ABSTRACT: The falling ball viscosimeter (Hoppler) was modified to measure the rheological properties of non-Newtonian food fluids. The theory that formed the basis in developing this rheoviscosimeter was the analysis of slow falling of rigid spheres in the vicinity of non-Newtonian fluids. An electromagnetic device with automatic chronometry has permitted the determination of the corresponding shear stress and the shear rate through the terminal velocity measurements, including non transparent fluids. The falling ball rheoviscosimeter demonstrated precision when tested with a Newtonian product (glycerol) and gave flow constants not significantly different from those obtained using rotoviscosimeters for the evaluation of a non-Newtonian product (carrageenan). The rheological parameters (K, n) of carageenan, in this work, demonstrated agreement when compared with results obtained with different rheoviscosimeters. Although the modified Hoppler viscosimeter as reported here does not encompass as wide a range of shear rates as some of the more expensive instruments, its range is sufficiently large to include most food fluids in respect to the stimulations associated with the sensory evaluation of viscosity.

KEY-WORDS: reology, rheovisimeter, non-Newtonian food fluids, viscosity, shear stress, shear rate.

1 - INTRODUCTION

Food scientists and technologists, even not directly involved in food rheology, have in some moment their interest led toward the determination of flow characteristics of food fluids which they work with. These characteristics are essential in plant design, quality control and consumer acceptance.

Non-Newtonian fluids do not have a linear relationship between shear stress (τ) and shear rate (γ), that provide the absolute viscosity (μ). The relationship between (τ) and (γ) for a non-Newtonian fluid is named apparent viscosity (η) (LEWIS, 1987).

Many food fluids with non-Newtonian characteristics require rheological measurements over ranges of shear rate, temperature and concentration, to be described appropriately (BRODKEY, 1967).

The rotoviscosimeters are the most frequently used equipments in rheiological determinations. Meanwhile, the high cost of these equipments has limited their use (SHOMAEKER et al., 1987).

The theoretical analysis of the flow of non-Newtonian fluids in the vicinity of rigid spheres infers the possibility of rheological measurements of food fluids, employing a simple system of falling balls (MASSARANI & TAN, 1977).

In this work, a falling ball viscosimeter, known as Hoppler or Stokes viscosimeter (MLW, Typ. BH2 Nr. 16691), was modified and combined with an electromagnetic device to obtain an accurate, versatile and low cost system to study characteristics of food fluid flow.

2 - MATERIALS AND METHODS

2.1 - Theoretical basis

In a slow translational movement of a sphere in a fluid, in steady state regime, the shear stress (τ) is the only relevant material function in process (MASSARANI & TAN, 1977). Therefore, the shear rate:

\[ \gamma = \frac{v}{d} \cdot \theta \left( \frac{d}{D} \right) \]
3 - EXPERIMENTAL

An ultrathermostatic bath FANEN, model 111, controls the temperature of the sample. The sample volume is about 40 mL An electromagnetic device is constituted of field coil (source: 1.5 V) inserted into a "ferrite" bar. The stainless steel spheres (specific gravity 7.7734 g cm\(^{-3}\)) are held in place due to the magnetic field. A key cuts the magnetic field which releases the sphere and puts in motion a digital chronometer at the same time. The operation is finished when the sphere touches the bottom electric sensor (copper sheet in X shape) built in a "tecnil" (polyamide-Rhodia) cylindrical piece. The experimental data were obtained changing the diameter of the spheres (0.1585 to 0.7420 cm) (YABE, 1989), being 1.60 cm the internal tube diameter.

The device was tested at 25°C with a Newtonian fluid (glycerol) and a non-Newtonian (carrageenan solution at 1%) (CIALGAS, SP, BR) to evaluate their performance when applied to food fluids.

The rheological parameter n (flow behavior index) and K (consistency index) of the power law model \( (\tau = K \cdot \dot{\gamma}^n) \) were obtained by non-linear regression (SAS, 1985).

4 - RESULTS AND DISCUSSION

A comparison was conducted between the modified viscosimeter and a traditional Hoppler viscosimeter (Table 1). For this, glycerol (25°C), recommended substance to standardization (LANGE, 1973), was used.

The \( t \)-test to \( t_{0.02} \) (SPIEGLER, 1979) was used to compare the absolute viscosity obtained by the modified and traditional devices. The differences are non significant \( (p < 0.05) \) as were expected.

The report of a cooperative study under auspices of European Cooperation in Scientific and Technical Research (COST, 90) (PRENTICE & HUBER, 1983), about food rheology, indicated that a carrageenan gum (1%) solution at 25°C, showed more consistent results between the non-Newtonian food gums. The carrageenan is a hydrocolloid used as ingredient in food formulation, due to their functional properties such as thickening, gelling and emulsifying agent (FROST et al., 1984). It is used in the production of pudding, ice cream, jelly and other foodstuffs.

The European scientists (COST, 90) data to carrageenan 1% at 25°C, were modeled in this study employing the power law model through non-linear (NLIN) regression (SAS, 1985).

The experimental data \( (\tau \text{ vs } \dot{\gamma}) \) obtained in our laboratory are shown in Figure 2. These data processed by NLIN (SAS, 1985), showed a typically pseudoplastic behavior through the power law model.

For comparison purposes on evaluation of the rheoviscoimeter, we used rheological parameters \( (K, n) \) obtained at University of Nottingham, Department of Applied Biochemistry and Food Science, using the same gum and similar conditions, that may be observed in Table 2.

Both consistency indexes \( (K) \) could be inserted at confidence interval (99%) each one (SAS, 1985), and the same occurred with both flow behavior indexes \( (n) \). On the other hand, the European Commission (COST, 90) (PRENTICE & HUBER, 1983) showed that for the rheological analysis, even with carrageenan solution (1%, 25°C), conducted in several laboratories and instruments, could be expected a variation as 2.54 \( K \geq 0.91 \) and 0.61 \( n \geq 0.43 \). The rheological behavior of carrageenan gum solution (1%, 25°C reported here, employing the power law model, was in agreement with the data published in the literature (PRENTICE & HUBER, 1983).

Therefore, the evidences presented indicate a real
FIGURE 1. 1. sensor; 2. field coil + "ferrite" bar; 3. amplifier and control; 4. digital chronometer; 5. source (continuous current); 6. thermostat; 7. thermometer; 8. rod

TABLE 1 – MODIFIED VISCOSIMETER EVALUATION TO NEWTONIAN FLUID (GLYCEROL, 25°C)

<table>
<thead>
<tr>
<th></th>
<th>Traditional device (Höppler viscosimeter)</th>
<th>Modified device (falling ball rheoviscosimeter)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Absolute viscosity, mPas or cP</td>
<td>Mean 773.27</td>
<td>787.07</td>
</tr>
<tr>
<td></td>
<td>CV(^a) (%) 1.42</td>
<td>2.32</td>
</tr>
</tbody>
</table>

\(^a\) CV - coefficient of variation

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FIGURA 2. Carrageenan 1% solution at 25°C - Rheogram using the falling ball rheoviscosimeter.

TABLE 2 - RHEOLOGICAL PARAMETERS (POWER LAW MODEL) OF CARRAGEENAN GUM
1% SOLUTION (25°C, $\rho = 1.0027$ g/cm$^3$)

<table>
<thead>
<tr>
<th>Equipment</th>
<th>K (sd)</th>
<th>n (sd)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1) rotoviscosimeter$^b$</td>
<td>1.95 (0.13)</td>
<td>0.51 (0.02)</td>
</tr>
<tr>
<td>(2) rheoviscosimeter$^c$</td>
<td>1.64 (0.13)</td>
<td>0.56 (0.03)</td>
</tr>
</tbody>
</table>

$^a$ (sd) standard deviation  
$^b$ Concentric-cylinder viscosimeter and cone and plate instrument at Univ. of Nottingham  
$^c$ Modified Hoppeler viscosimeter in Univ. of Londrina laboratory
sensory evaluation of viscosity. Correlations between instrumental and organoleptic measurements may be established, if known the prevalent conditions during the sensory evaluation (RAO, 1977).

In fact, Theological behaviors were established for pear pure, sweet condensed milk and orange honey, employing the falling ball rheoviscosimeter, which showed an agreement with the data published in literature (YABE, 1989).


RESUMO: O viscosímetro de queda de esferas (Höppler) foi modificado para medir as propriedades reológicas de fluidos alimentares não-newtonianos. A base teórica que fundamentou o desenvolvimento deste reoviscosmetro foi a análise do escoramento lento de fluido não-newtoniano na vizinhança de esferas rígidas. Um dispositivo eletromagnético com cronômetro automático, permitiu a determinação da tensão cisalhante e da taxa de distensão correspondente através de medidas da velocidade terminal, inclusive para fluidos não transparentes. O reoviscosmetro de queda de esferas mostrou ser reproduzível quando testado com um fluido newtoniano (óleo) e fornecê parâmetros reológicos que não diferenciaram significativamente daqueles obtidos por rotoviscosimetros na avaliação de produto não-newtoniano (solução de carregena 1%, 25°C). Foram estabelecidos comportamentos reológicos para o purê de pera, leite condensado e mel de flor de laranjeira, com o emprego do modelo da lei de potência, os quais apresentaram boa concordância com os dados da literatura. A faixa da taxa de distensão (°), embora não tão ampla como em alguns instrumentos muito mais custosos, é suficiente para incluir a maioria dos produtos alimentares fluidos no que diz respeito à identificação dos estímulos associados à avaliação sensorial da viscosidade.

PALAVRAS-CHAVE: reologia, reoviscosmetro, fluidos alimentares não-newtonianos, viscosidade, taxa de distensão, tensão de cisalhamento.

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