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Crystallization behavior of polypropylene/calcium carbonate nanocomposites

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Abstract - Recently calcium carbonate nanoparticles have been used to enhance impact resistance of polymeric materials. Besides their toughening effect calcium carbonate nanoparticles affect crystallization of semicrystalline polymers. The aim of this work is to study the effects of calcium carbonate nanoparticles in the crystallization of polypropylene. The experimental includes DSC analyzes of isothermal and non-isothermal crystallization, optical microscopy, and X-Ray Diffraction. The results showed that the incorporation of nanoparticles increases the crystallization temperature, and the kinetics constant (k). Moreover the half crystallization time and spherullites diameter were reduced. Finally we found the presence of PP β phase in the nanocomposites.

Four compositions of PP/$CaCO_3$ nanocomposites with calcium carbonate content of 3, 5, 7 and 10%wt were prepared in a co-rotational twin screw extruder machine Werner&Pfleirer ZSK-30, with temperature profile of 170/190/190/190/190/195^oC, and screw speed of 100rpm. DSC analyzes were conducted by heating the samples from 30^oC to 200^oC at a heating rate of 10^oC/min, keeping the sample at this temperature for 2 minutes and than cooling down from 200^oC to 30^oC with a cooling rate of 10^oC/min. The isothermal analyzes and optical microscopy analyzes were conducted by heating the sample at the same conditions and than cooling from 200^oC to their crystallization temperature, maintaining at this temperature for 15 minutes. X-Ray diffraction analyses were conducted in a Rigaku Geiger Flex equipment using Cu Kα radiation, with 2θ varying from 5 to 90^o. All the samples were obtained from injection molded tensile specimens.
The results showed the presence of β phase through DSC and X-Ray diffraction analyzes of the nanocomposites which is the result of the nucleation effect of $CaCO_3$ nanoparticles in PP crystallization process [1] (Figure 1). The non-isothermal analyzes showed that the melting temperature is not affected by $CaCO_3$ nanoparticles but the crystallization temperature and crystallinity degree increase with the addition of calcium carbonate content (Table 1). Isothermal analyzes showed that the incorporation of $CaCO_3$ nanoparticles reduces the half crystallization time ($t_{1/2}$) and increases the kinetics constant (k) which means that the nanocomposites crystallizes faster than neat PP due to the nucleation effect of the nanoparticles. [2] (Table 1) Optical microscopy results show a reduction in the spherullites size with the incorporation of nanoparticles. From the results of this work it's clear that $CaCO_3$ nanoparticles affect the crystallization process of PP by changing its phase formation, crystallization temperature and kinetics and spherulitic morphology.

Table 1: Thermal properties of polypropylene and the nanocomposites.

Sample	$T_{melting}$ (^oC)	$T_{Crystal.}$ (^oC)	Crystallinity Degree (%)	$t_{1/2}$ (s)	k (s^{-1})
PP	167	117	38	27	$8{,}52 \times 10^{-5}$
PPC3	167	126	40	25	$3{,}42 \times 10^{-4}$
PPC5	166	124	45	25	$2{,}95 \times 10^{-4}$
PPC7	167	124	46	24	$5{,}64 \times 10^{-4}$
PPC10	167	124	44	23	$4{,}44 \times 10^{-4}$

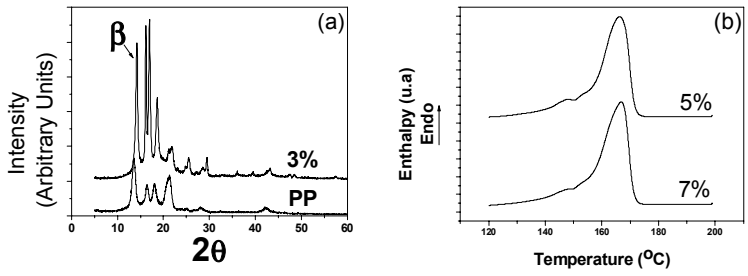

Figure 1: (a) X-Ray diffraction spectra of neat PP and nanocomposite with $CaCO_3$ content of 3%wt and (b) Heating curves of PP/$CaCO_3$ nanocomposites with calcium carbonate content of 5 and 7%wt.

References

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