

TORQUE VISCOMETRY OF MOLTEN POLYMERS AND COMPOSITES

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ABSTRACT:

A systematic approach for collecting data from a torque rheometer is described, and the Bousmina et al. model is evaluated for HDPE, LDPE, LLDPE and HDPE/filler composites. The torque rheometer results are in good agreement to capillary and parallel plate viscometer measurements for neat polymers, when the torque values measured are corrected for the temperature rise due to viscous dissipation. For the composites, the torque and capillary results virtually coincide, but the viscosities are lower than those measured with the parallel plate instrument, in oscillatory mode, because the Cox-Merz rule is not valid. Although there are some limitations at high shear rates, due to viscous dissipation, the present work provides a proof and a methodology for the practical utility of torque rheometers for viscosity measurement in highly viscous systems.

ZUSAMMENFASSUNG:

Ein systematischer Ansatz, um Daten mit Hilfe eines Drehmomentrheometers zu erhalten, wird beschrieben und das Modell von Bousmina et al. für HDPE, LDPE, LLDPE und HDPE/Füllstoff-Komposite ausgewertet. Die Ergebnisse des Drehmomentrheometers stimmen für reine Polymere gut mit Messungen eines Kapillar- und eines Parallele-Platten-Rheometers überein, falls die gemessenen Drehmomentwerte bzgl. der Temperaturerhöhung aufgrund der Dissipation korrigiert werden. Für die Komposite stimmen Drehmoment- und kapillarrheometrische Resultate virtuell überein. Jedoch sind die Viskositäten geringer, als die mit dem Parallele-Platten-Rheometer im oszillatorischen Modus gemessenen Werte, da die Cox-Merz-Regel nicht erfüllt ist. Obgleich bei hohen Schergeschwindigkeiten aufgrund von Dissipation Limitierungen existieren, beschreibt diese Arbeit eine Begründung und eine Methode für die praktische Anwendung eines Drehmomentrheometers für Viskositätsmessungen von hochviskosen Systemen.

RÉSUMÉ:

Une approche logique pour la collection de données provenant d'un rhéomètre de torsion est décrite, et le modèle de Bousmina est calculé pour des composites de HDPE, de LDPE, de LLDPE et de HDPE avec charges. Les résultats du rhéomètre de torsion sont en accord avec les données obtenues avec un viscosimètre capillaire et un viscosimètre à plateaux parallèles pour les polymères seuls, lorsque les valeurs mesurées pour le couple sont corrigées en tenant compte de l'élévation de température due à la dissipation visqueuse. Pour les composites, les résultats des mesures en viscosimétrie capillaire et de torsion coïncident pratiquement, mais les viscosités sont plus basses que celles mesurées avec l'instrument aux plateaux parallèles, dans le mode oscillatoire, parce que la loi de Cox-Merz n'est pas valide. Malgré les limitations à grandes valeurs de taux de cisaillement, dues à la dissipation visqueuse, ce travail fournit une démonstration et une méthodologie pour l'utilisation pratique des rhéomètres de torsion dans les mesures de viscosités de systèmes très visqueux.

KEY WORDS: viscosity, viscous dissipation, Cox-Merz rule

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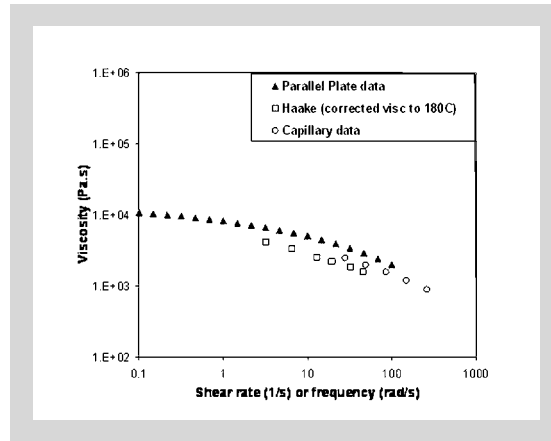
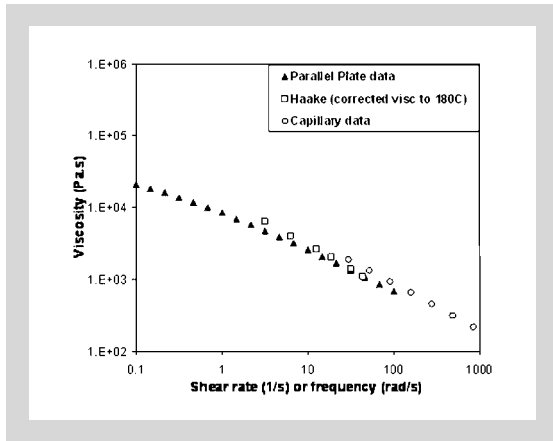
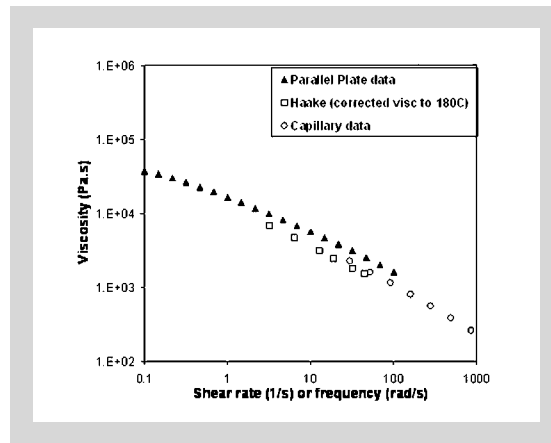
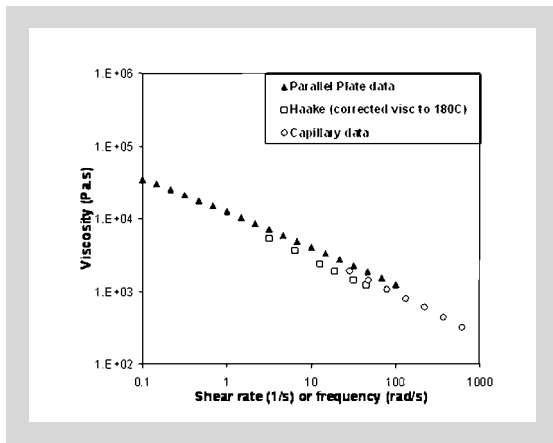


Figure 4 (left above):
Viscosity results for HDPE₁.

Figure 5 (right above):
Viscosity results for HDPE₂.

Figure 6 (left below):
Viscosity results for LDPE.

Figure 7 (right below):
Viscosity results for LLDPE.

mer degradation and the temperature rise too high, and the possibility of large errors in the torque correction procedure that was described earlier.

The dynamic and capillary data of Figures 4 to 7 show that the Cox-Merz rule is generally obeyed for HDPE, LDPE and LLDPE. This rule states that the shear rate dependence of the steady state viscosity η is equal to the frequency dependence of the complex viscosity η^* [15]; that is,

$$\eta(\dot{\gamma}) = |\eta^*(\omega)| \quad \text{with } \dot{\gamma} = \omega \quad (9)$$

Some deviations from an absolutely perfect match are probably due to wall slip phenomena that were observed, but were not taken into consideration in plotting of the capillary viscosity data. The torque rheometer data are also in very good agreement with those of the capillary and the parallel plate and these results prove that the procedures followed for the determination of an equivalent internal diameter and the correction for temperature rise due to viscous dissipation are valid.

The results for the three composite formulations studied are shown in Figures 8 to 10. The torque rheometer data are in excellent agreement with the capillary data. However, the dynamic viscosity values are much higher than the steady viscosity values, which mean that the

Cox-Merz rule is not obeyed for these filled systems. This was also noted by Hristov *et al.* [16] for wood fibre polymer composites and other filled systems [17, 18]. This behaviour can be attributed to wall slip phenomena, filler orientation and filler-filler interactions, but it is beyond the scope of the present investigation.

For comparison purposes the power-law parameters from best fitting of the capillary and torque rheometer data are shown in Table 4. It can be seen that although the shear rate range of the capillary data is much larger, the parameters obtained from the torque rheometer are very close to those from the capillary, and this is an indication of the practical utility of such an instrument.

4 CONCLUSIONS

Experiments were carried using a torque rheometer with the objective of assessing the suitability of this instrument for viscosity measurements. Four different low melt index polymers and composites of HDPE with wood flour, CaCO₃ and talc at 40 % by weight were used. For determination of the viscosity and shear rate, equations proposed by Bousmina *et al.* [7] were found to be suitable. The experiments show significant temperature rise due to viscous dissipation at high rotational speeds. To obtain viscosity versus shear rate curves the measurements were cor-

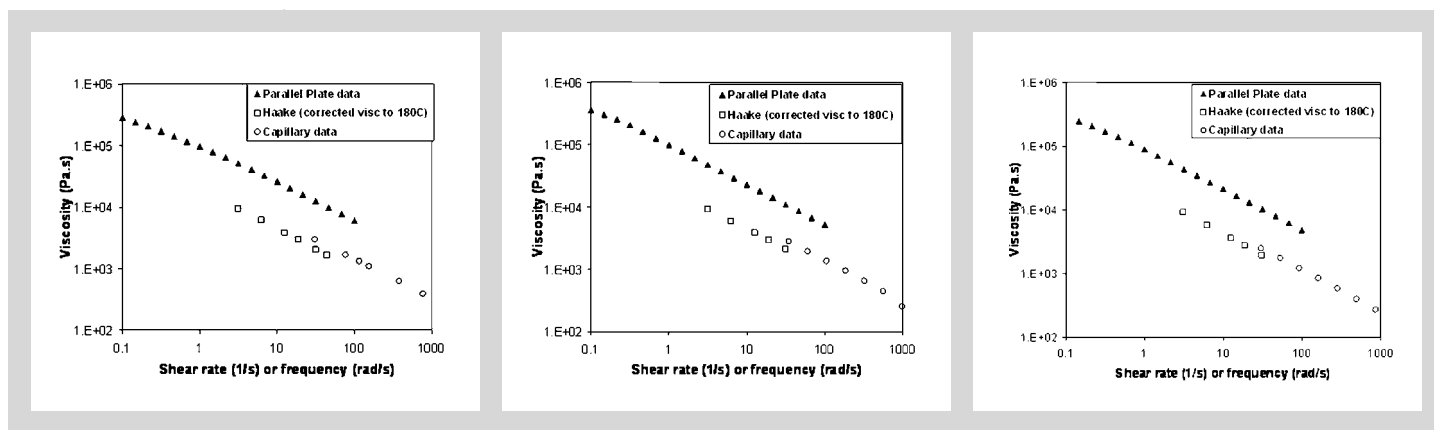


Figure 8 (left): Viscosity results for composite HDPE1/Wood flour.

Figure 9 (middle): Viscosity results for composite HDPE1/CaCO₃.

Figure 10 (right): Viscosity results for composite HDPE1/Talc.

rected for the torque reduction due to viscous dissipation. The torque rheometer results are in very good agreement to capillary and parallel plate viscometer measurements for neat polymers. For the composites, the torque and capillary results virtually coincide, but the viscosities are lower than those measured with the parallel plate instrument, indicating that the Cox-Merz rule is invalid. Although there are some limitations at high shear rates, due to viscous dissipation, the present work provides a proof and a methodology for the practical utility of torque rheometers for viscosity measurement.

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