



Rheological study of orange juices for a better knowledge of their suspended solids interactions at low and high concentration



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ABSTRACT

A better comprehension of the interactions between suspended solids is necessary to optimize the performance and reduce energy requirement during different operations in fruit juice processing (thermal treatment, sedimentation, centrifugation, filtration, spray drying, etc.). In this context, the aim of this study was to propose a rheological approach to get more insight into the energy of cohesion of orange juice suspended solids (SS). The specific energy of cohesion of suspended solids was obtained from rheological measurements in dynamic mode carried out on orange juices fractions with different suspended solids concentrations and size-classes. Results showed that the specific energy of cohesion increased as the SS content increased, independently of the particles size. However, the solid-like behavior of the juice was enhanced by the presence of large particles. The results obtained show that particles network strength and the viscoelastic behavior of juice result of complex interactions between all juice particles. Even if it was demonstrated that these complex interactions depend on the size of the particles, they might depend also on the physicochemical nature of these latter. The knowledge, for the studied fruit juices, of the evolution of their viscoelastic behavior as function of the SS content might be useful to control and optimize the different operations of their transformation chain.

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1. Introduction

Fruit juices are complex and heterogeneous suspension of insoluble large particles, supra-colloidal and colloidal materials that are dispersed throughout a continuous medium rich in soluble compounds, including sugars, organic acids, soluble pectins, phenolic compounds and salts. The heterogeneous dispersed matter is mainly formed by cellular tissues fragments produced during the juice elaboration (Filippi et al., 2008).

In such dispersion of solid particles in sugar solution, particles–particles, particles–water and particles–sugar interactions, govern the stability and the rheological behavior of the suspension (Benítez et al., 2009). Moreover, the physicochemical interactions that can be established between the particles are closely linked to the biochemical composition of the fruit juice, more particularly to

the presence of polysaccharides, such as pectins, cellulose, hemicelluloses (Klavons et al., 1994; Sila et al., 2009), proteins (Corredig et al., 2001; Galant et al., 2014), lipids and low molecular weight solutes, such as sugars and ions (Niu et al., 2008; Simpkins et al., 2000).

Furthermore, the interactions between juice particles become more complex during fruit juices processing. Indeed, after being obtained by pressing, fruit juices can undergo different operations (thermal treatment, spray drying, membrane separation, resin adsorption, sedimentation, centrifugation, etc.) in order to ensure their sterilization, concentration, clarification and to recover some of their functional compounds (Abadio et al., 2004; Carabasa et al., 1998; Santhirasegaram et al., 2015; Ushikubo et al., 2007; Vaillant et al., 2005). During these operations, the juice characteristics mainly the particles concentration can evolve (Shamsudin et al., 2013), resulting in a progressive evolution of the type and the intensity of the initial interactions between suspended particles. For instance, during membrane separation (notably microfiltration and ultrafiltration) the concentration of suspended insoluble solids in

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the bulk suspension and in the fouling cake increases, leading to significant changes in some operational effects such as the diminution of the overall performance of the filtration process (permeate flux decay) (Vaillant et al., 2001).

In this context, a better knowledge of the interactions between juice suspended solids is necessary to optimize the performance and reduce the energy requirement during different operations in fruit juice processing. It is well known that Information concerning the rheological properties is useful to develop, monitor and optimize transformation processes of various food (Magerramov et al., 2007). However, most of the reported works concerning fruit juices, focused on their viscosity or on rheological measurements such as their flow behavior study (Dahdouh et al., 2015; Magerramov et al., 2007; Shamsudin et al., 2013). Advanced researches concerning the evolution of fruit juice rheological characteristics during processes still limited. As far as the current authors are aware, no published data is yet available concerning the effect of their concentration on the viscoelastic behavior of fruit juices evaluated through dynamic rheological tests. Therefore, the aim of this study was to propose a rheological approach to measure of the interactions strength between fruit juices suspended solids during their concentration. Thus the viscoelastic behavior of three orange juices was studied, notably through the evolution of the specific energy of cohesion of their suspended solids as function of their concentration. This specific energy of cohesion was obtained from rheological measurements in dynamic mode carried out on orange juices fractions with different suspended solids concentrations and particles size-classes. These rheological properties might be useful in predicting, controlling or optimizing fruit juices processing (filtration, thermal treatment, centrifugation, mixing, pumping, etc.).

2. Materials and methods

2.1. Fruit juices

Three orange juices were chosen as test matrix two commercial pasteurized juices (J_{C1} and J_{C2}), purchased from a local market, and a raw juice (J_R). J_R was prepared by squeezing Lane late oranges (*Citrus sinensis*) in a semi-industrial extractor (Automatic orange juicer, model 32, SANTOS, Vaulx-en-Velin, France) and filtered through a stainless steel sieve (0.8 mm mesh size). pH and degrees Brix of the three juices were close to 3.7 and 11, respectively.

All juices were stored at $-20\text{ }^{\circ}\text{C}$ and thawed before use at $35\text{ }^{\circ}\text{C}$ for 20 min.

2.2. Juices characterizations

2.2.1. Measurements of solids content

Dry matter (DM) was determined after two successive drying of an aliquot of fruit juice (5 g), at $50\text{ }^{\circ}\text{C}$ under vacuum for 24 h, then at $103\text{ }^{\circ}\text{C}$ for 24 h. The content of suspended solids (SS) for each juice was determined by centrifuging 40 g of homogenized juice at 18,000 g/60 min (Eppendorf Model 5810 R centrifuge, Hamburg, Germany). The supernatant was removed and the settled solids obtained were resuspended in 40 g of distilled water. The suspension was centrifuged again at 18,000 g/60 min. This washing procedure was repeated twice. The content of settled solids (g/100 g of juice) was recorded after drying at $70\text{ }^{\circ}\text{C}$ under vacuum for 48 h. Turbidity was measured in nephelometric turbidity units (NTU) at 90° light scattering and 890 nm, using a turbidity meter (Hanna LP 2000, Hanna instruments, Szeged, Hungary). The turbidimeter was calibrated using two formazine standard solutions, 0 and 10 NTU; turbidity measurements were performed on water diluted juices to fall in the turbidimeter range. For all these physical analyses, measurements were performed at $25\text{ }^{\circ}\text{C}$ and values provided are

the average of three replicates.

2.2.2. Measurement of solids size

Particles size distribution was determined by LASER diffraction using a Mastersizer (model 3000, Malvern Instruments Limited, Worcestershire, UK), in a range between 10 nm and $3500\text{ }\mu\text{m}$. Measurements were carried out in a wet-mode using water as the suspension medium under controlled conditions (obscuration of 42% and stirring of 1500 rpm) (Dahdouh et al., 2015). The values 1.73 and 1.33 were attributed to the refractive indices of cloud and dispersant phase (water), respectively, and 0.1 was used for the absorption index of cloud particles (Corredig et al., 2001). For each measurement, size distributions (volume density against particles size) were provided and statistical volume diameters, D_{10} , D_{50} and D_{90} were given (D_x indicates a particles size for which x% of the particles are below that size). The particles size polydispersity (Wu et al., 2009), characterized by the size distribution span (σ_D) was calculated according to Eq. (1).

$$\sigma_D = \frac{D_{90} - D_{10}}{D_{50}} \quad (1)$$

Moreover, the surface area average diameter $D[3.2]$ (Sauter mean diameter) was calculated according to Eq. (2).

$$D[3.2] = \frac{\sum_i n_i d_i^3}{\sum_i n_i d_i^2} \quad (2)$$

with n_i the number of particles of diameter d_i .

This mean diameter is defined as the diameter of a sphere that has the same volume/surface area ratio as the set of particles (Filippa et al., 2012); it is widely used in the characterization of suspension since it links the area of the suspended phase to its volume and hence to mass transfers and chemical interactions with the surrounding environment (Pacek et al., 1998). Furthermore, Dahdouh et al. (2015) demonstrated that Sauter mean diameter is among the five reliable variables used to predict fruit juices filterability.

2.3. Experimental strategy

The specific energy of cohesion of suspended solids was obtained from rheological measurements in dynamic mode carried out on fruit juices fractions with different suspended solids concentrations. Methodologies associated to fruit juices concentration and rheological measurements are given below.

2.3.1. Fruit juices concentration

The procedure was performed in two steps: (i) a separation of suspended solids from the juice suspension (step 1) and (ii) a dilution of these suspended solids in the liquid phase (aqueous solution) of the juice in order to obtain suspensions with increasing suspended solids content (step2).

In step 1, for the separation of suspended solids (large particles, supra-colloidal and colloidal materials); aliquots of 40 mL of homogenized juices (J_{C1} , J_{C2} and J_R) were centrifuged at 18,000 g for 60 min. In these centrifugation conditions, it is theoretically possible, according to Stokes' law (with an approximate density of 1100 kg m^{-3} for the solids and 1040 kg m^{-3} for the juice), to isolate particles with diameter approximately above $0.2\text{ }\mu\text{m}$. This assumption was subsequently verified through a particles size characterization by Dynamic Light scattering (DLS) (Fig. 1). The supernatant containing mainly soluble compounds (sugar, ions, etc.) was removed and the settled insoluble solids were drained for 5 min.

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