

THE CAPABILITY OF THE VISCOSITY MEASUREMENT PROCESS

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Abstract

The aim of the paper is to evaluate the quality of measurement process of the dynamic viscosity using the Measurement Systems Analysis and the analysis of uncertainty. The viscosity of a fluid is defined as a measure of its resistance to gradual deformation by shear stress or tensile stress. Viscosity is a property arising from collisions between neighboring particles in a fluid that are moving at different velocities. Dynamic or absolute viscosity is a measure of the internal resistance of the fluid. The measurement process is realized which of ten samples of vegetable and mineral oils, four operators and the viscometer "Rheotest-2". The temperature of individual oil was according to customer requirement. The process of the measurement according to the Measurement Systems Analysis is capable because the value of index %GRR is 8.85 %. Operators do not have a statistically significant effect on the measured viscosity or its uncertainty. The paper presents a procedure to calculate the uncertainty of the measured values of dynamic viscosity. In addition to possible sources of uncertainty that are reported in the available literature, the calculation is supplemented by further sources, also important according to authors.

Keywords: liquids, dynamic viscosity, oils, rotation viscometer, Measurement Systems Analysis

1 Introduction

The viscosity of the fluid is an important property in the analysis of liquid behavior and fluid motion near solid boundaries. It is a measure of a fluid's resistance to flow. The resistance is caused by intermolecular friction exerted when layers of fluids attempt to slide by one another. The knowledge of the viscosity is needed for proper design of required temperatures of the fluid. There are two related measures of fluid viscosity - dynamic and kinematic viscosity.

The paper deals with measurement system of dynamic viscosity, which is a measure of the internal resistance. It is the tangential force per unit area required to move one horizontal plane with respect to the other at unit velocity when maintained a unit distance apart by the fluid.

Viscosity is one of the main properties of the oil. Its value should vary as little as possible in operation, under all engine-operating conditions [1], a change of viscosity is linked to physicochemical oil properties [2]. In the food industry, viscosity is one of the most important parameters required for the design of technological process. The viscosity slightly decreases with increased degree of unsaturation and rapidly increases with polymerization [3]. Viscosity can change with temperature, pressure, and concentration of fluids; theoretical equations can model all these changes, for example, the Arrhenius equation [2]. When heat is applied to fluids,

molecules can then slide over each other more easily making the liquid become less viscous [4, 5]. The optimum design of heating and cooling systems for cooking and frying, and the fundamental understanding of cooking and frying processes requires that the thermo-physical properties of the major ingredients involved oil in these processes be known. Two of the essential thermo physical properties are viscosity and specific heat. The dimensioning and selection of pumps and pipes for handling hot oil also require that the viscosity of the oil was known [6, 7, 8].

1.1 The capability of the measurement process

The aim of the measurement management system according to standard STN EN ISO 10012 [9] is to regulate the hazard that the measurement equipment or measurement process could yield incorrect results. False results negatively affect the final quality of products, as a rule, with consequential economic or moral damages (e.g. the loss of manufacturer's goodwill). The incorrect results of measurement can eventually affect the health, safety, human environment, governmental interests. Although we can suppose, from experience, that confirmed (calibrated and verified) measurement equipment would be accurate also at the end of the calibration interval; there is an obvious danger of equipment misdirection. The probable consequence of measurement equipment misdirection is measuring of incorrect values even with the most accurate and truest (with the most freedom from bias) measurement equipment. The misdirection can be a result of incorrect measurement method, the environment of measurement or incompetent operators.

The measurement system with high variability is not suitable for the analysis, since its variability can mask the variability of measured manufacturing process. The capability of measurement system (and also it of process) is defined by statistical properties of multiple measurements obtained from the measurements, working in stable conditions.

In the contemporary industrial and manufacturing world, it is essential to have measurement systems that are capable of providing reliable and accurate data to comply with ever stricter regulations and satisfy customer demands. One of the most widely used methodologies for the initial validation of measurement systems and continuous follow up is Measurement System Analysis (abbreviation MSA) defined, for example, in Automotive Industry Action Group (abbreviation AIAG) [10]. This methodology establishes certain analysis and validation criteria for variables and attributes measurement systems analysis, basing decisions on data analysis from designed experiments that consider the main factors affecting measurement systems [11].

Measurement Systems Analysis is an experimental and mathematical method of determining how much the variation within the measurement process contributes to the overall process variability. The measurement process, running in capable measurement system is capable as well. Measurement Systems Analysis used, above all, in the automotive industry helps to conform to STN P ISO/TS 16 949 [12], as well as AIAG standards requirements.

The analysis of variance (ANOVA) is one of the Measurement System Analysis methods. Its advantages are that it is capable of handling any experimental set-up, can estimate the variance accurately and extracts more information from the experimental data. The disadvantages are more complex numerical computations, and users require a certain degree of statistical knowledge to interpret the results [10].

Simpler method of Measurement System Analysis, Gauge Repeatability and Reproducibility (GRR) is a system designed to help engineers and quality professionals assess, monitor, and reduce measurement system variation.

In regard to more simply approach, the GRR was used for estimation the capability of the measurement process of dynamic viscosity. The calculation was carried out in accordance with [10], the software QUANTUM XL with the significance level $\alpha = 0.01$, confidence level $\alpha = 0.01$ and 6.00σ was used.

2 Experimental materials and methods

The aim of the paper is to evaluate the quality of the measurement system in which the measurement process of dynamic viscosity is carried out using Measurement Systems Analysis and the analysis of uncertainty. The values of dynamic viscosity μ (mPa.s) of ten oils (glycerine, hemp, linseed, olive, walnut, paraffin, wheat sprout, colzaseed, sesame, and sunflower) were measured with rotation viscometer at different temperatures according to customer demand. The measured oils were of food or pharmacist's quality. The study was performed with "Rheotest-2" viscometer (Germany). It is dual system equipment containing cylindrical measuring device (Couette), and it is suitable for determination of dynamic viscosity of solutions exhibiting Newtonian behavior.

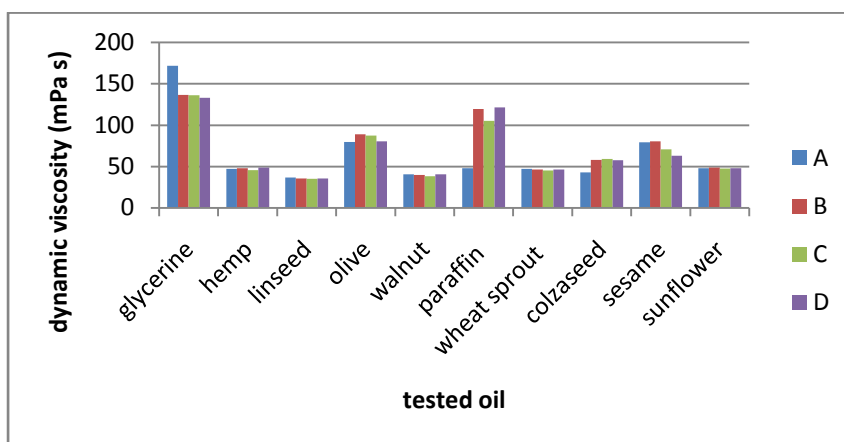


Fig. 1 Average values of the viscosity measured by individual operators

The standardization of rotational viscometers covers, with few exceptions, only general specifications concerning the flow pattern, range of shearing stresses to be used and velocity gradient as well as specification relating to specific substances. The procedure recommended in the standard STN EN ISO 3219 [13] was used in the measurement and calibration.

The viscometer measures the torsion moment appearing due to the ring-shaped oil layer between a fixed cylinder and inner cylinder rotating at a constant angular velocity [14].

The rheological characterization of oil samples was performed under thermostatic conditions, using a cylinder N suitable for the viscosity range of these fluids. The cylinder was filled with 10 ml $\pm 5\%$ of measured oil. The range I. with revolutions changed between 5/9 and 243 per minute with 24 points in steps of approximately the square root of three was used for all oils. The viscometer was calibrated prior to measurement. The results of the calibration are presented in section 3.2.

Three replications (trials) per sample were done, and the results were subsequently averaged. The measurements were carried out by four operators in random order. Average values of viscosity, measured by individual operators A, B, C and D are in (Fig. 1).

According to the two factors ANOVA (Analysis of Variance) without replication, the operator has not ($p = 0.78876$) and the oil has ($p = 5.41 \cdot 10^{-11}$) statistically significant effect on the measured value of dynamic viscosity.

The normality and the outliers were determined for the files of 120 values. Grubbs' test (significance level $\alpha = 0.05$) was used for detection of statistical outliers. Their presence indicates measurement process suffering from special disturbances and out of statistical control. The normality was determined by Freeware Process Capability Calculator software (Anderson – Darling test). The value “p” for the files with a normal distribution is above 0.05. As it can be seen in Tab. 1, the normality was confirmed only for the values of hemp, linseed and walnut oil. The standard methods of Measurement Systems Analysis used to assess the capability of measuring process, assume normal probability distribution. Otherwise, there is an overestimation of incapacity (increasing value of % GRR).

Table 1 Tested oils, ambient temperatures, number of outliers and p value of the normality test for all 120 measurements on individual oil

Oil	glycerine	hemp	linseed	olive	walnut	paraffin	wheatsprout	colzaseed	sesame	sunflower
T (°C)	12	20	24	14	24	14	23	20	15	23
Outliers	0	1	0	0	0	0	3	0	0	0
p-value	0	0.09309	0.11663	0	0.10763	0	0	0	0	0

3 Results

3.1 Measurement Systems Analysis

The first step of Measurement Systems Analysis is to estimate whether the discrimination is sufficient. The general rule of thumb is that the discrimination ought to be at least one - tenth the process variation (i. e. one- tenth of process variation or smaller) [10]. The standard deviation of the hemp oil is 28.1 mPa.s (the results of all measurements of all four operators were regarded) and the discrimination (the value of one scale division of the potentiometer) is equivalent to 3.29 mPa.s in average with exclusion of outlier values, for example. The resolution of used equipment reaches the limit. The situation is similar for other tested oils.

Analyzed measurement system is not under statistical control. Only the results of the operator A are inside control limits in the range control chart (R). The X-bar control chart provides an indication of “usability” of the measurement system. The area within the control limits represents the measurement sensitivity (“noise”). If less than half fall outside the control limits the measurement system, lacks adequate, effective resolution between the viscosity of individual oils. Analyzed system has sufficient sensitivity – the most of the measurements fall outside control limits.

The index %EV = 3.89 % represents cumulative influence of the suitability of the measurement equipment, method and environmental conditions on the variability. It is a function of the average range of trials of all operators.

The index %AV = 7.95 % represents the influence of operators on the variability, for example, their reliability (responsibility) and competence. It is a function of the maximum average operator difference. Low value of the index confirms the competence of all operators.

The %PV is a function of the range of individual measurements of samples. It is sensitive to variability of the viscosity of different oils. The values of %PV indirectly specify the suitability

of used measurement equipment for particular measurement. The value of %PV above 99 % is for excessively accurate equipment, between 90 % and 99 % for suitable, between 70 % and 90 % for satisfactory and between 50 % and 70 % for inaccurate one. Therefore used equipment with %PV = 99.61 % is very accurate [15, 16].

The number of distinct categories (“ndc”, based on Wheeler's discrimination ratio) is related to the resolution of measurement equipment. It indicates the number of various categories, which can be distinguished by the measurement systems. It is the number of non-overlap 97 % confidence intervals, which cover the range of expected variability of product. The “ndc” is greater than or equal to 5 for capable processes; results with “ndc” values between 2-5 may be conditionally used for rough estimations. The “ndc” value of the analyzed system is 15.

The %GRR index represents the process capability in practice. %GRR < 10 % is considered to be an acceptable measurement system; %GRR > 30 % is considered to be not acceptable. Analyzed measurement process is considered to be acceptable – capable, %GRR = 8.85 %.

As was remarked above the normality of most files (values measured for one oil) has not been established. Thus, the actual process capability is higher than the calculated one.

3.2 Calibration and uncertainties

Calibration of the rotational viscometer shall be verified using viscosity standards prior to use at each site [17]. The tester was calibrated before the measurement using the certified reference material (abbreviation CRM).

NF-60 (7178.76) with nominal value of dynamic viscosity $\mu = 74.223$ mPa.s, density $\rho = 0.860$ g.cm⁻³, with maximal permissible error (abbreviation MPE) 1 % at 20°C (manufacturer Rheotest Medingen GmbH / Fischer Scientific). Nominal viscosity of the standard was chosen close to the average value of the viscosity of all measured oils ($\mu = 67.24$ mPa.s). The recommendations [18] and [19] were used to prepare the calibration procedure.

The cylinder N (filled with 10 ml $\pm 5\%$ of the standard) and range I. with three replications were used. The calibration was carried out the most experienced operator B. The outlier values were eliminated by Grubbs test. The file of 18 values with normal distribution ($p = 0.5885$), average value of $\mu = 71.13$ mPa.s and standard deviation SD = 8.41 mPa.s is the result. The A type standard uncertainty [20], $u_A = 1.982$ mPa.s was calculated using the standard deviation.

Identified sources of B type standard uncertainty:

1. The uncertainty as a result of the systematic error (bias), the difference between the average value and nominal value of the standard was calculated according to the method referred in article [21], $u_{B_{\text{syst}}} = 1.857$ mPa.s.
2. The uncertainty as a result of possible error of nominal value of the standard was calculated using MPE of the standard (0.742mPa.s) according to equation (1). The coefficient $\chi = \sqrt{3}$ for assumed rectangular distribution, $u_{B_{\text{standard}}} = 0.429$ mPa.s.
3. The uncertainty as a result of possible error of the scale was calculated using MPE of the scale (1.5 %) of the viscometer according to equation (1.), $\chi = \sqrt{3}$, $u_{B_{\text{scale}}} = 1.612$ mPa.s. The manufacturer of the standard does not provide its dependence of its viscosity on the temperature. The ambient temperature during the calibration was 20°C without recorded changes. The factor of temperature was, therefore, not considered as a source of uncertainty.

$$u = \frac{MPE}{\chi} \quad (1)$$

Combined standard uncertainty of the viscometer $u_{\text{tester}}=2.817$ mPa.s, relative expanded uncertainty $U_{\text{rel}} = 7.92$ %. Expanded uncertainty is the quantity defining an interval about the result of a measurement containing a true value of measured quantity with probability 95 % (coverage factor $k = 2$).

Relative error of the viscometer $E_{\text{rel}} = -2.53$ % does not exceed the maximal permissible error (MPE) of the viscometer guaranteed by the manufacturer (± 3 %).

Relative uncertainty U_{rel} of measured value of the viscosity of different oils, measured by different operators A, B, C and are explained in (Fig. 2). Its sources were u_{tester} and the A type standard uncertainty of individual oils, based on the standard deviation of replicated measurements. According to the two factor ANOVA without replication, the operator has not ($p = 0.48643$) and the oil has ($p = 8.99 \cdot 10^{-11}$) statistically significant effect on the value of relative expanded uncertainty U_{rel} of the measured values of dynamic viscosity.

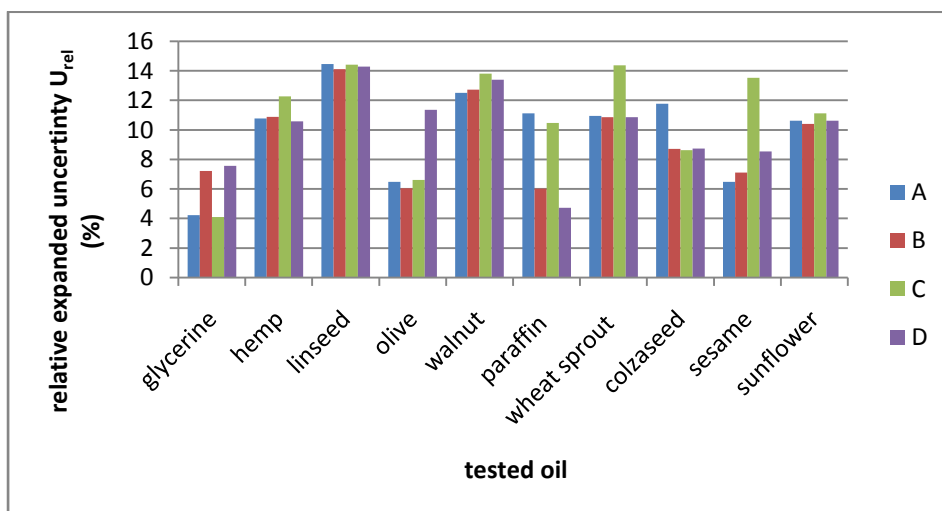


Fig.2 Relative expanded uncertainty of the measured values of the viscosity

4 Discussion

Measurement Systems Analysis has been developed primarily to evaluate the capability of the process of dimensional measurements. Previously published works pointed to its suitability for evaluating measurement processes of physic - chemical parameters, but also the mechanical and technological tests [22-24]. There are exceptions, such as a low capability when measuring hardness [25]. Possibility of interconnection of Measurement Systems Analysis and uncertainty analysis, outlined in the abovementioned work [21], but also in more recent works [26, 27] indicates future direction of evaluating the quality of measurement processes.

Particular problem is the lack of a defined procedure for calculating the uncertainty of rotational viscometers in standards. Since the procedures referred to in the scientific literature take into account only several sources of uncertainty, final value of uncertainty is likely smaller than it is in fact. Two sources of the uncertainty are recommended to taken into account in [19] for example. They are A type of the uncertainty and the uncertainty of used standard $u_{\text{Bstandard}}$.

The calculation of uncertainty was supplemented, based on literature data and experience, by two other sources in this paper. They are the uncertainty as a result of the systematic error (bias),

the difference between the average value and nominal value of the standard found during the calibration and the uncertainty as a result of possible error of the scale, calculated using MPE of the scale of the viscometer. These sources are significant and their omissions significantly distort the overall value of uncertainty in the opinion of authors.

This fact, of course, led to an increase in uncertainty. Expanded uncertainty of the resulting viscosity of oils increased between 18.5 % (glycerine oil) and 27.8 % (colzaseed oil). This increase of the uncertainty to be negative at first sight, but the opposite is true. Just this increase reduces the risk that the true value of the viscosity measured by analyzed process in the analyzed system will be found outside the interval and thus will be out of control.

5 Conclusion:

1. The process of the measurement of dynamic viscosity is capable, %GRR = 8.85 %.
2. Despite some occurrence of outliers, operators do not have a statistically significant effect on the measured viscosity or its uncertainty.
3. Used tester seems to be less sensitive about the discrimination. On the contrary, the index EV% lower than the% AV index and high value of index % PV show the opposite. Also, the analysis of the X-bar control chart confirmed the sufficiently sensitive tester. It is obvious that the possibility of using multiple evaluation methods reduces the probability of an erroneous conclusion.
4. The error of the tester found in the calibration does not exceed its maximum value (MPE) guaranteed by the manufacturer.

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