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ORIGINAL ARTICLE

Shear Viscosity Investigation on Mango Juice with High Frequency Longitudinal Ultrasonic Waves and Rotational Viscosimetry

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Abstract Ultrasonic velocity and attenuation measurements were performed on mango juices at 25 MHz in order to estimate longitudinal viscosity. Juices were extracted from fruits, removed periodically from fruit batches undergoing ripening for 3 weeks under controlled conditions. The correlation between longitudinal viscosity and apparent dynamic shear viscosity, obtained from flow tests, showed that up to 12–13 wt.% of Soluble Solids Content (SSC), the juices presented a Newtonian behavior. In this case the relation between longitudinal viscosity measured by ultrasound and shear viscosity measured by flow tests was very simple leading to the conclusion that ultrasound could replace rotating viscosimeters for specific applications. Over this limit, the results were also clearly correlated but the correlation depended on the shear rate because of the shear thinning behavior of the juices certainly due to soluble pectins. The use of longitudinal ultrasonic waves as a tool for viscosity determination on large batches of samples is discussed at the end of this communication.

Keywords Ultrasound · Mango juice · Viscosity · Soluble Solids Content · Shear Thinning

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Introduction

Ultrasonic investigation of food properties is a very important subject which has been widely studied. The growing interest for high frequency approaches these last two decades is essentially due to large progress in data acquisition, electronics, and signal processing. Bibliography concerning the subject is enormous but one can refer to some very relevant works [1–5] made by Saggin, Coupland, Garbolino and Ziegler. They used both longitudinal or shear waves in order to correlate longitudinal velocity, longitudinal or shear reflectance to food composition or processing. In these works, correlations to "classical" rheology measurements are also proposed. Furthermore, McClements and coworkers studied the ultrasonic velocities in sugar solutions and fruit juices in great detail [6, 7]. A very good overview can be found in Povey's book where McClements and Povey describe classical ultrasonic methods for food properties investigation in a simple and rapid manner [8]. The most recent review concerning the subject has been written by Awad and co-workers [9].

Looking into the details of these studies, one can notice that ultrasonic attenuation of longitudinal waves in food investigation is generally used for particles sizing in emulsions but is less used for detailed rheological behavior analysis.

As mentioned by Dukhin and Goetz [10], shear ultrasonic attenuation is preferred but is so high that it is never directly measured in liquids even if it is easily linked to rheological shear modulus. On the other hand, longitudinal attenuation is simply accessible but can be due to several processes and the link between longitudinal attenuation and rheological behavior is not as simple as one can think. So, for juice

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investigation, scientists are generally focusing their attention on ultrasonic velocity [11, 12].

On whole mango fruit work has already been done by Mizrach [13, 14], but to our knowledge, few results exist on mango juice [15, 16]. In their work, Singh and Eipeson [15] worked on clarified and free from soluble pectins mango juices. They observed a Newtonian behavior and they recalled that, for pulp, the behavior was not the same.

Our work aimed to give complementary results on centrifuged raw mango juice using longitudinal ultrasound as a tool for rheological behavior investigation.

In this study we propose to use ultrasonic longitudinal waves and to show that if the frequency, the sensor, and the signal processing are well chosen it is possible, under some assumptions to estimate the shear viscosity of juices or sugar solutions. The preliminary work on (water / sucrose) mixtures, will be used to qualify the experimental protocol. In a second time the approach will be applied on mango juices.

The approach is not novel, has been successfully applied by Dukhin and Goetz [10] on various chemical products but has not been previously applied to fruit juices in the literature. Therefore, it has practical applicability given the online process control abilities of ultrasonic sensors in a non-destructive and food-grade manner.

At last, regarding works of Greenwood et al. [17], who performed both ultrasonic and flow tests, on binary sucrose / water solutions using shear reflectance in order to estimate shear viscosity, the agreement between flow tests and ultrasonic tests was correct except for small amount of sugars (< 20 %). Such an element is not surprising, considering the fact that for small amount of sugar the reflection coefficient at an interface ultrasonic sensor / sucrose solution is quite equal to one. So, our paper also aimed to demonstrate that ultrasonic longitudinal rheology method based on velocity and attenuation to estimate the shear viscosity is preferable for small amount of sugar and so for juice investigation.

Materials and Methods

Fruit Ripening and Juice Preparation [18]

Fruit Ripening

Two batches of Kent variety (respectively 41 and 25 fruits) and one of Keitt variety (42 fruits) were purchased from a mango producer in the province of Andalusia, Spain. Fruits were picked during the growing season 2010 either at the beginning or the end of the harvesting period. Fruits were harvested at their commercial maturity stage ("hard green"). The fruits of each batch were split into different cardboards which were placed into a ripening chamber (Binder KBF 720) of 720 L. Temperature and humidity were respectively set to 22 °C and 80 % RH for the 3 weeks that necessitated the complete softening of all the fruits. Five to six fruits were periodically drawn at random every 2 or 3 days and analyzed in order to get fruits having a wide range of fruit maturity.

Juice Preparation

After peeling and cutting, all the pulp pieces were introduced into a juicer (Whole Fruit Juicer LTK7189) to separate juice from solid pulp fraction. The resulting juice was then centrifuged (Avanti-JE, Beckman Coulter Inc.) during 15 min at 10,000 rpm (g-force ranging from 5,000 to 15,000) in order to remove cell debris and small particles from the clear supernatant. The supernatant samples were collected and frozen to be later analyzed. Soluble solids content (SSC in wt. %) recorded as °Brix was measured by refractometry at 20 °C by using a handheld digital refractometer, Atago (Tokyo, Japan) ranging from 0.0 to 33.0 °Brix.

Flow Tests with a Rheometer

Apparent shear viscosity measurements were carried out using a Physica MCR301 Rheometer (Anton Paar, Germany) equipped with a Couette flow measuring cell (Ref. DG27/T2000/SS). Sample temperature was achieved at 20 ±0.1 °C using a Peltier system and a fluid circulator Viscotherm VT 2 controlled directly from the Physica MCR. After 5 min of thermal stabilization, each 11 mL sample was submitted to a flow test in the 10 to 400 s^{-1} shear rates range.

Ultrasonic Method: Protocol, Signal Processing [19]

Starting from an arbitrary distance z_o (along a vertical axis) between an ultrasonic plane transducer generating longitudinal waves and the bottom of a tank containing the juice, the distance between the sensor and the bottom of the container was gradually reduced using a high resolution stepper motor. At each step, the ultrasonic echo on the bottom of the container after propagation in the juice was acquired with a GPIB / USB interface. At the end of the experiment we obtained a number of n echoes corresponding to n distances z between the sensor and the bottom of the container. As the distance was reduced during each step of the measurement, the time of flight (time needed to propagate in the juice) of successive echoes was also reduced. In addition, the amplitude of the echoes grew during the experiment because the distance was reduced and the ultrasonic signal propagated on a smaller distance in the absorbent juice. On a practical point of view, we worked with a 25 MHz Panametrics VM 324-SM sensor excited

with a Sofranel Panametrics 5,800 pulse generator. All the displacements were ensured with a Microcontrol© stepper motor with an accuracy of ±1 μm. The echoes were displayed on a LT374M Lecroy© oscilloscope. For each echo 20,000 points were acquired with a time base of 0.5 μs leading to a sampling step of 0.25 ns and a sampling frequency of 4 GHz. For each experiment the total motor displacement was 6 mm and we acquired 30 echoes in 30 steps. At each step the motor was stopped, and the echo (averaged 256 times with the oscilloscope) was acquired. So at each step the acquisition time is around 4 s. The system (juice - container - sensor-motor) was put in a Binder KB 53 incubator in order to obtain a very good temperature regulation at 20±0.1 °C.

Since juices and sugar solutions are viscoelastic materials, all the ultrasonic parameters can depend on frequency. Hence, one has to extract the elastic parameters for a unique frequency. The ultrasonic transducer used is efficient from 20 to 30 MHz with a central frequency of 25 MHz. So, the following signal processing has been used to extract all the information for the 25 MHz frequency. In a first step the Fast Fourier Transform (FFT) of each echo acquired was performed leading to the modulus and the phase of the FFT. Let (A) be the amplitude of the FFT modulus for the chosen frequency (f). We plotted 20.Log(A) versus the distance z. Then the slope is equal to $-2.\alpha_{dB}$ were α_{dB} is the attenuation in $dB.m^{-1}$. For the frequency (f) we also plotted the FFT phase divided by 2πf versus z. The slope of this linear trend is then equal to $2/V_L$ where V_L is the longitudinal velocity. The time needed to perform the measurement and the signal processing is around 2 min and so, this approach is very interesting for large batches of samples analysis or for samples which could rapidly evolve.

Theoretical Aspects: Longitudinal and Shear Viscosity Estimation from Ultrasonic Data

Ultrasonic attenuation observed in liquids is caused by many factors: viscous losses, losses due to thermal conductivity, diffraction due to non-infinite dimensions of the ultrasonic transducer, scattering by small particles in suspension [20, 21]. Losses due to thermal conductivity are negligible but for diffraction and scattering losses, the choice of frequency is fundamental. If the frequency is too low given the size of the sensor, the diffraction losses will predominate over viscous losses. If the frequency is too high, losses due to particle diffusion is so high that the estimation of small viscosities can become tricky. A frequency around 25 MHz was chosen because, given the size of transducer (6 mm diameter) one can demonstrate [22] that diffraction losses are negligible, which provides a direct measurement of the attenuation without having to correct the sensor effect. Furthermore, using the areas defined by Povey [8], if scatters are less than ten microns, the measurement is performed in "long wavelength region" and, in this case, scattering by particles are negligible. This allows working on centrifuged juice samples instead of juice samples obtained from filtering through membranes which makes the experimental protocol simpler.

On a theoretical point of view [10], in order to take into account damping, the ultrasonic displacement (u) of a monochromatic wave of frequency (f) propagating in (z) direction can be written as: $u \sim e^{-\alpha z} e^{j(\omega t - kz)}$. In this expression, (α) is the ultrasonic wave attenuation (longitudinal or shear), (ω) the radial frequency ($\omega = 2\pi f$), $k = \omega/V$ where (V) is the velocity (shear or longitudinal) of the mechanical wave. This is generally re-written as $u \sim e^{j(\omega t - k^* z)}$ with $k^* = (\omega/V) - j\alpha$. Then, if k* is defined with the help of a complex velocity as $k^* = \omega/V^*$, on can define a complex elastic modulus as ρV^{*2} where (ρ) is the density. If we consider a shear wave, $V = V_s$ and the modulus will be the shear modulus $G^* = G' + jG''$. If now we take a longitudinal wave, $V = V_L$ and α_L represents the longitudinal attenuation. So, the modulus is a longitudinal elastic modulus called M* and equal to $M' + jM''$.

Such an approach is not new and has been first introduced by Litoviz et al. [23–25] or Piccirelli et al. [26]. By analogy with the definition of the shear viscosity $\eta_s(\omega) = G''/\omega$, the longitudinal viscosity is defined as:

$$\eta_L = \frac{M''}{\omega} \tag{1}$$

For longitudinal waves, the attenuation is generally small in liquid [10, 21] and one can demonstrate that the longitudinal viscosity is simply expressed by:

$$\eta_L \sim \frac{2\alpha_L \rho V_L^3}{(2\pi f)^2} \tag{2}$$

The details of calculations can be found in the works of Dukhin and co-workers [10].

In this relation, one has to keep in mind that α_L is expressed in m^{-1} (or Neper). The link between α_{dB} (measured) and α_L is $\alpha_{dB} = [20/\ln(10)].\ \alpha_L$.

When viscosity is not frequency dependant (Newtonian liquids) it can be demonstrated [10, 23–26] , that this longitudinal viscosity can be linked to the apparent shear viscosity (dynamic shear viscosity) thanks to the following relationship:

$$\eta_L = \eta_b + \frac{4}{3}\eta_s, \tag{3}$$

where η_s is the "classical" dynamic shear viscosity, η_b is the bulk viscosity and is generally proportional to η_s. So, let's write $\eta_b = \Delta\eta_s$. The factor Δ is generally ranging between 1 and 3. For instance [10, 21, 25, 27] for water, (Δ) is around 3 and is equal to 1 for glycerol. But it seems [10] that this rule is not universal and for specific liquids (Δ) could be greater.

Results and Discussion

Ultrasonic Investigation of Water/Sucrose Mixtures

As (water / sucrose) mixtures are generally simple models for juice representation in a very first approximation [11] we decided to investigate such mixtures. According to Greenwood et al [17]. who performed both ultrasonic and flow tests, such binary solutions behave as Newtonian liquids up to 60 wt.%. After velocity and attenuation measurements, the longitudinal viscosity was calculated with the relationship (2). For density we used the relationship given by Contreras et al [11]. We have decided to work with small volumes (around 1 ml) in order to validate the method for small amount of juices. Such a point is important when many analyses (physical, chemical…) have to be performed. Consequently the total displacement of the sensor is small and for low sugar contents the variation of amplitude is very small. Hence the accuracy on longitudinal viscosity estimation with ultrasound was ±0.9 mPa.s. This accuracy could be largely improved with higher volumes because in this case the propagation distance would be higher, leading to a bigger attenuation and to a better accuracy on its evaluation. In Fig. 1 we have plotted the measured longitudinal viscosity of (water / sucrose) solutions versus their shear viscosity given in literature [17, 28, 29]. From 0 to 40 wt.% we observed a clear proportionality validating the Newtonian behavior of the solution. Over 40 %, the linear trend was not verified. So, we decided to work between 0 and 40 wt.%. The adjustment gave $\eta_L = (4.39)\eta_s$ (see Eq. 3), leading to $\Delta = 3.05 \pm 0.09$. So, for (water / sucrose) mixture up to 40 wt.% one can take $\Delta = 3$ (as for water) in order to deduce η_s from η_L. The fact that we found $\Delta = 3$ as for water means that the internal structure of the fluid was not modified in depth and justified the term of "light liquid" used by Greenwood et al. [17]. for their binary mixtures of (water / sucrose). We think that the discrepancies observed up to 40 % in our case could reveal a beginning of non Newtonian behavior.

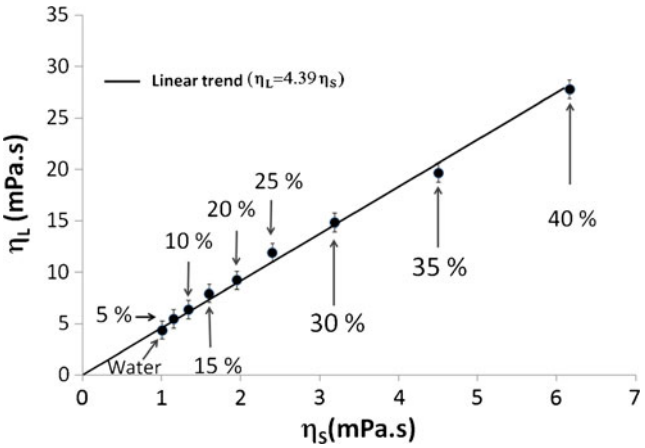

Fig. 1 Longitudinal viscosity estimated with the ultrasound method versus shear viscosity (from bibliography) of (water/sucrose) mixtures in the range 0–40 wt.%

Ultrasonic Velocity Versus Soluble Solids Content

The evolution of ultrasonic longitudinal velocity at 20 °C versus SSC of mango juice is reported Fig. 2. For water we found 1,482±2 $m.s^{-1}$. Such a result was in good agreement with the value recommended for water by Briggs [27]. For mango juices, accuracy was also 2 $m.s^{-1}$ leading to the conclusion that the variability observed Fig. 2, for a chosen SSC, was not due to the experimental protocol but due to juices composition changes. On a qualitative point of view one can observe that for SSC greater than 12–13 % the slope of the plot - velocity vs. SSC- was higher than the one observed for SSC lower than 12–13 wt.%. According to Contreras et al. [11] if the numbers and type of sugars are exactly known, the link between velocity and composition is correct. In the case of real juices, extracted from fruits from various stages of ripening, it can be more difficult [30] because other substances such as soluble pectins can highly influence the results. Furthermore as soluble pectins have got a very big influence on viscosity it seems natural to examine ultrasonic attenuation which is linked to viscosity. This is the reason why we decided to focus our attention on attenuation . The velocity analysis will remain here qualitative. The main conclusion of the data behavior observed in Fig. 2 was that it seemed that after 12–13 wt.% of SSC, another parameter which is not a simple sugar included in SSC was influencing the data.

Attenuation Versus Soluble Solids Changes and Viscosity Evaluation

Figure 3 shows the plot of attenuation in $dB.m^{-1}$ versus soluble solids content for all the juice samples. For water,

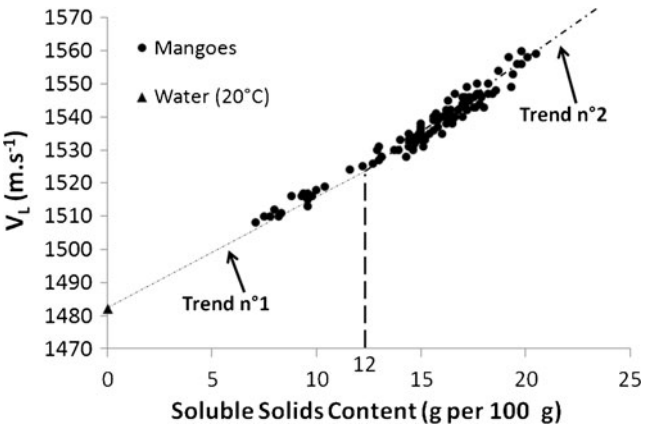

Fig. 2 Ultrasonic longitudinal velocity in mango juice versus Soluble Solids Content

we found 148 dB.m^{-1} which was in line with value recommended by Briggs [27]. Attenuation measurements are given with a mean accuracy of ±30 dB.m^{-1}. Here again, two major zones were clearly defined. From 0 to 12–13 wt.% SSC, attenuation was not increasing a lot. Above 12–13 wt.% SSC, the variability was very high and for a given SSC, attenuation could be multiplied by a factor two depending on the sample analyzed. Regarding these results and considering that changes in attenuation mainly result from the effect of viscosity in our juices, soluble pectic substances occurring during the ripening of mangoes, due to pulp softening and cell walls changes [31], and which are known to dramatically increase the viscosity, can be suspected to be the cause of this dispersion.

In order to go further in these investigations, and to confirm that the high variability were really due to juice composition and not to another parameter (for instance scattering by small particles), flow tests were performed for the selected samples reported in Fig. 3. In particular, two samples having SSC=18 % and 19.8 % can seem outlier and the results with flow tests will be very interesting. The selected samples for flow tests were chosen in such a way that, for a given SSC, they covered the variability observed on the measured attenuation with ultrasound. For samples with SSC less than 13 % the behavior was Newtonian. On the contrary, over 13 % SSC all the samples analyzed for shear rates between 10 and 400 s^{-1} exhibited a shear thinning behavior confirming the role played by soluble pectic substances. Examples of plots of apparent shear viscosity versus shear rate are presented in Fig. 4 for various mango juices with a SSC ranging from 7 to 19.8 %.

To give a link between ultrasonic approach and flow tests we have plotted for all selected samples the longitudinal viscosity estimated with ultrasound versus apparent viscosity measured with flow tests. Figure 5 is devoted to Newtonian selected samples. One can observe that the 6 points are not very far from the line $\eta_L=4.39\eta_s$ (model for water / sucrose mixtures), but a better adjustment led to $\eta_L=3.96\eta_s$, giving a value of $\Delta=2.63\pm0$. With this value of Δ, it is possible to estimate the shear viscosity from ultrasound via the calculation of the longitudinal viscosity with a maximum error of 15 % compared to the value given by flow tests.

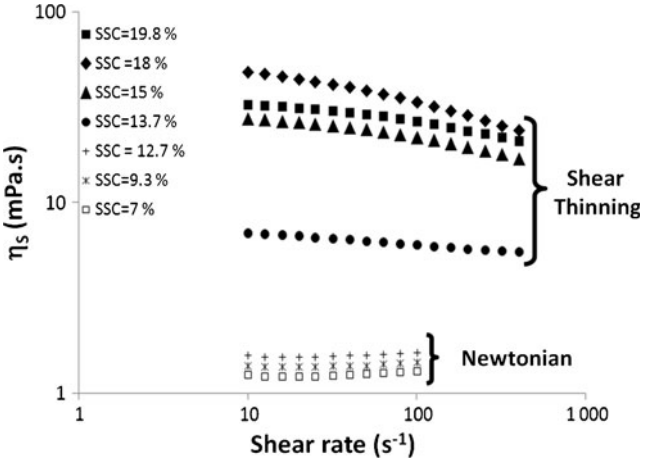

Fig. 4 Apparent shear viscosity estimated with flow test versus shear rate for some selected samples

For the samples having a shear thinning behavior, the shear rate has to be taken into account. In Fig. 6 we have reported the longitudinal viscosity versus the apparent shear viscosity for shear rates of 10 s^{-1} and 400 s^{-1}. It is clear on this graph that for the two shear rates the two types of viscosities could be linearly correlated. Two linear regressions are proposed on this graph. As shear rate increased the global trend seemed to tend to the Newtonian behavior represented by $\eta_L=3.96\eta_s$.

Hence in a first time, longitudinal viscosity could be directly used to follow the evolution of a juice for instance here versus SSC. Secondly, as ultrasonic and flow viscosities were linearly correlated, the variability observed on longitudinal viscosity was also observed on shear viscosity and was not due to an experimental artifact but to mango

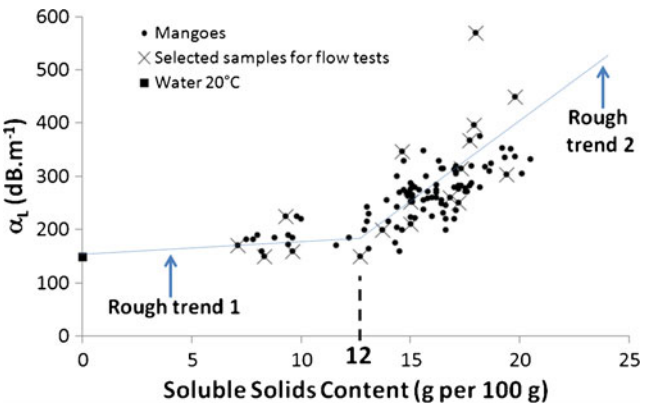

Fig. 3 Ultrasonic longitudinal attenuation in mango juice versus Soluble Solids Content

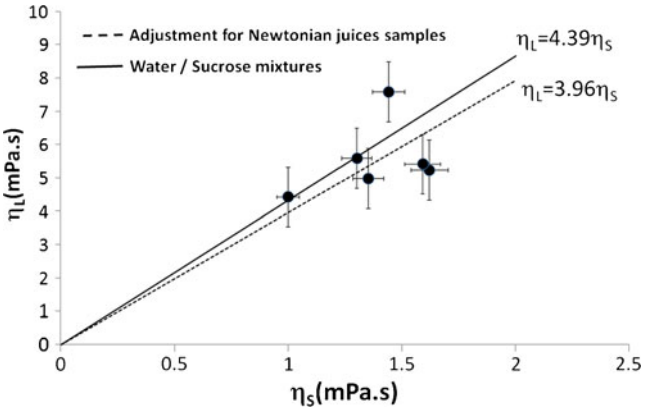

Fig. 5 Longitudinal viscosity versus apparent shear viscosity for the selected Newtonian samples

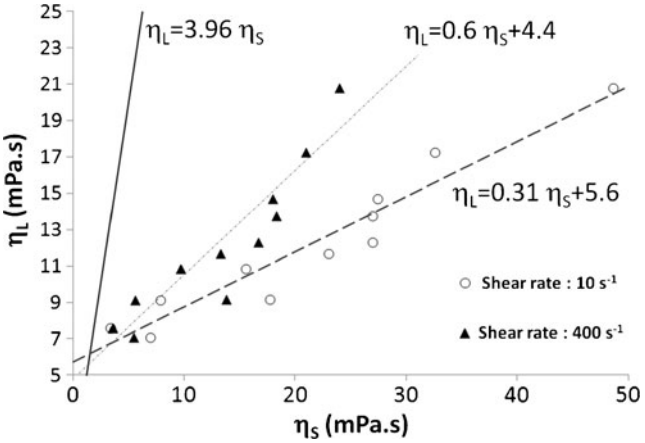

Fig. 6 Longitudinal viscosity versus apparent shear viscosity and shear rate for the selected Shear Thinning samples

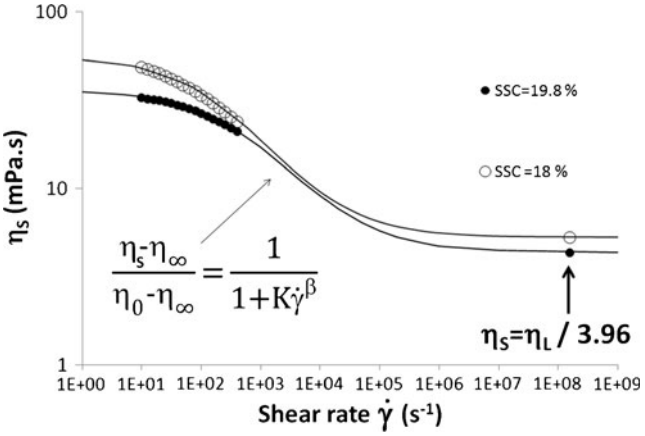

Fig. 7 Examples of large bandwidth rheograms

juice chemical composition which was not taken into account by SSC.

Considering the fact that the global behavior seemed to converge to Newtonian behavior when the shear rate increased and considering the fact that, with ultrasound, the shear rates tended to infinity, we assumed that with ultrasound we were in fact analyzing the second Newtonian plateau of the shear thinning law. In order to rely flow tests to oscillatory ultrasonic experiments we have used Cox-Merz rule which is generally used for small shear rates but can also sometimes be applied at high shear rates [32]. Following this law, we have considered in a first approximation that the shear rate can be taken equal to the radial frequency. So for ultrasonic tests it would lead to a shear rate of 1.57.10 [8] s^{-1}.

Hence we propose to adjust our flow curve with classical shear-thinning model written as follows:

$$\frac{\eta_s - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{1 + K\dot{\gamma}^{\beta}} \quad (4)$$

In this expression, $\dot{\gamma}$ represents the shear rate, η_o the shear viscosity for $\dot{\gamma} \to 0$ and η_∞ the shear viscosity for $\dot{\gamma} \to \infty$. K and β are parameters to be adjusted. We propose to take $\eta_\infty = \eta_L/3.96$ (measured with ultrasound). With this asymptotic value, the adjustments were very good and the rheological behavior of the juices is assessed on more than 8 decades. In Fig. 7, such an adjustment is presented for two specific samples with SSC=19.8 % and 18 %. These samples are very important because on attenuation results they appeared as outliers. Furthermore SSC was not a good indicator because the attenuation (and longitudinal viscosity) was higher for the sample having a smaller SSC. In fact flow tests lead to the same conclusions. At last, the rheograms obtained on a large bandwidth show that the sensitivity was depending on the shear rate. Indeed around 1,000 s^{-1} the viscosities calculated by the model were not significantly different.

Works are going on in our labs in order to precisely correlate these rheological results to biochemical analysis. In particular a first observation concerning a small molecule, myo-inositol has drawn our attention: its concentration which remained stable from 0 to 13 % SSC rapidly decreased between 13 and 20 % SSC with a very large variability. As this molecule is involved in pectins metabolism [33], we are now trying to see in which extend the ultrasonic method could become a tool for prediction of the amount of pectins in fruit juice.

Conclusion

In this work we developed an ultrasonic protocol to obtain very precise estimation of velocity and attenuation of centrifuged mango juices. The estimation of shear viscosity by this ultrasonic method was proved to be reliable for Newtonian sucrose solutions. On mango juice, the comparison of apparent shear viscosity estimated by ultrasound and flow tests revealed a Newtonian behavior for juices having a SSC less than 12.7 %. Over this limit the juices exhibited a non Newtonian Shear Thinning behavior certainly due to the increase of soluble pectic substances resulting from the mango pulp softening and more peculiarly due to cell walls modifications. Finally, the viscosity estimated with ultrasound was very useful for large bandwidth rheological behavior estimation. Regarding these results, the ultrasonic method based on attenuation interpretation, never applied on juices in literature is more relevant for juices having a small amount a sugar compared to shear reflectance which is preferable for high sugar contents (Honey…). So, it could be used for investigation of viscosity of fruit juices. The experiment is rapid, can be applied on large batches of juices. It could be a preliminary test before using more complex and expensive tools such as rheometers. The experiments could be simplified for an industrial use. At last, as glass, metal are non opaque to ultrasound one could imagine applications through bottles,

pipes… An implementation of the high frequency measurements on pocket refractometer is also possible due to the small size of the ultrasonic sensors.

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