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A novel tool to determine nucleus density in colored system with NA

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Introduction
Adding nucleating agent (NA) into a crystallizable polymer system is a common way, applied in polymer industry, to control crystallization and tailor mechanical and optical properties of products. These properties are closely related to the resulting nucleus density. For colored NA systems, nucleus density becomes too large and subsequently changes the optical properties, which will both make quantification of nucleus density difficult with optical microscopy. The aim of this project is to develop a new method to determine the nucleus density for colored NA systems with rheometry.

Materials and Experiments
An isotactic polypropylene (Borealis HD120MO, Mw = 365 kg/mol, PDI = 5.4) was compounded with the organic NA (U-Phthalocyanine, bulk density = 310 kg/mol) at a concentration of 0.2 wt%, by which the system was colored blue.

A Rheometrics ARES rheometer was used with a plate-plate geometry for small-amplitude oscillatory measurements at an angular frequency of 5 rad/s and a strain of 0.5%.

Method
1) Obtain raw data from rheological measurements.

2) Derive space filling from dynamic moduli.
A linear viscoelastic version of the three dimensional generalized self-consistent method (3D GS CM) of Christensen and Lo[1] was used. This model has been validated by experimental data in our previous work[2]. If spherulites are formed, the relative dynamic modulus, \( f^* G = G^* / G^*_{0} \), is obtained from

\[
A^* f^* + B^* f^* + C^* = 0
\]

with \( G^* \) the effective dynamic modulus and \( G^*_{0} \) the dynamic modulus of the continuous phase. The complex coefficients \( A^* \), \( B^* \) and \( C^* \) depend on space filling \( \phi \), \( \mu^* = G^* / G^*_{0} \), which is the ratio of the complex moduli of the continuous phase \( (G^*_{0}) \) and dispersed phase \( (G^*_{1}) \), and \( \nu_0 \) and \( \nu_1 \), being the Poisson ratios of both phases.

In this case, space filling is unknown and is obtained from the measured \( f^* G \) by minimizing eq. (1). Results obtained by fitting are shown in Fig. 2.

3) Use space filling to determine nucleus density.
Kinetics of space filling at different temperatures depend on both nucleus density and growth rate. Growth rate is a known exponential function of temperature[3],

\[
G(T) = G^*_{ref} \exp \left[ -c_{G}(T - T^*_{T^*_{ref}})^2 \right]
\]

where for HD120MO, parameters \( G^*_{ref} \), \( c_{G} \), \( T^*_{T^*_{ref}} \) are \( 3*10^{-6} \) m/s, \( 2.3*10^{-3} \) and \( 363 \) K, respectively. And then, nucleus density could be obtained using Avrami equation[4]:

\[
N(T) = \frac{-\ln(1 - \phi)}{4\pi G(T)\phi^3}
\]

Conclusion
A suspension-based rheological method to determine the nucleus density is applied to colored NA filled systems, where optical microscopy is not possible. Nucleus densities of this system and pure iPP, crystallizing at various temperatures, determined by this method are shown in Fig. 3.

References