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Steady state analysis of structured liquids in a penetrometer

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ABSTRACT

A penetrometer pushes a solid object into a product and measures the resistance against deformation or flow. Many factories of semi-liquid/semi-solid foods or pastes use penetrometry devices for quality control, typically measuring the resistance force at a single penetration velocity. Furthermore, the physical texture characterization obtained by penetrometry analysis has been found to correlate well with sensory attributes. Boujlel and Coussot (2012) have shown that a simple penetrometer can also be used to obtain relevant rheological parameters, by combining data from different penetration velocities or from the relaxation of the signal after the probe has stopped inside the product. In the present paper their approach is extended to a grid-shaped probe, which is suitable for semi-liquid products like mayonnaise. We have tested its use for our purposes for various fluids, from Newtonian to thixotropic viscoplastic fluids. These fluids were also characterized by conventional rheometry for comparison. The probe of the penetrometer was a grid comprising of woven metal cylinders. The probe was simplified in a CFD simulation which provided a correlation from which the rheological parameters could be extracted. The viscosity of Newtonian fluids was determined satisfactorily from these correlations. Likewise the flow index of shear thinning liquids could be computed relatively well. The yield stress of this set of selected materials was better determined from the flow curves obtained by the penetrometer than by the relaxation method. In general, the analysis presented in this work confirms that useful rheological information can be obtained from penetrometry.

1. Introduction

Rheometry is a very valuable tool in the food industry for the development and production of for example: mayonnaise, ketchup, yoghurt and spreads. However, in practice rheometers are hardly available outside the laboratory. Instead devices such as penetrometers are installed in factories, which are economically more attractive and relatively easy to operate.

A penetrometer measures the resistance against deformation or flow. A penetrometer pushes a solid object into the product of study at a constant speed and reports the force, or measures the depth or velocity at a constant force. It is a widely applied tool to recover information about *e.g.* the mechanical properties of dairy products (Campos et al., 2002; Wright et al., 2001), cookie dough texture (Booth et al., 2003), texture attributes of mayonnaises (Liu et al., 2007), or salt contents in polymer melts through estimation of the viscosity (McLin and Angell, 1996).

The penetrometry tests developed in industry often limit the measurement to merely obtaining the resistance at one probe velocity at a certain depth. Comparing all products from a single production line to one specific set point is useful. However, it has been shown that more information can be extracted from penetrometry tests. For example, the viscosity of Newtonian liquids was computed via an empirical relation for conical shaped probes (McLin and Angell, 1996). Characteristics of non-Newtonian liquids were also determined using a rectangular plate as probe (Boujlel and Coussot, 2012). With a well defined rectangular plate it was possible to compute the stresses on the plate and to relate these stresses to the measured drag forces. The yield stress was determined satisfactorily from a relaxation test with the penetrometer as well as the flow index from penetration runs at multiple speeds (Boujlel and Coussot, 2012; Tikmani et al., 2013).

In this study we consider the extraction of non-Newtonian flow parameters from penetrometry tests with a probe described by

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Anton et al. (2013) and Kuil et al. (2004), see Fig. 1. This grid is used for industrial penetrometry tests of mayonnaises to determine the Stevens value which is a measure for firmness (Anton et al., 2013; Kuil et al., 2004). Firmness or hardness represents the force required to penetrate a sample (Mohamed and Morris, 1987; Batista et al., 2006). Note that the force needed to pull up the probe is a measure for cohesiveness or stickiness (Liu et al., 2007). The area under the penetration curve is a measure for consistency and the negative area for the work of adhesion or adhesiveness. The attributes defined by the penetration curves appear to say something about the sensory perception of the product and indeed correlations have been found between the parameters from the penetration curves and panel scores of *e.g.* mayonnaises (Liu et al., 2007; Worrasinchai et al., 2006).

What is lacking is a clear description of the penetrometry measurement in terms of rheological parameters from the constitutive equations describing the materials. Two major challenges are foreseen. First, the complex geometry of the probe makes it hard to interpret the flow field. The stresses will be non-homogeneously distributed around the grid as opposed to conventional rheometry where the stresses are constant at a single deformation rate. Second, the liquids under study are non-Newtonian and the flow is often time dependent. It is likely that the flow will not be fully developed for some fluids during the course of a penetrometry test.

Therefore, we propose the following approach: the steady-state flow curves of many pre-treated semi-solid/semi-liquid food stuffs in a rheometer (uniform shear rate) can be captured by a Herschel-Bulkley (HB) model. We therefore propose to apply the HB model locally in our case, i.e. to couple the local shear stress to the local shear rate via the HB model, assuming that the model parameters are material properties (so valid throughout the fluid volume). The spatial variation of the shear rate (in steady state) is handled by using Computational Fluid Dynamics (CFD) to calculate the flow field around the probe and the total drag force exerted on it by the fluid. The calculated drag coefficient can be linked to dimensionless numbers based on geometry-, process, and HB parameters (*e.g.* Oldroyd number) via mastercurves. Comparing experimental data with the mastercurves, values for the HB parameters can be deduced.

2. Experimental section

2.1. Material preparation

2.1.1. Newtonian liquids

300 ml nano pure water and 700 g sucrose (PFEIFER & LANGEN) were mixed. The mixture was heated to 60 °C to produce a supersaturated sucrose solution. It was then cooled slowly to 0 °C. The clear liquid in the upper layer was sampled for the measurement. The viscosity of the saturated sugar solution at 0 °C was 1.5 Pa s over a shear rate range from 0.1 s^{-1} to 100 s^{-1}. Glycerol (≥99.5% purity, Sigma-Aldrich) was measured to have a constant viscosity of 1.2 Pa s at 20 °C over the same range of shear rates.

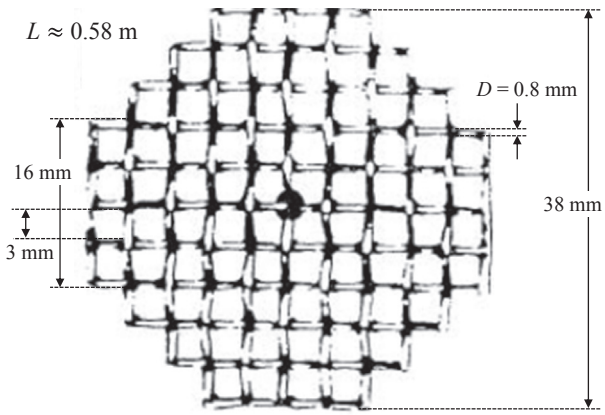

Fig. 1. The grid acting as a probe for penetrometry tests. *L* equals the combined length of all the bars in the grid.

2.1.2. Non-Newtonian liquids

3 g Carbopol (U10, Lubrizol) was dispersed in 1000 ml water by a Silverson High Shear Mixer (L4RT-A, Silverson Machines, Buckinghamshire, UK) at 800 rpm. Sodium hydroxide was added to reach a pH of 6, while the sample was stirred by a paddle impeller. The solution was then centrifuged at 4000 rpm for 10 min to release the air bubbles.

The preparation of the Xanthan solutions began with mixing respectively 10 g, 20 g and 30 g Xanthan Gum (CPKELCO) with 2 l water. A paddle mixer was used to homogenize the mixture with a rotational speed of 200 rpm at room temperature. The samples were agitated for at least 2 h before they were sent to a centrifuge operating at 4000 rpm for ten minutes to remove the air bubbles. The 0.5, 1, and 1.5 wt% Xanthan solutions were then kept at rest for at least 24 h before measurement, in order to guarantee a high level of hydration. The samples were stored at 5 °C and were warmed-up to room temperature before measurement.

Four types of oil-in-water emulsions were made with various oil and or surfactant concentrations as well as various processing conditions resulting in various droplet sizes. Sodium Dodecyl Sulphate (SDS, Sigma-Aldrich) was added to stabilize the emulsions. For the preparation of emulsion A, 3.2 g SDS (10 mM) was dissolved in 250 ml water at room temperature. 833 ml rapeseed oil was then added slowly into the solution while mixing with a Silverson Mixer at 3500 rpm with an emulsor screen workhead with openings of 1.5 μm. The Sauter mean droplet size was 6.4 μm determined by static light scattering techniques (Mastersizer 2000, Malvern Instruments Ltd, Worcestershire, UK). Emulsion B was prepared using the same procedure as emulsion A only the Silverson mixer speed was maintained at 4500 rpm resulting in a Sauter mean droplet size of 4.9 μm. Emulsion C was prepared at 3500 rpm but contained 5 wt % less oil. The resulting average droplet size was 4.9 μm. The fourth emulsion D, was prepared according to the recipe of emulsion A only the concentration of SDS was doubled. The Sauter mean droplet size of emulsion D was 4.5 μm.

Three mayonnaise samples were prepared with the same ingredients but processed under different conditions. To begin with 47 g NaCl (AkzoNobel), 65 g sugar (Suiker Unie), and 0.375 g EDTA (AkzoNobel) were dissolved in 615 g water and mixed with 380 g egg yolk (92/8 liquid egg yolk-salt, Bouwhuis-Enthoven) by using a spatula. 3.765 kg sunflower oil was then added slowly to the container equipped with a Silverson Mixer running at a rate of 3500 rpm. Finally 125 g vinegar (10% spirit vinegar, Carl Kühne) was added. This mayonnaise will be referred to as premix for the remainder of the text and had a droplet size of 12.6 μm. Two thirds of the mayonnaise was further processed with an IKA mixer (MKO module). Half of it was treated at 3500 rpm, resulting in an average droplet size of 6.4 mm and will be referred to as Setting 1. The other half was processed at 4500 rpm which gave an average droplet size of 4.3 μm and will be referred to as Setting 2.

2.2. Rheological measurements

The torque was measured at fixed rotational speeds by a parallel plate rheometer (AR, 2000ex, TA instruments, Delaware, USA) which had sand blasted surfaces and a diameter of 40 mm. The true stress response of the material was obtained through the

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