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Additional Information

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# RHEOLOGICAL BEHAVIOUR OF AN INSOLUBLE LEMON FIBRE AS AFFECTED BY STIRRING, TEMPERATURE, TIME AND STORAGE.

Arturo Córdoba, María del Mar Camacho, Nuria Martínez-Navarrete

Food Technology Department. Universidad Politécnica de Valencia. Spain.

Corresponding author: Nuria Martínez-Navarrete. Food Technology Department.  
Universidad Politécnica de Valencia. Camino de Vera s/n, 46022 Valencia, Spain.  
Tel: +34 963879362, Fax: +34 96387736, e-mail: nmartin@tal.upv.es

**Abstract** In this work, the effect the preparation method (two different stirring systems at two temperatures and times), fibre concentration (between 2 and 3 %), temperature (between 10 and 50 °C) and storage time (between 24 h and 50 days at 5 °C) had on the flow properties of a suspension of dietary lemon fibre prepared with a 45 °Brix sucrose solution were evaluated. This information will be helpful in order to discover the best possibilities of using fibre to increase the viscosity of certain kinds of products, depending on the processing, storage and consumption conditions. The obtained results indicate that all the aspects which contribute to increase component solubilisation and the interaction of the insoluble fraction with the aqueous phase when preparing the suspension, such as a decrease in particle size, a rise in temperature or a longer homogenization time, entail a higher apparent viscosity. Moreover, favouring the solubilisation in the preparation process leads to a more stable rheological behaviour of the suspension during storage. As expected, the apparent viscosity of suspensions was dependent on the shear rate, concentration and temperature. A thixotropic behaviour of fibre suspension was only observed at a very low shear rate ( $5 \text{ s}^{-1}$ ).

**Key Words:** Fibre, fibre suspension, flow curve, yield stress, thixotropy, homogenization.

**Running Title:** Rheological behaviour of an insoluble lemon fibre.

## Introduction

Food rheological behaviour is related to its structure, temperature and composition. In the case of viscous or low-gelling products (such as nectars, jams, jellies...), the naturally-occurring soluble and insoluble components will condition their rheological behaviour. There are several additives commonly used at low concentration levels in the food industry to help achieve the characteristic viscosity of these products. This is the case of some hydrocolloids, such as starch, gelatine, guar gum, locust bean gum, pectin, carrageenan, xanthan gum, cellulose derivatives, arabic gum, agar, etc. Pectin, which is considered as soluble dietary fibre, is also an interesting ingredient from a nutritional viewpoint. Nevertheless, other types of less soluble fibres can also be used to help to increase the viscosity.

The fibre incorporated into formulated foods changes the consistency, texture, rheological behaviour and sensory characteristics of the final products (Grigelmo-Miguel et al., 1999a). Furthermore, the way in which it is incorporated into such products may offer new opportunities for use in the food industry. Moreover, apart from its nutritional properties, the fibre can be used with different economic and technological ends in mind, as an agent to increase the volume and as a fat substitute. However, not all fibres can be incorporated in the same way (concentration and type) and into the same kinds of products (beverages, dairy products, soups, jams, sauces, meat, snacks, pasta or bakery products). Their functional properties are the factors that determine their use (Guillon & Champ, 2000). These properties depend on the intrinsic characteristics of the fibre, the way in which the product is processed and its final structure or the spatial arrangement of the structural elements and their interactions. In this sense, the particle size, the porosity and chemical characteristics of the surface area, the hydration capacity, the rheological and fat retention properties and the colour, aroma and flavour greatly influence the functionality of the fibre.

Insoluble fibres, as compared with soluble ones, take up little water and form low-viscosity mixtures. From this point of view, they involve a smaller increase in volume and leave no feeling of satiety. However, the presence of a high proportion of insoluble solids can result in a sandy texture and a change in the functional properties of food that should be taken into account. Raghavendra et al (2006) conclude that fibre hydration properties (water retention and swelling capacity), particularly in the case of coconut residue, depend on the particle size, which has a positive or negative effect: a

1 particle size reduction of up to 550  $\mu\text{m}$  results in an increase of these properties, but  
2 smaller particle sizes lead to a decrease. On the other hand, Lario et al (2004), Sangnark  
3 & Noomhorm (2003) and Sakata & Saito (2007) found a direct relationship between  
4 these two parameters, although they suggest that some pre-treatments, such as stirring,  
5 may produce significant changes in fibre behaviour that could modify its hydration  
6 properties. Nevertheless, Jongarrontapnagsee et al. (2007) did not find any effect of the  
7 particle size or the temperature of hydration on the water holding capacity of fibre  
8 obtained from lime or cabbage leaves.  
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14 In this work, the effect the method used for preparing dietary lemon fibre  
15 suspension had on the flow properties and also how they were affected by the fibre  
16 concentration, the temperature and the storage time have been evaluated. The variables  
17 considered for the preparation of suspensions were homogenization intensity,  
18 temperature and time of mixing.  
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## 25 **Material and Methods**

### 26 27 28 29 **Lemon fibre**

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33 The raw material used for this study was lemon fibre (Herbacel AQ Plus Citrus Fiber,  
34 from Herbstreith&FoxKG, Barcelona) which contains 88-93% of dietary fibre and is  
35 75-83% insoluble in water.  
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### 40 **Preparation of fibre suspension**

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43 Different concentrations of fibre suspensions were prepared for the different studies in a  
44 45° Brix sucrose solution. Concentration levels of between 2 and 3% (w/w) were  
45 selected to obtain suspensions with viscosity levels equal to or greater than those shown  
46 by a sucrose solution of 45 °Brix and 2.5% pectin, which is the consistency of many  
47 products similar to jam (Saldaña et al., 2000).  
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53 Lemon fibre suspensions were prepared at 2.5% (w/w) in order to study the  
54 influence of the preparation method and storage time on the rheological behaviour of  
55 the samples. Two sets of equipment were used to get the fibre dispersion in the 45° Brix  
56 sucrose solution: an Ultra-Turrax Eurostar digital homogenizer (8000 rpm) and a  
57 Heidolph RZR 2020 stirrer (300 rpm). The estimated shear rates generated in the  
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1 suspensions were 115 and 25 s<sup>-1</sup>, respectively. In each case, the stirring process was  
2 performed by keeping the sample in a bath at 25° and 60° C for different time periods,  
3 as established by Table 1. The fibre suspensions prepared using the Ultraturrax  
4 homogenizer were submitted to 50 mbar vacuum pressure for 10 minutes to remove air  
5 bubbles. All the samples were rheologically characterized, as described below, in the 24  
6 hour period following preparation and after different storage times at 5° C (between 22  
7 and 50 days).  
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12 To study the microstructure of fibre suspensions, the thixotropy and the  
13 influence of fibre concentration and temperature on their rheological behaviour, they  
14 were homogenized with the Ultraturrax (8000 rpm) for 7 minutes at 25° C. Microscopy  
15 was performed on suspensions prepared at 3% fibre concentration. For the thixotropic  
16 study, lemon fibre suspensions were prepared at 2.0, 2.5 and 3.0%. To analyse the  
17 influence of fibre concentration, samples were prepared at 2, 2.25, 2.5, 2.75 and 3 g  
18 fibre/100g suspension and rheologically characterized, as described below, after 24  
19 hours and 20 days of storage at 5° C. A 2.5% suspension was used to study the influence  
20 of temperature (10, 20, 30, 40, 50 ° C) on rheological behaviour.  
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### 31 Microscopy of fibre and fibre suspension

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34 Lemon fibre and a 3% lemon fibre suspension, prepared as described in the above  
35 paragraph, were observed by optical microscopy (Leica DMLM). Toluidine blue 0.1%  
36 was used for staining samples.  
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### 42 Rheological characterization

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45 Measurements to obtain the lemon fibre flow curves were taken at 25° C (and at 10, 20,  
46 30, 40 and 50° C for the suspension used to study the influence of temperature on the  
47 rheogram) in a Plate Rheometer (Model Rheolab MC100, Paar Physica, Austria)  
48 working with a gap of 1 mm and a plate of 50 mm in diameter. Temperature  
49 measurement was controlled through a thermostated bath (Physica Viscotherm VT10).  
50 The rheograms were obtained by applying a gradient sweep at a shear rate of 0 to 100 s<sup>-1</sup>  
51 in 1 minute. All the samples were characterized in triplicate.  
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1 To study the thixotropic nature of lemon fibre, the shear stress variation with  
2 regard to time was obtained when applying a constant shear rate gradient. The applied  
3 gradients were 5, 10, 30, 50 and 100 s<sup>-1</sup> for 600 s.  
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5 The rheological data were modelled by means of a non-linear regression  
6 procedure, at a 95% probability level, using the program Statgraphics Plus v. 5.1. To  
7 analyze the effect of the different studied factors on the model fitted parameters or on  
8 the viscosity of the suspensions, analyses of variance (ANOVA) were carried out using  
9 the same program.  
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## 14 **Results and discussion**

### 15 **Microscopy study.**

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23 Fig. 1 shows the micrographs obtained for fibre (a) and fibre suspension (b). The image  
24 corresponding to the lemon fibre shows a significant amount of small, globular particles  
25 stained magenta or dark purple, mixed with elongated fibrous structures. According to  
26 the literature, the former may correspond to remnants of pectin and the latter to traces of  
27 cellulose and lignin (Flint, 1996). In the case of lemon fibre suspension, there were  
28 fewer observed globular particles. This would result from the dissolution in water of the  
29 soluble pectin which forms a part of this fibre. The globular particles remaining in  
30 suspension may correspond to the insoluble pectin, while the fibrous structures may be  
31 in response to the presence of insoluble cellulose and hemicellulose.  
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42 Effect of the preparation method on the rheological behaviour of lemon fibre  
43 suspensions.  
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47 Fig. 2 shows some of the rheograms obtained of each sample using both stirring systems  
48 (Ultraturrax and Heidolph) at different homogenization temperatures. The behaviour  
49 differed from case to case depending on the preparation method. For both stirring  
50 systems, the longer the homogenization time and the higher the temperature at which  
51 the fibre dispersion was carried out, the more evident was the upward flow curve  
52 movement. This difference was much less marked when the Heidolph homogenizer was  
53 used. Using the Ultraturrax homogenizer, on the other hand, entails a greater upward  
54 curve movement.  
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1 For a more thorough comparison between the treatments, the rheological  
2 behaviour of the different samples was characterized by applying the Herschel-Bulkley  
3 (HB) model (eq. 1) or the Power Law (PL) (eq. 2) when the yield stress was not  
4 statistically significant. Table 2 shows the obtained parameters.  
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$$\sigma = \sigma_0 + k\dot{\gamma}^n \quad (1)$$

$$\sigma = k\dot{\gamma}^n \quad (2)$$

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11 where  $\sigma$  is the shear stress (Pa),  $\sigma_0$  is the yield stress (Pa),  $k$  is the consistency index (Pa  
12  $s^n$ ),  $\dot{\gamma}$  is the shear rate ( $s^{-1}$ ) and  $n$  is the flow behaviour index.  
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15 Both model adjustments led to quite a high coefficient of determination, ranging  
16 from 0.9893 to 0.9986. The standard error of the estimate concerning  $\sigma$  ranged from 0.3  
17 to 2.1 Pa. In Fig. 2, the dotted lines show the close fit of the models to the experimental  
18 values. For samples prepared at slower stirring speeds, by using the Heidolph  
19 homogenizer, the yield stress values obtained when applying the HB model were not  
20 significant. In this case, the modelling was carried out by using PL. The plastic nature  
21 of those samples prepared using the Ultraturrax was detected, since the yield point was  
22 significant at both 25 and 60° C. As may be observed, both longer time periods and  
23 higher temperatures caused an increase in  $\sigma_0$ . It is important to take the yield point into  
24 account if it is significant when modelling the product behaviour, because it is closely  
25 related to the minimum initial energy for the material movement (Duran & Costell,  
26 1982; Mizrahi, 1979) as well as to product sensory perceptions (Akdogan & McHugh,  
27 2000). As regards the  $k$  and  $n$  values (Table 2) and as expected from the flow curves,  
28 preparing a fibre suspension by applying both the Ultraturrax system and higher  
29 temperatures for longer leads to higher consistency index levels and lower flow  
30 behaviour index values. The action of Ultraturrax can be related to its mechanical effect;  
31 since a breaking of the fibrous insoluble particles and a disaggregation of initially  
32 aggregated globular particles may occur. Such aspects could involve a greater  
33 interaction between the insoluble fraction and the aqueous phase, as well as a more  
34 intense solubilisation of the other fraction. Both aspects would increase the suspension's  
35 apparent viscosity (higher  $k$  value) and lead to the sample's behaviour varying more  
36 greatly from that of a Newtonian fluid (lower  $n$  value), while contributing to the yield  
37 stress occurrence mentioned in the previous paragraph.  
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57 On the other hand, applying higher temperatures for a longer time when  
58 preparing the fibre can lead to a greater solubilisation of soluble components and  
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1 consequently an increase in the consistency along with a rise in the plastic and pseudo-  
2 plastic nature of the dispersion. The most marked effect of using Ultraturrax for  
3 preparation indicates that the above-mentioned particle fragmentation and  
4 disaggregation make the interaction between them and the aqueous phase somewhat  
5 easier.  
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9 On the other hand, to distinguish the effect of the homogenization time from that  
10 of temperature, samples prepared by applying 7, 15 and 20 min of homogenization at a  
11 constant temperature of 25° C (samples U<sub>25-7</sub>, U<sub>25-15</sub> y U<sub>25-20</sub>) can be compared. Fig. 3  
12 shows the different rheograms for the three preparation times along with the line  
13 corresponding to the HB model fitted for each particular case. Table 2 shows the  
14 corresponding parameters. Although an increase in the consistency of the samples can  
15 be detected when they are dispersed for longer, an equilibrium value tendency seems to  
16 be proven, as differences between the curves obtained at 7 and 15 min are greater than  
17 between those at 15 and 20.  
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20 In fact, according to the ANOVA, differences for k and n parameters were  
21 statistically significant (p<0.05) between 7 and 15 min but not between 15 and 20 min,  
22 whereas for the yield point, they were not significant between 7 and 15 min, but they  
23 were so for 20 min preparation time. Such results confirm that it is not only the  
24 temperature during the mixing but also the homogenization time that contribute to  
25 achieve dispersions with higher apparent viscosity. Incidentally, if sample U<sub>60-10</sub> is  
26 included in the above-mentioned ANOVA, it can be grouped along with those prepared  
27 by applying a longer homogenization time at 25° C. Therefore, this indicates that the  
28 same apparent viscosity can be developed in lemon fibre suspensions by homogenizing  
29 them for longer or by preparing the suspension at a higher temperature.  
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45 Effect of storage time on the rheological behaviour of preparations.  
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48 Rheograms of samples H<sub>25-30</sub>, H<sub>60-45</sub>, U<sub>25-7</sub> and U<sub>60-10</sub> were obtained again after being  
49 stored for a particular period. The obtained flow curves were modelled by following the  
50 same procedure as that for the samples after 24 hours' preparation. Fig. 4 shows the  
51 evolution of model parameters. As can be observed, storage time had the most marked  
52 effect on the  $\sigma_0$  parameter. Although the samples prepared using the Heidolph  
53 homogenizer initially showed no yield stress, this developed throughout storage; the  
54 longer the storage time, the greater the yield stress. This  $\sigma_0$  increase with time was  
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1 significant ( $p < 0.05$ ) for H samples and also for the one obtained using Ultraturrax at a  
2 lower temperature, while remaining stable for the sample prepared using Ultraturrax but  
3 at a higher temperature. This result will be coherent with an increase in the progressive  
4 solubilisation of the suspension components, which will take place during storage  
5 provided that maximum component solubilisation was not reached at the moment of  
6 preparation. This last phenomenon is what seems to be obtained with samples U<sub>60-10</sub>.  
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10 As regards the other two parameters,  $k$  and  $n$ , a significant increase in  $k$  and a  
11 significant decrease in  $n$  were only detected for sample H<sub>25-30</sub> and just until the yield  
12 stress appeared (after 22-days' storage). From the results commented on previously, this  
13 would be the sample that showed the lowest component solubilisation during the  
14 suspension preparation.  
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20 From this viewpoint, if what is intended is a stable product that does not evolve  
21 over time, an intense homogenizer system should be used, such as that provided by the  
22 Ultraturrax at 8000 rpm. Furthermore, it should be prepared at 60°C for ten minutes,  
23 which involves the maximum component solubilisation when preparing the suspension  
24 and, consequently, its maximum apparent viscosity, with no subsequent changes during  
25 the storage period. Nevertheless, according to the above mentioned results (Table 2), the  
26 same effect is also likely to be obtained at 25° C and by applying a sample  
27 homogenization time of between 15 and 20 min using the Ultraturrax system.  
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36 Thixotropic behaviour of lemon fibre.  
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40 Fig. 5 shows the results of the study analysing the effect of shearing time on the  
41 rheological behaviour of lemon fibre preparations at three concentration levels: 2.0%  
42 (a), 2.5% (b) and 3.0% (c) and at different shear rates (5, 10, 30, 50 and 100 s<sup>-1</sup>). Only  
43 when the lowest shear rate was applied, 5s<sup>-1</sup>, was the shear stress observed to decrease  
44 over time, until an equilibrium value was reached. This phenomenon seems to be more  
45 marked at the highest concentration level (3.0 %). The effect of time on rheological  
46 behaviour is related to a progressive change in the product structure. In this particular  
47 case, it is probably based on the progressive orientation of the fibrous particles of lemon  
48 occurring in the flow direction, which entails a system viscosity decrease with shearing  
49 time. This is the typical behaviour of thixotropic products. When all the particles have  
50 been orientated, there are no more structural changes and, consequently, no more  
51 viscosity variations, so the time effect is no longer detectable. The fact that this effect is  
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1 not observed at high shear rates means that, at over  $5 \text{ s}^{-1}$ , an instantaneous particle  
2 orientation occurs.

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4 Time-dependent behaviour may be modelled by considering the kinetics of the  
5 structural destruction that takes place in the product due to the flux. The Hahn Empirical  
6 model (eq.3) (Hahn et al., 1959) assumes a first order kinetic for structural destruction,  
7 considering that this destruction corresponds to the change of the shear stress from time  
8 zero to a given time.  
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$$11 \quad \text{Ln}(\sigma - \sigma_e) = p - at \quad (3)$$

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13 where  $\sigma$  is the shear stress (Pa), the  $p$  constant corresponds to the initial stress value for  
14 structural degradation (Pa), the  $a$  constant is related to the specific rate for structural  
15 rupture ( $\text{s}^{-1}$ ),  $\sigma_e$  is the stress value (Pa) when the flow achieves equilibrium and  $t$  is the  
16 flux time (s). In this sense, greater  $p$  and  $a$  values will be related with a more viscous  
17 product that suffers a faster structural rheodestruction, the greater the  $\sigma_e$ , the more  
18 resistant the structure is to the rupture.  
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26 The Hahn Empirical model was fitted to the specific  $\sigma$  variation with time  
27 obtained at  $5 \text{ s}^{-1}$ . For this purpose, the equilibrium stress value ( $\sigma_e$ ) was the average  
28 stress value taken in a time period ranging between 400 and 600 s at a 2.0 %  
29 concentration level ( $\sigma_e = 20.88 \text{ Pa}$ ) or between 500 and 600 s for 2.5 % ( $\sigma_e = 34.64 \text{ Pa}$ )  
30 and 3.0 % ( $\sigma_e = 42.49 \text{ Pa}$ ). Similar  $\sigma_e$  values can be found in studies on baby food,  
31 where  $\sigma_e$  values correspond to 31 and 45.5 Pa for vegetable-based and fruit-based baby  
32 food, respectively (Alonso et al., 1995).  
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39 Table 3 contains the parameters obtained from fitting the Hahn model. The  $p$   
40 increase related to concentration entails a higher resistance or stress needed to initiate  
41 structural degradation as the concentration rises, but with such degradation occurring  
42 more slowly, as deduced from a value decrease. The values obtained for these samples  
43 were in the range of those found for similar products. In this respect, Alonso et al  
44 (1995) found maximum  $p$  values of 1.93 Pa for baby food, which is similar to that  
45 obtained for 2.0% lemon fibre preparation. However, the rate of breakage of the  
46 structure of lemon fibre dispersions was lower than that of the commercial baby-food  
47 products assessed by Alonso et al (1995), in which the shear rates ranged between  $0.019$   
48  $\text{s}^{-1}$  for fruit-based products and  $0.031 \text{ s}^{-1}$  for meat-based products. Even higher were the  
49 rate values for the structural breakage described by Paredes et al (1988) working on  
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1 salad dressing products, with the maximum parameter value ranging between 0.13 and  
2 0.14 s<sup>-1</sup> at 2°C.  
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5 Effect of fibre concentration on rheological behaviour.  
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9 The rheograms obtained for different lemon fibre concentrations at initial status and  
10 after 20-days' storage are represented in Fig. 6. A more marked plastic behaviour was  
11 detected in samples when the storage time and fibre concentration increased. Likewise,  
12 in both cases, the flow curves moved upwards denoting an increase in the consistency of  
13 the samples.  
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18 In these cases, fitting the Herschel-Bulkley model always gave a significant yield  
19 stress. Table 4 shows the parameter values obtained for all the samples. This table  
20 reveals the accuracy of the fitting, with low values for the standard error of estimation  
21 and high ones for the determination coefficient. Likewise, Fig. 6 shows that the fitted  
22 model reproduces the experimental behaviour.  
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27 To analyze the effect of concentration and storage time on the value of the  
28 parameters of the fitted model, an ANOVA was performed with both factors. The  
29 results of this ANOVA revealed a significant effect (p<0.05) of concentration and  
30 storage time on  $\sigma_0$ , with no significant interaction between them, but only of  
31 concentration on k and n. When increasing the concentration, a significant rise (p<0.05)  
32 in the yield stress and the consistency index occurs, whereas a significant decrease in  
33 the flow behaviour index takes place. This is related to the higher viscosity of the  
34 suspension and to the fact that it is less Newtonian in nature. The same relationship has  
35 been detected in dietary peach fibre suspensions (Grigelmo-Miguel et al., 1999b) and in  
36 nutritional wheat oat fibre supplements (Mahmoud & Fugitt, 1996).  
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45 For the consistency of these samples to be compared with that of other products, the  
46 work of Guerrero & Alzamora (1998) on papaya and mango purees can be cited. In this  
47 case, at 25° C, the yield stress for papaya puree ( $a_w = 0.93$ ; 41.9 °Brix) was  $1.0 \pm 0.2$  Pa  
48 and that for mango puree ( $a_w = 0.93$ ; 41.9 °Brix) was  $9.5 \pm 0.6$  Pa, values included in the  
49 range we found for our samples (between 1.8 and 14.44 Pa). The authors indicate a  
50 consistency index of  $2.2 \pm 0.6$  Pas<sup>n</sup> for the same mango puree and 0.7 Pas<sup>n</sup> for the  
51 papaya one, although the latter increased up to 15.3 Pas<sup>n</sup> in 51.2° Brix concentrated  
52 puree (fruit content of 24%).  
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On the other hand, the viscosity change that took place when the fibre concentration was varied can be appreciated in Fig. 7, where an exponential relationship was established. The viscosity values were calculated at two extreme shear rates from among those used to obtain the flow curve: 1 and 100 s<sup>-1</sup>. To this end, equation 4 was used, where the  $\sigma$  value was calculated from the Herschel-Bulkley model parameters (Table 4).

$$\eta = \frac{\sigma}{\dot{\gamma}} \quad (4)$$

Effect of temperature on the rheological behaviour of a lemon fibre suspension at 2.5%.

Fig. 8 shows how the flow curve shifts upward when the temperature decreases, i.e., the values of shear stress go up and, therefore, the apparent viscosity increased. All the curves obtained at different temperatures were fitted to the HB model, except the one at 50°C, at which yield stress was not significant and PL was used for the adjustment of experimental values. Table 5 includes the values corresponding to the obtained parameters. In general, a decrease in  $\sigma_0$ , k and n can be detected for the samples characterized at between 10 and 40°C as the temperature rises. The greater mobility of particles as the temperature rises can contribute to a reduction in the energy needed to achieve the flow. Consequently,  $\sigma_0$  decreases to the extent that it is not noticeable at 50°C.

In order to know the extent to which the temperature changes affect the fibre dispersion's apparent viscosity, models fitted to experimental data were used to calculate the apparent viscosity, according to eq.4, at two shear rates: 1 and 100 s<sup>-1</sup>. The apparent viscosity values obtained are shown in Fig. 9 based on temperature (°C), where an Arrhenius exponential relationship (eq.5) was detected. Such an adjustment of the equation to the data (with R<sup>2</sup> values of 0.962 and 0.963) led to the obtaining of the corresponding activation energies, their values being 12.847 kJ/mol at 1s<sup>-1</sup> and 11.232 kJ/mol at 100 s<sup>-1</sup>. These values are included among those reported by Grigelmo-Miguel et al (1999b) for a dietary peach fibre at concentrations of 5 % (Ea = 11.7 a 14.3 kJ/mol), 7% (Ea = 6.4 a 15.1 kJ/mol) and 10% (Ea =10.2 a 16.4 kJ/mol).

$$\eta = A \exp\left(\frac{E_a}{RT}\right) \quad (5)$$

1  $\eta$ = apparent viscosity (Pa.s), A=model constant (dimensionless),  $E_a$ =activation energy  
2 (kJ mol<sup>-1</sup>), R= universal gas constant (8.314 10<sup>-3</sup> kJ K<sup>-1</sup> mol<sup>-1</sup>) and T= temperature (K).  
3

4  $E_a$  calculated from such expressions proves the extent to which temperature  
5 variations affect viscosity changes. In this respect, if  $E_a$  is high, the viscosity change  
6 arising from a variation in temperature is higher than for lower  $E_a$  values. In this case,  
7 the fact that the obtained  $E_a$  values were very similar at both shear rates means that the  
8 influence of temperature is similar in both cases, with no additional contribution to be  
9 noticed as for shear rate regarding the interaction between the particles responsible for  
10 the flow.  
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## 18 **Conclusions**

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22 When preparing a suspension, all the aspects which contribute to an increase in  
23 component solubilisation and insoluble fraction interaction with the aqueous phase  
24 (particle size decrease, temperature rise or homogenization time increase) lead to a  
25 higher viscosity level. The effect of these variables can be related to a mechanical effect  
26 of breakage affecting the fibrous particles and the disaggregation of the globular  
27 particles which compound lemon fibre. This would imply a higher interaction between  
28 the soluble components and water, as well as a rise in the number of insoluble particles,  
29 both phenomena contributing to the viscosity increase. During mixing, these effects are  
30 enhanced as the stirring gets more intense, the time period is extended and the  
31 temperature used rises. Moreover, favouring the solubilisation in the preparation  
32 process leads to stability in the rheological behaviour of the suspension during storage.  
33 On the other hand, the apparent viscosity of the suspensions proved to be dependent on  
34 shear rate, shear time (only at low shear rate), concentration and temperature.  
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**Figure 1** Micrographs of lemon fibre (a) and 3% lemon fibre suspension (b) (10x).

**Figure 2** Examples of rheograms obtained for lemon fibre suspensions under different preparation conditions: U<sub>25-7</sub>: Ultraturrax, 25 °C, 7 min; U<sub>60-10</sub>: Ultraturrax, 60 °C, 10 min; H<sub>25-30</sub>: Heidolph, 25 °C, 30 min; H<sub>60-45</sub>: Heidolph, 60 °C, 45 min. Dotted lines correspond to fitted model (Table 2).

**Figure 3** Average rheograms obtained for 2.5% concentration samples. Treatment times are: 20 min, 15 min and 7 min. Lines correspond to the behaviour predicted by the Herschel-Bulkley fitted model.

**Figure 4** Variation of yield stress  $\sigma_0$ , consistency index k and flow behaviour index n during sample storage. U<sub>25-7</sub>: Ultraturrax, 25 °C, 7 min; U<sub>60-10</sub>: Ultraturrax, 60 °C, 10 min; H<sub>25-30</sub>: Heidolph, 25 °C, 30 min; H<sub>60-45</sub>: Heidolph, 60 °C, 45 min.

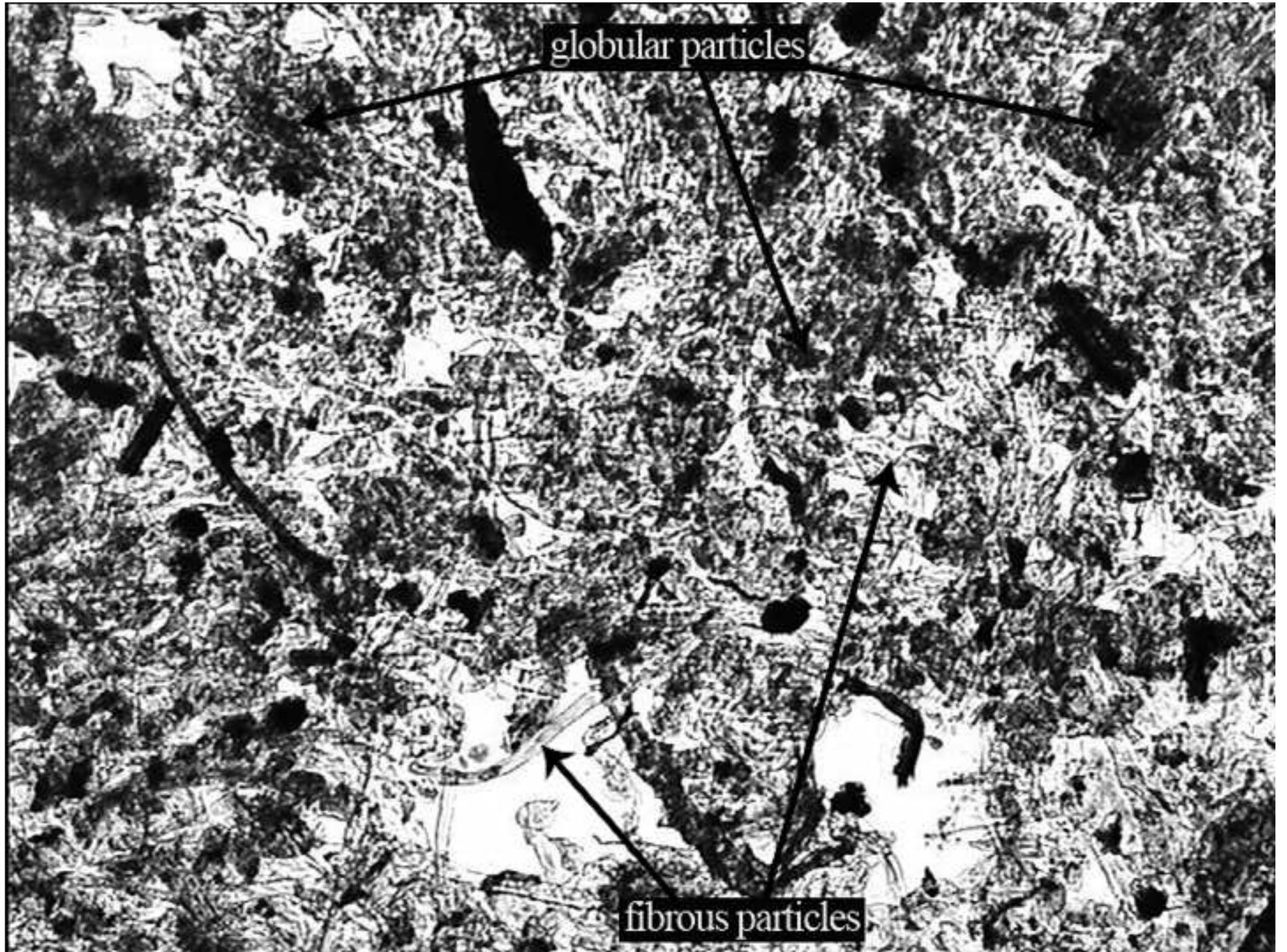
**Figure 5** Shear stress variation measured in lemon fibre preparations at 2.0% (a), 2.5% (b) and 3.0% (c) with time at shear rate of  $5\text{s}^{-1}$  ( $\diamond$ ),  $10\text{s}^{-1}$  ( $\blacksquare$ ),  $30\text{s}^{-1}$  ( $\blacktriangle$ ),  $50\text{s}^{-1}$  ( $\times$ ) and  $100\text{s}^{-1}$  ( $+$ ).

**Figure 6** Average rheogram obtained at 25°C for 2.0% ( $t_0\blacklozenge$ ,  $t_{20}\blacklozenge$ ); 2.25% ( $t_0\blacksquare$ ,  $t_{20}\square$ ); 2.5% ( $t_0\blacktriangle$ ,  $t_{20}\triangle$ ); 2.75% ( $t_0\bullet$ ,  $t_{20}\circ$ ) and 3.0% ( $t_0*$ ,  $t_{20}\times$ ) lemon fibre dispersions.  $t_0$ : 24 hours after preparation;  $t_{20}$ : after 20 days' storage. The lines correspond to the fitted Herschel-Bulkley model.

**Figure 7** Viscosity ( $\eta$ ) evolution with fibre concentration at  $1 \text{ s}^{-1}$  and  $100 \text{ s}^{-1}$ . The line corresponds to the fitting of exponential data:  $\eta(1\text{s}^{-1}) = 0.0053 e^{2.1996C}$ ,  $R^2=0.8961$ ;  $\eta(100\text{s}^{-1}) = 0.014 e^{1.5184C}$ ,  $R^2= 0.9756$ .

**Figure 8** Average rheograms obtained for lemon fibre dispersion at 2.5% and different temperatures between 10 and 50°C. The lines correspond to the fitted Herchel-Bulkley or Power Law model.

**Figure 9** Viscosity representation ( $\eta$ ) according to temperature (T) obtained at  $1\text{s}^{-1}$  and  $100\text{s}^{-1}$ . The line corresponds to the exponential model adjusted to viscosity variation that takes place with temperature.



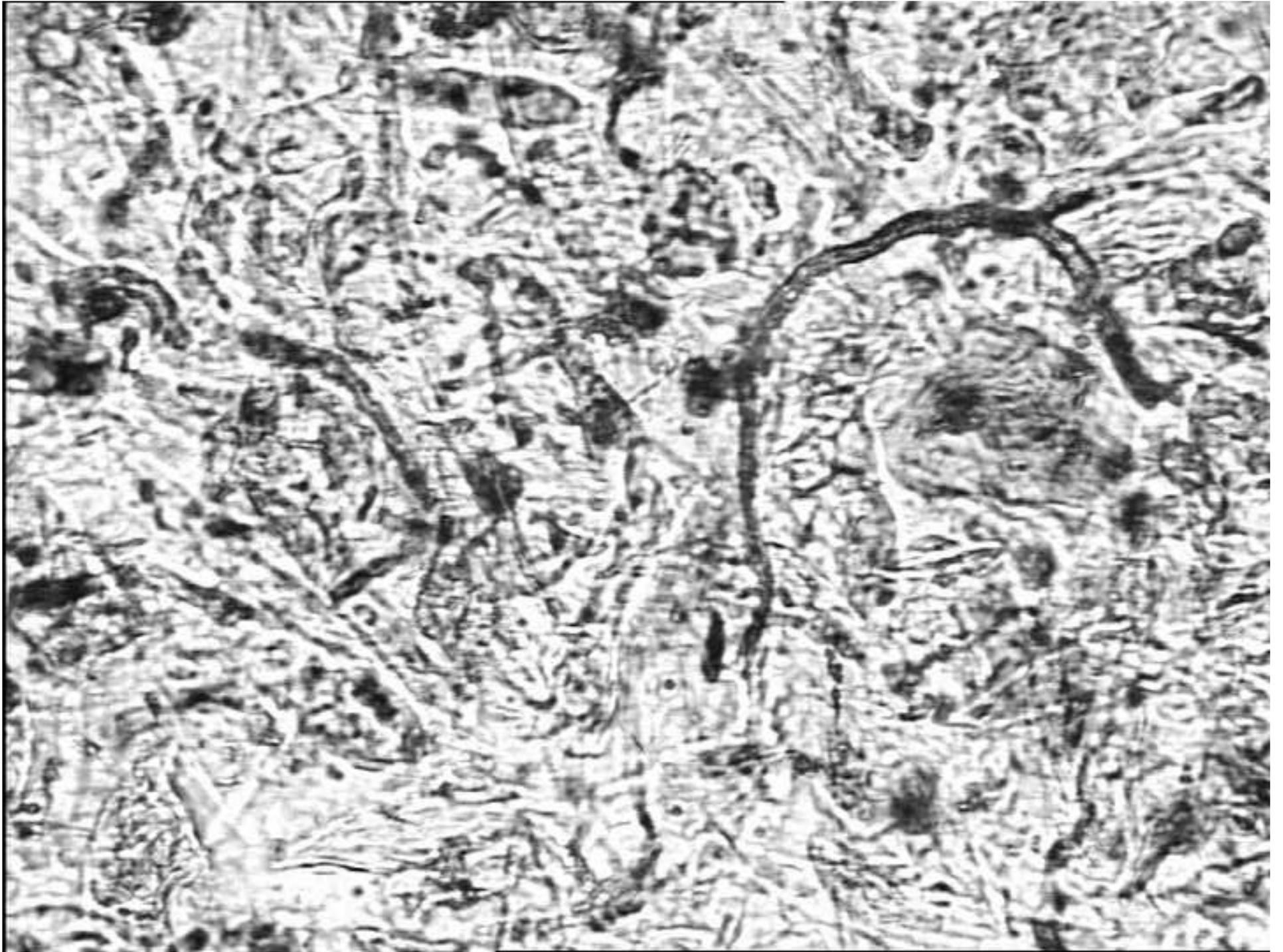


Figure 2

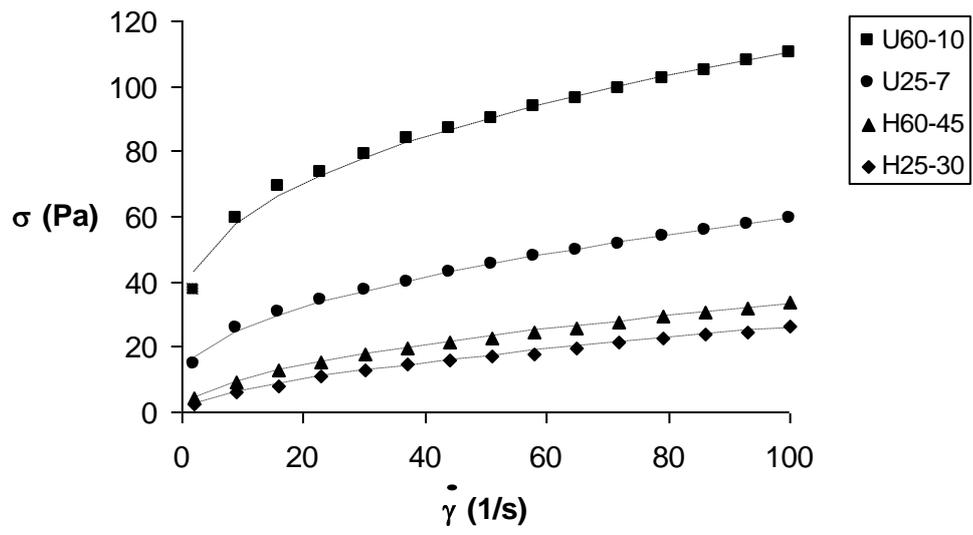


Figure 3  
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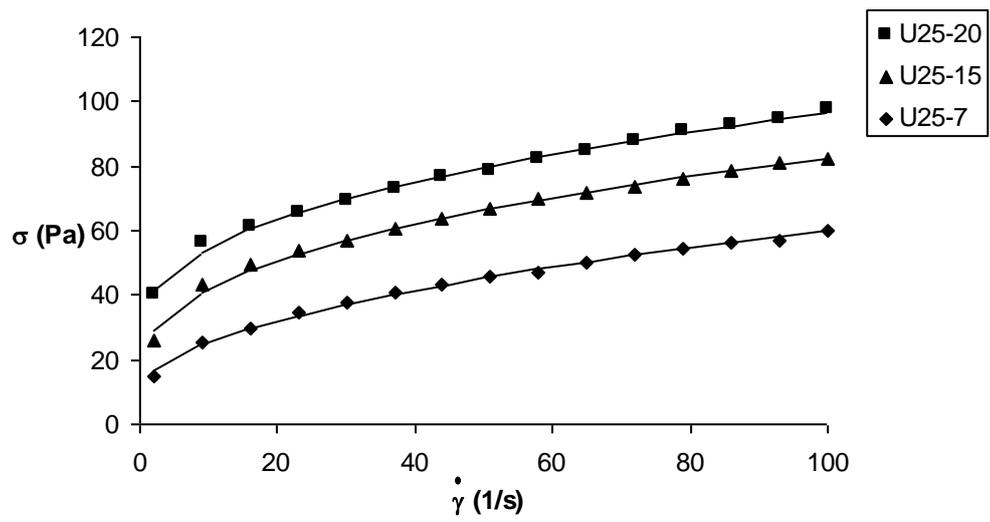


Figure 4

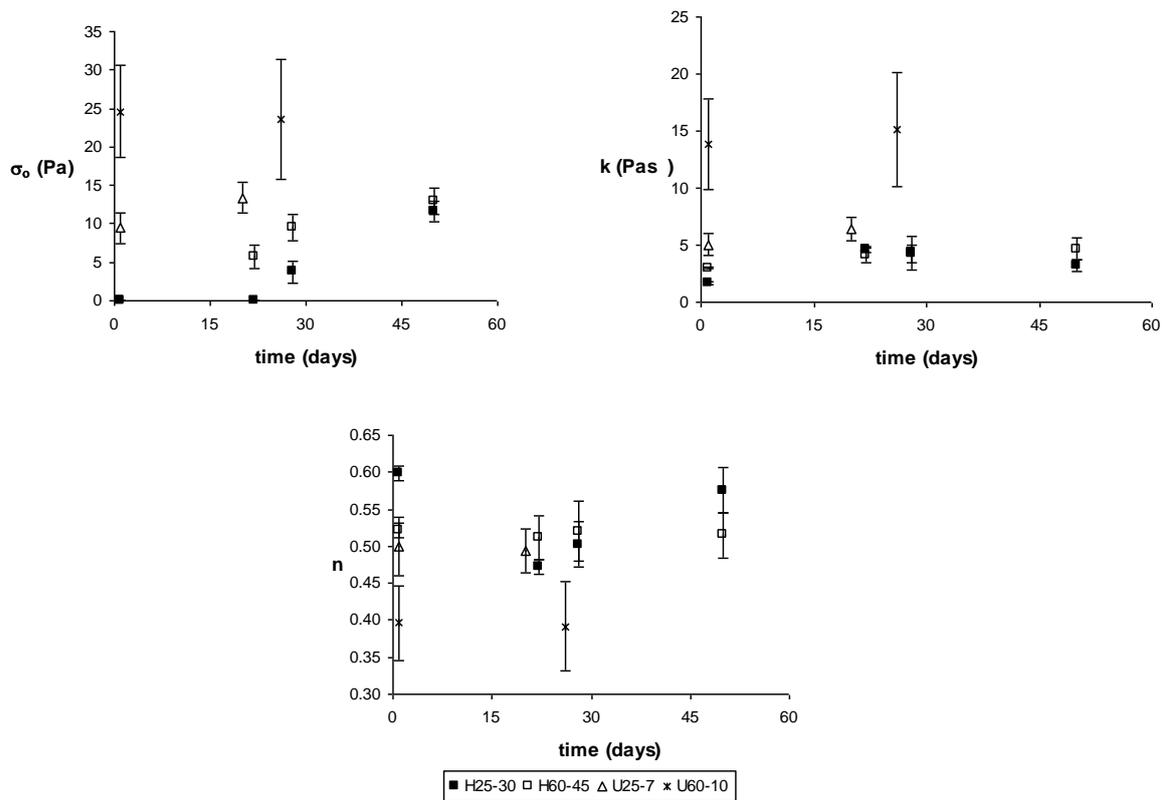


Figure 5

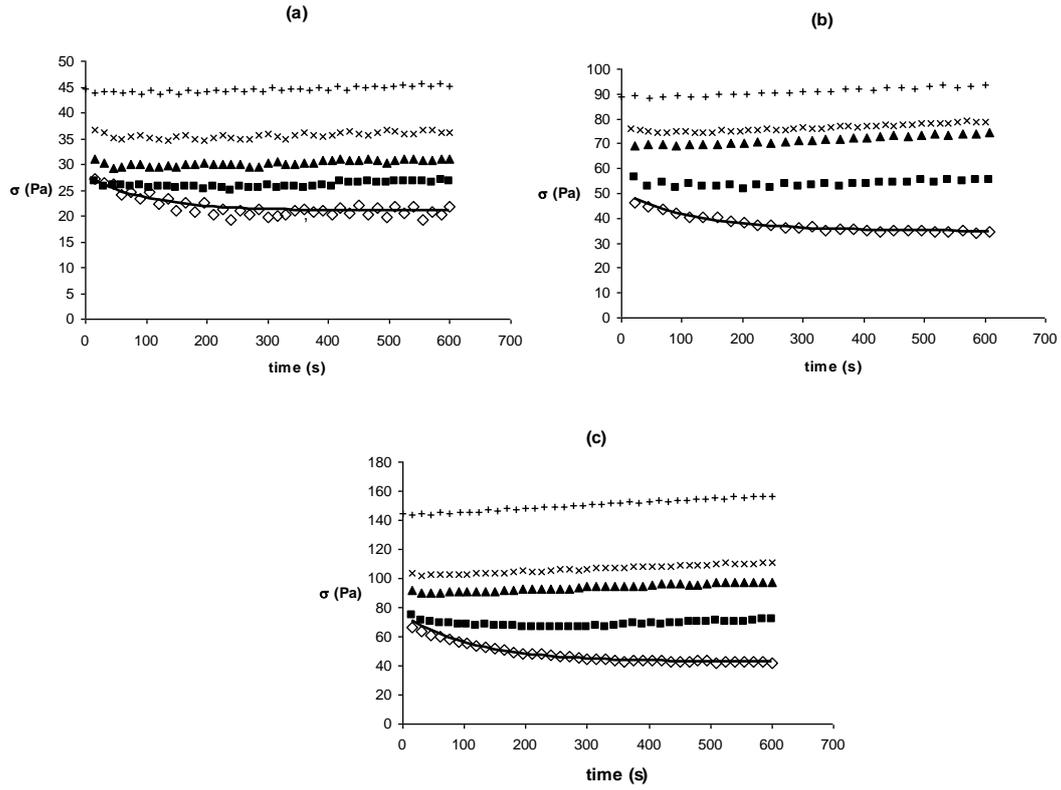


Figure 6

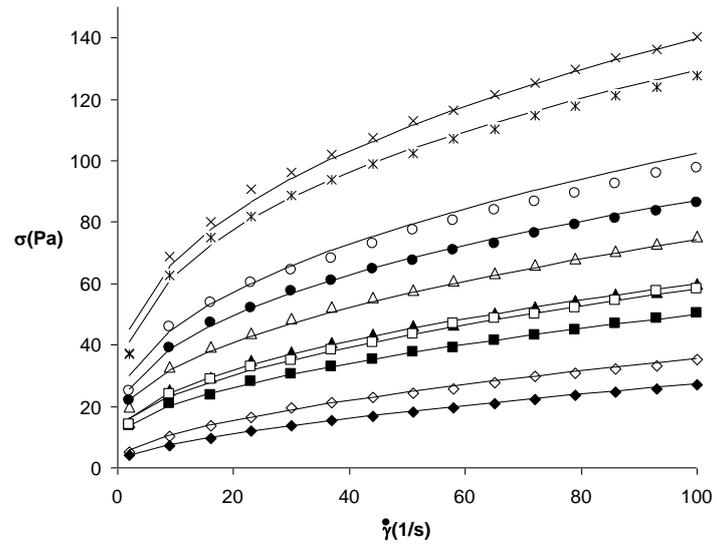


Figure 7

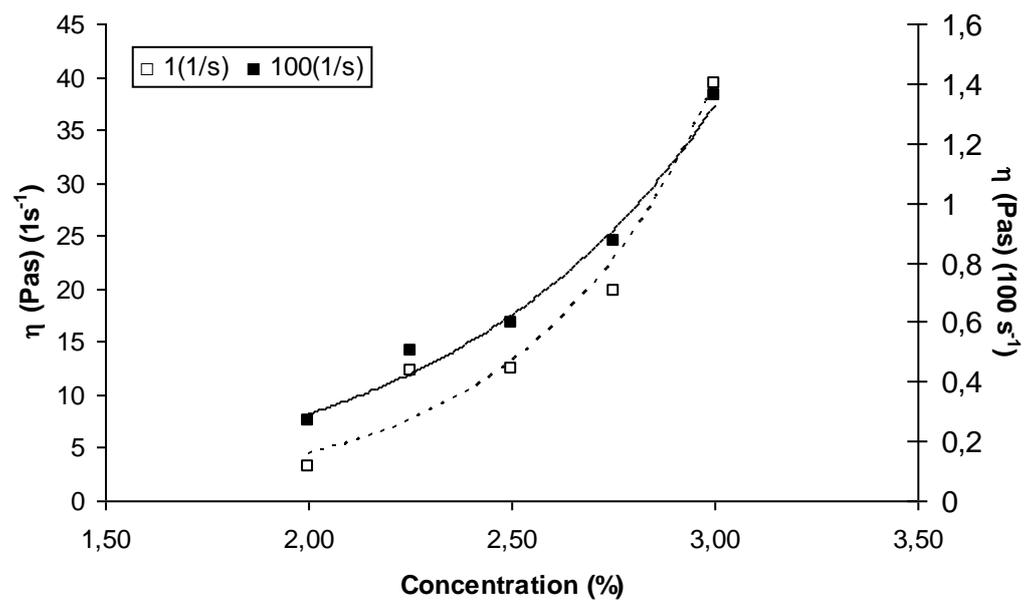


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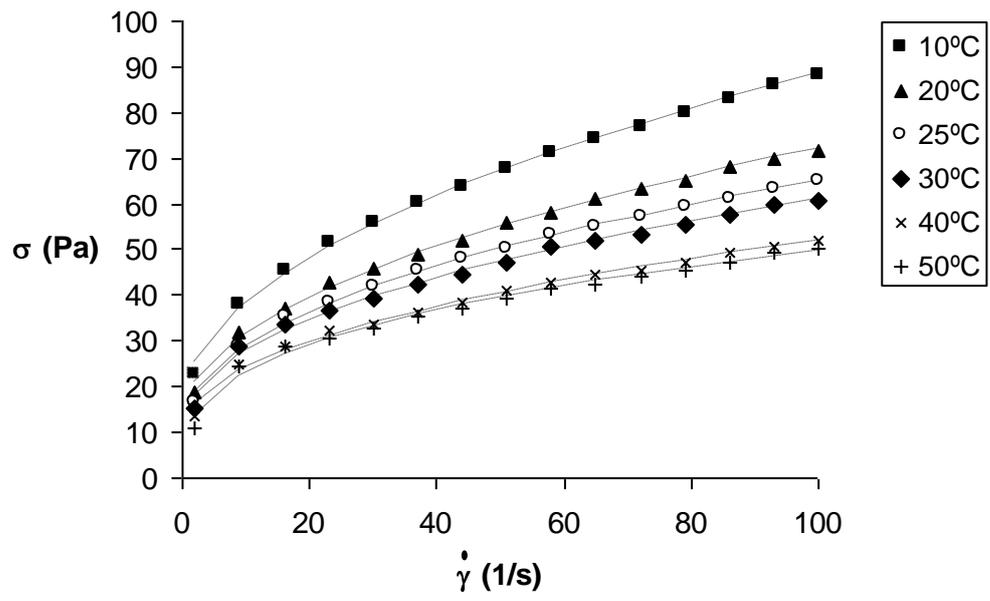
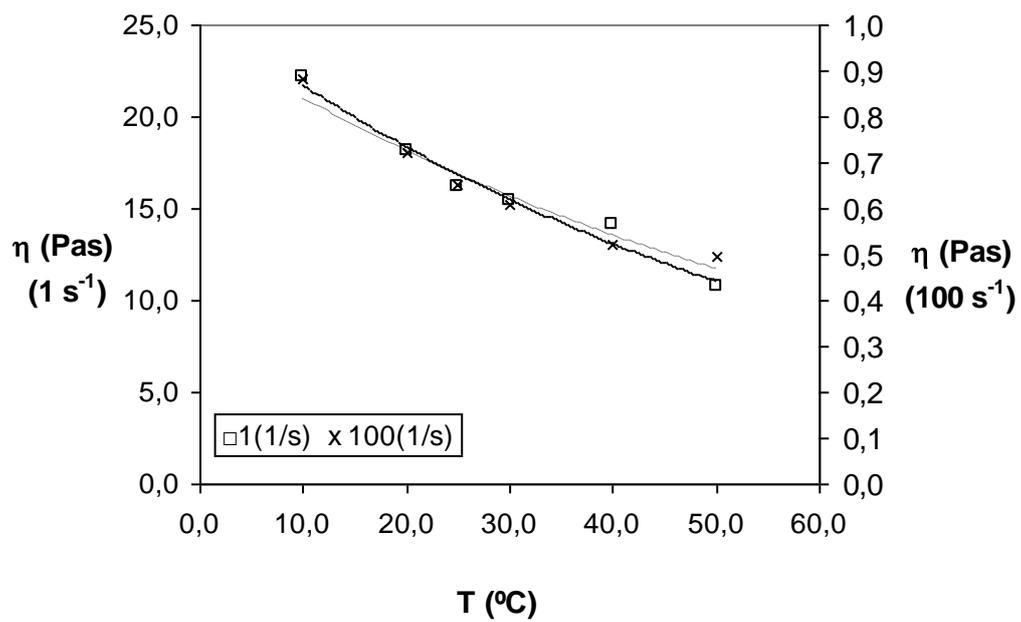


Figure 9



**Table 1** Stirring time (min) for samples prepared at different temperatures (25 and 60 °C) and by using different homogenizers (Ultraturrax and Heidolph). The code identifying each one has been placed in brackets.

**Table 2** Average and standard deviation of the Herschel-Bulkley or Power Law parameters obtained from the different lemon fibre suspensions.

**Table 3** Average values and standard deviation for parameters of the Hahn model fitted to the stress values obtained at a constant shear rate of  $5 \text{ s}^{-1}$  for a maximum time period of 600 s.

**Table 4** Average values and standard deviation for the Herschel-Bulkley model parameters fitted to the values of flow curves obtained from lemon fibre dispersions at different concentration levels, 24 hours ( $t_0$ ) after preparation and after a 20-day storage period ( $t_{20}$ ).

**Table 5** Average values and standard deviation for parameters of the Herschel-Bulkley or Power Law models fitted to experimental rheograms corresponding to lemon fibre obtained at different temperatures (T).

Table 1  
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Temperature (°C)	Homogenizer process	
	Time (min)	Time (min)
	Ultraturrax	Heidolph
25	7 (U <sub>25-7</sub> )	30 (H <sub>25-30</sub> )
	15 (U <sub>25-15</sub> )	
	20 (U <sub>25-20</sub> )	
60	10 (U <sub>60-10</sub> )	45 (H <sub>60-45</sub> )

Table 4

Concentration (%) and time	$\sigma_0$ (Pa)	k(Pas <sup>n</sup> )	n	Adjustment	
				SEE	R <sup>2</sup>
2.0 (t <sub>0</sub> )	1.8±0.3 <sup>(a)</sup>	1.41±0.13 <sup>(a)</sup>	0.63±0.02 <sup>(a)</sup>	0.21	0.9991
2.0 (t <sub>20</sub> )	2.4±0.7 <sup>(b)</sup>	2.3±0.3 <sup>(a)</sup>	0.58±0.03 <sup>(a)</sup>	0.42	0.9978
2.25(t <sub>0</sub> )	8.6±0.3 <sup>(c)</sup>	3.6±0.2 <sup>(b)</sup>	0.53±0.02 <sup>(b)</sup>	0.382	0.9988
2.25(t <sub>20</sub> )	10.4±1.6 <sup>(d)</sup>	3.8±0.7 <sup>(b)</sup>	0.55±0.04 <sup>(b)</sup>	0.852	0.9956
2.5 (t <sub>0</sub> )	9±2 <sup>(c)</sup>	5.1±1.0 <sup>(c)</sup>	0.50±0.04 <sup>(c)</sup>	0.89	0.9953
2.5(t <sub>20</sub> )	13±2 <sup>(d)</sup>	6.4±1.1 <sup>(c)</sup>	0.49±0.03 <sup>(c)</sup>	0.92	0.9966
2.75(t <sub>0</sub> )	11±3 <sup>(c)</sup>	11±2 <sup>(d)</sup>	0.42±0.03 <sup>(d)</sup>	1.20	0.9956
2.75(t <sub>20</sub> )	15±5 <sup>(d)</sup>	11±3 <sup>(d)</sup>	0.45±0.05 <sup>(d)</sup>	1.81	0.9922
3.0(t <sub>0</sub> )	14±6 <sup>(e)</sup>	21±4 <sup>(e)</sup>	0.37±0.03 <sup>(e)</sup>	1.75	0.9952
3.0(t <sub>20</sub> )	20±8 <sup>(f)</sup>	19±5 <sup>(e)</sup>	0.40±0.05 <sup>(e)</sup>	2.81	0.9903

SEE: Standard error of the Estimation; R<sup>2</sup>: Coefficient of determination.

Within a column, values with the same following letter do not differ significantly from each other (p>0.05)

**Table 5**

<b>T (°C)</b>	<b><math>\sigma_0</math> (Pa)</b>	<b>k (Pas<sup>n</sup>)</b>	<b>n</b>	<b>Adjustment</b>	
				<b>SEE</b>	<b>R<sup>2</sup></b>
10	14±2 <sup>(a)</sup>	7.8±1.1 <sup>(a)</sup>	0.49±0.03 <sup>(a)</sup>	0.93	0.9976
20	12±2 <sup>(a)(b)</sup>	6.7±1.1 <sup>(a)</sup>	0.48±0.03 <sup>(b)</sup>	0.88	0.9967
25	9±2 <sup>(b)</sup>	7.2±1.3 <sup>(a)</sup>	0.44±0.03 <sup>(b)</sup>	0.87	0.9960
30	8±3 <sup>(b)</sup>	7.2±1.9 <sup>(a)</sup>	0.43±0.05 <sup>(c)</sup>	1.20	0.9911
40	9±3 <sup>(b)</sup>	5.6±1.4 <sup>(a)</sup>	0.44±0.05 <sup>(b)</sup>	0.97	0.9918
50	n.s.	10.7±0.5	0.333±0.012	1.14	0.9884

SEE: Standar error of the estimate; R<sup>2</sup>: Coefficient of determination.

Within a column, values with the same following letter do not differ significantly from each other (p>0.05)

**Table 2**

<b>Sample<sup>(1)</sup></b>	<b><math>\sigma_0</math> (Pa)</b>	<b><math>k</math> (Pa·s<sup>n</sup>)</b>	<b><math>n</math></b>	<b><math>R^2</math><sup>(2)</sup></b>	<b>SEE<sup>(3)</sup></b>
H <sub>25-30</sub>	n.s.	1.64±0.08	0.599±0.011	0.9975	0.36
H <sub>60-45</sub>	n.s.	2.91±0.09	0.521±0.006	0.9986	0.33
U <sub>25-7</sub>	10±2 <sup>(a)</sup>	4.8±0.9 <sup>(a)</sup>	0.51±0.04 <sup>(a)</sup>	0.9954	0.87
U <sub>25-15</sub>	13±4 <sup>(a)</sup>	12±3 <sup>(b)</sup>	0.38±0.04 <sup>(b)</sup>	0.9935	1.26
U <sub>25-20</sub>	27±4 <sup>(b)</sup>	11±2 <sup>(b)</sup>	0.40±0.04 <sup>(b)</sup>	0.9950	1.15
U <sub>60-10</sub>	25±6 <sup>(b)</sup>	14±4 <sup>(b)</sup>	0.40±0.05 <sup>(b)</sup>	0.9893	2.07

<sup>(1)</sup>H: Heidolph; U: Ultraturrax; 1<sup>st</sup> number refers to temperature and 2<sup>nd</sup> one to time.

<sup>(2)</sup> $R^2$ : coefficient of determination

<sup>(3)</sup>SEE: standard error of the estimate

n.s.: not statistically significant ( $p>0.05$ )

Within a column, values with the same following letter do not differ significantly from each other ( $p>0.05$ )

**Table 3**

<b>Parameter</b>	<b>2.0%</b>	<b>2.5%</b>	<b>3.0%</b>
<b>p (Pa)</b>	1.9±0.2	2.7±0.2	3.49 ±0.10
<b>a (s<sup>-1</sup>)</b>	0.0097 ±0.0009	0.0080 ±0.0006	0.0089± 0.0004
<b>R<sup>2</sup>*</b>	0.8852	0.8878	0.9474
<b>SEE*</b>	0.4386	0.4066	0.2632

\*SEE = Standard Error of the Estimation; R<sup>2</sup>: coefficient of determination