



## Structural parameters that determine the rheological properties of apple puree



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### ABSTRACT

The objective of this work is to better understand how the structural parameters (particle content, particle size and serum viscosity) influence the rheological properties of apple purees. An apple puree (called “native”) was ground to obtain dispersions with three different particle size distributions. This mechanical treatment induced the separation of parenchyma irregular cell clusters into regular single cells, modifying both the morphology and the particle size distribution of purees. A separation–reconstitution step made it possible to obtain samples with a wide range of insoluble solids (8–24 g/kg). Pectin was added to some of the samples in order to increase the viscosity of the continuous phase. The rheological behaviour and structural properties of the modified apple purees were investigated using flow and oscillatory rheological measurements, particle size measurements and confocal laser scanning microscopy. Rheological properties such as apparent viscosity, yield stress and elastic modulus decreased as particle size decreased and they increased as insoluble solids content increased. Modelling of rheological properties with master curves made it possible to take into account the apparent relative volume occupied by the particles of different sizes.

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### 1. Introduction

In recent years there has been a great interest in consuming foods containing dietary fibre, due to its great potential for beneficial effects in health and well being. Plant-based foods are one of the most important platforms because the plant cell walls, which are their major structural component and can be present in high concentrations, are dietary fibres (Redgwell and Fischer, 2005; Sun-Waterhouse, 2010).

During processing, plant cell walls undergo modifications in terms of their physical state, macrostructure, microstructure, and composition, as well as structure-dependent changes in their functional and material properties. Fruit purees are obtained after a thermal (cooking) and mechanical (grinding) treatment of the fruits. These treatments will considerably influence the structure and the material properties of the system (Kunzek et al., 1999).

From a physical point of view, fruit purees can be considered as concentrated dispersions of soft and deformable insoluble particles

that are dispersed into an aqueous solution of sugars, organic acids and pectic substances called the serum (Cepeda and Gomez, 2002; Rao, 1999). The solid insoluble particles in plant food dispersions are of various shapes and have multimodal size distributions. They are constituted of cell wall “ghosts” of cells and their aggregates from parenchyma tissue. Their constitutive units are thus the cell walls of the corresponding fruit. The parenchyma tissue of the apple contains cells with irregular shapes and diameters between 50 and 200  $\mu\text{m}$ , which are connected by the pectin of the middle lamella (Khan and Vincent, 1993; Kunzek et al., 1999).

Rheological parameters can provide analytical tools to yield meaningful insight on the structural organisation of food (Ahmed and Ramaswamy, 2007). Fruit purees behave as non-Newtonian fluids: in general, purees of fruits and vegetables are shear thinning fluids (Rao, 1992) presenting a yield stress. Plant food dispersions have usually been described by a power law model and also by different models that include the yield stress as a fitting parameter, such as the Herschel–Bulkley and Casson models (Colin-Henrion et al., 2009; Qiu and Rao, 1988). The properties of different plant food dispersions depend on the raw material and are influenced not only by the concentration but also by the particle size, stiffness, composition, and elastic properties (Day et al., 2010; Den Ouden and Van Vliet, 2002; Kunzek et al., 1999, 1997; Maceiras et al.,

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2007; Nindo et al., 2007). The rheological properties are expected to depend on both the soluble solids in the serum phase and the particle volume fraction of insoluble solids (Lopez-Sanchez et al., 2011; Rao, 1992; Vitali and Rao, 1982), contributing to the complex rheological behaviour of the plant food dispersions. The volume fraction occupied by the cell wall particles in suspension remains difficult to estimate as it depends on the arrangement of the soft particles, which in turn is determined by the particle shape, stiffness and size distribution (Cepeda and Gomez, 2002; Hemar et al., 2011). Recently, Day et al. (2010) have attempted an estimate of the volume fraction of particles in dispersions of carrot and broccoli using dynamic oscillatory measurements, in our knowledge no information is available on the volume fraction of apple puree.

In our previous work (Espinosa-Muñoz et al., 2012) we studied the effect and the importance of the structural parameters (particle size, solids content and serum rheology) for the sensory perception of apple puree's texture. We now study and evaluate the influence of these structural parameters on the rheological behaviour of apple purees. A mechanical treatment was used to modulate the particles size and shape, separation/reconstitution to modulate solids content, and addition of pectin to modify serum viscosity. Structural parameters of apple purees were thus modified in a controlled manner to investigate their impact on the dynamic and steady-flow rheological characteristics. Microscopy techniques were also used to characterise the structure of the apple purees.

## 2. Material and methods

### 2.1. Plant material and processing conditions

A single batch of fresh mature apple Golden Delicious was industrially processed into puree by Conserves France according to industrial best practices. Apple pieces were refined (sieve opening of 1.2 mm) and cooked (98 °C, 4 min). Finally purees were conditioned in hermetically sealed bags. This sample is called Native Puree (NP).

### 2.2. Preparation of apple purees varying particle size and solids content

In order to vary the particle size and to study the impact of mechanical treatment, NP was ground in a Grindomix GM 200 (Retsch GmbH, Germany) at 5000 rpm for 15 s, sample denoted as MG (Medium Ground), and at 10,000 rpm for 3 min, sample called HG (Highly Ground).

To obtain samples with a wide range of solids content, the pulp and the serum from NP, MG and HG purees were separated by centrifugation at 5000g for 2 h at 20 °C in a 3.18 K centrifuge (Sigma GmbH, Germany). After the separation, samples with varying cell wall content were prepared by weighing and mixing the pulp and serum in different ratios (Table 1).

To modify the viscosity of the serum, 1% of amidated pectin (Herbstreith & Fox KG: Pectin Amid CF 005-D E440) was dispersed directly in purees, mixed at ambient temperature with a pale flat stirrer (Bioblock, Scientific) for 15 min at 200 rpm. A test was carried out using the rheometer MCR-301 (Anton Paar, Physica) to verify complete dissolution of pectin.

### 2.3. Dry insoluble solids content (ISC)

Apple purees were separated by centrifugation as described in Section 2.2. The insoluble material was then prepared from the pulp as alcohol insoluble solids (Renard, 2005) with a supplementary first "washing" step. This step consisted of a centrifugation after the addition of a buffer solution (50 mM sodium acetate,

**Table 1**  
Sample coding and processing conditions.

Sample	Solids concentration (g dry insoluble solids/kg puree)	Grinding	Pectin added (g/100 g)
NP8	8	–	0
NP	11	–	0
NP16	16	–	0
NP21	21	–	0
MG7	7	5000 rpm/15 s	0
MG	11	5000 rpm/15 s	0
MG18	18	5000 rpm/15 s	0
MG20	20	5000 rpm/15 s	0
HG	11	10,000 rpm/ 3 min	0
HG16	16	10,000 rpm/ 3 min	0
HG24	24	10,000 rpm/ 3 min	0
NP8 + P	8	–	1
NP16 + P	16	–	1
HG + P	11	10,000 rpm/ 3 min	1
HG16 + P	16	10,000 rpm/ 3 min	1

adjusted to pH 3.5 with acetic acid) to the pulp to eliminate the soluble pectin trapped by the particles in the serum. Approximately 5 g of pulp were suspended in the buffer solution (15 mL), mixed in a vortex and centrifuged (13,200g, 12 min, 10 °C). The sediment was washed again with the buffer solution, two washes were judged to be significant (elimination of approximately 15 sixteenth of the serum) while leading to an acceptable risk of pectin extraction from the cell walls. The second sediment was blended with 96% ethanol (1:3), and stirred at 4 °C over 12 h. The next steps followed the procedure described in Renard (2005) which included washing by 70% ethanol and then drying by solvent exchange. Insoluble solids content was expressed as the relation between the dry materials weight and the initial sample weight.

### 2.4. Pulp content

The pulp content of the samples was determined in triplicate by centrifugation at 5000g for 2 h at 20 °C. The pulp content was expressed as the relation between the pulp weight ( $W_p$ ) and the initial sample weight ( $W_s$ ) (Qiu and Rao, 1988).

$$\text{Pulp \%} = \frac{W_p}{W_s} \times 100$$

### 2.5. Particle size distribution

The particle size distribution (PSD) of the samples was measured using a laser diffraction analyser (Master Sizer, Malvern Instruments Ltd., UK), applying the Fraunhofer optical model. The  $d(0.9)$  represents the diameter above which there is only 10% (in volume) of bigger particles. Analysis was done on the diluted samples in distilled water; each sample was run in triplicate.

### 2.6. Confocal scanning laser microscopy

Confocal laser scanning microscopy was used to visualise the dispersed particles by colouring the cell wall material. Samples were diluted 15 times with serum obtained previously by centrifugation (part 2.2) and they were stained with 5 drops of the fluorescent dye congo red (10 g/L in water, Sigma Aldrich) at 20 °C for 3 h.

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