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Measurement of rheology in reaction chamber by immersion type EMS system

EMSシステムによる反応容器内における粘弾性の連続測定

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1. Introduction

Viscosity and elasticity give us various information on the state of materials. Measurement of the rheological properties can be employed as a monitoring system over long-time industrial processes, such as aging, fermentation and synthetic. Electro magnetically spinning (EMS) visco-elasticity measurement system developed by us has an advantage that the rotational probe can be set in reaction chambers of the above processes, since the driving torque is remotely conducted to the rotor from outside of the chamber. The driver applies a rotating magnetic field to the space around the rotor made of electrically conductive materials. The interaction between the rotational magnetic field and the induced Lorentz current generates torque, which rotates the probe so that it follows the rotation of the magnetic field. The magnitude of the driving torque is proportional to the deference in the rotational speeds of the magnetic field and the rotor, and the viscosity is determined as the ratio between the difference and the rotational speed of the probe rotor giving the shear deformation rate.

In our previous work, we proposed an auto-standing type rotor, which keeps the rotation axis into the vertical direction with the aid of the gravity and buoyancy; the gravity applied to the pivot of the rotor can be reduced and the rotational friction becomes ignorable. It has been successfully employed for the in-line monitoring, however, some problems arise when it is applied as the immersion type in the long-time industrial process. One is that the pivot of the probe rotor, which determines the vertical position of the rotor is immersed in the sample liquid; precipitation and/or aggregation of the ingredients to the pivot might occur, which leads to the excess frictional force and unstable rotation of the probe.

2. Floating type Auto-standing rotor

To settle the above problem, we made a new type of the auto-standing rotor, which has a pivot above the probe. Figure 1 shows a schematic view of the rotor.

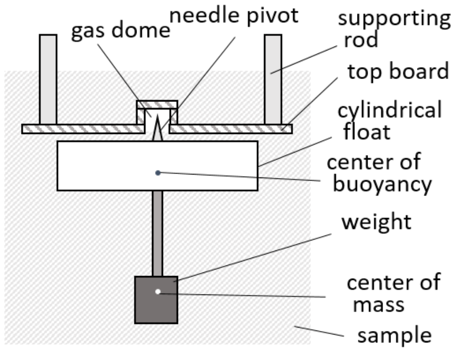

Fig.1 Schematic view of floating type auto-standing rotor employed in the immersion type EMS system.

The auto-standing type rotor is composed of two parts; one has low density and contributes to the buoyancy, which is located upward, and the other has high density located downward, which works as a weight. For the floating type, the summation of the gravity and the buoyancy applied to the whole probe is designed to be almost the same but slightly negative to float. Then the apparent load applied to the pivot of the rotor is decreased, which leads to the reduction of the mechanical friction against the probe rotation.

It is also important that the auto-standing rotor stably keeps its rotational axis even under fluctuation, which can be estimated by the restoring force against the incline of the rotor. For the conventional auto-standing cell with lower pivot, the buoyancy works as the restoring force, while the gravity causes instability. On the other hand for the floating type with upper pivot, the restoring force is given by the gravity and buoyancy causes instability. Therefore, to diminish the instability of attitude of the rotor, the distance between the upper pivot and the center of buoyancy should be satisfactorily small.

As shown in Fig. 1, the rotation probe is composed of a cylindrical float and a weight. A needle tip is set at the center top of the float, which fits in a small concave formed on the top plate, and determines the vertical position of the rotor. The contact point of the rotor pivot and the top board is in a small convex, which is filled with inert gas against the sample liquid.

The diameter and height of the cylindrical float is 40 mm and 10 mm, respectively. The weight is made of brass and positioned about 30 mm lower than the float. The distance between the pivot and the center of buoyancy is about 6 mm, while that between the pivot and mass center is 30mm, and the magnitude of the restoring torque is 5 times larger than that tends to incline the rotational axis

The gap between the top board and the upper surface of the disk float is much smaller than other distances between the rotor and walls of the sample container, therefore, major contribution of the viscous resistant force is brought about by the shear deformation of the sample liquid between the gap. In the present experiment, the top board and the upper surface of the float are flat and they compose plate-plate type rotational viscometer. Of course, the configuration of the cone-plate type rotational viscometer is available if either of the surface of the top board or the upper surface of the float is made to be conic. In this experiment, the gap between the upper and lower disk of the plate-plate type is set to be 1 mm.

Further, we have to pay attention when we newly design the rotational type viscometer, that is the part above the surface of the sample should be as small as possible so that the rotation of the part does not cause serious surface deformation. Actually, the top tip of the rotor is a needle with diameter of 1 mm and length of 4mm. The upper part of 2mm of the needle emerges above the sample surface.

3. Long time measurement of viscosity by immersion type EMS

Accuracy of the viscosity measurement by the floating type auto-standing probe was examined for lowly viscous liquids. Figure 2 shows the relation between the applied torque and the rotational speed of the probe, of which gradient is proportional to the viscosity. The samples are pure water and the aqueous solution of sucrose with the concentration

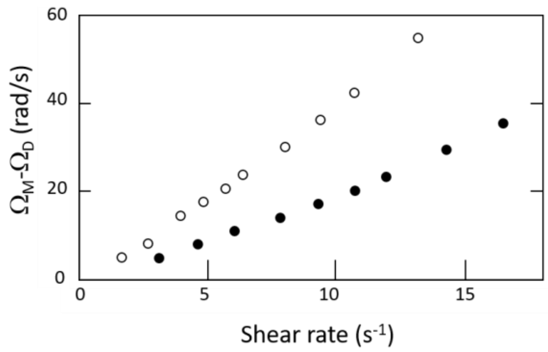

Fig.2 Relation between shear rate and applied torque obtained for pure water and 30 % aqueous solution of sucrose.

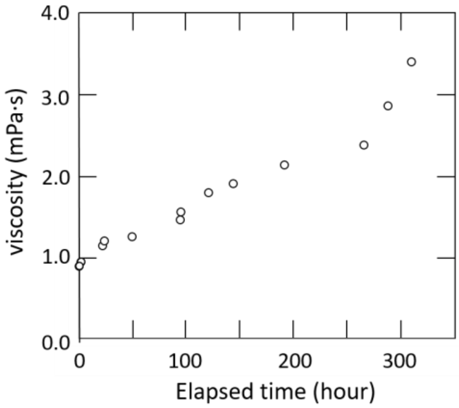

Fig.3 Viscosity change in dissolve process of crystal of sucrose into water.

of 30 wt%, of which viscosity is 2.35 mPa·s. As seen in the figure, we can distinguish the viscosity even for low viscosity region less than 10 mPa·s. The relation between the shear rate and the torque seems not to be straight especially for lowly viscous sample of pure water, which would be due to the effect of inertia, that can be normalized in terms of Reynold's number. The detailed analysis would be given in the presentation.

We then applied the system for the continuous measurement of viscosity. The sample prepared was rock sugar, which is commercially available crystal sucrose. Amount of 250 g was immersed in the water of 0.25 L. Sucrose is gradually solved into water and the viscosity of the solution changes in time region of weeks. Figure 3 shows the change of the viscosity of solution measured by the immersion type EMS viscometer. As seen, the viscosity increases as time elapses during over a week. The kink seen at about 250 hours would be caused by slight stirring of the sample, which homogenized the distribution of the local concentration in the sample container. The volume of rocks of sugar seems to decreases to almost half of the initial by visual observation, and the detailed results of the concentration change and its rheological analysis would be given in the presentation.

References

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