

## SIMPLIFIED MEASUREMENTS OF ABSOLUTE CHARACTERISTICS OF PASTING

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Key words: starch, pasting characteristics, viscometry

The construction and the principle of the operation of the rotor using of which significantly simplified the measurements of the absolute pasting characteristics are presented. The rotor can be also useful in rheology measurements of dispersion which sedimentate.

### INTRODUCTION

Viscosimetric studies of gelatinization of starch are based on the purely empirical, comparative method of the measuring of pasting characteristics. The latest presents a relation between indications of viscograph, temperature and time [1-4]. Unfortunately, complicated geometry and nonstationary flow of sample make impossible any attempt of the ascribing of the precisely defined meaning to the obtained results [5, 6]. For this sake, there is a possibility of neither correct interpretation of the characteristics of pasting nor comparison of results measured by instruments of different types.

The method of the measurements of the absolute pasting characteristics proposed by Winkler [7] and modified by Koubek [8] allows to ascribe a physical sense to the results of these measurements. The method involves a typical rotatory viscometer with co-axial cylinders. A preliminary gelatinization of suspension is achieved under conditions of a turbulent flow caused by rotation of the stirrer. The essential part of the measurement begins when the gelatinization holds up sedimentation of swollen starch grains. At this moment the former stirrer is replaced by the standard rotor in form of a cylinder. Since further gelatinization proceeds under conditions resembling simple shear indications of viscometer that may directly be expressed by the viscosity units. The device measures the magnitude close to the apparent viscosity of the paste.

However, this method is only randomly used in a laboratory because of the methodically troublesome change of the rotor on the course of the measurement. Also the moment most suitable for this change is not clearly defined. These facts prompted me to simplify the method by the use of specially constructed rotor. It combines the role of the measurement cylinder and stirrer. At high angular

velocity it acts as the centrifugal, hypotential stirrer providing circulation and homogeneity of starch suspension. For low velocity the rotor behaves like the typical rotating cylinder. In this manner the smooth, unperturbed run of the experiment is achieved.

## APPARATUS AND MEASUREMENT METHODS

### CONSTRUCTION OF THE ROTOR

Fig. 1 presents details of the construction of the rotor. It is a thin-wall cylinder [5] of 15 mm radius and 40 mm height, closed from the top with a cap [2] connected to the axis of viscometer. It has 8 square windows in its upper part which are positioned symmetrically round the rotor. A four-armed stirrer [4] is located inwardly. The chamber of the stirrer has the cylindrical shape and it forms in its lower part a suction channel [6]. The shape of the bottom of the cylinder [7] provides the formation of the air cushion. It eliminates a majority of so-called end effects of the measurement. The external wall of the cylinder [5] is rugged in 0.3 mm deep slots which cut the vertical the wall [2-3 slots per 1 mm of the perimeter].

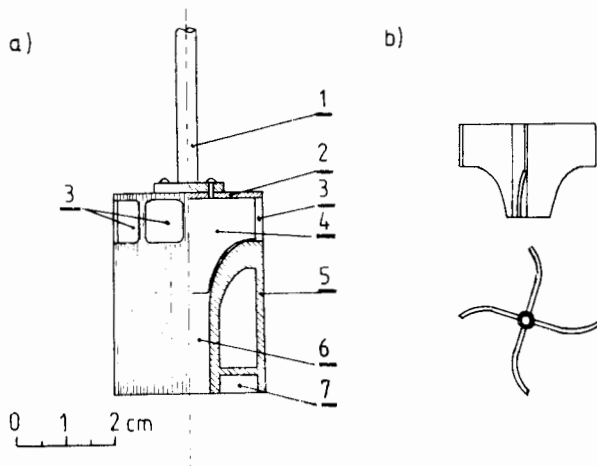


Fig. 1a. The construction of the rotor (a half-bisecting mode of presentation); 1 — the axis of rheometer, 2 — the cap, 3 — the admission window, 4 — the stirrer, 5 — the wall of cylinder, 6 — the suction channel, 7 — the chamber controlling the bottom effects;

Fig. 1b. The centrifugal stirrer (see Fig. 1a)

### PRINCIPLE OF THE WORK OF THE ROTOR

The rotor is fixed on the rotation axis of the viscometer in the stator. Investigated liquid fills the space between the rotor and the stator as well as interior of both the rotor and the suction channel. The circulation of the rotor causes the rotary flow of liquid in the gap between the stator and the rotor as well

as inside of the rotor. In a consequence this liquid is evacuated out of the rotor through its windows. It reduces the pressure inside the suction channel and therefore the liquid is uptaken into the rotor from the bottom of the stator. The circulation of the liquid is presented in Fig. 2.

For the high angular velocity of the rotor (over about 100 cycles per min) an additional circulation of the sample prohibits the sedimentation of the grains of aqueous starch suspension. As the velocity of the rotor decreases the effectiveness of the stirring is decreased. When the velocity reaches some scores of cycles per min the additional circulation ceases. Hence the rotor behaves as a typical cylindrical rotor.

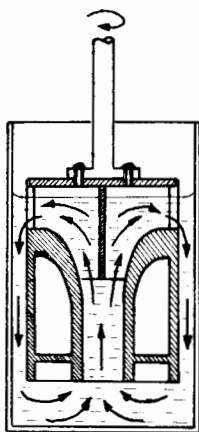


Fig. 2. The additional circulation of the sample evoked by the move of the rotor

## RESULTS AND DISCUSION

### CONTROL RUNS

#### *Viscosity measurement*

Control runs were carried out for a few selected Newtonian liquids of known viscosity. The obtained results by means of the standard cylinder and tested rotor closely follow one another in a wide range of angular velocity in the area of the laminar flow. The deviations reaching few percent appear just at the end of the laminar flow when the effect dealing with the additional circulation caused by the construction of the rotor becomes essential. Thus, using this rotor at low velocity an absolute run of viscosity may be carried out. The value of terminal velocity may be calculated by means of the Wein's and Mitschka's version of the Taylor's criterion [9].

#### *Wall effect*

The multiphase structure of gels formed has some specific attributes influencing the measurements. Among them the separation of phases has to be

taken into consideration as it causes an effective slipping at the surface of the rotor. The results univocally demonstrate such slipping at the surface of non-rugged rotor which takes place on the runs of characteristics of pasting as well as flow curves. The nature of observed differences was rather clear in the measurements of flow curves. The results for 2.5 % potato starch paste at 40° C are shown as an example in Fig. 3.

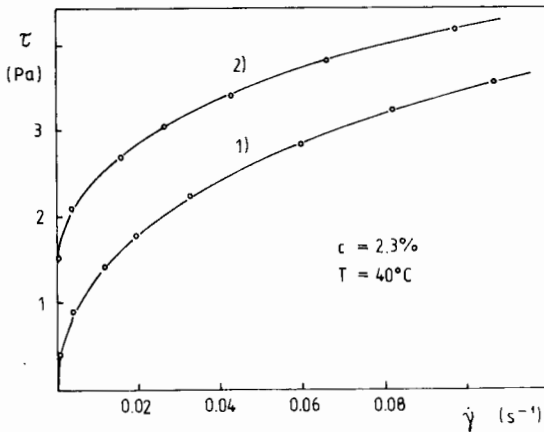


Fig. 3. The influence of the slipping on the pattern of the flow curve; 1— with the slipping (the non-gritty rotor), 2 — without slipping (the rugged rotor)

It may be seen that effective slipping introduces essential error in the evaluation of the rheological properties of gel. The pattern of the flow curve found with the rotor of blank surface is typical for non-Newtonian shear-thinning fluids although as a matter of fact gel is viscoplastic liquid with clearly observable yield stress  $\tau_y$ .

#### *Absolute characteristics of pasting*

In typical rotatory viscometers working at constant angular velocity  $\Omega$  the change of the function of the rotor (stirring — measurement) is accompanied by the change of the velocity  $\Omega$ . The optimal temperature for the most suitable moment of this change is set experimentally. The run is still simpler in a case of the use of the rotatory viscometer working with constant torque [10]. The adjustment of proper torque providing high preliminary velocity of the rotor is a sufficient manipulation in this case. The decrease of the velocity  $\Omega$  at the beginning of the proper measurement takes place automatically as the viscosity of gelating suspension increases. The obtained results with such viscometer [11] are shown in Fig. 4. The characteristic of pasting was run for the 2.3% suspension of potato starch at constant torque  $1.5 \times 10^{-4}$  Nm. In range of the laminar flow the characteristic shows changes of viscosity determined as a ratio of the shear stress  $\tau$  to the Newtonian rate of shear  $\dot{\gamma}_N$ . Unfortunately, it is not

strictly the apparent viscosity of the studied gel. For non-Newtonian liquids a determination of the reliable rate of shear  $\dot{\gamma}$  value by the one-point measurement method is impossible [12].

It should be mentioned that the described rotor may be suitable for rheological studies of the other dispersions which sedimentate during the long measurement. Up to date solutions of this problem were based on the capillary viscosimetry [13].

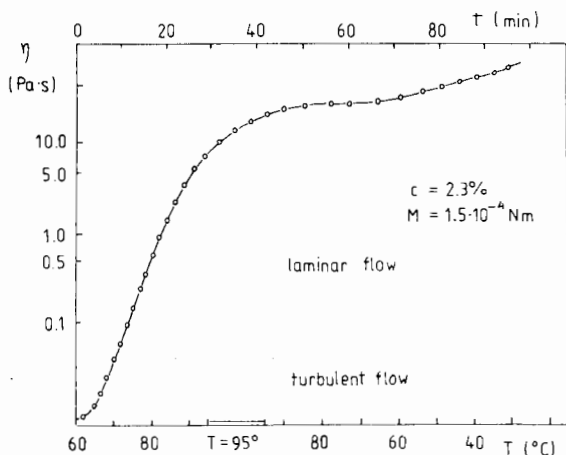


Fig. 4. The characteristic of pasting of potato starch measured by means of viscometer with constant torque and the rotor described in this paper

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## UPROSZCZENIE METODYKI POMIARU ABSOLUTNYCH CHARAKTERYSTYK KLEIKOWANIA

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### Streszczenie

Wyniki pomiarów charakterystyk kleikowania skrobi, prowadzonych wiskozymetrem rotacyjnym o współosiowych cylindrach mogą być wyrażone w absolutnych jednostkach lepkości. Przy wykorzystaniu rotora o przedstawionej konstrukcji, mogącego pełnić funkcję tak mieszadła jak i cylindra pomiarowego, metodyka pomiaru charakterystyk ulega znacznemu uproszczeniu. Zmiana warunków pracy rotora (mieszanie-pomiar) odbywa się wyłącznie przez zmianę jego prędkości obrotowej bez konieczności ingerencji w układ pomiarowy.

Rotor może być stosowany również w badaniach właściwości reologicznych układów dyspersyjnych ulegających procesowi sedymentacji.