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Evaluation of Thermal and Thermo-mechanic Properties of Composites Based on Styrene-butadiene Copolymer (SBS)-seed Shell Particles

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***Abstract**: Nowadays, the use of agricultural wastes is a high impact research area, due the fact that this kind of materials can provide some interesting properties to a polymer matrix. Among these materials, the seed shell are materials with no industrial applications, so it is necessary to find a use in the aim to avoid the pollution. The Styrene-butadiene copolymer (SBS) is a wide used material due it thermoplastic behavior. In present work, the inclusion of two different species of seed shell particles: (jatropha curcas seed particles (JCSP) and pistachio (PSP)) into a Styrene-butadiene copolymer (SBS) and their effect on the thermal behavior of the resulting composites have been studied. The composites were prepared by melting fussion process at different concentrations ranging from 2 to 10 phr (parts per hounded of resin). The obtained composites were characterized by means of Thermogravimetric analysis (TGA), Dynamic Mechanical Analysis (DMA) and infrared Spectroscopy (FTIR).*

***Keywords**: seed shell particles, thermal stability, viscoelastic behavior, SBS copolymer*

1. Introduction

The use of waste materials in industrial applications, has gained special relevance in recent years, since it represents an option to reduce pollution and to enhance the properties of other materials at the same time. This practice is useful to both the compliance of environmental regulations and the production of composite materials with improved properties that can find a place in many interesting applications. Several waste materials from agricultural sources have been studied by various research works [1-4] to obtain composite materials using a polymeric matrix.

A composite material is based on two parts: a matrix, which provides elasticity, flexibility, strength, as well as other physical properties, and a filler, whose function is to confer or improve specific properties of the matrix, e.g. thermal, physical, mechanical, etc.

Jatropha curcas is a species of flowering plant in the spurge family, native to the American tropics, mainly located in México and Central America [5]. The fruit of this plant is commonly used for the obtention of biofuels, but the shell has no application so far, so it is considered a waste material. The dry *jatropha curcas* fruit is about 35-40% shell and 60-65% seed, and its chemical composition is mainly constituted by crude fat, crude fiber, ashes and proteins [6]. Today, the main application of the seed relies on its potential use as biofuel, but the search for useful applications for the shell is still ongoing. A few works have attempted to use shell particles as reinforcement in epoxy and polyurethane polymers reporting improvement in mechanical properties and tribological behavior [7-10].

Another plant in a similar situation is the pistachio, which is a small plant native from Central Asia and Middle East that belongs to the cashew family [11]. Pistachio fruit is a highly consumed food, and it is easily acknowledged by its characteristic beige-greenish color which can also be red sometimes [12]. The pistachio shell represents a considerably high amount of garbage, normally used to produce

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charred material [11], fungicidal oil [13] and some decorative items. Just a few studies evaluate the addition of PSP as additive in composites. Karaagac reports the evaluation of the addition of PSP as a substitute of carbon black in natural rubber /Styrene-butadiene rubber (SBR) blends [14]. Alsaadi et al [15] report that adding Pistachio shell to a polyester polymer improves its tensile, flexural and impact properties at concentrations up to 5%, and it also informs that a good dispersion of particles is observed. However, the authors highlight the relevance of the shell particle size, as it plays an important role in the interfacial adhesion between particles and polymer matrix. Also report chemical composition of Pistachio shell is about 42% cellulose, 13% lignin, 0.18% extractables, 1.26% ashes, 3.11% lingo-cellulose [15]. Najafabadi et al [16] report the use of shell flour for the preparation of composites based on High density Polyethylene (HDPE) and nanoclays. It was found that, when pistachio was added to the composite, the tensile modulus decreased. The authors also warn about the fact that moisture absorption affects this property. Polymethyl methacrylate (PMMA) is also reinforced by Pistachio powder, especially for dental applications [17].

The block elastomer styrene-butadiene (SBS) is an important material with wide range of application areas due to its thermoplastic behavior, which means that under certain conditions it shows a plastic response, otherwise it displays an elastic behavior. This kind of material may be arranged in a variety of structures, such as linear, radial or multi-radial block copolymers [1]. There are several studies of composite materials based on SBS reinforced with natural or animal waste materials [18-21] finding that its properties can be modified with the addition of this kind of fillers.

The present research is an exploratory work to obtain composite materials using seed shells particles (SSP) from *jatropha curcas* and pistachio as SBS fillers. The use of plant waste, seed husks, among others, has been taken as an option to obtain low-cost bioadditives, in addition to the fact that they can impact from an economical and environmental point of view. Also, theses kind of waste materials can improve the abrasion resistance, thermal, mechanical and structural properties of polymeric matrices, so based on that, the present work may contribute to the exploitation of these materials that have not been reported yet as fillers in SBS matrix, which can be an interesting application for these agricultural wastes with such good properties. Thermal properties were evaluated by means of TGA and DMA in SSP concentrations from 2 to 10 phr.

2. Materials and methods

2.1. Materials

Pistachio and jatropha curcas shells were collected from domestic sources. The shells were milled in an IKA analytical mill, to decrease their particle size to about 500 μm (ASTM sieve #35). Particles were identified as Pistachio shell particles (PSP) and Jatropha Curcas shell particles (JCSP). The SBS used is a block copolymer with styrene content of 30% with radial structure.

2.2. Composites preparation

The composites preparation was made by melt mixing in a Brabender Plastograph, under the following conditions: 90ºC, blades speed 50 rpm for 20 min, using roller blades. Different shell particles (SP) concentrations were studied: 2, 4, 6, 8 and 10 phr (parts per hundred of resin), the nomenclature was: SBS-JCSP-2PHR, SBS-JCSP-4PHR, SBS-JCSP-6PHR, SBS-JCSP-8PHR and SBS-JCSP-10-PHR for composites with JCSP, and SBS-PSP-2PHR, SBS-PSP-4PHR, SBS-PSP-6PHR, SBS-PSP-8PHR and SBS-PSP-10PHR, for composites with PSP, the number indicate the SP content. Once the materials were mixed, they were hot pressed to form plate-like samples. The press conditions were: 10 min under 0 t, 5 min under 5 ton, and 10 min under 10 t, all at 210ºC.

2.3. Composites characterization

The characterization of composites was carried out through the following analytical techniques: 1) Infrared spectroscopy, using a Spectrum One equipment using an ATR accessory with Se-Zn plate, from 4000 to 600 cm^{-1}, 12 scans and resolution of 4 cm^{-1}; 2) Thermogravimetric analysis (TGA), which was

carried out in a Q600 Series Simultaneous Thermal Analyzer (SDT) DSC/TGA TA Instruments equipment, in a temperature range from 30 to 600ºC with a heating rate of 10ºC/min, using an alumina pan, and 10 mg size of sample, under nitrogen atmosphere with a 100 mL/min flow rate, and 3) Dynamic Mechanical Analysis (DMA), that was carried out using a Q800 series TA Instruments equipment, using a dual cantilever clamp and sample size about 20X15X3 mm^3, from -90ºC to 150ºC with a heating rate of 5ºC/min. Optical microscopy was carried out in the aim to corroborate the dispersion of SP in SBS matrix, for this an upright ligh microscope Zeiss Axioscope, model Axis Lab A1 was used, collecting images at 100X of magnification. The images were collected installing a camera on the top of the optical microscope and images were collected using the Motic Images plus 3.0 software.

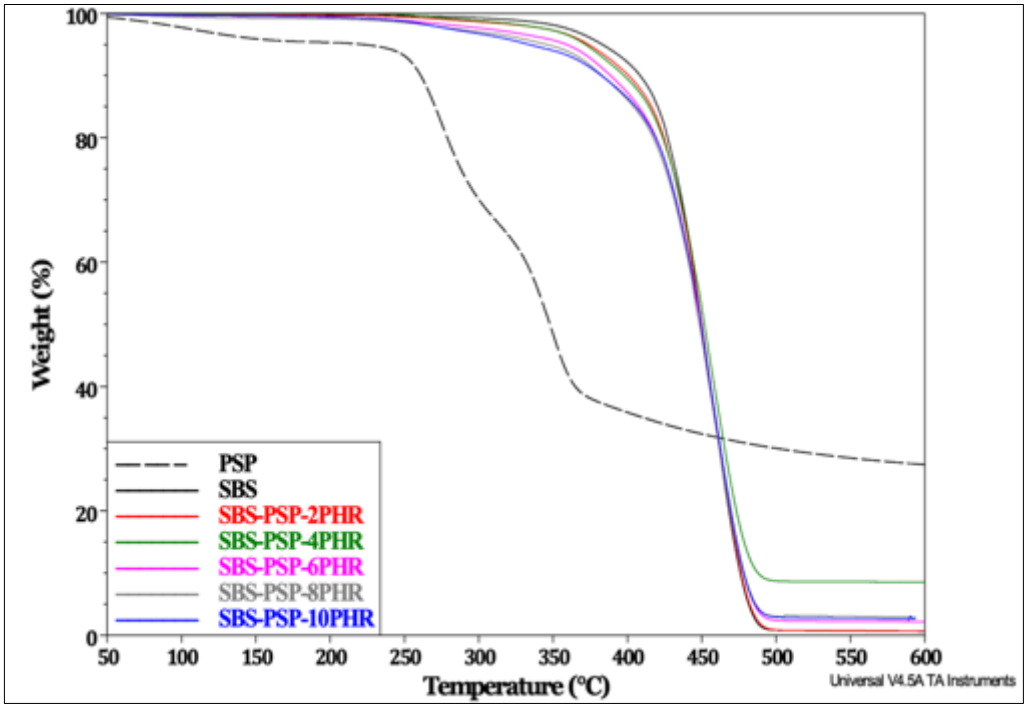

Figure 1. TGA thermogram for SBS and SBS-PSP composites

3. Results and discussions

3.1. Thermogravimetric analysis (TGA)

The Figure 1 shows the TGA thermogram for SBS, PSP and SBS-PSP composites with different content of PSP. The SBS shows a typical single decomposition stage around 450ºC [22]. Previous works report that PSP has two main decomposition stages: the first one around 300ºC, that corresponds to the hemicellulose decomposition and the second one about 350ºC, that is related to the cellulose decomposition which is identified as a narrow peak in the DTG curve [23]. It can be observed that the addition of PSP modified the thermal stability of the SBS matrix, by decreasing the onset temperature which is associated to the decomposition of the backbone. Accordingly, as the PSP content increases, the onset temperature is displaced to a lower temperature. Apaydin-Varol et al [12], report that Pistachio shows its main decomposition stage around 280ºC, which is close to the temperature at which the degradation of the SBS-PSP composites begins. Other works report that the addition of PSP negatively affects the thermal stability of natural rubber and styrene-butadiene rubber composites at concentrations up to 10 phr [14].

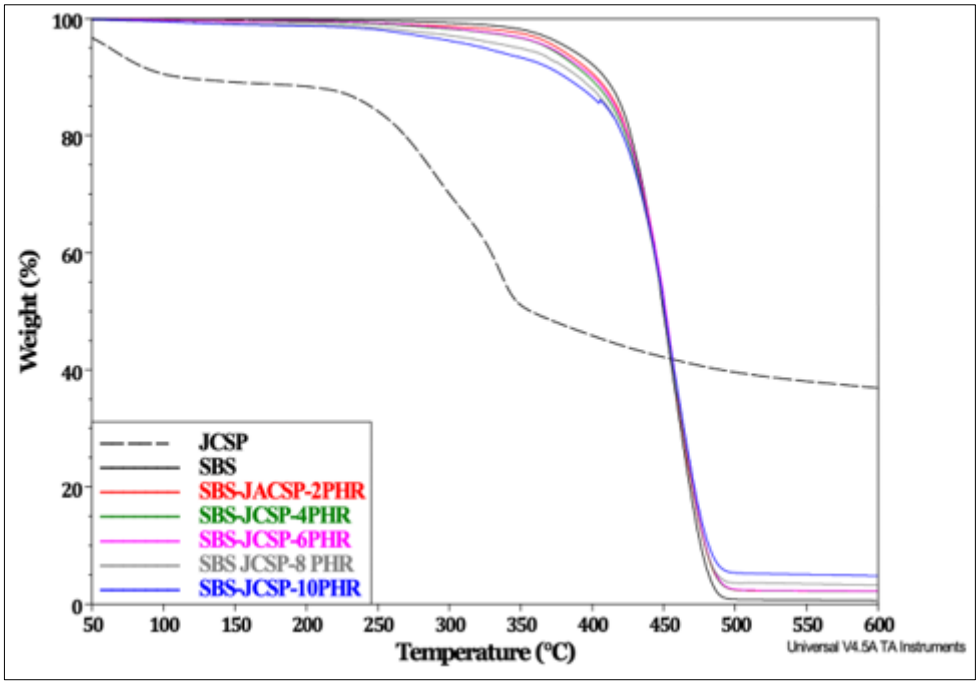

Figure 2. TGA thermogram for SBS and SBS-JCSP composites

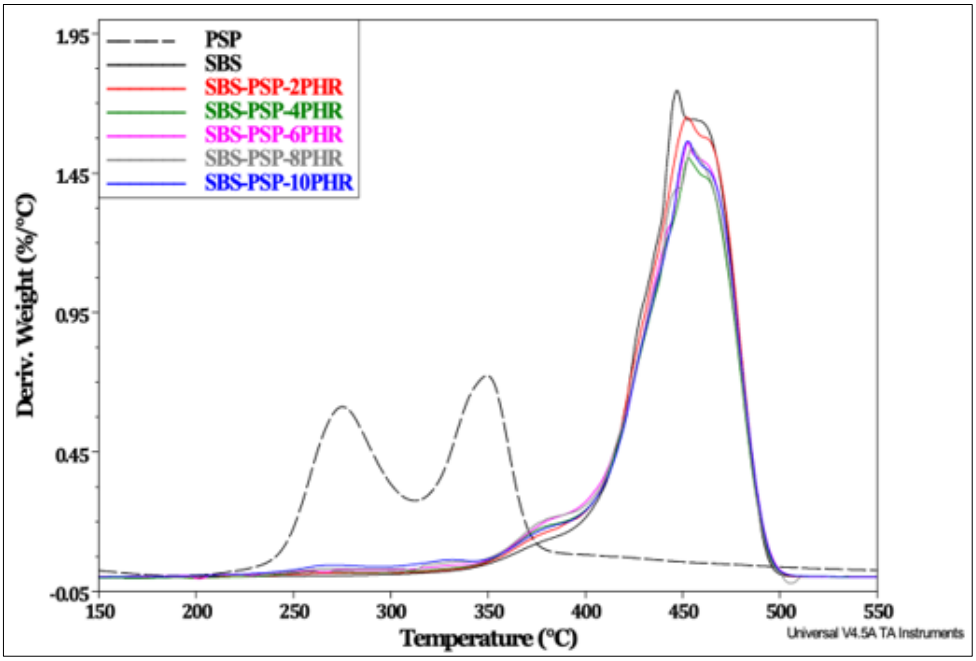

Figure 3. DTG thermogram for SBS and SBS-PSP composites

The same behavior was observed for composites prepared with JCSP (Figure 2). The change in the thermal stability of the composites is attributed to the JCSP main components, i.e. cellulose and hemicellulose, which present decomposition temperatures around 300-350ºC and around 450ºC (related to lignin decomposition) [5, 24]. This could be an interesting finding considering that the inclusion of a natural material is intended as filler or reinforcer in a synthetic polymer matrix.

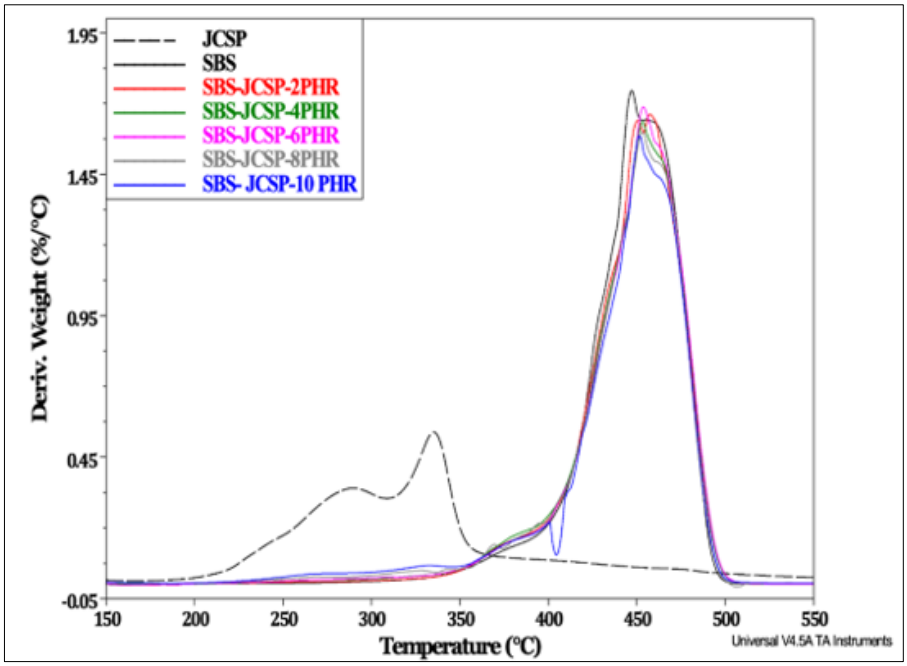

Figure 4. DTG thermogram for SBS and SBS-JCSP composites

Figures 3 and 4 show the Derivative TGA (DTG) curves for the SBS-PSP and SBS-JCSP composites, respectively. It can be observed, for SBS-PSP composites that the main decomposition temperature is almost the same, but the quotient of the derivative slightly decreases with increasing PSP, and the peak around 450ºC is slightly displaced to higher temperature, this behavior has not been reported before for composites based on SBS elastomers. It is also possible to confirm the two stages of PSP decomposition at 280ºC and 350ºC. A similar behavior was observed for composites with JCSP, which is indicative that material the is less susceptible to degradation. Besides, there is a slight displacement of the main decomposition temperature peak to higher temperatures, suggesting a possible physical interaction between elastomeric matrix and SSP [19]. Puttaswamy et al [25] report the obtention of cellulose microfibers from JCSP and how they modified the thermal stability of Polyvinyl Alcohol (PVA) considering that this kind of material can be an interesting filler for a polymer matrix.

3.2. Infrared spectroscopy (FTIR)

Figure 5 shows the FTIR spectra for SBS and SBS-PSP 10 phr composite. The main functional groups of SBS are present at 3005, 2916, 2844, 1800 and 1639 cm^{-1} due to unsaturated carbons, stretching of methyl and methylene groups and aromatic rings, respectively [18, 26]. In SBS-PSP composites spectra, it is evident the peak at 1742 cm^{-1}, which is attributed to C=O groups, as well as the signal at 3690 cm^{-1} from –OH groups and the peak at 1163 cm^{-1} associated to aliphatic ethers. The described bands have been previously reported as characteristic signals of Pistachio shell [23].

The infrared Spectra for SBS-JCSP 10 phr composite and pure SBS is reported in Figure 6. *Jatropha curcas* presents small peaks at 3686, 1716, and 1638 cm^{-1} attributed to –OH, C=O, and oxygen groups in carboxyl groups, respectively [24]. There is not a significative evidence of presence of JCSP in composite, the JCSP signals are not easy to identify in the composite spectrum. According to the FTIR spectra, there is no evidence of a chemical reaction between SBS and seed shells particles.

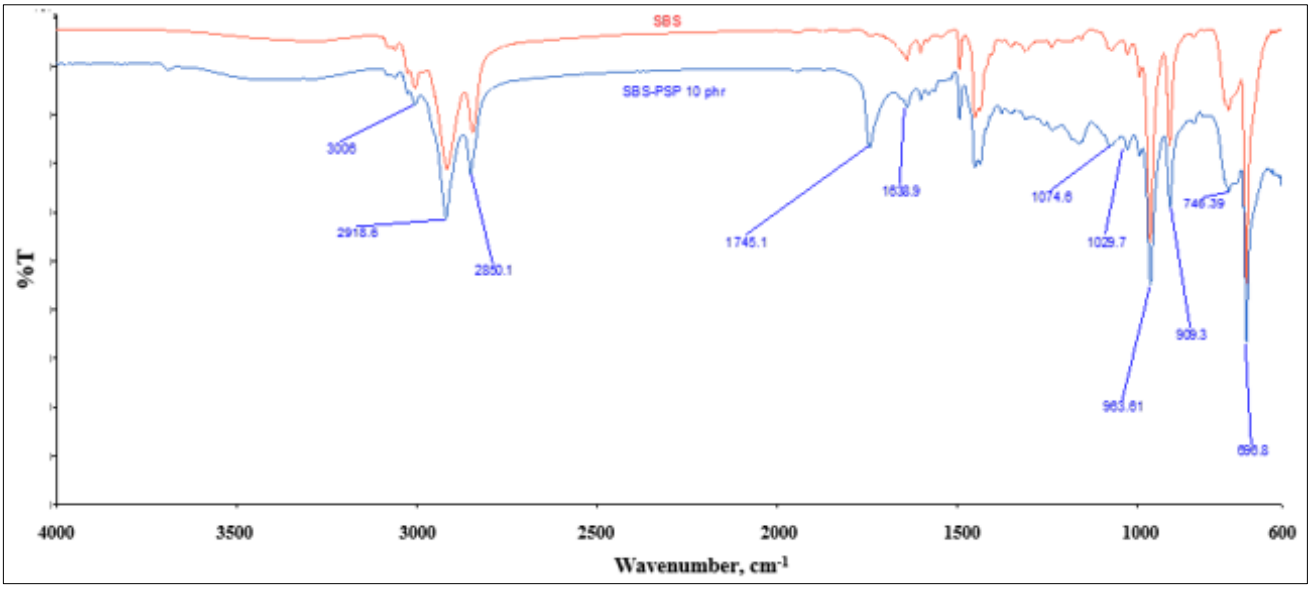

Figure 5. IR spectra for SBS and SBS-PSP composites

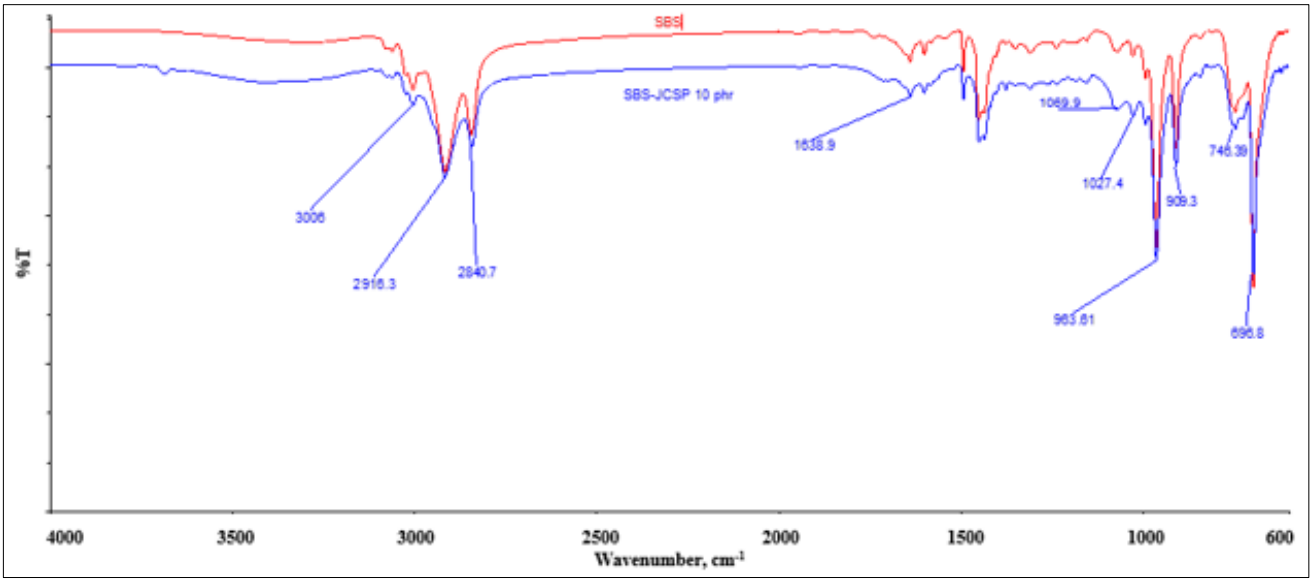

Figure 6. IR spectra for SBS and SBS-JCSP composites

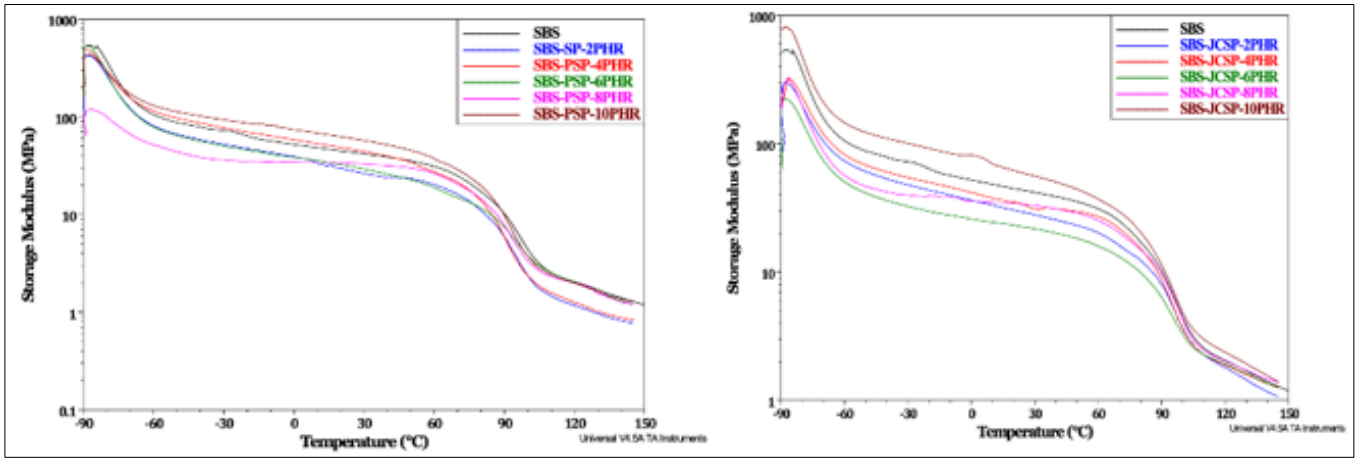

Figure 7. Storage modulus curves obtained by DMA for a) SBS and SBS-PSP, and b) SBS-JCPS composites

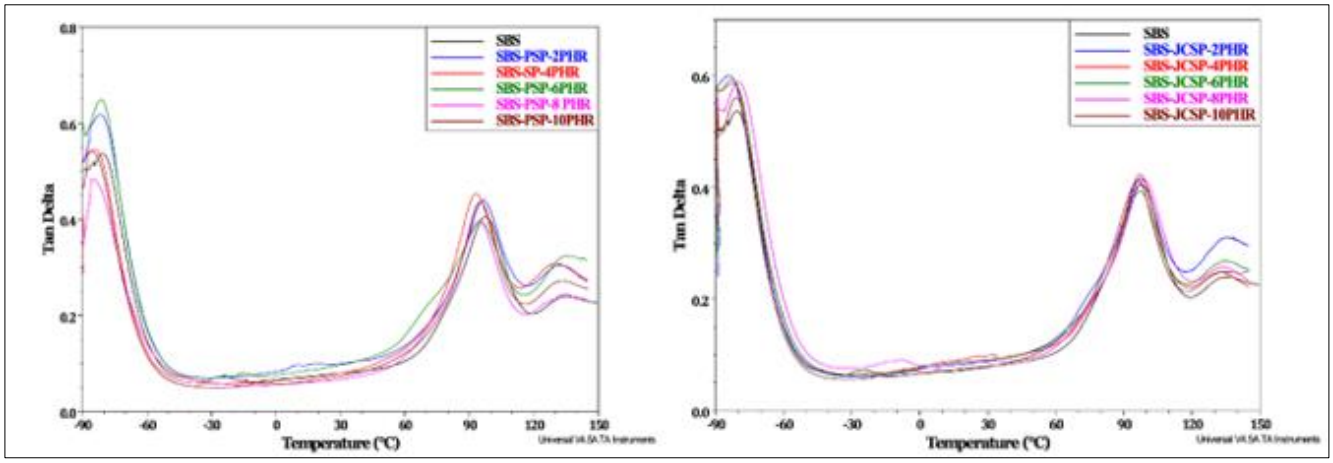

Figure 8. Tan δ curves obtained by DMA for a) SBS and SBS-PSP, and b) SBS-JCPS composites

3.3. Dynamic mechanical analysis (DMA)

DMA is a powerful technique to evaluate the viscoelastic behavior in composite materials. Through the storage modulus and Tan δ, it is possible to identify the reinforcer dispersion and the degree of matrix modification. Figure 7a shows the Storage modulus obtained from DMA thermogram for SBS and SBS-PSP composites. It is observed that at low temperatures the addition of PSP affects the viscoelastic behavior in composite, as the storage modulus decreases with increasing PSP concentration, except for SBS-PSP 10 phr composite. This indicates that the enhanced stiffness the material decreases the free movement of particles in the polymer backbone. This behavior is common for two-block copolymers, since one of the blocks possesses higher compatibility with the filler, therefore, that specific block is likely to be more affected than the other one. In this case, the polybutadiene (PB) block is usually the PSP-rich phase [19]. In addition, around 80ºC the SBS-PSP composites behave as pure SBS, because this temperature is close to polystyrene (PS) glass transition, which suggests that PSP has better interaction with the PB block than with the PS block. Salasinka and Ryszkowska [27] report a similar behavior of HDPE composites with PSP, attributing the decrease in storage modulus to poor adhesion between the particles and the polymer matrix, that is more evident at higher particles concentrations.

For SBS-JCSP composites (Figure 7b), the behavior of Storage Modulus was similar, but in this case SBS-JCSP with 10 phr content shows an increase of storage modulus. For the composites with lower content of JCSP the storage modulus is reduced with respect to SBS, as observed in SBS-PSP composites. This suggests that it is possible to use higher concentrations of SP to get a higher storage modulus and improve the stiffness of the composites.

To evaluate how the inclusion of PSP affects the SBS matrix, the Figure 8a and b the Tan δ for SBS and SBS-composites, where two peaks are observed due to the PB block (around -85ºC) and PS block (arpund 90ºC) transitions. It can be observed in Figure 8a that the quotient value is affected with the addition of PSP. The PB block appears to be the most influenced by the addition of PSP at 2 and 6 phr, concentrations at which the Tan δ value increases, indicating a good particles-matrix interaction and a reinforcing effect at these concentrations. On the other hand, a decrease in Tan δ is related to the restriction of the polymer chains mobility because of the presence of particles that hinder the movement of the matrix molecules [28]. Additionally, the PS block is also affected by the incorporation of PSP, as the composites show a variation in the quotient and a slight displacement of the peak to lower temperatures, which means that PSP cause a plasticizing effect.

The Tan δ can be related to the impact resistance of material, decreasing of quotient indicates the damping reduction. The level effect of filler or reinforcers in a polymer matrix, on depends of concentration, size and shape particle in matrix. For SBS-JCSP composites (Figure 8b), it can be observed that the Tan δ value at low temperatures is higher in all the SBS-JCSP composites with respect to SBS, indicating an improvement in damping of the PB block in composites. Oppositely, the PS block

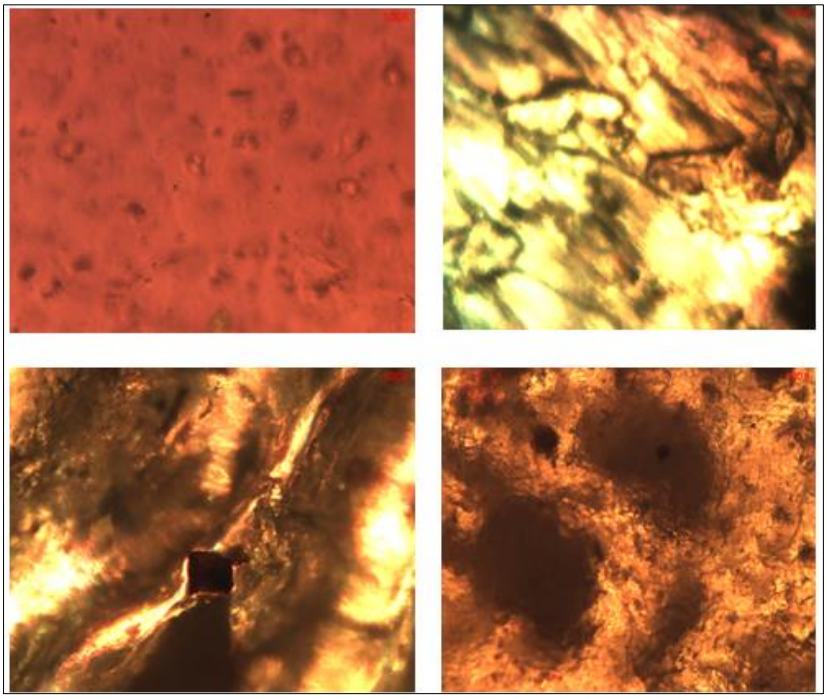

Figure 9. Optical Microscopy images for SBS (a) and SBS-PSP composites with 2 PHR(b), 6 PHR (c) and 10 PHR (d)

peak does not suffer any temperature displacement and the Tan δ value shows a minimal change, which means that the presence of JCSP mostly affects the soft block in SBS copolymer.

3.4 Optical microscopy (OM)

Figure 9 shows the OM images of SBS (a) and SBS-PSP-2 PHR (b), 6-PHR (c) and 10 PHR(d). The optical microscopy allows to observe the state of dispersion of SP on SBS matrix. Figure 9a shows the SBS surface that is smooth with some points that seem to be imperfections on material, in figure 9b, c and d, present the images for composites with 2, 6 and 10 phr, it is possible to distinguish that particles are distributed in SBS matrix and there is an increase of presence of PSP according with content, and there is not presence of agglomerates zones of PSP, which can create weak point in matrix [15]. This behavior is evidence that PSP are well distributed in matrix which is associated with good dynamical mechanical behavior, as was discussed previously in section 3.3.

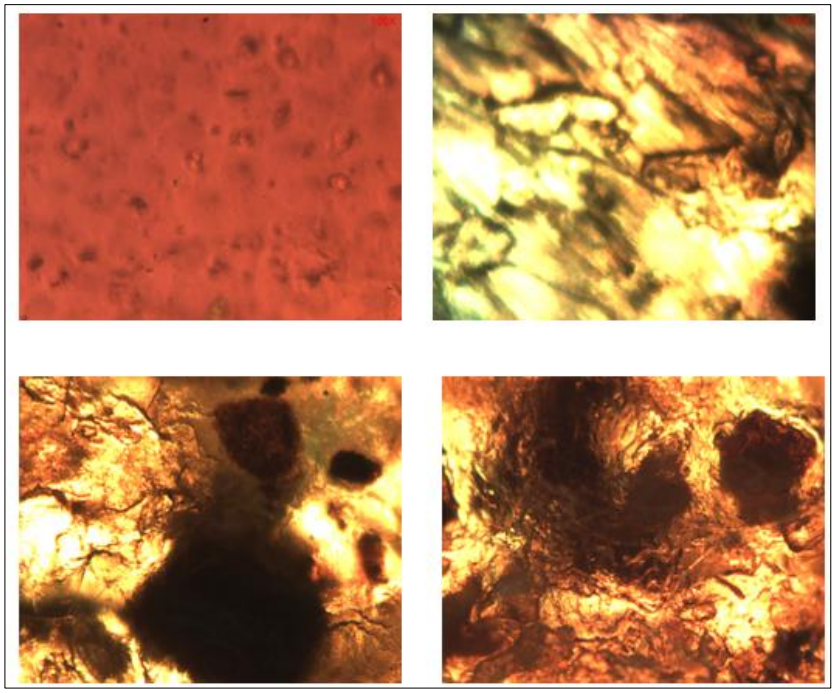

Figure 10. Optical Microscopy images for SBS (a) and SBS-JCSP composites with 2 PHR(b), 6 PHR (c) and 10 PHR (d)

A similar behavior was observed in figures 10 a, b, c and d, that reports the OM images for SBS, and composites with 2, 6 and 10 phr of JCPS respectively, where it is possible to observe that SP are well distributed in SBS matrix and that according with content increase, the presence of particles is more evident.

4. Conclusions

According with obtained results, there is an effect in thermal stability of Composites was modified with the presence of SP particles, it is interesting due decrease the onset temperature of composites compared with SBS matrix. The viscoelastic behavior was modified in presence of SP, resulting the major affectation the soft block of SBS copolymer, and with not affectation in transition temperatures. The IR results show not evidence of a reaction between particles and elastomeric matrix. This is an interesting founding considering that SP are consider waste materials with no application, and results of this explorative work can help to taking advantage of these materials in the aim to avoid pollution, for solve environmental duties furthermore to produce composite materials with improved properties that can find an interesting application in a wide options areas.

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