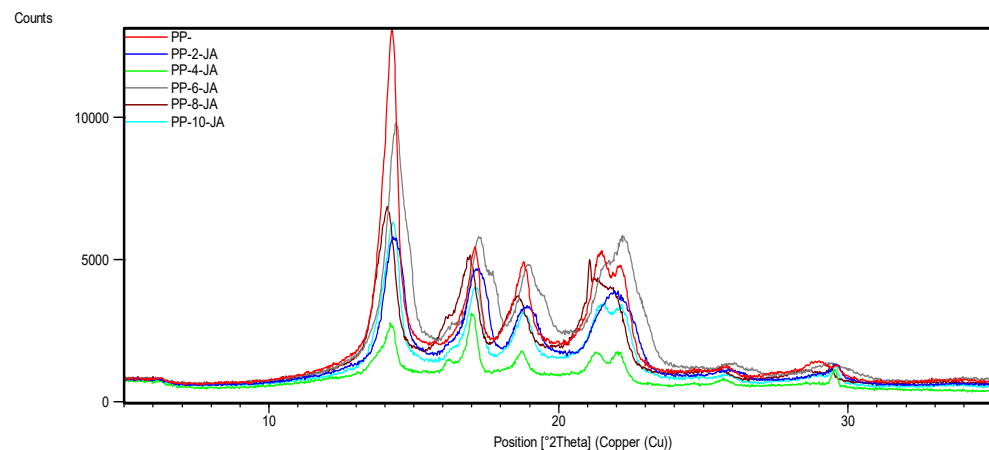


Figure 7. XRD pattern for PP and PP-JA composites.

Figure 8 illustrates the XRD pattern for PP and PP-PI composites; there is similar behavior to PP-JA composites, with the presence of a peak at 16° , which is associated with the presence of the β -type crystals of PP with 300 planes. This peak is more evident for composite PP-4-PI, as this material shows the highest % crystallinity calculated by DSC. Some works report that β -crystals of PP melts at 150°C , but others report the presence of β -crystals with the presence of a peak at 16° [28]. Others attribute the presence of the β -form of PP to the shearing forces between the polymer matrix and particles, which can be reached during composites preparation.

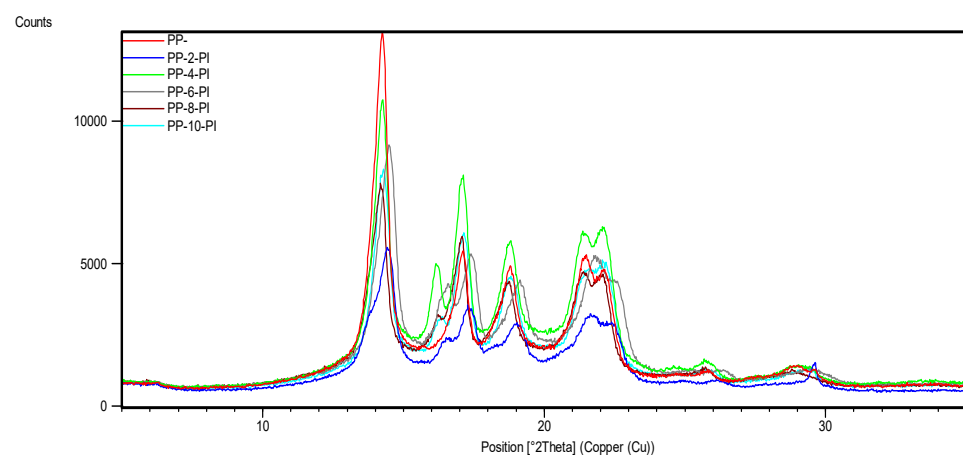


Figure 8. XRD pattern for PP and PP-PI composites.

4. Conclusions

Based on the findings in this study, the use of *Jatropha curcas* and pistchio shell particles are demonstrated to be a good options as bioadditives for polymeric matrix. The crystallinity of PP was improved with low content of JA until 6 phr and PI up to 4 phr, and higher content causes a contrary effect attributed to particle agglomeration.

Additionally, the crystallization process was not affected by the presence of shell particles, but the T_m value decreases with the addition of shell particles; however the crystallinity % increase in JA and PI is more significant for PI compared with JA. The thermal stability of composites increases and susceptibility to decomposition decreases according to DTG analysis. XRD results show the formation of β phase in PP-JA composites, which can be favorable for improving of mechanical properties. According to DMA results, the damping factor increases with shell particles due the fact that the presence of particles generates a higher capacity for energy dissipation. Finally, JA and PI were demonstrated to be new promising new options for materials to be used as bioadditives in different polymer materials in the aim to improve thermal, structural, and mechanical properties.

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Conflicts of Interest: The authors declare no conflict of interest.

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