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STUDY OF THE RHEOLOGICAL PROPERTIES OF SOME ESSENTIAL OILS USED IN COSMETICS AND PHARMACEUTICALS

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Abstract: The processing of various liquids used in industrial production is accompanied by complex physicochemical, biological and mechanical processes, the detailed study of which allows the organization of objective control and management of the technological cycle of production. The rheological characteristics of cosmetics and pharmaceuticals fluids are important for production, packaging, filling, and storage. The course of these processes, the design parameters of the working bodies of the relevant machines and devices, as well as the quality of the products obtained to a significant extent depend on the rheological properties of the processed materials. The main processes in which the rheological properties of the processed materials should be most fully taken into account are forming, mixing and transportation. When studying the indicated processes, a relationship is established between the rheological characteristics of the material and the technological parameters of its processing. One of the most significant properties of continuous media is viscosity. The article considers the main methods of measuring the viscosity of liquids with a rotational viscometer. This article presents results of a study of the rheological properties of essential oils /rose oil, lavender oil and peppermint oil/. Thermo Scientific HAAKE MARS iQ Rheometer Air-bearing was used to determine the dependences of viscosity change on temperature. Currently, in some reference materials, approximate values of the dynamic viscosity of essential oils can be found, but the exact numerical values at different temperatures are missing. The results obtained have practical value in technological processes and the design of machines and mechanisms that use essential oils.

Keywords: viscosity measurement, temperature, rheological properties, essential oils.

1. INTRODUCTION

Fluids are substances that do not have their own shape but take on the shape of the container they are placed in.

Any material medium, whether it be a liquid or a gas, possessing the properties of continuity and mobility, is called a fluid. Fluids include all liquids and gases. Unlike solid objects, fluids undergo significant changes in their shape, even when subjected to minor external forces. The difference in the mechanical properties between fluids and solid objects lies in their molecular structure. In solid objects, the forces of attraction between molecules are significant, and they occupy specific positions within the volume of the body.

During deformation, the distance between the molecules of a fluid changes, but it is restored after the removal of external forces. In liquids, the intermolecular and cohesive forces are significantly weaker, and under the influence of external forces, the molecules change their positions. After the force is removed, they assume new equilibrium positions without being able to regain their original shape [1], [2].

The processing of various liquids used in industrial production is accompanied by complex physicochemical, biological and mechanical processes, the detailed study of which allows the organization of objective control and management of the technological cycle of production. The rheological characteristics of cosmetics and pharmaceuticals are important for production, packaging, filling, and storage. The course of these processes, the design parameters of the working bodies of the relevant machines and devices, as well as the quality of the products obtained to a significant extent depend on the rheological properties of the processed materials. The main processes in which the rheological properties of the processed materials should be most fully taken into account are forming, mixing and transportation. When studying the indicated processes, a relationship is established between the rheological characteristics of the material and the technological parameters of its processing.

Viscosity is the property of fluids to resist the force that causes them to flow. Viscosity is divided into two types: dynamic viscosity and kinematic viscosity. Unlike kinematic viscosity, dynamic or absolute viscosity does not depend on the density of the fluid since it determines the internal friction within the fluid. Absolute viscosity is often related to shear stress, which is caused by a force acting parallel to the cross-sectional area of the body or, in our case, the liquid.

Essential oils are concentrated aromatic liquids extracted from plants that possess a specific aroma and therapeutic properties. They are also known as volatile oils and are widely used in aromatherapy, cosmetics and medicine.

Rheology describes the deformation of a body under the influence of stresses. “Bodies” in this context can be either solids, liquids, or gases.

Ideal solids deform elastically. The energy required for the deformation is fully recovered when the stresses are removed. Ideal fluids such as liquids and gases deform irreversibly – they flow. The energy required for the deformation is dissipated within the fluid in the form of heat and cannot be recovered simply by removing the stresses.

The real bodies we encounter are neither ideal solids nor ideal fluids. Real solids can also deform irreversibly under the influence of forces of sufficient magnitude – they creep, they flow.

The resistance of a fluid against any irreversible positional change of its’ volume elements is called viscosity. To maintain flow in a fluid, energy must be added continuously.

While solids and fluids react very differently when deformed by stresses there is no basic difference rheologically between liquids and gases; gases are just fluids with a much lower viscosity, for example hydrogen gas at 20°C has a viscosity a hundredth of the viscosity of water.

Instruments which measure the visco-elastic properties of solids, semi-solids and fluids are named “rheometers”. Instruments which are limited in their use for the measurement of the viscous flow behavior of fluids are described as “viscometers”.

When discussing viscosity, two types of fluids are distinguished: Newtonian and non-Newtonian. The viscosity of Newtonian fluids does not depend on the force acting upon them. In the case of non-Newtonian fluids, the situation is more complex because their viscosity can vary depending on the magnitude of the applied force and the manner in which it is applied.

Under normal conditions, they are nearly completely free of viscosity. Their viscosity does not change, even if a small force is applied to them, such as slowly stirring them with a spoon. The viscosity determined by the internal friction within the fluid is called dynamic viscosity η and is measured in Pascal seconds (Pa.s).

The viscosity of liquids changes with variations in temperature and pressure $\eta=(T, p)$. The higher the temperature of a liquid, the lower its viscosity [1], [2], [4], [5].

2. VISCOSITY MEASUREMENT WITH ROTATIONAL TEST – BASIC TERMS

There are two basic types of flow, these being shear flow and extensional flow. In shear flow fluid components shear past one another while in extensional flow fluid component flowing away or towards from one other. The most common flow behavior and one that is most easily measured on a rotational rheometer or viscometer is shear flow and this viscosity introduction will focus on this behavior and how to measure it [1], [4], [6], [7].

2.1. The basic law

The measurement of the viscosity of liquids first requires the definition of the parameters which are involved in flow. Then one has to find suitable test conditions which allow the measurement of flow properties objectively and reproducibly. Isaac Newton was the first to express the basic law of viscometry describing the flow behaviour of an ideal liquid:

$$\tau = \eta . \frac{dv}{dy} \tag{1}$$

shear stress = viscosity x shear rate

Shear flow can be depicted as layers of fluid sliding over one another with each layer moving faster than the one beneath it. The uppermost layer has maximum velocity while the bottom layer is stationary. For shear flow to take place a shear force must act on the fluid.

2.2. Shear stress

Shear induced flow in liquids can flow between two parallel flat plates, when one plate moves and the other is stationary. This creates a laminar flow of layers which resembles the displacement of individual cards in a deck of cards. A force *F* applied tangentially to an area *A* being the interface between the upper plate and the liquid underneath, leads to a flow in the liquid layer. This external force takes the form of a *shear stress* (τ) which is defined as the force (F) acting over a unit area (A) as shown in *Figure 1*.

$$\tau = \frac{F}{A} \tag{2}$$

The velocity of flow that can be maintained for a given force is controlled by the internal resistance of the liquid, i.e. by it's viscosity.

In response to this force the upper layer will move a given distance *x*, while the bottom layer remains stationary. Hence, we have a displacement gradient across the sample (x/h) termed the *shear strain (deformation)* - (γ). For a solid which behaves like a single block of material, the strain will be finite for an applied stress – no flow is possible. However, for a fluid where the constituent components can move relative to one another, the shear strain will continue to increase for the period of applied stress. This creates a velocity gradient termed the *shear rate* or *strain rate* ($\dot{\gamma}$) which is the rate of change of strain with time ($d\gamma/dt$).

$$\dot{\gamma} = \frac{d\gamma}{dt} = \frac{dv}{dy},\ 1/s \tag{3}$$

$$\tau = \eta . \frac{dv}{dy} = \eta . \frac{d\gamma}{dt} = \eta . \dot{\gamma},\ \text{Pa} \tag{4}$$

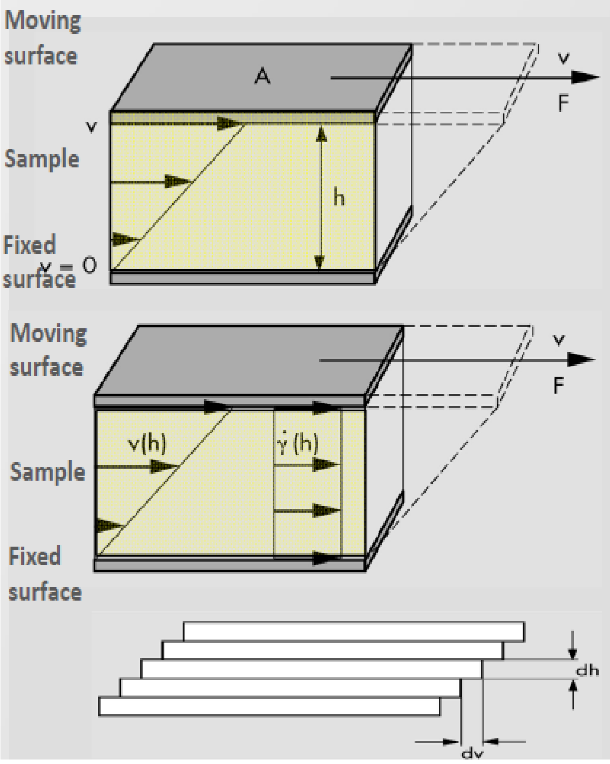


Figure 1. *Flow between two parallel flat plates, when one plate moves and the other is stationary.*

2.3. Dynamic viscosity

The (dynamic) viscosity describes the mathematical relationship between shear rate and shear stress of fluids. This is also known as Newton's law of fluid motion [1], [4], [5], [8]. Solving equation for the dynamic viscosity η gives

$$\eta = \frac{\tau}{\dot{\gamma}} \tag{5}$$

The unit of dynamic viscosity η is the "Pascal second" [Pa.s]. It is worthwhile noting that the previously used units of "centiPoise" [cP] for the dynamic viscosity η are interchangeable with [mPa.s], 1 mPa.s = 1 cP.

2.4. Kinematic viscosity

The kinematic viscosity puts the viscosity of a fluid in relation to its density and is a measure of how “pasty” a fluid is. It is determined by the quotient of dynamic viscosity and density:

$$v = \frac{\eta}{\rho}, \text{ [m}^2\text{/s]; [mm}^2\text{/s]= 1 cSt, Stokes” [St] or “centi Stokes” [cSt].} \qquad (6)$$

2.5. Flow and viscosity curves

The correlation between shear stress and shear rate defining the flow behavior of a liquid is graphically displayed in a diagram of τ on the ordinate and $\dot{\gamma}$ on the abscissa. This diagram is called the “Flow Curve” – fig. 2, a. Another diagram is very common - η is plotted versus $\dot{\gamma}$. This diagram is called the “Viscosity Curve” fig. 2, b:

A fluid which does not obey Newton’s law of viscosity is called a non-Newtonian fluid. Non-Newtonian fluids are classified into three types based on their characteristics: Bingham fluid, pseudoplastic fluid, and dilatant fluid. The relations between velocity gradient and shear stress of each fluid are shown in Figure 3.

As for a Bingham fluid, shear stress acts even when a Bingham fluid remains stationary. Thus, a Bingham fluid does not flow until shear stress reaches a certain value, and starts to flow when shear stress exceeds a certain value. The shear stress when a Bingham fluid starts to flow is called Bingham yield value. Toothpaste and butter are familiar examples of Bingham fluids.

Some examples of Newtonian fluids include water, organic solvents, mineral and vegetable oil. For those fluids viscosity is only dependent on temperature. As a result, if we look at a plot of shear stress versus shear rate (See Figure 3) we can see a linear increase in stress with increasing shear rates, where the slope is given by the viscosity of the fluid [6], [8]. This means that the viscosity of Newtonian fluids will remain a constant no matter how fast they are forced to flow through a pipe or channel (i.e. viscosity is independent of the rate of shear).

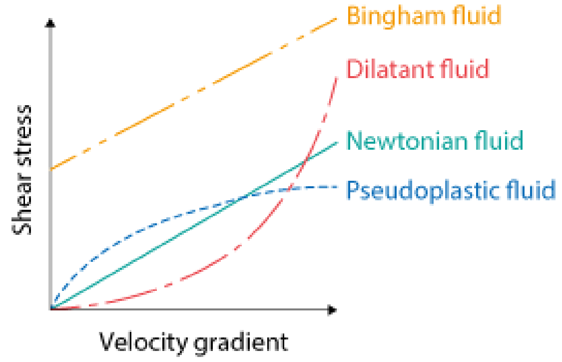
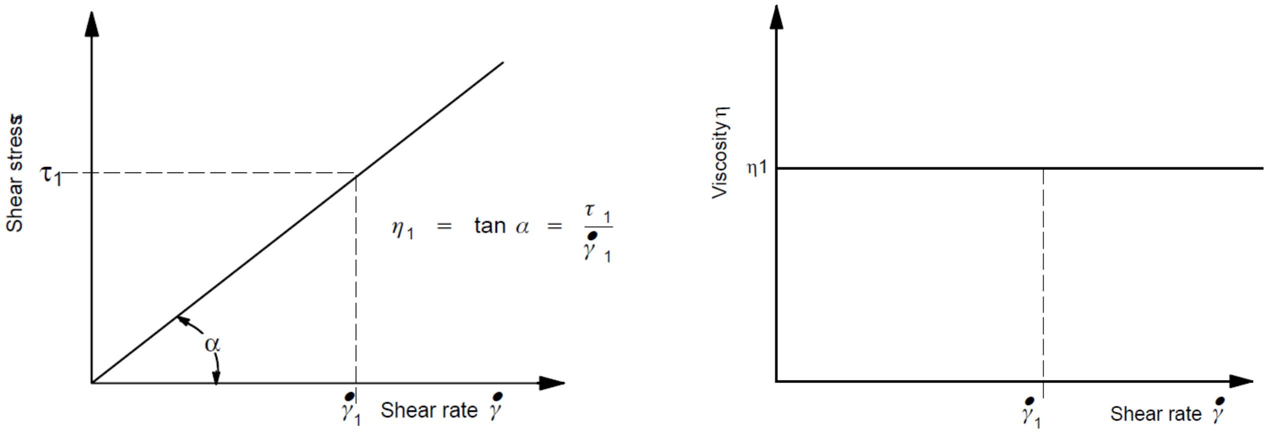


Figure 3. *Non-Newtonian fluids.*

An exception to the rule is Bingham plastics, which are fluids that require a minimum stress to be applied before they flow. These are strictly non-Newtonian, but once the flow starts they behave essentially as Newtonian fluids (i.e. shear stress is linear with shear rate).

2.6. Rotational viscometer

Viscosity can be measured with a rotational viscometer by assessing the resistance of a fluid to flow when subjected to a rotational force. The basic principle behind this method is that as the viscometer’s spindle or rotor rotates within the fluid, it expe-



a *b*

Figure 2. *Flow Curve – a, Viscosity Curve – b.*

riences resistance due to the fluid's internal friction (viscosity). This resistance is then used to determine the fluid's viscosity.

Key Components of a Rotational Viscometer are – fig.4: Rotor (Spindle): This is the part that rotates in the fluid. The shape and size of the rotor vary based on the type of fluid being tested; Motor: The motor applies a rotational force to the rotor; Torque Sensor: Measures the torque (rotational resistance) that the fluid exerts on the rotor as it spins; Viscosity Display/Output: This is the final measurement, often in units like centipoise (cP), pascal-seconds (Pa.s), or poise (P) [3].

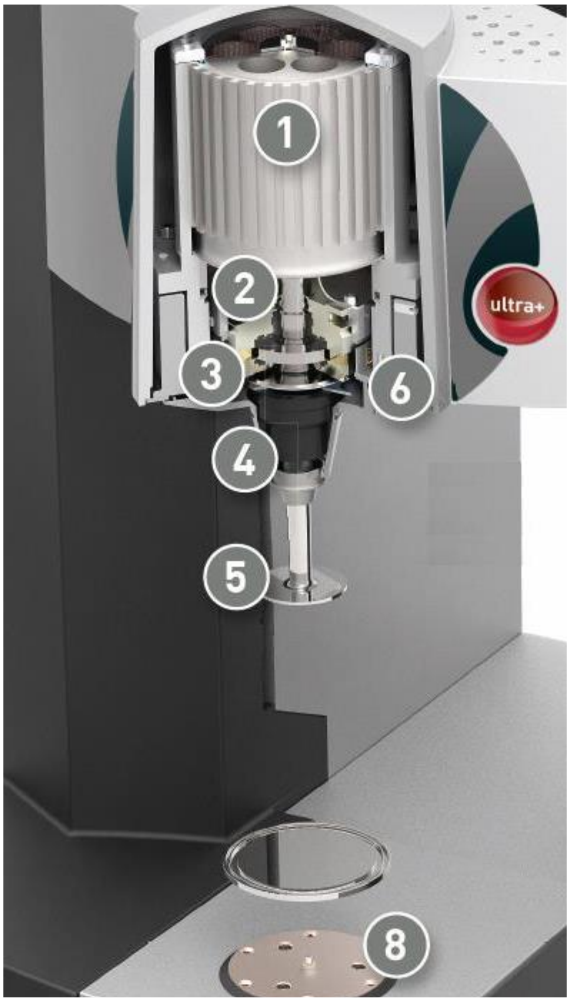


Figure 4. *Thermo Scientific HAAKE MARS iQ Rheometer Air-bearing Principles –Measurement 1. Drag Cup Motor = Stress, 2. Air Bearing; 3. Position Sensor = Shear Rate (Strain); 4. Geometry Recognition & Chuck; 5. Measurement Geometry / Measuring System; 6. Strain Gauge = Normal Force & Automatic Gap; 8. Temperature Control*

Viscosity is determined by shearing a sample and measuring its resistance to that shear. The sample is placed in a stationary container and a moving spindle (bob, cone, plate), which is attached to the rheometer's motor, is lowered into or onto the sample. The spindle is moved at a fixed speed (shear rate) and the force (shear stress) required to move that speed is measured [3], [8].

2.7. Geometry factors for calculating the stress, strain and shear-rate

In rotational rheometers the shear stress τ is given by the equation,

$$\tau = A.M_d, \tag{7}$$

here M_d is the torque applied or measured by the rheometer measuring head and A is a geometry factor.

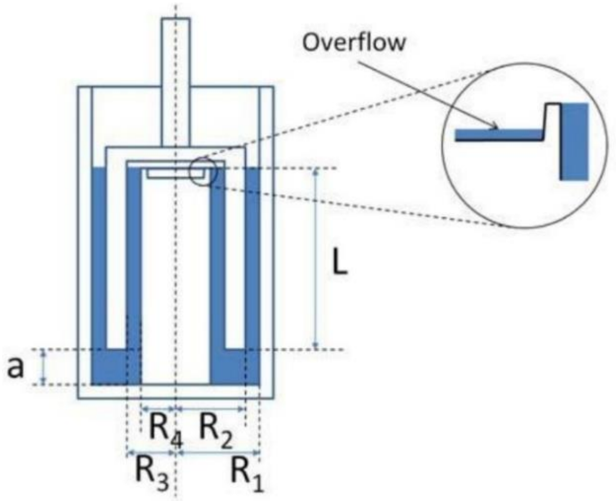
The strain γ and the shear rate (or strain-rate) $\dot{\gamma}$are given by the equations,

$$\gamma = M.\varphi \tag{8}$$

$$\dot{\gamma} = M.\Omega \tag{9}$$

here φ is the angle and Ω the angular-velocity applied or measured by the rheometer measuring head and M is a geometry factor. The geometry factors A and M depend on the type and the dimensions of the measuring geometry. The equations for the geometry factors as well as the (default) values of A and M for the standard measuring geometries are given in HAAKE documents. The viscosity η is defined by equation (5).

For the experiment we use approximately 7 ml of essential oil. It is placed inter (cup) CCB41 DG, and then the coaxial cylinder CC41 DG/Ti. Equations below in fig. 5 are used to calculate the two geometry factors A and M for the recessed ends coaxial cylinder geometries. These equations are based on the equations for the recessed end coaxial cylinder geometries described in the DIN 53019 (part 1) standard [3], [8].



R_1 =	inner radius of cup
R_2 =	outer radius of rotor
$\delta_{12} = R_1/R_2$ =	radius ratio of outer coaxial
R_3 =	inner radius of rotor
R_4 =	outer radius of stator
$\delta_{34} = R_3/R_4$ =	radius ratio of inner coaxial cylinder
L=	height of cylindrical part of rotor
a=	axial gap between rotor and cup

$$A = \frac{1}{2 \cdot \pi \cdot R_2^2 \cdot L + 2 \cdot \pi \cdot R_3^2 \cdot L}$$

$$M = \frac{2 \cdot \delta 2}{\delta^2 - 1}, \ \delta = \frac{R_1}{R_2} \cong \frac{R_3}{R_4}$$

Measuring geometry

CC41 DG CC41 DG/Ti

Geometry factor A = 3701 (Pa/Nm)

Geometry factor M = 72.67 (s^{-1}/rad^{-1})

Figure 5. *Geometry factors calculation for CC41 DG CC41 DG/Ti*

3. THE VARIATION OF NEWTONIAN VISCOSITY WITH TEMPERATURE

The viscosity of all simple liquids decreases with increase in temperature because of the increasing Brownian motion of their constituent molecules, and generally the higher the viscosity, the greater is the rate of decrease. For instance, while the viscosity of water reduces by about 3% per degree Celsius at room temperature; motor oils decrease by about 5% per degree, while bitumens decrease by 15% or more per degree. Many attempts have been made to describe the viscosity/temperature dependence mathematically. The most widely-used expression is that due to Andrade, the Frenkel-Andrade equation, or Frenkel-Andrade model, describes the temperature dependence of viscosity in liquids, particularly at higher temperatures. It posits that viscosity (η) is an exponentially activated process, meaning it increases with decreasing temperature. The equation is typically written as:

$$\eta = A.e^{E/k.T} \qquad (10)$$

where A is a pre-exponential factor, E is the activation energy, k is Boltzmann's constant 1.380649×10^{-23} J·K^{-1}, and T is the absolute temperature. The core idea is that viscosity changes exponentially with temperature due to the energy barrier (activation energy) that molecules need to overcome to move past each other in a liquid. activation energy (E) represents the energy required for a molecule to move from one position to another, and it's a crucial factor in determining how rapidly viscosity changes with temperature. Pre-exponential Factor (A) accounts for other influences on viscosity besides temperature, such as the liquid's molecular structure and density. In essence, the Frenkel-Andrade equation suggests that as temperature decreases, the exponential term (*e^(E/kT)*) increases, leading to a significant increase in viscosity. This model is useful for understanding and predicting the behavior of liquids, especially at higher temperatures where other models may not be as accurate.

In the present study of the viscous properties of essential oils - rose, mint and lavender, the model of and equation до Frenkel-Andrade is used.

4. EXPERIMENTAL INVESTIGATION OF VISCOSITY OF ESSENTIAL OILS WITH TEMPERATURE VARIATION

The dynamic viscosity of essential oils /rose, mint and lavender/ has determined using HAAKE MARS iQ Air rheometer and coaxial cylinder geometry. The coaxial cylinder configuration allows for precise measurement of the oil's dynamic viscosity by applying shear stress and measuring the resulting shear rate.

During the measurement, the temperature of the vegetable oil sample is maintained constant using a liquid thermal bath with an accuracy of 0.1 °C. For the mint and lavender oils, the temperature has changed from 5 to 80 °C. For the rose oil the oils the temperature has changed from 25 to 80 °C, because at 25 degrees, rose oil hardens and its structure changes.

Figure 6 shows the experimental result of the viscosity of lavender oil at a temperature of 5, 20, 40 and 80 °C. From the obtained result, it can be seen that the *shear stress* (τ) and dynamic viscosity (η) changes with varying the *shear rate* ($\dot{\gamma}$).

Using regression analysis, a straight line was determined that approximates the change in *shear stress* (τ) with Newton's law (5) and dynamic viscosity η have been obtained for the different temperature. The results are given in Table 1.

Table 1. *Dynamic viscosity* η *for lavender, mint and rose oil for different temperature*

Temp. °C	**Viscosity *η*, Pa.s**			
	Lavender Oil	**Mint Oil**	**Temp. °C**	**Rose Oil**
5	0.004483	0.0123	25	0.006933
10	0.003916	0.01058	30	0.005811
15	0.003418	0.008596	40	0.00434
20	0.003018	0.006903	50	0.003201
30	0.002372	0.004739	60	0.002457
40	0.001755	0.003537	70	0.001878
50	0.001492	0.002758	75	0.001666
60	0.001211	0.001979	80	0.001452
70	0.001054	0.001543		
80	0.000864	0.00112		

Figure 7 shows the experimental result of the viscosity of mint oil at a temperature of 5, 20, 40 and

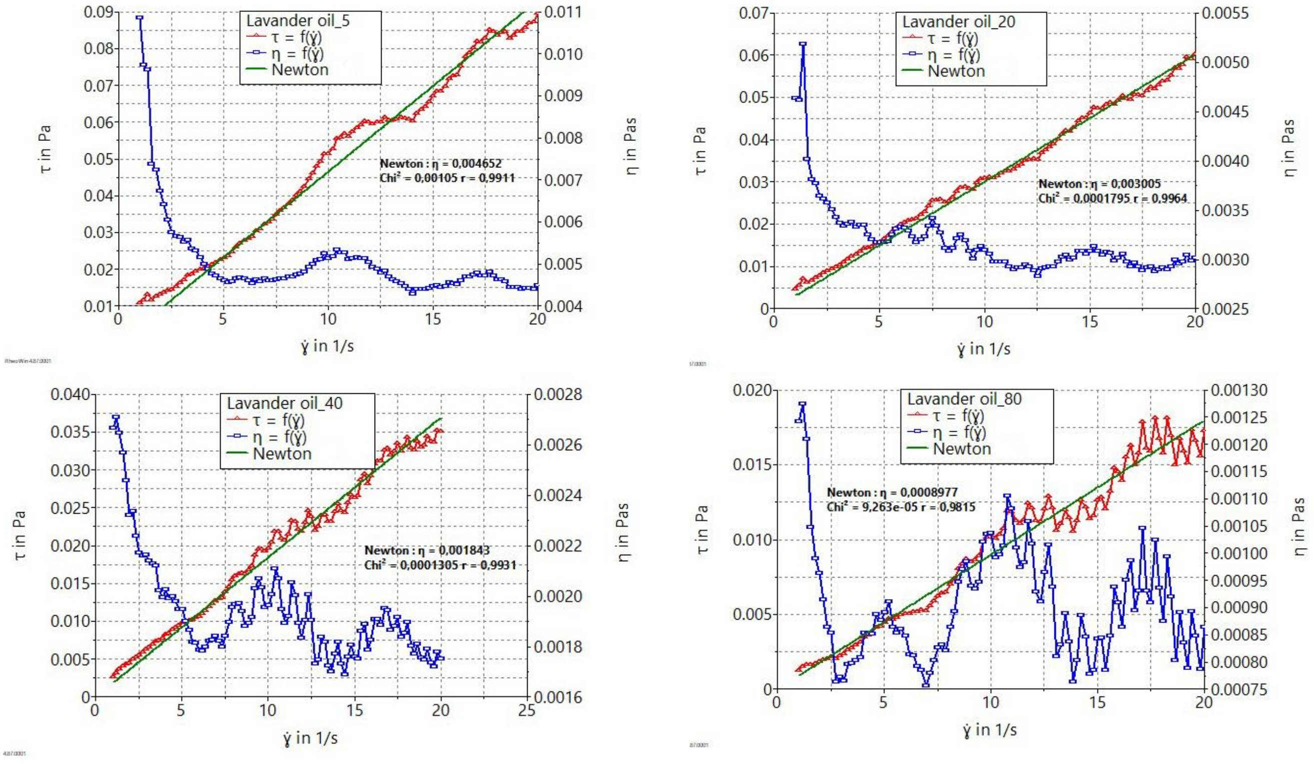


Figure 6. *Experimental result of the viscosity of lavender oil at a temperature of 5, 20, 40 and 80 °C*

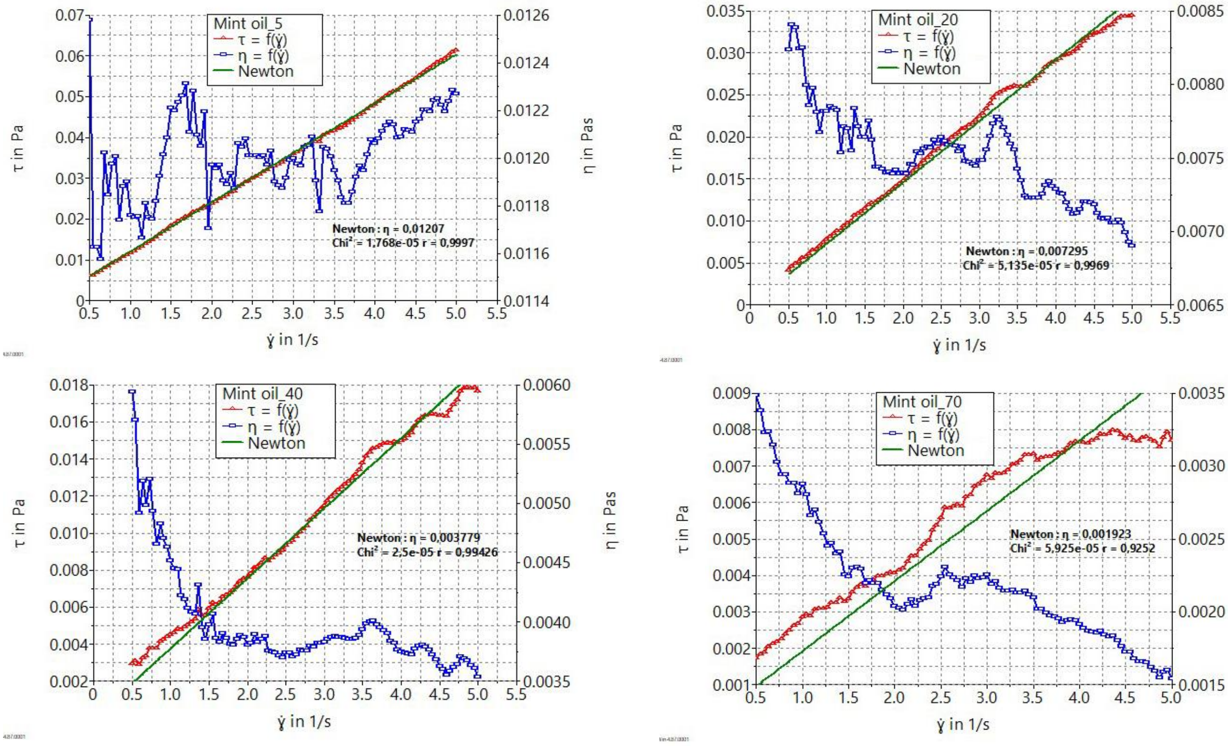


***Figure** 7. Experimental result of the viscosity of mint oil at a temperature of 5, 20, 40 and 80 °C*

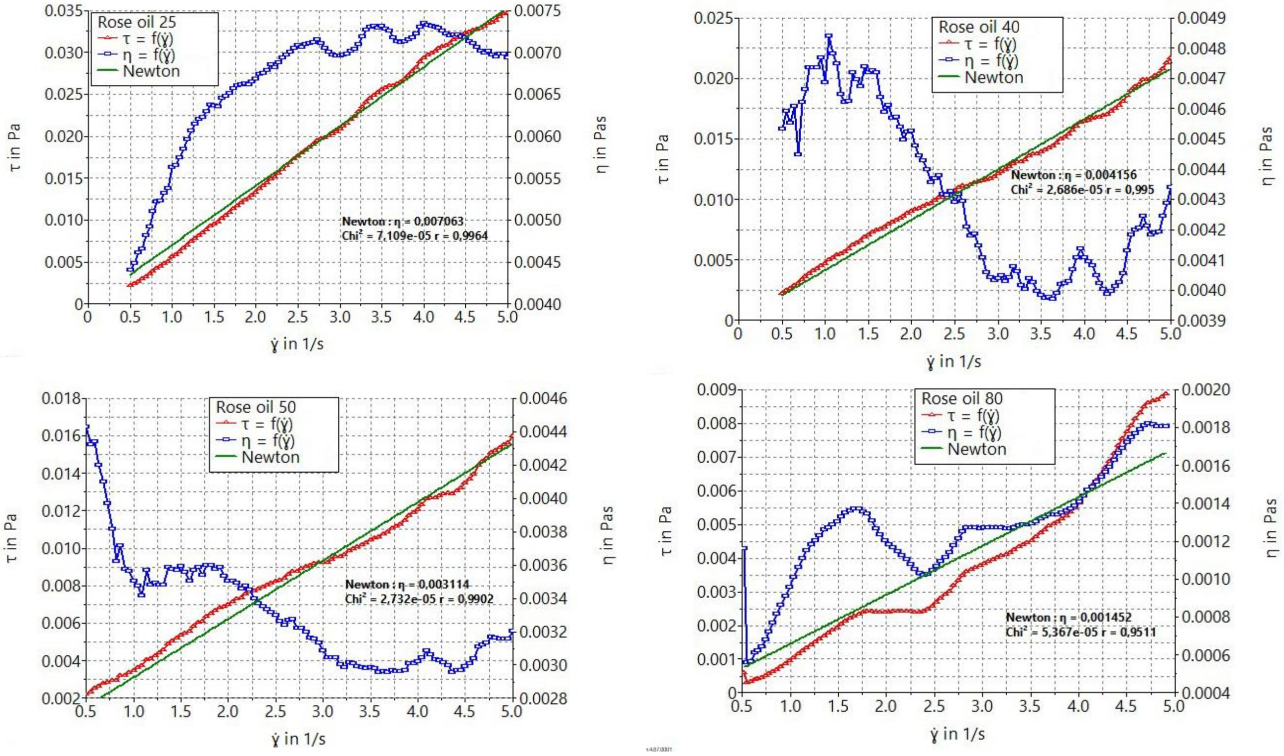


***Figure 8.** Experimental result of the viscosity of rose oil at a temperature of 25, 40, 50 and 80 °C*

80°C. From the obtained result, it can be seen that the *shear stress* (τ) and dynamic viscosity (η) changes with varying the *shear rate* ($\dot{\gamma}$). Using regression analysis, a straight line was determined that approximates the change in *shear stress* (τ) with Newton's law (5) and dynamic viscosity η have been obtained for the different temperature. The results are given in Table 1.

Figure 8 shows the experimental result of the viscosity of rose oil at a temperature of 25, 40, 50 and 80 °C. From the obtained result, it can be seen that the *shear stress* (τ) and dynamic viscosity (η) changes with varying the *shear rate* ($\dot{\gamma}$). Using regression analysis, a straight line was determined that approximates the change in *shear stress* (τ) with Newton's law (5) and dynamic viscosity η have been obtained for the different temperature. The results are given in Table 1.

Figure 9 shows the summarized experimental resultsof the viscosity η of lavender, mint and rose oil at different temperature. Research shows that peppermint oil has the greatest change in viscosity with temperature from 0.0123 to 0.00112 Pa.s, and to a lesser extent rose oil. Lavender oil has a smaller dependence on temperature in terms of viscosity change.

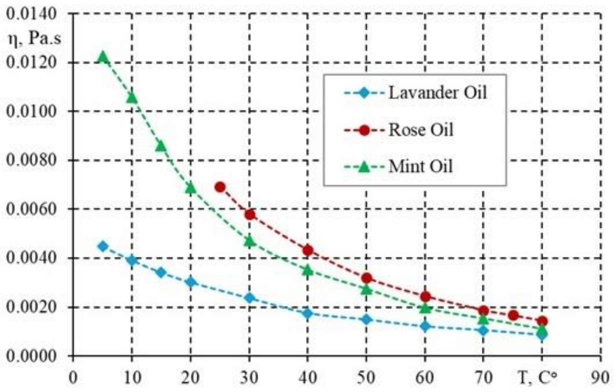


Figure 9. *Experimental result of the viscosity of lavender, min and rose oil at different temperature.*

5. THE FRENKEL-ANDRADE EQUATION MODEL IDENTIFICATION FROM EXPERIMENTAL DATA

The Frenkel-Andrade equation (9) is taken logarithmically and we get

$$ln\ (\eta) = lnA + {}^{E}/_{kT}. \tag{11}$$

The graph of the dependence ln(η) on *1/T* is a straight line. From the slope of this line, the value of the activation energy of the viscous flow activation energy *E* can be determined. The tangent of the angle of the slope is the ratio of the activation energy of the viscous flow divided by the Boltzmann constant *E/k*. The coefficient *A* can also be determined from the graph.

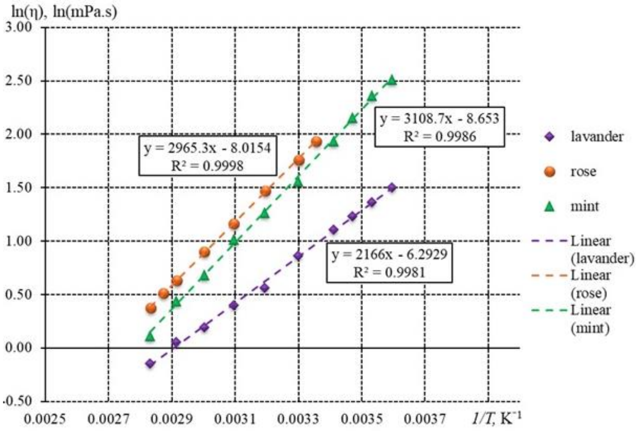


Figure 10. *Dependence of the logarithm of the dynamic viscosity ln(η) as a function of the reciprocal of the absolute temperature for the three types of essential oils*

The fig. 10 shows the dependence of the logarithm of the dynamic viscosity ln(η) as a function of the reciprocal of the absolute temperature for the three types of essential oils.

Table 2. *Values of E and A for various essential oils - lavender, mint and rose temperature*

Essential Oil	***E*, 10^{-20} J**	***A*, 10^{-5} mPa.s**
Rose oil	2.965	17.46
Mint Oil	4.094	33.033
Lavander Oil	2.99	184.94

Analysis of the data in Fig. 10 and Table 2 shows that the coefficients *A* for the different essential oils differ significantly, while the activation energy *E* has relatively close values.

6. CONCLUSIONS

The rheological characteristics of essential oils are important for production, packaging, filling, and storage. The course of these processes, the design parameters of the working bodies of the relevant machines and devices, as well as the quality of the products obtained to a significant extent depend on the rheological properties of the processed materials.

The results of investigation of essential oils exhibits the behavior of a typical Newtonian fluid. The dynamic viscosity of decreases with increasing temperature. The findings from the study of rheological characteristics can be utilized in determining hydraulic losses and hydroconveyer equipment of essential oils in various technological facilities.

From the study of the change in viscosity with temperature change of essential oils, the parameters of Frenkel-Andrade equation for lavender, mint and rose oil.

7. ACKNOWLEDGEMENTS

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ИСТРАЖИВАЊЕ РЕОЛОШКИХ СВОЈСТАВА НЕКИХ ЕСЕНЦИЈАЛНИХ УЉА КОЈА СЕ КОРИСТЕ У КОЗМЕТИЦИ И ФАРМАЦЕУТИЦИ

Сажетак: Прераду различитих течности које се користе у индустријској производњи прате сложени физичко-хемијски, биолошки и механички процеси, чије детаљно проучавање омогућава успостављање објективне контроле и управљања технолошким циклусом производње. Реолошке карактеристике флуида који се користе у козметици и фармацеутици од посебног су значаја за процесе производње, паковања, пуњења и складиштења. Ток ових процеса, конструктивни параметри радних тијела одговарајућих машина и уређаја, као и квалитет добијених производа, у значајној мјери зависе од реолошких својстава материјала који се обрађују. Основни процеси у којима је неопходно у највећој мјери узети у обзир реолошка својства обрађиваних материјала јесу обликовање, мијешање и транспорт. При проучавању наведених процеса успоставља се веза између реолошких карактеристика материјала и технолошких параметара његове прераде. Једно од најзначајнијих својстава континуалних медија јесте вискозност. У раду се разматрају основне методе мјерења вискозности течности помоћу ротационог вискозиметра. Представљени су резултати истраживања реолошких својстава етеричних уља (ружино уље, уље лаванде и уље нане). За утврђивање зависности промјене вискозности од температуре коришћен је реометар Thermo Scientific HAAKE MARS iQ са ваздушним лежајем. Тренутно се у појединим референтним изворима могу пронаћи приближне вриједности динамичке вискозности етеричних уља, али недостају прецизне нумеричке вриједности при различитим температурама. Добијени резултати имају практичну вриједност у технолошким процесима, као и у пројектовању машина и механизама у којима се користе етерична уља.

Кључне ријечи: мјерење вискозности, температура, реолошка својства, етерична уља.

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