

Theme	“Food products evaluation technology using viscosity”
Secondary title	(Measurement of the physical properties of thickening agents and powdered food products in relation to the evaluation of dysphagia)
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Keywords	Thickening, gelling, tuning fork vibro viscometer, static viscosity, viscosity of powders

1) Introduction

The word viscosity is not used very often day to day, however you will often hear words which express the viscous characteristics of liquids, such as watery or sticky. The history of measurement of viscosity, which expresses these characteristics of liquids, is long and stretches back to the Industrial Revolution. Oil was used to reduce friction in the axle bearings when the force of steam engines was converted to rotary movement; documents show that a need arose at that time to evaluate the viscosity of that oil.*¹ However, this use didn't become commonplace until the culmination of the Industrial Revolution with the mass production of the automobile. At this point, precise control of engine oil flow in a combustion engine, where it is of absolute necessity, became widespread as an essential part of the manufacturing process.

At first, viscosity of fluids was measured by the proportionate time taken to pass through the small opening at the bottom of a funnel. This method survives today with such devices as capillary type viscometers, with the time taken to pass through an orifice of set circumference being proportional to [viscosity ÷ density], allowing viscosity to be calculated. This measurement method was the first established as a standard to measure viscosity, together with water as a standard substance for measurement, due to its universal viscosity characteristics.

Following this development, further methods for measuring viscosity were established, such as the rotational method of immersing a rotor in a liquid to measure the torque required to rotate the rotor, with this torque being proportional to viscosity; or the falling ball method of dropping a metallic ball into a liquid with the time taken for the ball to drop also being

proportional to viscosity. These methods each had their own drawbacks and advantages, however in general all of them used large-scale devices which required significant amounts of liquid to be properly used. The range of viscosities which could be measured by these devices was quite small and they couldn't provide stable measurement results for liquids with a low viscosity such as water, a liquid used as a viscosity standard. They all also had the added problem of difficulties converting their values to digital signals.

In recent years, with the development of electronic parts using semiconductors, measurement and control technology has progressed rapidly. As a result, a new type of viscosity measurement was proposed about 50 years ago which differed from the three methods described above: the vibro viscometer, which was commercialized relatively recently.*² In the initial stages of selling vibro viscometers, as there had been no change in the field of viscosity measurement for many years there was a rejection of the proposal of a new method. However in recent years, as a result of the special features of vibro type viscometers earning a strong reputation, they have now joined the ranks of officially recognized standards in viscosity measurement, with their acceptance in the JIS Viscosity Standards Revision of 2011.*³ Further, prior to that acknowledgement, they were also designated as a JCSS-recognized device.*⁴

Viscosity measurement has grown in scope significantly in recent years beyond its traditional realm of heavy industry, where it is used to measure oil, petroleum, etc. It is now being put to use in many fields, including in the development of new materials such as nanocolloids, in ink for printing, adhesives or food products. Moreover, as we face the increasing problem of an aging population, tuning fork vibro viscometers are starting to be used in new applications relating to health, such as in the measurement of important indexes like "thickening" or "gelling temperature" which are vital in countermeasures to avoid the accidental ingestion of food or drink.*⁵ One reason the tuning fork vibro viscometer is chosen for research such as this is there was previously no such high-sensitivity viscosity measurement device with excellent repeatability available which could measure so simply and clearly the subtle differences in the sensations of

liquids that humans can distinguish. Another reason given is that alternative measurement devices can be extremely expensive.

This paper summarizes the results of tests measuring the viscosity of characteristic liquids and powders used in thickening and food products, etc., using a tuning fork vibro viscometer and tuning fork vibro rheometer with a low frequency drive of 30Hz. Using this method, it is now possible to evaluate the basic viscous properties of materials which could not be measured easily until now without causing damage to the material.

2) Principles of measurement for the tuning fork vibro rheometer

The difference between a tuning fork vibro viscometer and tuning fork vibro rheometer is whether the amplitude of the oscillators (which act as the sensor unit) is set at a fixed value or can be changed. Here the principles of the tuning fork vibro viscometer, which forms the basis of the sensing method, are explained.

○ Theoretical development of tuning fork vibro type

A tuning fork vibro viscometer, similarly to an actual tuning fork, has two oscillators, or sensor plates, that resonate in the direction parallel to their faces. The vibration energy needed to maintain the set amplitude of the two oscillators is generated by electromagnetic power. In other words, a drive force equivalent to the viscosity resistance of the liquid is exerted and as this drive force is proportional to viscosity resistance, the viscosity value of the liquid can be known. For vibro-type viscometers, in principle viscosity resistance is equal to [viscosity \times density]. We call this viscosity \times density value [static viscosity]*⁶ to differentiate it from the kinetic viscosity value given by capillary type viscometers and the dynamic viscosity value given by rotational types as well.

With tuning fork vibro method, by calculating the drive force necessary to resonate the oscillators of the tuning fork and the motion equation relating to the inertia term, viscosity term and elasticity term, it can be understood that the energy that drives the tuning fork oscillators is proportional to viscosity \times density.

Regarding the details of a theoretical model for a tuning fork vibro viscometer, please refer to a separate paper.*² This paper only explains the conclusions of related study. For the setup in Fig. 1, when the oscillators vibrate with a frequency of f , the mechanical impedance (R_z) received by the oscillators from the liquid can be calculated as:

$$R_z = A\sqrt{\pi f\eta\rho}$$

with each of the terms on the right side of the equation representing: f : vibration frequency (Hz); A : surface area of both sides of the oscillating sensor plates; η : viscosity of the liquid; ρ : density of the liquid. Here, if we make the force exerted on the oscillators by the electromagnetic drive unit F , and its resulting constant vibration velocity $Ve^{i\omega t}$, the equation can also be expressed as:

$$R_z = \frac{F}{Ve^{i\omega t}} = A\sqrt{\pi f\eta\rho}$$

From the equation above it can be understood that the force from the electromagnetic drive unit is proportional to static viscosity (the product of viscosity η and density ρ). In actual measurement, the proportionality of static viscosity [viscosity \times density] to electrical current is used to measure viscosity, as current is adjusted to a level necessary to control the torque generated for the electromagnetic drive unit to maintain a constant amplitude for the oscillators in the sample liquid.

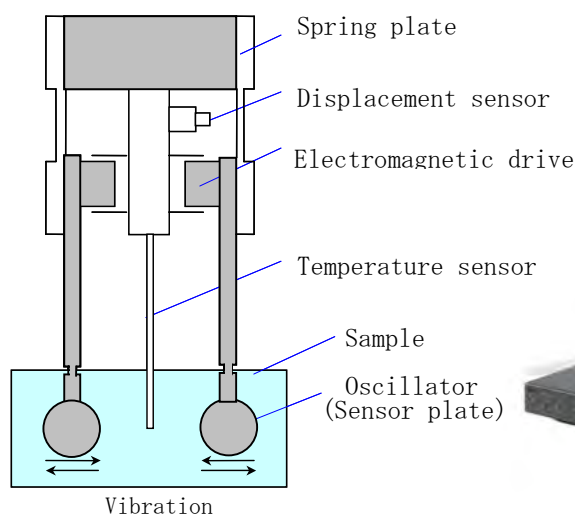


Fig. 1 Sensor unit configuration



Fig. 2 Photo of entire system

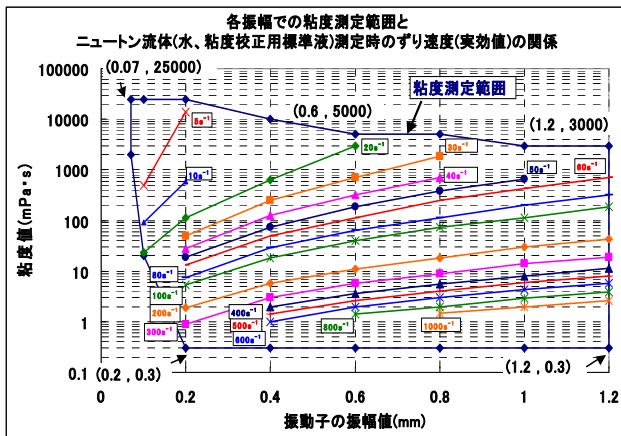


Fig. 3 Measurement range of viscosity

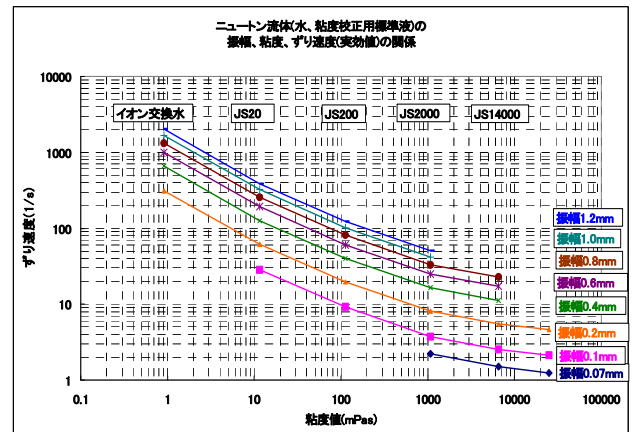


Fig. 4 Viscosity standards and shear rate distribution

○ Characteristics of tuning fork vibro method

The configuration of a tuning fork vibro viscometer's sensor unit is detailed in Fig.1. This method of using such a displacement sensor and controlling the acceleration force to keep that sensor's output (displacement) at a constant level using electromagnetic force has the same essential framework as the electromagnetic mass detection unit used in an analytical balance. Also, the spring plate which provides the natural vibration frequency also shares common elements with electromagnetic force balances. With this technology, the resolution of the tuning fork vibro viscometer calculated by dividing the maximum measurable viscosity of 10000mPa.s by the minimum sensitivity of 0.01mPa.s is as high as 1/1,000,000, equivalent to that of an analytical balance.

Fig.2 is a photograph of the full framework of a tuning fork vibro rheometer. From the left, the viscosity detection unit, control unit and special controller are displayed.

The graphs displayed in Fig. 3 and 4 are of data taken from the tuning fork vibro rheometer and show the distribution of shear rate. With the vibro method, only the oscillators are moving in the liquid and there is no opposing surface which could be used to determine the shear rate. On the other hand, even under the same measurement conditions, the distance at which shear occurs essentially differs depending on the viscosity value of each different liquid. Accordingly, even under the same acceleration

conditions or with the same rotary movement, the distance for shear rate differs depending on the viscosity of each liquid. Because of these reasons, the shear rate for the tuning fork vibro viscometer is calculated from the known viscosity value of a viscosity standard and the shear stress value, which is found by dividing the torque (force) needed to move the oscillators in the viscosity standard by the total wetted surface of the oscillators. Incidentally, viscosity could be defined by the following equation: [shear stress] = [viscosity] × [shear rate] and hence viscosity is a coefficient of shear stress divided by shear rate.*7

Regarding shear rate of the tuning fork vibro method, as the oscillators move in a back and forth motion, there is no constant value in relation to time. Therefore, shear rate is defined by its effective (root-mean-square) value.

In addition to the technical details of the tuning fork vibro viscometer described above, the fact that the resonance frequency is as low as 30Hz and the basic amplitude of the oscillators is as small as 0.4mm provides the reasons why high precision viscosity measurement is now possible with the minimum of changes to the structure of the object being measured.

3) Examples of measurement with tuning fork vibro rheometer

Results of measurements of various liquids using a tuning fork vibro rheometer or viscometer are detailed below. The y-axes of the graphs below, which should express static viscosity [viscosity × density], hypothetically take the density (specific gravity) of the liquid being measured as 1, so as to represent viscosity.

3-1) Measurement of Newtonian fluids

Fig. 5 & 6 show measurement results while changing the vibrational amplitude of the oscillators for water, a representative Newtonian fluid, and the viscosity standard JS2000.

It is clearly shown in the graphs that both water and JS2000 exhibit the Newtonian fluid characteristic of not changing viscosity in response to a change in vibrational amplitude.

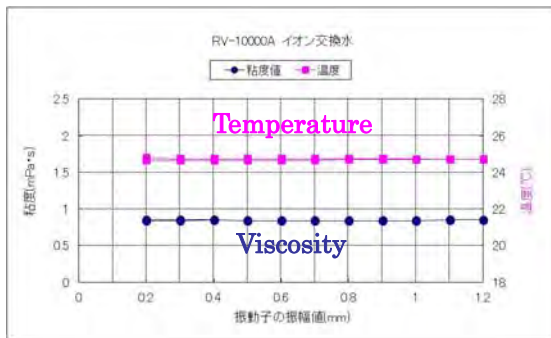


Fig.5 Ion-exchanged water

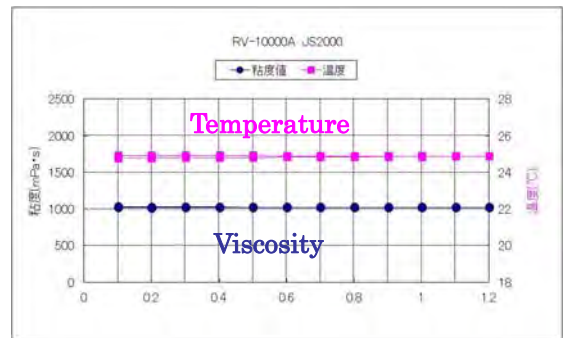


Fig.6 JS2000

3-2) Measurement of thickeners

Fig. 7, 8 & 9 show the results of measurements of typical thickener xanthane gum at different concentrations. The results show significant non-linearity of viscosity according to concentration and data was obtained confirming the non-Newtonian characteristics of these thickener fluids in relation to vibrational amplitude.

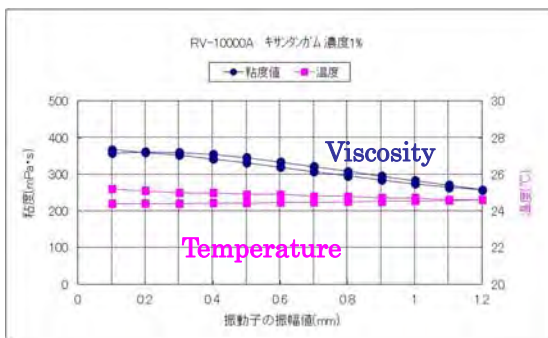


Fig.7 Xanthane gum 1%

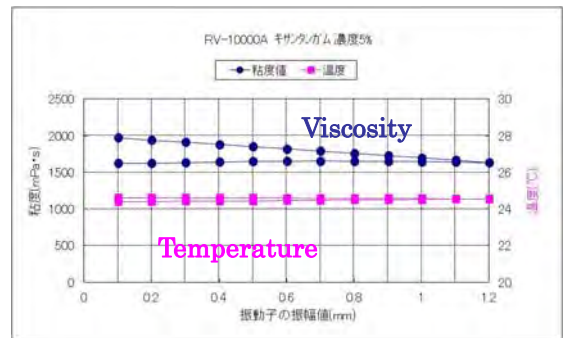


Fig.8 Xanthane gum 5%

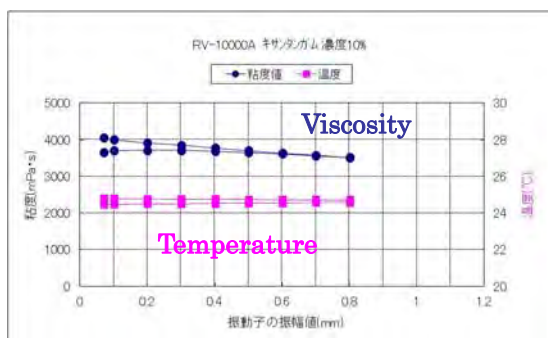


Fig.9 Xanthane gum 10%

3-3) Measurement of powders

The jury is out on whether the viscous resistance that occurs in powders should be called viscosity. However, using the tuning fork vibro method, it is now possible to measure, with good repeatability, the viscous resistance of powders which was previously difficult to quantify using other methods. Fig. 10 to 14 show measurement results for flour (both high gluten content and low gluten content) and starch in their original powder form, as well as low gluten flour in solutions with water.

For the viscous resistance of powders, the powder's "bulk density" is an influencing factor. Hence mass measurement was performed on the powders in a fixed measurement vessel after letting the powder freely fall into the vessel and then completely leveling them. They were then compacted until they reached a defined volume by applying a constant pressure by vibration. The bulk density value at that time was sought and viscosity measurement was then conducted.

When shear rate increased, the static viscosity of the powders suddenly decreased. The hysteresis relative to shear rate is almost uniform when increasing and when decreasing. However, only in the case of starch was the hysteresis large. Further, when the ratio of water mixed with the low gluten flour increased, hysteresis was confirmed relative to shear rate with a sudden drop in static viscosity simultaneously. A drop in static viscosity was also seen at a middling shear rate.

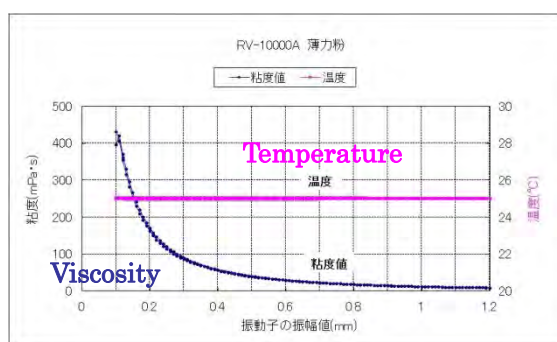


Fig.10 Low gluten flour
(bulk density 0.49g/cm³)

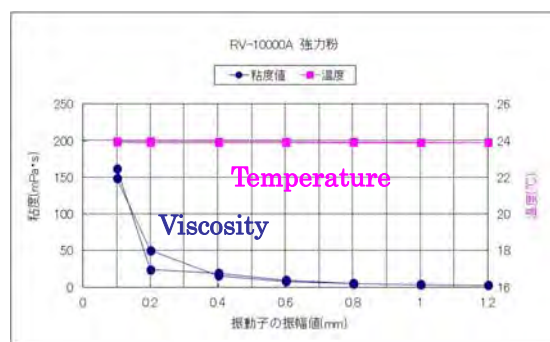


Fig.11 High gluten flour
(bulk density 0.56g/cm³)

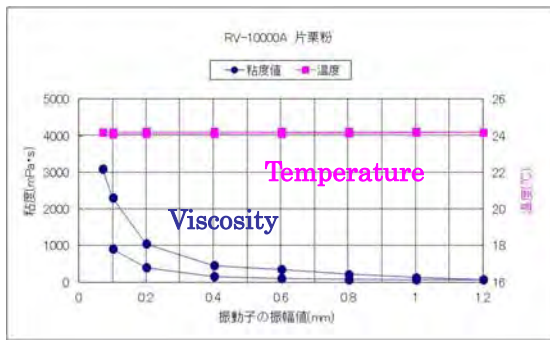


Fig.12 Starch
(bulk density 0.70g/cm³)

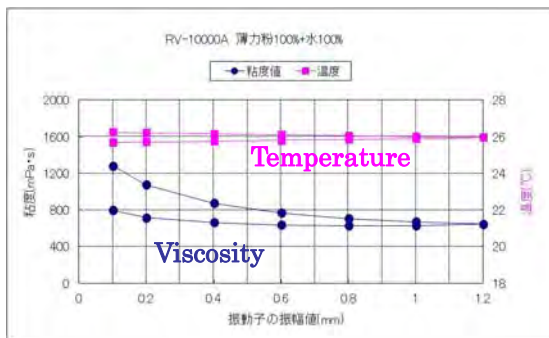


Fig.13 Low gluten flour 100% + water 100%

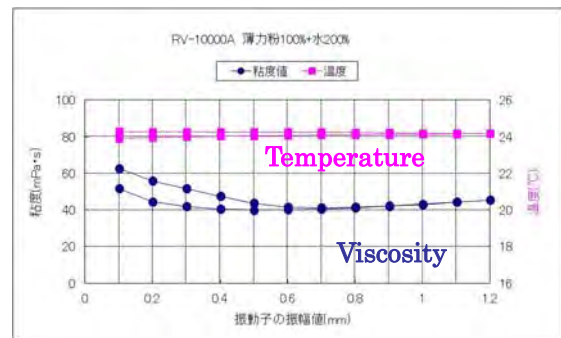


Fig.14 Low gluten flour 100% + water 200%

3-4) Other viscosity measurements

In addition to thickeners and powders, measurement results for food items with special characteristics, etc. are summarized below.

○ Measurement of gel point

Fig.15 graphs the change in viscosity for gelatin in relation to a change in temperature. With the vertical axis forming a logarithmic scale, it was confirmed that gel point differs according to density.

○ Measurement of the curing process of egg whites

Fig.16 shows the curing process of egg whites. Egg white consists of several different proteins which will change their structure at temperatures from 60°C or 70°C. This was confirmed by changes in their viscosity.

○ Measurement of the clouding point of non-ionic surfactant

Fig.17 shows the results of the measurement of clouding points. The clouding point, which occurs due to structural changes in micelles, is detected optically and measured by the temperature at that time. With the tuning fork vibro viscometer, it is also possible to capture this clouding point as a change in viscosity. For this measurement sample, the JIS standard mentioned above prescribes the clouding point of 35.9°C, and from a change in viscosity a clouding point of 35.4°C was confirmed.

○ Measurement example for oil

As a typical example, the temperature-viscosity curve for oil for a gasoline engine is presented in Fig. 18. It is said that if the viscosity of engine oil falls below 3mPa.s, burning occurs between the pistons and cylinders. It can be conjectured from the graph that the maximum temperature the oil can be used at is 110°C.

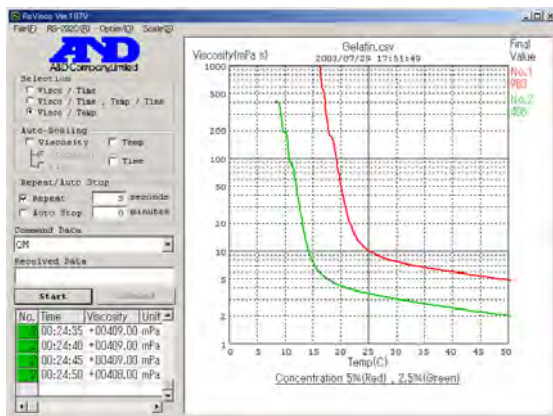


Fig.15 Gelatin

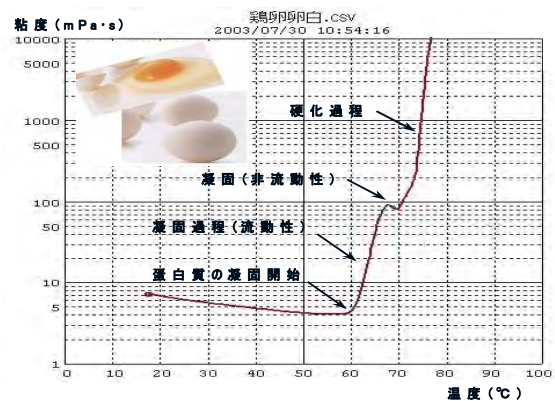


Fig.16 Egg white

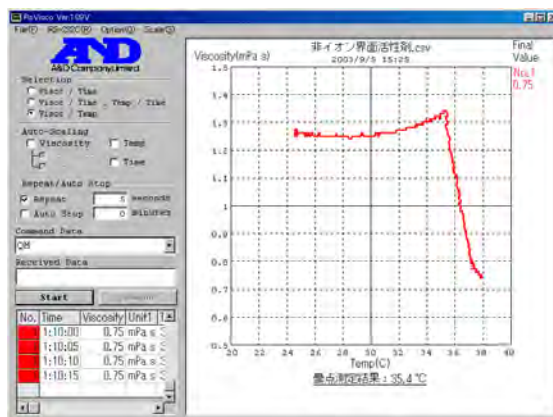


Fig.17 Non-ionic surfactant

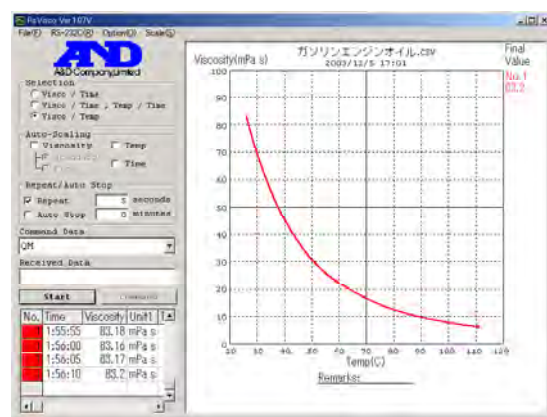


Fig.18 Oil for gasoline engine

4) Conclusion and considerations

- Evaluation results for thickeners

For recent thickening agents, a strong thickening effect is being demanded for a small amount of additive. This could be considered an appropriate development from an economic perspective, and in fact, when there was a change in the amount of thickener, the change in viscosity value was confirmed to be more than a one digit shift per 9% change in the proportion of thickener. This points to the extreme importance of volume management of thickeners when added to food products in the course of nursing care. Additionally, it has also been understood that for thickeners, the change in viscosity in relation to change in shear rate is small, as is the change in viscosity in relation to change in the speed the food product is drunk.

These special characteristics of thickeners can be considered a valuable capability at the place of care. Specifically speaking, the capability of the viscous resistance linearly changing relative to a change in shear rate suitably matches a person's actual physical sensations of viscosity. Accordingly, it can be surmised that for thickeners for the prevention of accidental ingestion or aspiration, not only is the capability to increase viscosity essential, but so too is having non-Newtonian characteristics as small as possible.

- Viscosity evaluation results for powdered food products

We presently cannot say that there is an established evaluation method for the special viscous characteristics of powders which are used as food products. That is, existing evaluation methods of the physical properties of powders are not represented as SI units (International System of Units). We could say this is clear from the fact that the data taken from testing machines for powders express the measurements in units particular to that testing machine.

In our measurements using the RV-10000A Tuning Fork Vibro Rheometer, the viscous resistance values for various powders for use as food products were obtained. The instrument was calibrated before the measurements were conducted by the viscosity values of Newtonian fluids which can act as viscosity standards. Because of this, these measurements can perhaps be

thought of as the first measurement and evaluation results of the viscosity of powders using an SI unit.

The viscous resistance figures taken from vibro viscometers are expressed by [viscosity \times density] in principle. Supposing that the viscous resistance of powders was expressed by viscosity and density (bulk density), this experiment can be judged to deliver precise measurement of the viscosity of powders by first seeking their bulk density.

- Shear rate

Nearly all naturally-occurring liquids are Newtonian fluids with a constant viscosity value in relation to shear rate. This means that the viscous resistance proportional to shear rate is obtainable. However, approximately 80% of the liquids which are synthesized are said to be non-Newtonian fluids, in which the viscous resistance is not proportional to shear rate. For these non-Newtonian fluids, as the amount of shear rate applied to the liquid differs depending on the viscosity measurement method, discrepancies in measurement results occur according to the measurement method used.

While the following is merely personal opinion, there are several points that should be given consideration with regards to shear rate. First of all, an assumption of the logic stated above is that when the internal and external walls of a container filled with a liquid is set into relative motion, a uniform shear rate is applied to the liquid enclosed by the internal and external walls, produced by the difference in rotation with the internal and external walls. However, for non-Newtonian fluids, it is not proven that the difference in rotation in a structure with internal and external walls, or top and bottom surfaces, exerts a uniform shear rate on the liquid which has been poured inside.

In other words, the shear rate applied to non-Newtonian fluids adheres to the principles of uniform shear rate applied to Newtonian fluids, and if it was the case that a uniform shear rate was not applied to a non-Newtonian fluid, this would not hold true.

The “shear rate” determined with existing rotational viscometers or

rheometers is determined from the above assumption of uniform shear rate, and the geometrical shape and relative motion between rotating bodies in relation to Newtonian fluids. However, with non-Newtonian fluids, the fact that the propagation range of the “shear rate” differs from Newtonian fluids under the same conditions can be thought of as inviting non-linearity in viscosity values in relation to change in “shear rate”. This makes it difficult not to doubt the adequacy of current viscosity assessment methods where a constant shear rate is assumed based on a constant rotation.

This can also be deduced from the fact that the validity of the phenomenon of a large change in the viscosity value of the material from only a slight difference in shear rate is not proven as a change in the physical properties of the material.

In the present experiment with thickeners, the changes in viscosity due to change in shear rate were comparatively small, which leads us to infer that until the shear rate becomes significantly high, a change in the bonding force from molecular chains or crosslinks will not ensue. From these inferences, it could be considered that the non-linearity of non-Newtonian fluids is caused by differences in the speed of the equipment, which generates shear rates. Such differences will result in changes in the reach of shear force and the distribution of shear rate, causing apparent changes in viscosity values.

With regards to these problems, the developer and supplier of viscosity evaluation devices should have a duty to users to provide a lot more information. Manufacturers, including A&D, need to increase their efforts to provide data and evidence to support such information.

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