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Synthesis and Characterization of Starch-Layered Double Hydroxide hybrid films

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Abstract – Novel layered double hydroxide (LDH) -based hybrid films were synthesized using the coprecipation method in the presence of gelatinized starch and aftermost solvent removal. X-ray diffraction (XRD) data of plasticized and unplasticized films reveal no peaks associated to the inorganic phase due to LDH delamination. TG-MS (thermogravimetry-mass spectrometry) curves also show a significant anticipation in the beginning of several degradation events associated to the decomposition of polymeric matrix after LDH loading with main release of fragments (m/z) 18 and 44, related to water and CO_2 species, respectively.

Organic-Inorganic hybrids consisting of cassava starch and layered double hydroxide (LDH) was synthesized employing coprecipitation method [1] to obtain the particles. At first, starch was gelatinized in deionized water at 75ºC for 1 hour with posterior addition of lactic acid to the gum and subsequent dropping addition of zinc and aluminum lactate solution (Zn^{2+}/Al^{3+} molar ratio = 2) to the system, controlling the pH around 7 with a NaOH solution. After addition of metals' solution, the system was kept under homogenization for 1 hour. Half of the formed gum was extracted from the system and poured into polystyrene recipients, while to the remaining part, a certain volume of glycerol was added. The remaining mixture was still kept under agitation for 1 hour and finally poured into a polystyrene recipient. Both materials were left at room temperature in order to promote the solvent removal.

XRD data of isolated materials showed that plasticized starch possess a significant amorphous structure, comprehending a broad peak between (2θ) 15-25º, even though small peaks can be observed in this range as well. Starch-LDH hybrid film without plasticizer exhibits a higher semicristallyne character than plasticized starch and also presents a peak around (2θ) 11.7º suggesting that a small part of the synthesized LDH was not delaminated in this system. The presence of plasticizer and delaminated LDH particles in the plasticized Starch-LDH causes a remarkable decrease in the crystallinity [2]. The low inorganic content over the synthesized hybrids can also be responsible for the non-visualization of LDH-Lactate diffraction peaks.

TG-MS curves reveal that all organic-based materials synthesized present three main events of decomposition, where the first one is related to the release of adsorbed water of the structure, occurring until 150 ºC. Second step is related to the non-oxidative decomposition of starch and it is remarkably anticipated due to the presence of LDH particles. The last decomposition process is related to the combustion of residual carbon and is clearly affected by the presence of temperature-modified LDH, shifting the beginning of the event for higher temperatures in both cases. The presence of glycerol also affects the shape of the last decomposition process probably due to the type of organization of the inner system and the presence of distinct reactive carbon species from different carbon sources after the non-oxidative event.

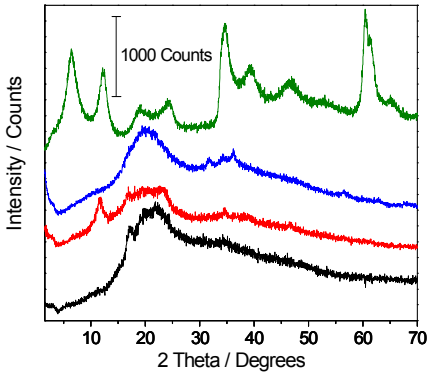

Figure 1: XRD data of plasticized starch (black), Starch-LDH without plasticizer (red), Starch-LDH glycerol-plasticized (blue) and LDH-Lactate (green).

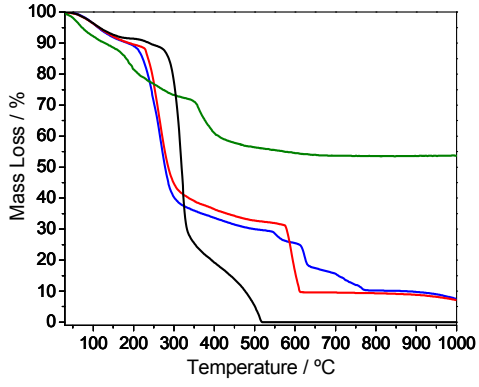

Figure 2: TG curves of plasticized starch (black), Starch-LDH without plasticizer (red), Starch-LDH glycerol-plasticized (blue) and LDH-Lactate (green).

References

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[2] C. Jaubertie, M. J. Holgado, M. S. San Román, and V. Rives, Chem. Mater. 18 (2006), 3114-3121.