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Correlating Melt Rheology of PET to Solution Intrinsic Viscosity<<

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1. INTRODUCTION

This work continues our study of the rheology of PET resins. Specifically this paper covers efforts made at improving capillary test methods and algorithms for estimating Intrinsic Viscosity (IV) from viscosity measurement in the melt.

Polymers have the unique ability to dramatically increase the viscosity of a liquid they are dissolved in, even at very low concentrations. Intrinsic Viscosity is a quantitative assessment of this ability. IV is not, however, directly related to molecular mass (molecular weight). A polymer chain with a branched structure will have a different IV than a linear chain with the exact same molecular mass. This is because the increase in viscosity is related more directly to the volumetric size of coiled polymer in the solvent. It is necessary therefore to develop empirical correlations between IV and molecular mass (obtained by some absolute method like light scattering, end group analysis etc.) for IV to predict molecular mass with accuracy. IV is used commercially to differentiate grades of resins along with other properties.

Most applications for polymers do not involve dissolution into solvent. More typically they are extruded, injection molded, calendered etc., all processed in the meeting of the application's specifications or production cost requirements. It makes sense therefore to test the material as close as possible to its final form since removal of these key components are critical to the final behavior of the material. Measuring solution viscosity, of course, necessitates the removal of these components whereas testing in the molten state does not. The first step in using melt rheology is to correlate neat (no additives) polymer IV to melt rheology. Correlations between IV and melt rheology for neat polymer exist for Polypropylene and PET though they have not received wide spread use. The two rheological parameters typically used for the correlation are the Zero Shear Viscosity and Intrinsic Melt Viscosity (IMV). Zero Shear Viscosity is used to describe a viscosity which has been obtained by extrapolating viscosity vs. shear rate data to zero rate. Like IV, the Zero Shear Viscosity can be related empirically to molecular mass. IMV is typically obtained from a constant shear rate test (or constant stress) where viscosity versus time is obtained for a degrading polymer and the data is extrapolated to obtain the viscosity at zero time (described in detail in the appendix of ASTM D3835). Previously we had shown a very clear correlation of IV to Zero Shear Viscosity, IMV and Melt Flow Rate for one particular vendor of PET using virgin materials. We will reiterate some of that work and append it with results from an additional vendor along with some recent model development work for obtaining a limiting viscosity at zero time.

CONCLUSIONS

When correlating melt rheology to IV, a multitude of potential problems can be remedied by "calibrating a system" with a known IV sample. The known IV sample adjusts the vertical offset value on the log-log plot to account for 1) systematic variations in the IV measurement (use of different IV test temperatures between vendors etc.) 2) systematic change in the polydispersity of a group of samples, and 3) minor inaccuracies in the test device. That is, since the slope of the Zero Shear Viscosity curve versus IV seems fairly robust, the known sample fixes the vertical position of a trace. For a single supplier of virgin material, results within +/- 0.01 dl/g are typically produced.

Assumptions include, that moisture level does alter the power dependence (slope of the line) significantly or that deviations are not overwhelming especially if the correlation is used over a limited window, near the reference point, as in inspection of incoming/outgoing material. Drying must be consistent.

Melt flow index can also be used though care must be taken to assure proper drying has occurred and an empirical rather than theoretical power dependence should be used. A reference sample of known IV is needed for each IV generated from different labs (vendors) unless consistent IV test methods are used.

- THE END –