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Test Method

Influencing factors of viscosity measurement by rotational method

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ABSTRACT

Rotational rheometer was used to measure the shear viscosity of a liquid reference material of viscosity named GBW13604. The influences of heat balance time, bath temperature, environment temperature and sampling volume on results of measurements were investigated. Furthermore, a comparison of accuracy and repeatability of shear viscosity results between two commonly used systems with cone–plate and coaxial cylinder geometry was performed. Rheological tests indicate that the coaxial cylinder method is applicable to tests requiring high accuracy, while the cone-plate method is suitable for measurements that demand low sampling volume and fast speed.

1. Introduction

Viscosity measurement is of great significance both in research and engineering applications on polymer materials with applications in realms as diverse as electronics, petrochemicals, foods, and healthcare technologies. For example, as to liquid crystal polymers, viscosity behavior in the nematic state plays an important role in the molecular dynamic characteristics, therefore viscosity is of major significance to rheology and commercial processing for such material [1]. Because of synthetic lubricants which use polymer as base stock has better performance than the mineral oil, thus, play more and more important role in modern industries. The viscosity of synthetic lubricants need to be finely controlled, thereby pumpability in the lubrication system, access nodes, and minimal frictional resistance during cranking can be ensured [2]. The viscosity variation with temperature is also important for synthetic lubricants application [3]. In oil recovery, water-soluble polymer solution uses its high viscosity and residual resistance coefficient to adjust the water injection profile thereby improving the water-oil mobility ratio and leads to enhanced oil recovery. Therefore, the study of viscosity measurement is also importance in enhanced oil recovery applications [4]. At present, methods for liquid viscosity measurements include capillary method, falling ball method, rotational method, vibration method and so on, among which rotational method is widely used because of its high level of automation, wide range of application and easy operation [5–8].

The measurement of viscosity by rotational method depends on rotor movement to generate shearing motion, derive liquid viscosity through measuring the viscous torque of liquid on rotors or measuring the rotational speed of rotors. Rotational method can be further employed in the measurements of parameters, such as modulus and viscoelasticity of polymer materials [9–13]. Since accuracy and repeatability of the measurements are not good enough, generating reliable viscosity measurements still remains a challenge [14]. In most cases, rotational method is not used as the primary standard in viscosity metrology system. But our researches showed that with accurate control over temperature, sampling volume, thermal equilibrium time and other factors during measurement, the accuracy of rotational method can be significantly improved. Thus, this method can meet the requirements of high-accurate measurements and became an important method for viscosity metrology of non-Newtonian fluid [15].

For the metrology of material, in addition to the accurate measurement of traceable input and traceable response, it is necessary to take into account the traceable characterization of material and the influence of the procedural aspects on the results [16]. In this paper, various factors that influence the measurement process using rotational rheometer were investigated. The results provide guidance to improve measurement accuracy of the rotational rheometer and expand the application of rotational rheometer to the measurement of polymer materials in related fields.

The rotational method uses different testing geometries, such as coaxial cylinder, cone-plate, double plates, double cones, hemisphere and so on. The measuring results from one type of geometries are not

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readily translatable into another. In this paper, we focused on the two most common used geometries: coaxial cylinder and cone-plate. The advantages and limitation of as well as scenario of application of these two setups were discussed.

2. Experimental

2.1. Materials

Reference material for viscosity, named GBW13604 with certificate value of 16.61 mPa s at 20 °C, was provided by the National Institute of Metrology, China.

2.2. Experimental apparatus

All experiments were performed on a rotational rheometer Discovery HR-2 by TA Instruments.

2.3. Experiment procedure

Two geometries were used for viscosity measurements:

Coaxial cylinder: minimum sampling volume is 22.4 ml; diameter of the rotor is 27.98 mm; inner diameter of the outer cylinder is 30.00 mm; length of the rotors is 42.15 mm; the distance from the top of rotor to the bottom of the outer cylinder is 5.91 mm during experiment.

Cone-plate: minimum sampling volume is 0.3 ml; diameter of the cone is 40.00 mm; cone angle between the cone and the plate is 1.011°. Except for experiments concerning the influence of sampling volume, scraping treatment of samples is necessary during experiments.

We used minimum sample volume in the series of experiments, 22.4 ml for coaxial cylinder system and 0.3 ml for cone-plate system, except the part which discussed the influence of sample volume.

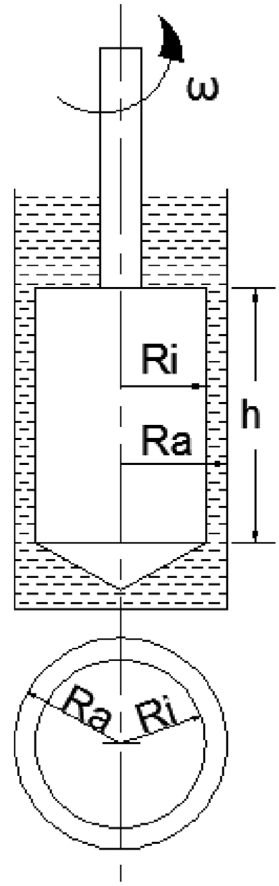

Fig. 1. Schematic diagram by coaxial cylinder.

3. Results and discussion

During measurements by coaxial cylinder, samples are filled into the concentric gap between outer cylinder and rotor. The radius of outer cylinder is set to be R_a. The radius of rotor is R_i. The immersion depth of rotor is h, as shown in Fig. 1.

When outer cylinder is stationary while rotor rotates with an angular velocity ω, the gradient of velocity can be derived as:

$$dv/dr = rd\omega/dr + \omega \tag{1}$$

where v is the linear velocity at a radius r. It is noteworthy that ω refers to the angular velocity of rigid rotator, which does not generate any shearing movements, so that its contribution to the shear rate is zero, and the shearing movement can only be generated by $rd\omega/dr$. That is, for rotational motions, the shear rate γ does not have the same meaning as the gradient of velocity.

In terms of measurements by cone-plate, samples are filled into the gap between cone and plate that are horizontally installed, as shown in Fig. 2.

When cone rotates against plate with the angular velocity ω, the shear rate at a radius of r can be calculated as:

$$\dot{\gamma} = \omega/\theta \tag{2}$$

where R and θ are radius of the cone and the cone angle (the angle between cone and plate as it shown in Fig. 2), respectively. Different from coaxial cylinder, the shear rate of cone-plate during experiments is only dependent on the cone angle and rotational speed, which is not related to radius.

GBW13604 is a Newtonian reference liquid mainly consists of mineral oil, with relatively ideal viscous properties. The reference material used in this paper has chain structure of hydrocarbon, which is very similar to the polymer chain structure in polymer solution. Moreover, it is a homogeneous Newton fluid and shear viscosity does not vary with the shear rate. The purpose of using this material is to make the comparison between the experiment data more meaningful without the interference of non-Newtonian characteristics of polymer solution. The characterization was performed at 20 °C, and the comparison of data obtained from cone–plate geometry and concentric cylinder geometry are shown in Fig. 3.

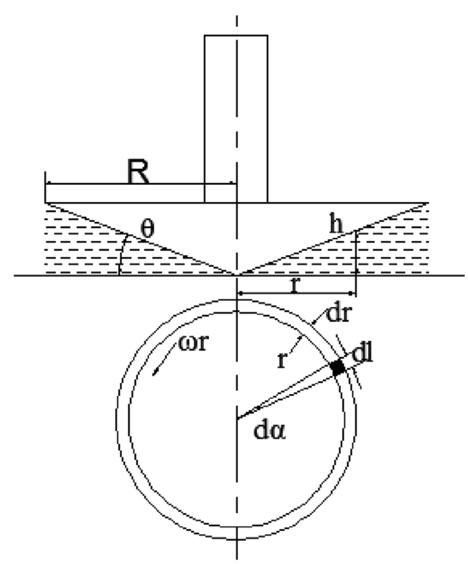

Fig. 2. Schematic diagram by cone-plate.

Due to the existence of system inertia of rotational rheometer, the total torque (M) consists of viscous torque (M_s), inertia torque (M_I) and residual torque (M_R). At relatively low shear rate, the major