Methods for the rheological characterization of food

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Abstract: *The present paper represents a summary of some rheological methods available for food characterization. Rheology, meaning "the science of the deformation and flow of matter", is studying the mechanical properties of gases, liquids, plastic, liquids etc.. The rheological data are very useful for food industry (calculates for a wide range of equipments, for the evaluation of food texture, shelf life testing of foods, intermediate or final control quality of food). The rheological methods can be divided into two major categories: rotational type and tube type. The tube type methods involves: glass capillary, high pressure capillary and pipe. The rotational type methods involve: cone and plate viscometry, parallel plate viscometry, concentric cylinder viscometry and mixer viscometry).*

Key: *rheology, viscometry, tube viscometry, rotational viscometry*.

1. Introduction

Rheology is now well established as the science of the deformation and flow of matter: It is the study of the manner in which materials respond to applied stress or strain [18].

Food rheology is the study of the manner in which food materials respond to an applied stress and strain. The food rheology has many applications in the fields of food acceptability, food processing, and food handling. Determination of rheological properties of foodstuffs provides an instrumental quality control of raw material prior to processing, of intermediate products during manufacturing, and on finished goods after production [1].

Rheology, meaning "the science of the deformation and flow of matter", is studying the mechanical proprieties of gases, liquids, plastics, asphalt materials, crystalline materials and others. So, the field of rheology is extends from the Newtonian fluids mechanic to the Hooke elasticity. The region between them, correspond to the flow and the deformation of all the liquid materials and suspensions [2].

The first use of the word "rheology" is credited to Eugene C. Bingham (circa 1928) who also described the motto of the subject as $\pi\alpha\upsilon\tau\alpha\rho\varepsilon\iota$ ("panta rhei", from the works of Heraclitus, a pre-Socratic Greek philosopher active about 500 B.C.) meaning "everything flows" [14].

Rheology has important applications in engineering, geophysics and physiology. In particular, hemorheology, the study of blood flow, has an enormous medical significance. In geology, solid Earth materials that exhibit viscous flow over long time scales are known as rheids. In engineering, rheology has had its predominant application in the development and use of polymeric materials (plasticity theory has been similarly important for the design of metal forming processes, but in the engineering community is often not considered a part of rheology).

Food rheology is the study of the rheological properties of food, that is, the consistency and flow of food under tightly specified conditions [12] The consistency, degree of fluidity, and other mechanical properties are important in understanding how long food can be stored, how stable it will remain, and in determining food texture. The acceptability of food products to the consumer is often determined by food texture, such as how spread able and creamy a food product is. Food rheology is important in quality control during food manufacture and processing [10].

Rheological properties of biological fluids can vary greatly, even within the same general product categories such as applesauce, ketchup or chocolate; hence it is important that rheological behaviour be carefully evaluated for all new products [17].

For the food industry, the rheological data are needed in numerous areas:

a. Process engineering calculations involving a wide range of equipment such as pipelines,

pumps, extruders, mixers, coaters, heat exchangers, homogenizers, calenders, and on-line viscometers;

b. Determining ingredient functionality in product development;

c. Intermediate or final product quality control;

d. Shelf life testing;

e. Evaluation of food texture by correlation to sensory data;

f. Analysis of rheological equations of state or constitutive equations [18].

1.1. Particular rheological materials

The simplest bodies studied by the rheology have just one property. Their behaviour is described with a linear law. These are the materials with unitary proprieties, with ideal behaviour:

- Newton fluid, pure viscous
- Hooke's solid, perfect elastic
- St. Venant's plastic, perfect plastic.

The bodies with unitary proprieties, under an acting deformation, deform. At constant strain, the Newtonian fluids flow with a constant speed. The deformation is unrecoverable. Hooke's solid has an elastic deformation, recoverable, proportional with the amplitude of the tension.

Over the stress threshold, the St. Venant's plastic is deforming unrecoverable. All the three materials have an ideal behaviour because the relationship between stress and strain, respective strain rate, is linear [19].

1.1.1. Newtonian fluid

The Newtonian fluid poses only viscosity. Under a deformation, it flows. The flow is a continuous deformation process with infinite shear rate. The law which describes the rheological behaviour includes viscosities coefficients and it is valid for steady flow [19].

1.1.2. Hooke solid

When force is applied to a solid material and the resulting stress versus strain curve is a straight line through the origin, the material is obeying Hooke's law. The relationship may be stated for shear stress and shear strain as:

$$\sigma = G\gamma \qquad (1)$$

where G is the shear modulus.

Hookean materials do not flow and are linearly elastic. Stress remains constant until the strain is removed and the material returns to its original shape. Sometimes shape recovery, though complete, is delayed by some atomistic process. This time-dependent, or delayed, elastic behaviour is known as inelasticity.

Hooke's law can be used to describe the behaviour of many solids (steel, egg shell, dry pasta, hard candy, etc.) when subjected to small strains, typically less than 0.01. Large strains often produce brittle fracture or non-linear behaviour [15].

1.1.3. St. Venant plastic

The perfect plastic materials deform unrecoverable after the effort has reached the stress threshold.

The plasticity represents behaviour of some materials. The metals are the materials which have elasticity and plasticity [15].

2. Methods for the rheological characterization of food

Common instruments, capable of measuring fundamental rheological properties of fluid and semi-solid foods, may be placed into two general categories (Fig. 1): rotational type and tube type. Most are commercially available; others (mixer and pipe viscometers) are easily fabricated. Costs vary tremendously from the inexpensive glass capillary viscometer to a very expensive rotational instrument capable of measuring dynamic properties and normal stress differences. Solid foods are often tested in compression (between parallel plates), tension, or torsion [15].

Experimental methods of measurement of rheological properties are defined by the general term rheometry, while, a more narrowly defined term, viscometry, is typically used in measurements of viscosity.

Classification of methods of viscosity measurement is based on the geometry of flow:

Three main cases of flow are possible:

- Flow of fluid between solid surfaces or through a hole in a solid body
- Flow of fluid around a solid body
- Free stream flow, relevant only to extension of a fluid stream [12].

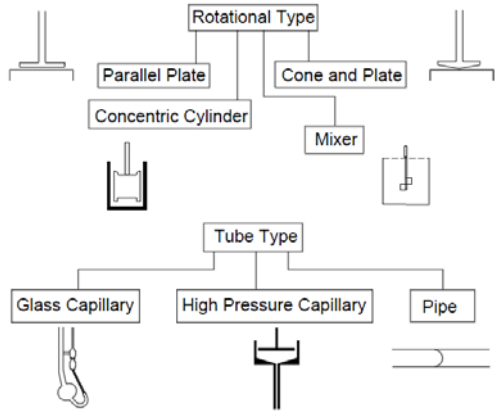

Fig. 1. Rheological instruments: Rotation Type and Tube Type.

Fluid flow between two solid surfaces can be realized in the following geometries:

- Fluid flow through a capillary with cross-section of capillary being usually, but not necessary, circular
- Rotational flow in which fluid is subjected to a circular motion in a gap between rotating cylinders, in a gap created by cone and plate or two conical surfaces, in a gap between two spherical surfaces, or other combinations of circular bodies
- Shear flow of fluid between two parallel plates
- Squeezing flow of fluid layer between two parallel plates approaching each other
- Indentation of a solid body into material

Viscosity measurement in fluid flowing around solid bodies is usually carried out according to the following schemes:

- Flow around a spherical or other surface moving in fluid with its resistance to flow depending on fluid viscosity. The space occupied by fluid may be restricted by solid walls or be infinite
- Indentation of a solid body (indentor) into the fluid layer with shapes of the indentor being different – conical, spherical, cylindrical, etc. [12].

2.1. Tube Viscometry

Tube viscometers are very useful in collecting rheological data. These instruments may be placed into three basic categories: glass capillaries, often called U-tube viscometers because of their resemblance to the letter U; high pressure capillaries; and pipe viscometers (Fig. 2). All establish a pressure difference to create flow [18].

2.1.1. Glass capillary viscometry

Capillary viscometry is the oldest and the most widely used method of qualitative estimation and viscosity measurement. The term ”capillary” usually means any tube (channel) with arbitrary length and cross-section, though, as a general rule, cylindrical tubes (capillaries) with large length-to-radius, L/R, (or diameter, D) ratio are used [12].

The determination of viscosity using a suitable capillary viscometer is carried out at a temperature of 20 ± 0.1 °C, unless otherwise prescribed. The time required for the level of the liquid to drop from one mark to the other is measured with a stop-watch to the nearest one-fifth of a second. The result is valid only if two consecutive readings do not differ by more than 1 per cent. The average of not fewer than three readings gives the flow time of the liquid to be examined.

Calculate the dynamic viscosity in millipascal seconds using the formula:

$$\eta = k\rho t \qquad (2)$$

k = constant of the viscometer, expressed in square millimetres per second squared,

ρ = density of the liquid to be examined expressed in milligrams per cubic millimetre, obtained by multiplying its relative density by 0.9982,

t = flow time, in seconds, of the liquid to be examined.

The constant k is determined using a suitable viscometer calibration liquid [5].

Glass viscometers are included in a special group because they are made out of glass and utilized are comparatively small, pressures, which cause flow. Many original constructions of capillary tube viscometers are known, but two basic versions of glass viscometers used in practice (Ostwald-Fenske viscometer and Ubbelohde viscometer) [10].

2.2. Rotational viscometry

Traditional rotational viscometers include cone and plate, parallel plate, and concentric cylinder units operated under steady shear conditions. They may also be capable of operating in an oscillatory mode which will be considered in the discussion of viscoelasticity. Cone and plate systems are sometimes capable of determining normal stress differences. Concentric cylinder systems have been used in research to evaluate these differences; however, commercial

instruments of this type are not available. Mixer viscometry, a "less traditional" method in rotational viscometry, is also presented because it has excellent utility in solving many rheological problems found in the food industry [18].

2.2.1. Concentric Cylinder Viscometry

In the concentric cylinder viscometer, a fluid is placed in the annular space between two coaxial cylinders, as Fig. 2. One of the cylinders is fixed, while to the other is applied a torque M. The performance of such an instrument will be considered when M is a constant. Let Ω be the angular velocity of the cylinder after reaching a stationary state. From the measurement of the relation between Ω and M, the flow curve of the liquid may be determined [7].

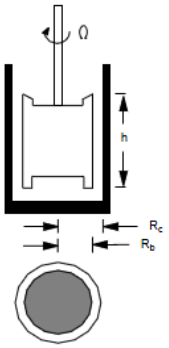

Fig. 2. Typical concentric cylinder viscometer.

It is easy to imagine that the process of loading a sample into the cup and insertion of the bob will either destroy or alter the structure of the test fluid. Also, it may require considerable effort to insert a bob into a cup containing a very high viscosity material, for example, dough. The bob is rotated and the drag of the fluid on the bob is measured by means of a torque sensor. In the Couette geometry, the cup is rotated and the main advantage is that higher shear rates can be obtained prior to the onset of turbulence due to Taylor vortices. By changing the rotational speed (shear rate) and measuring the resulting shear stress, it is possible to obtain viscosity data over a wide range of shearing conditions. In automated viscometers, the bob is programmed for an increase-in-speed ramp up to a predetermined shear rate and a decrease-in-speed ramp back to zero speed. In controlled-stress instruments, the variable controlled is the stress (torque), and the rotational speed is the response measured [15].

When the bob rotates at a constant speed and the cup is stationary, the instrument measures the torque (M) required to maintain a constant angular velocity of the bob (Ω). The opposing torque comes from the shear stress exerted on the bob by the fluid. A force balance yields

$$M = 2\pi r h r \sigma \tag{3}$$

where r is any location in the fluid, $R_b \le r \le R_c$.

Solving the above equation, for the shear stress shows that σ decreases in moving from the bob to the cup:

$$\sigma = f(r) = \frac{M}{2\pi h r^2} \tag{4}$$

The maximum value of the shear rate achievable with coaxial cylinder viscometers is usually not an instrument limitation but depends on the nature of the fluid sample. For high viscosity liquids, viscous heating may become a problem; for low-viscosity liquids, the upper limit may be set by the occurrence of secondary flows or the transition to turbulent flow [9].

The concentric cylindrical viscometers can be divided in two categories: Searle type and respectively Couette type. In the Searle systems (Fig. 3a), the inner cylinder is rotating with a defined speed, while the outer cylinder is fixed. The rotation forces the fluid to flow between the two cylinders, leading to a torque on the inner cylinder which counteract whit the engine torque [11]

This torque is recorded by a sensor, usually a spring, placed between the engine and the inner cylinder axle. The spring torsion is direct measure of the resistance opposite by the fluid to the flow, therefore its viscosity. The engine can be designed to ensure a certain speed, or speed area; in the latter case could be performed some rheological properties series.

In the Couette systems (Fig. 3b), the outer cylinder is rotating with a certain speed, the torque is transmitted to the inner cylinder, and the sensor measures the force necessary to maintain the inner cylinder steady. The inner cylinder can be connected to a torsion bar, the device can record the bar torsion [11].

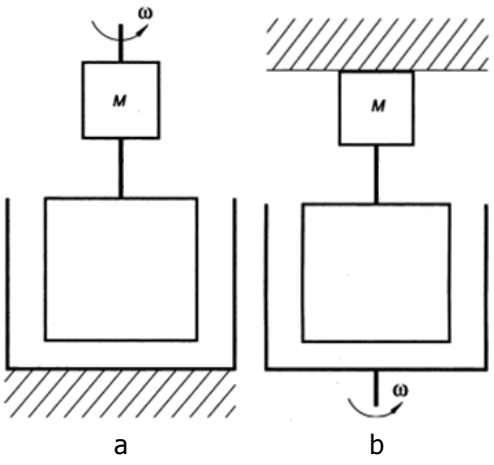

Fig. 3. Concentric cylinder viscometer a) Searle type, b) Couette type.

2.2.2. Parallel Plate Viscometry

The parallel plate system (Fig. 4.) has a constant, defined radius R and a variable plate gap H. The angular speed in the gap ω is constant in levels parallel to the plates and increases with the height:

$$\omega(h) = \omega \frac{h}{H} \tag{5}$$

The peripheral speed also depends on the height and also on the radius:

$$\omega(h) = r \cdot \Omega \frac{h}{H} \tag{6}$$

From this the shear rate is calculated:

$$\dot{\gamma} = \frac{r\Omega}{H} \tag{7}$$

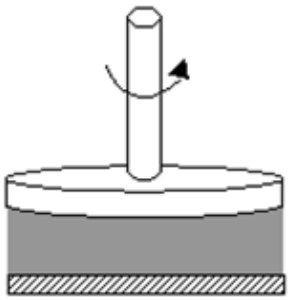

Fig. 4. Parallel Plate viscometer model**.**

In the case of the parallel plate viscometer, should be take care to ensure the gap does not become too small because then frictional effects would falsify the measuring results. As a rule of thumb, the gap should be at least five times larger than the largest particles contained in the sample. Consequently, the parallel plate viscometer is most suitable for semi-solid materials and has the added advantage of being easy to clean [4].

Equipment limitations and sources of error for the parallel plate viscometer are essentially the same as those for the cone and plate viscometer. When working with suspensions and emulsions, a common problem encountered with both types of instruments is the tendency of the sample to slip at the fixture walls; roughening the fixture surfaces or using serrated surfaces is the normal method of attempting to overcome this difficulty [4].

2.2.3. Cone and Plate Viscometry

Using a cone and plate apparatus (Fig. 5.), the shear stress versus shear rate curve may usually be obtained directly so the calculations are quite simple. In operating a cone and plate viscometer, the apex of the cone almost touches the plate and fluids fills the gap. The cone is rotate with a known angular velocity (Ω) and the resulting torque (M) is measuring on the fixed plate or through the cone. Some instruments are designed with rotating plates and fixed cones [18].

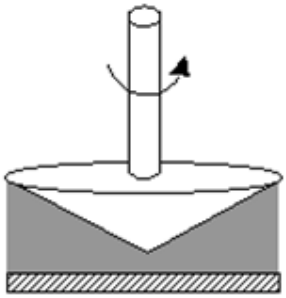

Fig. 5. Cone and Plate viscometer model.

Since the parallel plate viscometer model is more suitable for semi-solid substances and has the disadvantage of variable shear rates, it is legitimate to ask whether there are any measuring systems that do not have this disadvantage and can also measure liquids like water [4].

A major advantage of a cone and plate viscometer is the constant shear rate throughout all the liquid. This comes about become, at a fixed radial position, the circumferential velocity varies linearly across the gap between the cone and the plate. The constancy of shear rate is especially important, since it makes data analysis extremely straightforward [9].

This not only has the advantage that a constant rate prevails throughout the gap of the cone plate measuring system but also allows measurement of relatively high rates, small sample amounts and easy cleaning. But the cone plate model likewise has one minor disadvantage. Liquids like water are very difficult to handle on the bottom plate because they tend to run off the plate. During the measurement, at the latest, the sample will be expelled from the measuring gap by centripetal forces [4].

2.2.4. Mixer Viscometry

Extensive work has been conducted on mixer viscometry. The technique has been used mostly for nonreacting biological materials but also applied in evaluating the workability of fresh concrete, and to chemorheological studies involving starch gelatinization. Mixer viscometry may be useful to the food engineer in evaluating difficult fluids like those exhibiting slip or time-dependent behaviour, and those having large particles or particle settling problems. Some concepts, such as the matching viscosity method of determining the mixer viscometer constant, are also useful in developing models to simulate the shear history found in complex food processing equipment such as scrape-surface heat exchangers [18].

Mixing is a common unit operation found in the chemical and food processing industries. Mixing can be regarded as a complex process, and a thorough mathematical analysis can be difficult, rigorous, and time-consuming [6, 16].

Mixer viscometry techniques address mixer constant determination, and these procedures are useful for monitoring the viscosity of a material during a mixing process. Often, complex materials are difficult to analyze rheologically with conventional geometries. Brito De-La Fuente [3] demonstrated problems associated with measuring rheological properties of clay-particle suspensions; in particular concerns of slip can be addressed. Torque measurements were collected for helical ribbon agitators, cone and plate, and couette geometries immersed in these materials.

The conventional geometries produced unstable torque responses, while the helical ribbon generated smooth torque signals. These researchers attributed this instability to particle wall interaction and phase separation. A more effective bulk shear and improved homogenization were thought to occur with the helical ribbon agitator [8].

3. Conclusions

The rheological methods (tube or rotational type) are very useful for the food characterization. The capillary viscometry is used for the fluid with small viscosity, while the cone and plate, parallel plate, mixer viscometers are able to characterize the fluid with greater viscosity.

The cone and plate viscometer have many advantage starting with quantity needed (it is required a very small sample volume of fluid) and ending with the shear stress versus shear rate curve may usually be obtained directly so the calculations are quite simple.

The parallel plate system has the advantage of flexible gaps, which is useful for coarse dispersions.

The concentric cylinder viscometer advantage leads from being able to work with low viscosity materials and mobile suspensions. Their large surface area gives them sensitivity; therefore they will provide good data at low shear rates and viscosities.

The mixer viscometer, it is a less traditional rheological method, but it's useful to the food engineer in evaluating difficult fluids like those exhibiting slip or time-dependent behaviour.

The rheological data are very useful for food industry (calculates for a wide range of equipments, for the evaluation of food texture, shelf life testing of foods, intermediate or final control quality of food).

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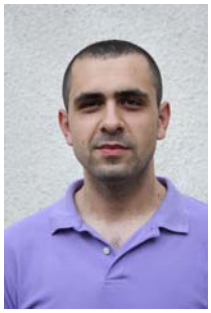

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