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Cup viscometer – A practical analytical tool

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Abstract: This paper focuses on helping smaller companies in the manufacturing industry who need to know the viscosity of their products or raw materials and do not have the funds to invest in expensive rotary rheometers. We developed a calibrated simple cup. It has replaceable nozzles designed so that dripping liquids cannot distort discharge times. The calibration of the cup is based on the relationship between kinematic viscosity and outflow time, which is valid for the laminar flow regimes. Here we described calibration and measurement procedures and methods for detecting non-Newtonian flow behaviour and the viscoelasticity of measured liquids. The maximum measurement error of 3% is defined for cases where all distractions are removed.

Keywords: kinematic viscosity; non-Newtonian behaviour test; outflow time

In the course of many years, inventions corresponding to the principle of the outlet cup have appeared. For example, patent Xie (2019) CN109357970 was presented. Patent Zhang et al. (2023) CN115728183 has fully automated the outflow viscometer function. Utility model Zhang et al. (2022) CN217033494 will provide us with an almost perfect tempering of the liquid, the viscosity of which we are trying to determine with the outlet cup. The invention given by Zhang et al. (2020) CN111595729 is interesting because it uses the weight of the flowing liquid for calibration, thereby eliminating the occurrence of unwanted bubbles and inaccurate observation of the volume of the flowing liquid. Other patents, e.g. Motoyama (2009) JP2009229338, Yasuda (2013) JP2013088166, and Xu et al. (2022) CN215414884 are focused on improving the accuracy of viscosity measurement related to the lowering and closing of the vent opening or it is about improving the shape of the container to enable the measurement of liquids that are difficult to measure.

We have developed a calibrated simple cup for operational purposes, where viscosity values are quickly needed. Although our paper is based on a publication from 1986, there are still simple devices, such as cups, that do not respect the rules for achieving correct viscosity values. Additionally, they do not have replaceable nozzles and are not designed so dripping liquids cannot distort discharge times. According to the ASTM D4212-16 (Standard Test Method for Viscosity by Dip-Type Viscosity Cups) standard, the exception is the Zahn cup, which has exchangeable outlet nozzles with different nozzle diameters.

The work of Šesták and Houška (1980) deals with the issue of unstable discharge, i.e. the dripping of vis-

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cous liquids from the nozzle orifice. This work applied existing theories for glass and revealed that this phenomenon is not caused by nozzle clogging but by the effect of surface tension at low flow rates. In this work, the condition for maintaining constant flow for an outflow velocity from a given diameter of a circular opening is:

$$\overline{u} > 0.42\left(D\,\rho\right)^{-0.4}\,\mu^{-0.21}\,\sigma^{0.6} \quad (1)$$

where: $\overline{u}$ – volumetric mean velocity of the liquid stream ($m \cdot s^{-1}$); D – nozzle inner orifice diameter (m); ρ – density ($kg \cdot m^{-3}$); μ – dynamic viscosity (Pa·s); σ – surface tension ($N \cdot m^{-1}$).

Theoretical relationships between discharge times and the kinematic viscosity of a liquid are known from the literature (Meskat 1957) and (Walters and Barnes 1980). According to these authors, the following relationship applies to the kinematic viscosity.

$$\nu = \frac{\mu}{\rho} \quad (2)$$

where: ν – kinematic viscosity ($m^2 \cdot s^{-1}$).

The relationship between kinematic viscosity and outflow time is valid for the laminar flow regime:

$$\nu = a\,t_\nu - \frac{b}{t_\nu} \quad (3)$$

where: a – empirical constant from Equation 2 ($m^2 \cdot s^{-2}$); t_ν – outflow time (s); b – empirical constant from Equation 2 (m^2).

This paper aimed to design a suitable shape for a cup viscometer, its calibration, and the prediction of unknown constants in Equation 3. Another goal of this paper is to measure and evaluate the data to eliminate possible errors, including dripping, to predict viscosity from discharge times.

This paper focuses on helping smaller companies in the manufacturing industry who need to know the viscosity of their products or raw materials and do not have the funds to purchase expensive rotary or capillary viscometers. This paper is based on a publication by Houška (1986) published in the Czech version of the journal 'Potravinářské vědy.' This journal is known as the Czech Journal of Food Sciences and publishes papers only in English.

MATERIAL AND METHODS

Cup viscometer design. The cup viscometer was designed based on our experience using similar standardised instruments. To eliminate dripping and extend the range of measured viscosities, we equipped our device with three replaceable nozzles with internal diameters of 2, 3, and 4 mm. The nozzles are closed with a conical plug on a cylindrical rod, which can be locked with a screw after lifting. The volume of flowing liquid was limited to 100 cm^3 and filled the upper cylindrical part of the cup. The dimensions are detailed in Figure 1. The device has two circular spirit levels, ensuring it is horizontal before the measurement starts. Leaking liquid was collected in a measuring container with a volume of exactly 100 cm^3. The outflow time of this quantity was measured with a stopwatch to an accuracy of 0.1 s.

Measurement procedure. After cleaning the cup, the device was placed in a horizontal state. The height between the base plate of the device and the upper edge of the cup was set at approximately 300 mm. After inserting a nozzle of the appropriate inner diameter (estimation of the size of the nozzle opening according to the estimation of the viscosity of the measured liquid) the inlet to the nozzle was sealed with a conical plug. Then, the measured liquid was poured into the cup. After equalising the temperature of the liquid inside the cup (measured using a digital thermometer with an accuracy of 0.1 °C), a cylindrical measuring vessel with a volume of 100 mL was placed under the nozzle. A stopwatch was then simultaneously started, and the nozzle cap was lifted and secured in the upper position with a screw. After an outflow of 100 mL, the stopwatch was stopped.

After that, the liquid was returned from the measuring cylinder back into the cup and, if necessary, topped up to the original level, i.e. to the lower edge of the reinforced upper part of the cup. We again measured the temperature of the liquid in the cup and repeated the measurement of the outflow time. We repeated measurements at least ten times without cleaning the cup between measurements. From these ten measurements, we excluded some measurements so that the maximum difference between the temperatures was 0.3 °C. Simultaneous with the measurement of the discharge times, we pycnometrically determined the density of the measured liquid for the average temperature of the measurement.

Instrument calibration. The measurement procedure described above was also used during the cali-

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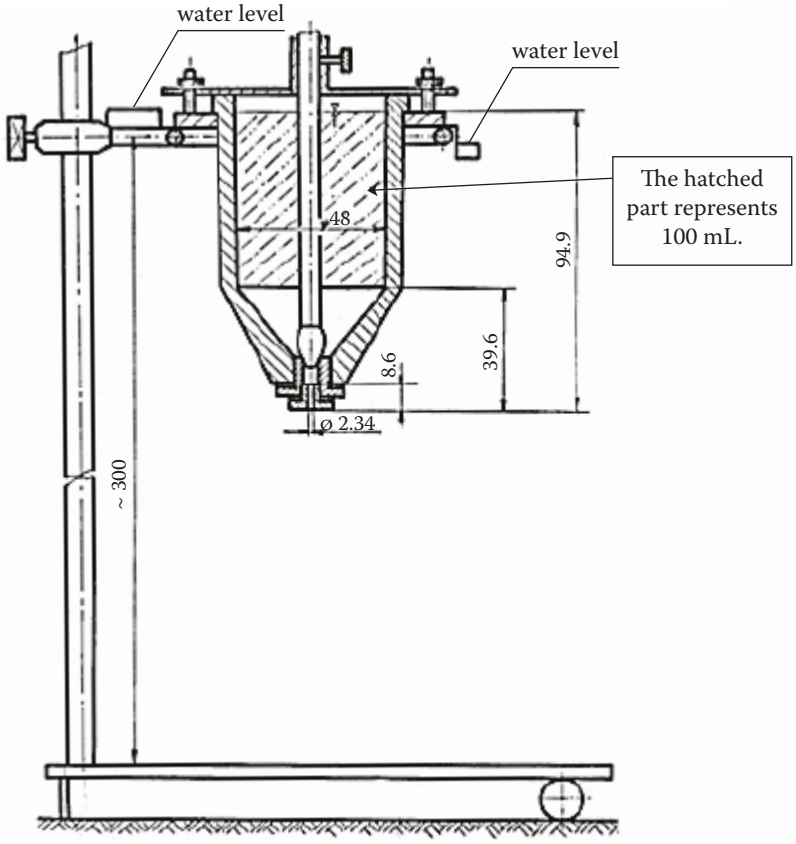

Figure 1. Basic cup dimensions

bration of the cup viscometer. The measurement was repeated with model liquids of known viscosity and density for the selected inner diameter of the nozzle so that the calibration liquid did not drip. A total of 11 model liquids with a viscosity from 1 mPa·s to 2.3 Pa·s were used for calibration. Their dynamic viscosity was exactly measured using a rotary rheometer (Haake RS-150; Gebrüder HAAKE GmbH, Germany) three times and mean values were used for cup calibration. The standard error (SE) of viscosity was around 3% of the mean value. The density of calibration liquids was measured pycnometrically in three replications, and arithmetic mean values were used. SE of density was around 2% of the mean value. These were mainly solutions of molasses in water, glycerin, mineral oil, and distilled water. The total number of calibration measurements was approximately 400 for the three nozzles. From the average flow times t_v corresponding to the measured viscosity and density at the measurement temperature, we determined the dependences between flow time and kinematic viscosity in the form of Equation 3, Meskat (1957), or Walters and Barnes (1980).

The calibration constants a and b are given in Table 1 for all tested nozzles. These constants were determined by the non-linear regression method of least squares using the strategy of Monte-Carlo (applied in Excel, Microsoft Office, version 365) and DataFit (version 6.1.10). A comparison of regression relation-

Table 1. Values of the calibration constants for Equation 3 and validity ranges

Nozzle diameter (mm)	$10^6 \times a$ ($m^2 \cdot s^{-2}$)	$10^6 \times b$ (m^2)	Validity ranges	
			t_v (s)	$10^6 \times \nu$ ($m^2 \cdot s^{-1}$)
2	0.248	301.2	36.9–500	1–124
3	1.092	244.7	15.4–550	1–600
4	2.641	172.4	8.2–600	1–1 600

a – empirical constant from Equation 3 ($m^2 \cdot s^{-2}$); b – empirical constant from Equation 3 (m^2); t_v – outflow time (s); ν – kinematic viscosity ($m^2 \cdot s^{-1}$)

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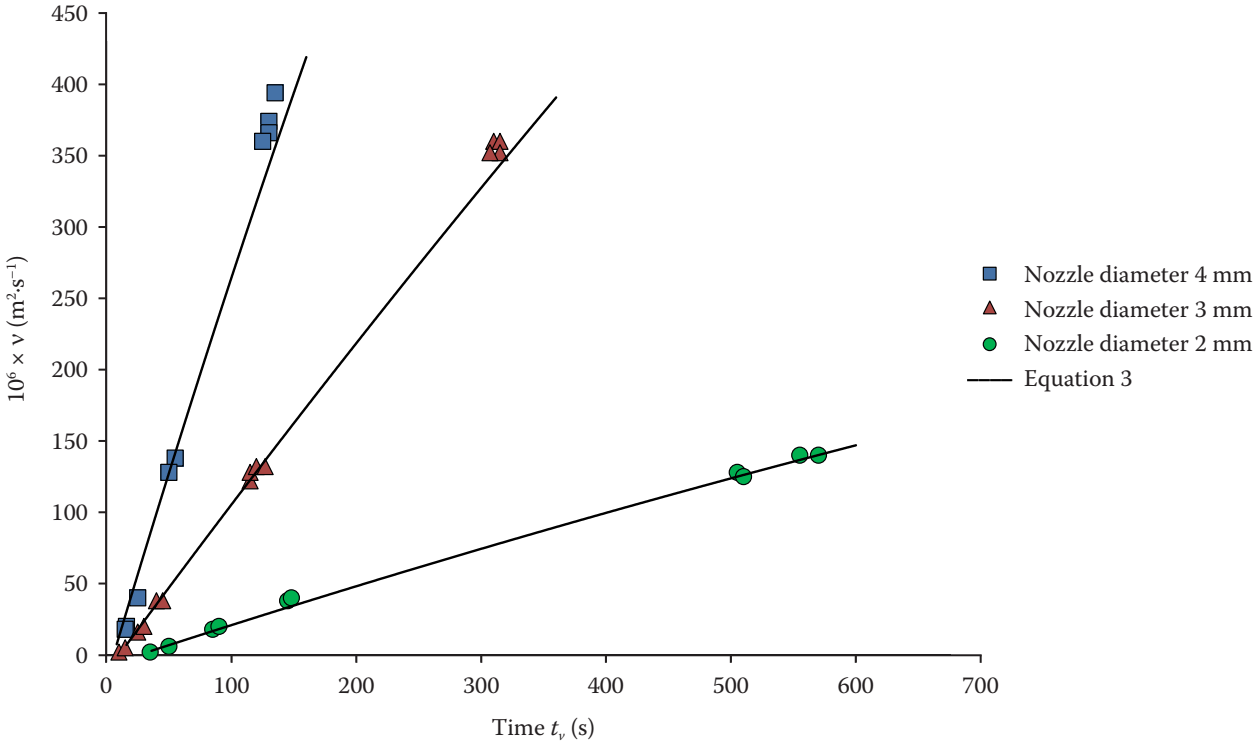

Figure 2. Cup calibration data for individual nozzles

t_ν – outflow time (s); ν – kinematic viscosity ($m^2 \cdot s^{-1}$)

ships with the measured data can be seen in Figure 2. The calibration of all nozzles was verified with three liquids of known pre-measured kinematic viscosity. The deviation of the prediction using t_ν and Equation 3 and the measured value by the rotary rheometer and pycnometer was around 3%.

RESULTS AND DISCUSSION

As shown in Figure 2, Equation 3 describes the measured data very well. The relationship to outflow time is not a linear function for low viscosities. This means that the often-performed procedure, where water is used to determine 'basic' outflow times and the ratio of the measured liquid's outflow time to the water's outflow time, is unsuitable. Therefore, we criticise using Engler grades (Engler viscometer, British standard BS2000), leading to underestimating changes, especially for lower viscosities. From the point of view of hydrodynamic forces, liquids flow out under the increased influence of inertial forces (transient flow regime), so flow time is no longer directly proportional to viscosity.

It must be emphasised that the characteristic dependences obtained in our paper apply strictly to the cup used, the indicated filling height, and the indicated amount of leaked liquid. These amounts ensure that the entire contents of the cup do not flow out and the measured time is not distorted by subjective evaluations of flow interruption, which can occur with other standardised devices. Even during calibration, we encountered disruptive influences, such as the transition from continuous to dripping flow, as mentioned in the introduction. Therefore, we had to cancel measurements and choose a larger nozzle diameter for the given liquid, which ensured a greater flow speed and fulfilment of condition (1).

Furthermore, it must be emphasised that the calibration dependences apply to determining the viscosity of Newtonian liquids, i.e. liquids for which the relationship between shear stress and shear rate (Equation 4) is valid.

$$\tau = \mu \dot{\gamma} \tag{4}$$

where: τ – shear stress (Pa); μ – dynamic viscosity (Pa·s); $\dot{\gamma}$ – shear rate (s^{-1}).

In our practice, however, we often encounter non-Newtonian liquids, and it is necessary to check wheth-

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er the given sample exhibits this type of rheological behaviour. The simplest rheological model for time-independent, non-elastic, non-Newtonian fluids is the well-known power-law model:

$$\tau = K\,\dot{\gamma}^{n} \tag{5}$$

where: K – consistency coefficient from power law flow model (Pa·s^n).

This model predicts that with increasing shear rate, there is a decrease in the apparent viscosity in case that $n < 1$ and an increase in the apparent viscosity in case that $n > 1$, as can be seen from the modified Equation 5.

$$\mu_a = K\,\dot{\gamma}^{n-1} \tag{6}$$

where: μ_a – apparent dynamic viscosity (Pa·s).

Our device can be used to indicate whether samples are non-Newtonian. The principle is to use multiple nozzles for the same liquid, which achieves a change in the shear rate since the liquid flows out at different speeds, and there is also a change in the shear rate in the nozzle. Then, by changing the inner diameter of the nozzle, there will be a significant change in viscosity (the difference in viscosities must be greater than the maximum measurement error, which is about 3% for the device we are presenting).

Walters and Barnes (1980) presented an empirical relationship for estimating the average shear velocity in a nozzle of radius r during the discharge of a liquid of volume V for the discharge time t_v.

$$\bar{\dot{\gamma}} = \frac{2V}{t_v r^3 \pi} \tag{7}$$

The relation is valid for inelastic non-Newtonian fluids. The measured viscosity for different nozzle inner radii and different average shear rates can be considered as the apparent viscosity μ_a.

When the non-Newtonian behaviour of the measured liquid is indicated by this method, it is recommended to use a much more accurate rotational rheometry for these substances.

Walters and Barnes (1980) proposed that significant changes in the height of the cup above the calibrated cylinder level indicate the measured liquid's viscoelasticity. Suppose the outflow times t_v differ for different cup heights above the surface. In that case, the measured liquid exhibits viscoelastic behaviour, and the cup cannot be used for viscosity measurements.

However, there are other limitations to the use of the cup. Above all, it is not possible to measure fast-sedimenting suspensions with particles whose size is comparable to the diameter of the nozzle (which creates a danger of clogging), and measurements at temperatures significantly different from the ambient temperature are not recommended since the described cup cannot be tempered.

If the measured liquids are Newtonian and we prevent all the interferences mentioned above, a maximum measurement error of ± 3% can be expected.

CONCLUSION

The described cup is a straightforward device. It can practically measure Newtonian fluid viscosity accurately if we follow the limiting conditions and avoid the errors associated with dripping flow, non-Newtonian behaviour, and viscoelasticity. The apparatus we have described can detect these deviations from Newtonian behaviour. The cup described can be used for comparative measurements of the viscosity of a liquid of the same composition to determine whether it has properties suitable for further processing. For example, a suitable viscosity of a yoghurt drink can be given in terms of optimal sensory consistency.

REFERENCES

Houška M. (1986): Calibration of cup viscometer (Cejchování výtokového viskozimetru). Potravinářské vědy, 4: 143–146. (in Czech)

Meskat W. (1957): Viskosimetrie. In: Hengstenberg J., Sturm B., Winkler O. (eds): Messen und Regeln in der chemischen Technik. Berlin-Heidelberg, Springer Verlag: 856–994.

Motoyama Y. (2009): Cup viscometer. JP2009229338.

Šesták J., Houška M. (1980): Modeling of conditions to estimate the stable technological regime of drawing glass tubes (Modelování podmínek k odhadu stabilního technologického režimu tažení skleněných trubek). Research report. Prague, Czech Technical University in Prague.

Walters K., Barnes H.A. (1980): Anomalous extensional flow effects in the use of commercial viscometers. Proceedings of 8th Congress of Rheology. Naples, Sept 1–5, 1980: 45–62.

Xie F. (2019): Outflow cup type viscometer. CN109357970.

Xu S., Liu J., Lei Y., Gao L., Shu H. (2022): Flow cup type viscometer capable of measuring viscosity of silica sol coating. CN215414884.

https://doi.org/10.17221/159/2023-CJFS

Yasuda T. (2013): Cup viscometer. JP2013088166.

Zhang K., Lin S., Sun M., Zhao H., Feng D., Han J., Xun Q., Liu X., Wang P., Song L., Yao X., Gong W. (2020): Method and device for calibrating outflow cup type viscometer. CN111595729.

Zhang K., Han J., Lin S., Ji K., Feng D., Yao X., Gong W., Lyu H., Liu X., Tuo R., Ren W. (2022): Constant temperature device for verification/calibration of outflow cup type viscometer. CN217033494.

Zhang K., Zhao H., Han J., Lin S., Gong W., Lyu H., Ji F., Liu X., Xun Q., Feng D. (2023): Full-automatic verification device and verification method for outflow cup type viscometer. CN115728183.

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