The method and the device for measuring thermophysical properties of liquids

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Abstract. This paper describes the new method and the device for measuring the effective thermophysical characteristics of the investigated fluid under shear flow conditions at a fixed shear rate in the gap between coaxial cylinders. The shear flow is provided by the rotation of the outer cylinder while the inner cylinder remains stationary. The measuring unit can measure the complex rheological and thermophysical characteristics of liquids in less than 30 minutes. This article describes a two-step measurement method. The first step involves the measurement of the excess temperature established because of the heat source action due to the viscous friction in shear flow of the test liquid in a layer between the cylinders; the function of the dissipative heat source in the fluid at this stage is based on the power-law. The second step includes the thermal effect on the liquid layer from the heat source (electric heater) in the inner cylinder and the registration of the temperature response in the inner cylinder layer near the heater. The experimental determination results of polyoxyethylene solution thermophysical characteristics with the addition of nanocarbon material "Taunit" are presented here.

1. Introduction

A lot of attention has been paid by scientists around the world to studying the heat transfer processes in suspensions containing nanoparticles. Scientists of Russia and CIS countries are actively working in this direction, in particular, at the Institute of computational modeling of the Siberian branch (SB) of the Russian Academy of Sciences (RAS), Siberian Federal University (Krasnoyarsk), at the S S Kutateladze Institute of Thermophysics (Siberian Division of the Russian Academy of Sciences, Novosibirsk, Russia). The analytical and numerical study of forced convection in nanofluid flows based on various liquid media has been of most importance for the research. Professor M M Safarov (Dushanbe, Tajikistan) and his followers investigate the influence of nanoparticles on the thermophysical properties of hydrazine and its derivatives depending on the concentration of nanoparticles and pressure.

The results of experimental determination of the suspensions’ thermal conductivity dependence on the shear rate was described in [1]. With an increase in the shear rate, the thermal conductivity of the liquid increases asymptotically, and the higher the volume of concentration and the larger the volume of particles, the more intense the increase in thermal conductivity. This effect is explained by the appearance of microconvection due to the increase in the angular velocity of the particle and by the appearance of "micro-swirls" around it. Thus, the measuring device used in the conditions of shear flow
allows to measure the effective thermal conductivity of the liquid with the presence of microparticles in it, which determines the heat transfer due to both – the thermal conductivity and the convection.

The intensity of heat exchange in liquids with nanoparticles at their boiling state was also confirmed in [2,3]. The effective thermal conductivity of suspensions based on oil and carbon nanotubes was studied in [4]. The authors of the article [4] found an abnormal increase in the effective thermal conductivity in comparison with the calculated values as compared with other nanosuspensions.

This paper discusses the method and the device, which differ from the known analogues in the ability of evaluating not only the thermal conductivity, as well as the thermal diffusivity, heat capacity, and rheological characteristics of the fluids, including the non-Newtonian ones.

2. Materials and methods

Figure 1 shows a diagram of the measuring device, which has two coaxial cylinders between which the test liquid is located. To ensure the measurements under conditions of high temperatures, the inner cylinder made of heat-resistant material – Polyether ether ketone (PEEK). It can withstand prolonged thermal exposure, while maintaining their mechanical properties, has resistance to thermal oxidative degradation, chemically inert, easily shaped and processed [5-7].

Cylinder 1 has primary and protective heaters, and its design provides the ability to measure the temperatures of these heaters [6]. The placement of the heaters in the inner fixed cylinder allows to stabilize the flow regime of the investigated liquid in the gap between the cylinders, while they are protected from possible impact from the investigated liquid by a sleeve made of heat-conducting material (aluminum) [8]. It should be noted that the thermal resistance converters capable of registering the temperature over time are located in the layers of the primary 5 and the protective 4 heaters.

![Diagram of the measuring device](image)

**Figure 1.** Diagram of the measuring device: 1 – the inner (stationary) cylinder; 2, 6 – protection sleeves; 3 – the investigated viscous liquid containing nanoparticles; 4 – protective heater; 5 – primary heater; 7 – the outer (rotating) cylinder.

The measurement of thermophysical characteristics of “a viscous liquid containing nanoparticles” (further nanofluid) were measured in several stages.

After establishing the temperature field that occurs due to the dissipative heating of the nanofluid layer during the shear flow (the first stage), the main heater is switched on, creating a temperature field in the measuring device, which is superimposed on the existing flow (the second stage) (figure 2) [7]. It is advisable to separate in time the onset moments action of these heat sources, applying the principle of superposition temperature fields [9-11].
At the preparatory step of measurement considering the outer cylinder rotating at a known angular velocity $\Omega$, the shear rate $\gamma$ can be estimated as follows:

$$\gamma \approx \frac{\Omega \cdot R_3}{R_3 - R_2}.$$  

After a certain interval of time $\tau_1$, a stationary temperature field is established in the layers of the measuring device, conditioned by the dissipation of the energy of the viscous friction forces and microconvection due to the availability of nanoparticles in the liquid layer during the shear flow [6,7]. We will consider this temperature field as the base one.

Differential equations describing the stationary temperature field in layers 6 (protective sleeve) and 3 (investigated nanofluid) of the measuring device (figure 1) in the steady-state stationary mode of the preparatory stage of measurements will take the following form [6]:

$$\begin{align*}
\frac{d}{dr} \left( r \frac{dt_1(r)}{dr} \right) &= 0, & R_1 < r < R_2, \\
\frac{d}{dr} \left( r \frac{dt_2(r)}{dr} \right) &= W(r), & R_2 < r < R_3.
\end{align*}$$  

(1)

The boundary condition is as follows:

$$-\lambda_1 \frac{dt_1(R_1)}{dr} = q_1(R_1) = 0,$$

(2)

$$t_1(R_2) = t_2(R_2), \quad \lambda_1 \frac{dt_1(R_2)}{dr} = \lambda_x \frac{dt_2(R_2)}{dr},$$

(3)

$$t_2(R_3) = t_0,$$

(4)

where $r$ is the radial coordinate:

$$W(r) = mr^{n+1} \left( 2\omega \frac{r^{-2n}}{m^r(R_s^{-2n} - R_3^{-2n})} \right)^{n+1}$$

– the function of the heat source due to the dissipation of viscous friction energy and microconvectionon account to the presence of nanoparticles in the layer of the studied liquid, subject to the power rheological law [7,8]; $m$ – the coefficient of consistency of a viscous liquid, Pa·s$^n$; $n$ – the index of the fluid flow [13].

The additional condition can be written like this:

$$t_1(R_1) = t_0.$$

(5)

Where $t_0$ is the experimentally determined temperature of the main heater winding.

The general solutions to the differential equations (1) obtained after integration can be expressed in the following form:
The following function is introduced:

$$\lambda_\kappa = \frac{C_1 \lambda_1}{C_3 - R_2^{2n}} \Phi(\omega)(2/n + 1).$$

(7)

Applying the general solutions (6) and the boundary conditions (2)-(4) we obtain the calculated dependence for the thermal conductivity coefficient of a viscous non-Newtonian fluid:

$$\lambda_\kappa (\tau) = \frac{C_1 \lambda_1}{C_3 - R_2^{2n}} \Phi(\omega)(2/n + 1).$$

(8)

The results of thermal conductivity measurements calculated by this formula have significant uncertainty (error) in the case of small values of the consistency coefficient of the liquid material.

The non-stationary temperature field in the measuring device after switching on the heater will be described by the following mathematical model:

$$\tau_1, 0 \leq \tau_1, (1, \lambda), T_{r_1}, (1) = 0,$$

$$\tau_2, 0 \leq \tau_2, (1, \lambda), T_{r_2}, (1) = 0,$$

where $\tau_1$ – conventionally taken as the starting point ($\tau_1 = 0$).

The boundary condition is as follows:

$$-\lambda_1 \frac{dT_1(R_1, \tau)}{dr} = q_1(R_1, \tau) = \frac{P}{S},$$

(11)

Where P is the heater power, W; S – surface area of the main heater, m².

$$T_1(R_1, \tau) = T_2(R_2, \tau), \quad \lambda_1 \frac{dT_1(R_2, \tau)}{dr} = \lambda_2 \frac{dT_2(R_2, \tau)}{dr},$$

(12)

$$T_2(R_3, \tau) = 0.$$  

(13)

The additional condition is given in the following form:

$$T_1(R_1, \tau) = T_1(\tau).$$

(14)

Using the integral Laplace transform $T^*(r, p) = \int_0^\infty T(r, \tau) \cdot e^{-pr} d\tau$, this model can be reduced to:

$$pT_1^*(r, p) = a_1 \frac{d}{dr} \left[ r \frac{dT_1^*(r, p)}{dr} \right], \quad R_1 < r < R_2,$$

$$pT_2^*(r, p) = a_2 \frac{d}{dr} \left[ r \frac{dT_2^*(r, p)}{dr} \right], \quad R_2 < r < R_3.$$
\[ T_2'(R_2, p) = 0, \]

where \( T^* \) – time integral temperature characteristic, °C·s; \( p \) – Laplace transform parameter, s\(^{-1}\).

The additional condition represents the result of experimental temperature measurement in a layer of the internal cylinder with radius \( R_1 \):

\[ T_1'(R_1, p) = T'_e. \]

The General solution of differential equations (15) can be written according to [12]:

\[ T_i(r, p) = C_{2i-1}I_0(r \sqrt{ \frac{p}{a_i} }) + C_{2i}K_0(r \sqrt{ \frac{p}{a_i} }), \tag{15} \]

where \( C_1, C_2, \ldots C_4 \) – constant coefficients; \( I_0(x), K_0(x) \) – modified zero-order Bessel and Hankel functions, and \( I'_0(x) = I_1(x), K'_0(x) = -K_1(x) \).

After substituting into the boundary and additional conditions (9–15), the following equations can be obtained:

\[
\begin{align*}
\lambda_x \cdot (C_2K_1(R_1 \sqrt{ \frac{p}{a_1} }) - C_1I_1(R_1 \sqrt{ \frac{p}{a_1} })) &= \lambda \cdot \frac{q_1}{p}; \\
C_1 \cdot I_0(R_2 \sqrt{ \frac{p}{a_1} }) + C_2K_0(R_2 \sqrt{ \frac{p}{a_1} }) &= C_1I_0(R_2 \sqrt{ \frac{p}{a_2} }) + C_4K_0(R_2 \sqrt{ \frac{p}{a_2} }); \\
\lambda_x \cdot [C_1I_1(R_2 \sqrt{ \frac{p}{a_1} }) - C_2K_1(R_2 \sqrt{ \frac{p}{a_2} })] &= \lambda \cdot \frac{q_1}{p} - C_4K_1(R_2 \sqrt{ \frac{p}{a_2} }); \\
C_3 \cdot I_0(R_3 \sqrt{ \frac{p}{a_3} }) + C_4K_0(R_3 \sqrt{ \frac{p}{a_3} }) &= 0; \\
C_1 \cdot I_0(R_1 \sqrt{ \frac{p}{a_1} }) + C_2K_0(R_1 \sqrt{ \frac{p}{a_1} }) &= T'_e.
\end{align*}
\]

This system of five equations has six unknowns: \( C_1, C_2, C_3, C_4, \lambda_x, a_x \). Given that the characteristics \( \lambda_x, a_x \) should not depend on the parameter \( p \), it’s possible to set two values \( p_1 = p \) and \( p_2 = kp, k > 0 \), to obtain ten equations with ten unknowns: \( C_1(p_i), \ldots C_4(p_i), \lambda_x, a_x \), where \( i = 1, 2 \) and solve this system with respect to \( \lambda_x \) and \( a_x \).

At the stationary step of the measurement, the desired thermal conductivity of the liquid can be estimated by solving the following inverse boundary value problem:

\[
\begin{align*}
\frac{\partial}{\partial r} \left( r \frac{\partial T_1}{\partial r} \right) &= 0, \quad R_1 < r < R_2, \\
\frac{\partial}{\partial r} \left( r \frac{\partial T_2}{\partial r} \right) &= 0, \quad R_2 < r < R_3.
\end{align*}
\]

The boundary condition is as follows:

\[ -\lambda_x \frac{\partial T_1}{\partial r}(R_i) = q_i(R_i) = \frac{P}{S}, \]

\[ T_1'(R_2) = T_2'(R_3), \quad \lambda_x \frac{dT_1}{dr}(R_2) = \lambda_x \frac{dT_2}{dr}(R_3), \]

\[ T_2(R_3) = T_e = 0. \]

The additional condition can be expressed like this:
The solution of this problem gives the following calculated relationships for thermal conductivity:

\[
\lambda_x = \frac{R_0 \lambda_1 \cdot \ln \left( \frac{R_2}{R_1} \right)}{R_1 \ln \left( \frac{R_1}{R_2} \right) - ST \lambda_1} \quad (16)
\]

The method for evaluating the thermal conductivity and thermal diffusivity values of the test material at a given shear rate according to the experimental data of the non-stationary measurements phase can be described as follows:

1. Setting the input variables: \(R_1, \lambda_1, a_1, R_2, P, I, R_3, p, k\).

2. Calculating the parameters (functions):

\[
g_{11}(p) = R_1 \cdot \frac{p}{a_1}; \quad g_{21}(p) = R_1 \cdot \frac{p}{a_1}; \quad h = R_3/R_2; \quad q = P/\pi \cdot 2 \cdot R_1 \cdot l.
\]

3. Reading temperature values from an array file \(T_j(\tau_i)\), where \(j = 1, 2; i = 1, 2, \ldots, n\); \(n\) – number of measurements.

4. Calculating the time integral characteristics of the temperature \(T^*_1(R_1, p) = \int_0^{\infty} T(R_1, \tau_i) \cdot e^{-p \tau} d\tau\),

\[
T^*_2(R_1, kp) = \int_0^{\infty} T(R_1, \tau_i) \cdot e^{-kp \tau} d\tau \quad (i = 1, 2, \ldots, n)
\]

using the Simpson method:

\[
T^*_j(R_1, p_j) = \frac{\Delta \tau_2}{3} \left( T(R_1, 0) e^{-p \tau_{j0}} + T(R_1, \tau_{j0}) e^{-p \tau_{j1}} + \sum_{i=2}^{n/2} (4T(R_1, \tau_{2i-1}) e^{-p \tau_{2i-1}} + 2T(R_1, \tau_{2i}) e^{-p \tau_{2i}}) + \frac{T(R_1, \tau_{2n}) e^{-p \tau_{2n}}}{p_j} \right), \quad j = 1, 2, \quad p_1 = p, \quad p_2 = kp.
\]

5. Calculating the function values: in order to simplify the calculation, we set

\[
f(x) = I_0(x) \cdot K_0(x) + I_1(x) \cdot K_1(x),
\]

where \(I_0, I_1\) – modified zero-order and first-order Bessel functions; \(K_0, K_1\) – modified Hankel functions of zero and first order; \(x\) – is a variable, \(x = p, kp\).

\[
C_1(p) = -\frac{R_1 \cdot q_1 \cdot K_0(g_{11}(p)) - T^*_1(R_1, p) \cdot p \cdot \lambda_1 \cdot g_{11}(p) \cdot K_1(g_{11}(p))}{p \cdot \lambda_1 \cdot g_{11}(p) \cdot f(g_{11}(p))},
\]

\[
C_2(p) = \frac{R_1 \cdot q_1 \cdot I_0(g_{11}(p)) + T^*_1(R_1, p) \cdot p \cdot \lambda_1 \cdot g_{11}(p) \cdot I_1(g_{11}(p))}{p \cdot \lambda_1 \cdot g_{11}(p) \cdot f(g_{11}(p))},
\]

\[
C_1(kp) = -\frac{R_1 \cdot q_1 \cdot K_0(g_{11}(kp)) - T^*_2(R_1, kp) \cdot p \cdot \lambda_1 \cdot g_{11}(kp) \cdot K_1(g_{11}(kp))}{k \cdot p \cdot \lambda_1 \cdot g_{11}(kp) \cdot f(g_{11}(kp))},
\]

\[
C_2(kp) = \frac{R_1 \cdot q_1 \cdot I_0(g_{11}(kp)) + T^*_2(R_1, kp) \cdot k \cdot p \cdot \lambda_1 \cdot g_{11}(kp) \cdot I_1(g_{11}(kp))}{k \cdot p \cdot \lambda_1 \cdot g_{11}(kp) \cdot f(g_{11}(kp))}.
\]

The \(g_x\) parameter is calculated as the root of the equation:

\[
\frac{C_2(p) \cdot K_1(g_{21}(p)) - C_1(p) \cdot I_1(g_{21}(p))}{C_4(p) \cdot K_1(g_x) - C_3(p) \cdot I_1(g_x)} \times \frac{C_4(kp) \cdot K_1(h \cdot g_x \cdot \sqrt{k}) - C_3(kp) \cdot I_1(h \cdot g_x \cdot \sqrt{k})}{C_2(kp) \cdot K_1(g_{21}(kp)) - C_1(kp) \cdot I_1(g_{21}(kp))} = 1.
\]
The desired values of the thermal diffusivity and the thermal conductivity are defined as follows:

\[ \alpha_x = \frac{p \cdot R_x^2}{g_x}, \quad (17) \]

\[ \lambda_x = \frac{C_4(p) \cdot K_1(g_21(p)) - C_1(p) \cdot I_1(g_21(p))}{C_4(p) \cdot K_1(g_21) - C_3(p) \cdot I_1(g_21)}. \quad (18) \]

The effective thermophysical characteristics of the fluid under study are determined under shear flow conditions and are related to a fixed shear rate.

3. Results and discussion

Using the described measurement setup and measurement method were investigated the thermophysical and rheological characteristics of a 10 % solution of polyoxyethylene in water containing 0.5 % of carbon nanomaterial "Taunit" which is a quasi-one-dimensional, nano-scale, filamentary formations polycrystalline graphite predominantly cylindrical shape with an inner channel (carbon nanotube). The inner diameter of the nanotubes is 10-20 nm, the outer diameter is 40-50 nm, and the length is more than 2 \( \mu \)m.

Polyoxyethylene (POE) at room temperature – is a flexible thermoplastic white color with a melting point \( \approx 70 ^\circ C \). It is produced by catalytic polymerisation of ethylene oxide \( C_2H_4O \). Used as binder and modifier for different materials. At high shear rates, the destruction of molecular chains is observed.

When measuring the test material with a volume of 25±1 ml was placed in the gap between the inner and outer cylinders of the measuring device, after which the measurements were performed based on the technique described elsewhere [6]. For each shear rate, a series of three measurements were carried out, the results of which were calculated arithmetic mean values of thermal conductivity and thermal diffusivity.

Joint measurement of the shear rate \( \gamma \) and the torque transmitted to the inner cylinder due to forces of viscous friction of the investigated liquid material allow us to find the dependence of the shear stress \( \sigma \) versus \( \gamma \), which makes possible the determination of the rheological characteristics (parameters of power-law): the consistency coefficient \( m \) and the flow index \( n \), using method of approximation. The shear rate of the investigated fluid in the gap between the cylinders varied from 0 to 150 s\(^{-1}\). The dependence of the shear stress \( \sigma \) versus shear rate in rheology is called the "flow curves". Their analysis for the test material leads to the conclusion that the fluid has significant non-Newtonian properties and flow curves can be approximated by the exponential equation \( \sigma = m \cdot \gamma^n \). Here are the values of \( m \) and \( n \) for POE solution at a temperature of 30° with concentrations of nanomaterials "Taunit" 0 % and 1.5 % (Table 1).

| Table 1. Rheological characteristics of the investigated liquid |
|---------------------------------------------------------------|
| Concentration of "Taunit, % | The consistency coefficient \( m, \text{Pa} \cdot \text{s}^n \) | The flow index \( n \) |
|---------------------------|--------------------------|-------------------|
| 0                         | 20                       | 0.92              |
| 1.5                       | 31                       | 0.85              |

Figure 3 shows the dependences versus the shear rate of the thermal conductivity calculated by the formula (15). The values calculated by the formula (18) had a much larger spread in the sample of three dimensions in the series.
The thermal diffusivity values of the studied material at zero shear rate, calculated by the formula (16), for the "Taunit" solutions with concentration 0 % and 1.5 % were equal to $(1.2\pm0.1)\times10^{-7}$ m$^2$/s and $(1.3\pm0.1)\times10^{-7}$ m$^2$/s, respectively. The dependence type of the thermal conductivity versus the shear rate (at various shear rates) qualitatively coincided with the dependencies shown in figure 3. Such dependence pattern confirms the measurement results of the suspensions’ thermal characteristics at shear flow [1].

The effective values increase of the thermophysical characteristics with the growing shear rate can be explained by the heat transfer intensification in the liquid layer due to violation of the laminarity flow and the convective component flow. The presence of suspended particles (carbon nanotubes) in the liquid layer increases the value of the apparent thermal conductivity, which can be used to increase the efficiency of heat transfer from heated surfaces in the heat exchangers.

The analysis of arising measurement errors was carried out taking into account the results of experimental determination of thermophysical and rheological liquid characteristics with well-known thermophysical and rheological characteristics (glycerin, distilled water, 96 % ethyl alcohol), as well as the results of extended uncertainty (error) measurement evaluation. The results of this analysis showed that the developed measuring device has the following metrological characteristics, as presented in Table 2.

The thermal conductivity calculation using the formula (7) is possible only for fluids, as their flow curves follow the power law, and the heating of the liquid due to forces of viscous friction is greater than 2 °C. This is possible for liquids with high consistency and at significant shear rates. The measurement error in this case will be so significant that makes this method applicable only for preliminary evaluation of the thermal conductivity coefficient.

The thermophysical characteristics calculation using the formulas (15-18) is possible for liquids with any flow law. A significant measurement time remains as one of the disadvantages (at least half an hour).

Figure 3. The dependence of the modified polyoxyethylene solution’ thermal conductivity versus the shear rate.
Table 2. Metrological characteristics of the measuring unit.

| Measured physical quantity                                      | Duration of measurement | Measuring range | δ, % |
|------------------------------------------------------------------|-------------------------|-----------------|------|
| Thermal conductivity (non-stationary stage) – \( \lambda \), \( W/(m \cdot K) \) | up to 20 min            | 0.1-0.8        | ±9   |
| Thermal conductivity (stationary stage) – \( \lambda \), \( W/(m \cdot K) \) | up to 60 min            | 0.1-0.8        | ±6   |
| Coefficient of thermal diffusivity, – \( a \cdot 10^{-7} \), \( m^{2}/sec \) | up to 20 min            | 0.8-5        | ±7   |
| Volumetric heat capacity– \( c_p \cdot 10^{6} \), \( J/(m^{3} \cdot K) \) | up to 20 min            | 1-5          | ±10  |
| Shear stress– \( \sigma \), Pa                                | up to 1 sec             | 10^{-10^{4}}  | ±0.5 |
| Temperature – \( T \), °C                                       | -                       | 20-250     | ±0.5 |
| Shear rate – \( \gamma \), s^{-1}                             | -                       | 0-150      | ±5   |

4. Conclusion
Thus, hus, a method and apparatus have been developed which allow measuring with an error of not more than 10% the effective thermophysical and rheological characteristics of liquid phase materials and suspensions under shear flow conditions. Experimental data are presented for 10% polyoxyethylene solution containing additives of carbon-containing nanomaterial “Taunit”, as well as without them.

The results of experimental studies show an increase in effective values of thermal conductivity and coefficient of thermal conductivity at shear flow and at presence of nanoparticles in the liquid layer.

The presented method and measuring device are relevant for enterprises, technological processes of which include non-isothermal flows of liquid materials with an unstable structure through channels of different shapes.

Knowledge of thermophysical characteristics of processed materials will help to optimize these processes.

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