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X-ray characterization of thermoplastic starch and clay nanocomposites

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Abstract – TPS-montmorillonite Cloisite-Na $^{+}$ nanocomposites were prepared by melt processing in a Haake Rheocord mixer. X-ray diffractions were used to investigate the clay exfoliation and intercalation degrees in thermoplastic starch (TPS) matrix. At lower clay content (1 and 2 wt %), it was observed a good intercalation and complete exfoliation of the clay platelets in polymeric matrix. The appearance of the new peak at 2 θ = 4.86° and at 2 θ = 4.96° for 3 and 5% clay indicates the intercalation of TPS in the gallery of the silicate layer of the clay; however, in these conditions the exfoliation degree was not very efficient.

One of the most studied and promising raw materials for the production of biodegradable plastics is starch [1]. One major problem with granular starch composites is their limited processibility. Therefore, it is difficult to make blown thin films for package applications. For this reason, thermoplastic starch (TPS) has been developed by gelatinizing starch with heat and pressure [2]. A recent and innovating area of composites called nanocomposites, which is a combination of biodegradable polymer matrix and nanosized filler, has been proposed. The main reason for such improvement compared to conventional composites is the large surface area resulting in high interactions between the polymer matrix and the nanofillers when theses nanoparticles are well dispersed. The most intensive researches are focused on layered silicates, and especially on montmorillonites (MMT) (such as Cloisite-Na⁺), as the reinforcing phase due to their availability, versatility, and respectability [3]. In this work, the TPS-MMT Cloisite-Na⁺ nanocomposites were prepared by melt processing in a Haake Rheocord mixer with controlled parameters, i.e.: residence time, temperature and rotor speed. X-ray diffractions powder (XRD) pattern were collected using a Rigaku D/Max 2500PC X-ray diffractometer with a rotary anode using Cu Ka (k = 1.5406 Å). The clay interlayer spacing values (d₀₀₁) were calculated from the MMT diffraction peak with the use of the Bragg's law. Figure 1 shows the XRD patterns of the Cloisite-Na⁺, TPS, and TPS-Cloisite-Na⁺ nanocomposites prepared at different clay content (1-5 wt %). Cloisite-Na⁺ shows an intensive peak at 2 θ = 7.80°, corresponding to an interlayer basal spacing (d_{001}) of 1.14 nm. For nanocomposites with 1 and 2% clay, no diffraction peak between 2 θ = 3-12° was observed, indicating a good nanodispersion (intercalation) and complete exfoliation of the clay platelets, i.e., separation of platelets from one another and dispersed individually in the TPS matrix. It was also observed the appearance of the new peak at 2 θ = 4.86° (d₀₀₁ = 1.82 nm) and at 2 θ = 4.96° (d₀₀₁ = 1.78 nm) for 3 and 5% clay (wt %) indicates the intercalation of TPS in the gallery of the silicate layer of the clay; however, in these conditions the exfoliation degree was not very efficient. These diffraction peaks may be caused by the higher clay content of this sample, which could lead to the formation of some clay aggregates, but according to the low intensity of this diffraction peak, the aggregation degree should be rather low [3].

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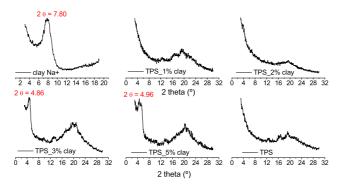


Figure 1: XRD patterns of the Cloisite-Na⁺, TPS, and nanocomposites prepared at different clay content (1-5 wt %).

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