Polymer Nanocomposites: Influence of Processing on Product Stability

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Polymer processing is a critical step in the manufacture of polymer articles. This step is even more critical when dealing with inhomogeneous polymer nanocomposites systems. Proper selection and tuning of the process variables should, in principle, lead to improved characteristics of the fabricated product. With multiphase systems, however, this is not straightforward and it is often the case that the process conditions are chosen initially to improve one or more desired properties at the expense of others. My aim in this lecture is to show how processing conditions affect thermoxidative stability of polypropylene nanocomposites (PPNCs) and how, in turn, aspects of stability may be used to better define these conditions so as to improve the manufacturing process of a specific formulation.

The melt blending approach is generally adopted for manufacturing polymer nanocomposites in which twin-screw extrusion plays a central part to achieving efficient mixing and compounding in a continuous production process. Co- and counter- rotating twin-screw extruders (TSE), with specially designed screw configurations, are used in a reactive processing approach to produce a product with optimal melt properties and characteristics suitable for further downstream conversion processes. For a specific screw size and design, temperature and screw speed can be used to control the production process for delivering the desired quality of the extrudate for a set of starting components. A systematic approach to studying the effects of these two process variables should, therefore, lead to a better definition of the process conditions required to produce polymer nanocomposites with target end-use properties. A stabilised PPNC system (containing Cloisite 15A & 20A) was used to perform the work reported here.

The design-of-experiment study was performed using a TSE fitted with an intermeshing co-rotating screw. Both temperature (210^{o}C & 230^{o}C) and screw speed (100 rpm & 300 rpm) were varied during the production process which was carried out under similar output process power so as to maintain similar stress levels on the melt. Profiles of properties of the extrudates were developed and related to their processing conditions. Properties examined include the thermoxidative stability (melt and solid) and impact strength of the extrudates.

A melt-recirculation (MR) test protocol using a micro-extruder with conical co-rotating screw geometry was used as a guide to assessing the melt stability. **Figure 1** shows changes in the torque in PPNCs under fixed conditions of shear (355 s^{-1}) and temperature (190^{o}C) with limited air ingress. It is clear that under both extrusion temperatures and screw speeds the PPNCs samples containing C20A nanoclay showed higher melt stability than those containing C15A.

Examination of the long term thermal stability (LTTS) of PPNCs containing C20A and C15A nanoclays, using air-circulating single-chamber Wallace Oven tests, shows that films containing C20A outperform also their C15A counterparts produced under the different extrusion conditions (see **Fig. 2a and b**) which agrees with the order of their melt oxidative stability described above. However, higher screw speeds under both extrusion temperatures tested gave much higher thermal stability (in both Wallace oven test in the solid state and DSC - OIT test in the melt), see for example **Fig. 2c**.

The organically modified C15A nanoclay contains more of the thermally unstable QUAT (quaternary ammonium surfactant). The higher amounts of the QUAT in the C15A nanoclay may contribute, at least in part, to the lower overall LTTS observed with PPNC-C15A films. However, there are some major differences in the distribution (surface vs bulk) of these two clays in these films which would influence their response to thermoxidative stresses. The structural differences of the PPNCs containing the nanoclays and the effect of the processing conditions on their overall behaviour will be elaborated.

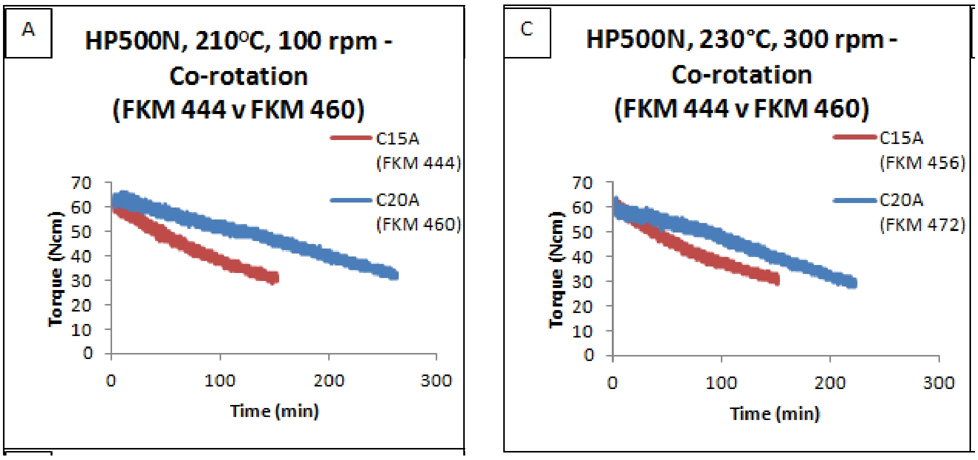

Fig. 1. Torque - time history from MR tests for C15A- (lower curves, in red) and C20A- PPNCs produced under different processing conditions.

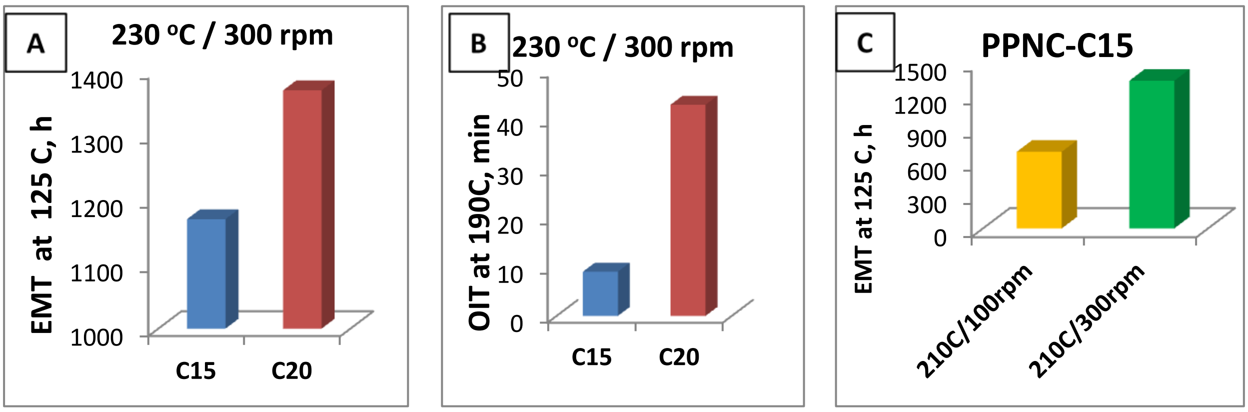

Fig. 2. Time-to-embrittlement from LTTS tests of PPNCs produced under different extrusion conditions.