Rheological and thixotropic behavior of banana (Musa cavendishii) puree

Ibarz, A., Falguera, V. and Garvín, A*

Departament de Tecnologia d'Alimentos UTPV-XaRTA, Universitat de Lleida,
Av. Rovira Roure, 191, CP 25198 Lleida – España.

Comportamiento reológico y tixotrópico del puré de plátano (Musa cavendishii)

Comportament reològic i tixotròpic del puré de plàtan (Musa cavendishii)

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RESUMEN

Se han estudiado las propiedades reológicas del puré de plátano. Para cuantificar el comportamiento tixotrópico, se han ajustado los datos experimentales a las ecuaciones de Hahn, Weltman i Figoni-Shoemaker. El estudio de la tixotropía se realizó en un rango de temperaturas entre 5 y 75ºC y se aplicó un gradiente de velocidad de 10 y 20 s^{-1}, encontrando en todos los casos que la ecuación de Figoni-Shoemaker describe mejor la dependencia con el tiempo.
El comportamiento al flujo se ajustó utilizando el modelo de Herschel-Bulkley en un rango de temperaturas entre 5 y 75ºC, y para concentraciones entre 12 y 22 ºBrix, obteniendo que el coeficiente de consistencia disminuye con la temperatura y aumenta con el contenido en sólidos solubles, el índice de comportamiento al flujo disminuye con el contenido en sólidos solubles y la viscosidad aparente disminuye con la temperatura y con el contenido en sólidos solubles. Se obtuvo la energía de activación al flujo para diferentes contenidos en sólidos solubles mediante el ajuste de la variación de la viscosidad aparente con la temperatura a una ecuación de tipo Arrhenius.

Palabras clave: Reología, tixotropía, viscosidad, puré, plátano.

SUMMARY

Some rheological properties of banana puree were investigated. Experimental data was fitted to Hahn, Weltman and Figoni-Shoemaker equations in order to quantify the thixotropic behavior. Thixotropy measurements were carried out in a range between 5 and 75ºC and applying shear rates of 10 and 20 s^{-1}, finding that, in all cases, the Figoni-Shoemaker relationship was the best one to model the time dependency response.
The flow behavior was well fitted by the Herschel-Bulkley model in a range between 5 and 75ºC, and between 12 and 22 ºBrix, obtaining that consistency coefficient decreased with temperature and increased with soluble solids content, flow behavior index decreased with soluble solids content, and apparent viscosity decreased with temperature and increased with soluble solids content. Adjusting the variation of apparent viscosity with temperature by means of an Arrhenius-type equation, the activation energy was obtained for different soluble solids content.

Keywords: Rheology, thixotropy, viscosity, puree, banana.

RESUM

S'han estudiat les propietats reològiques del puré de plàtan. Per tal de quantificar el comportament tixotròpic, les dades experimentals s'han ajustat a les equacions de Hahn, Weltman i Figoni-Shoemaker. L'estudi de la tixotropia es van realitzar en un rang de temperatures entre 5 i 75ºC i aplicant un gradient de velocitat de 10 i 20 s^{-1}, trobant que en tots el casos, l'equació de Figoni-Shoemaker descrivia millor la dependència amb el temps.
El comportament al flux es va ajustar segons el model de Herschel-Bulkley en un rang de temperatures entre 5 i 75ºC, i per concentracions entre 12 i 22 ºBrix, obtenint que el coeficient de consistència disminueix amb la temperatura i augmenta amb el contingut en sòlids solubles, l'índex de comportament al flux disminueix amb el contingut en sòlids solubles, i la viscositat aparent disminueix amb la temperatura i amb el contingut en sòlids solubles. Es va obtenir l'energia d'activació per a diferents continguts en sòlids solubles ajustant la variació de la viscositat aparent amb la temperatura mitjançant una equació de tipus Arrhenius.

Paraules clau: Reologia, tixotropia, viscositat, puré, plàtan.

Corresponding authors: aibarz@tecal.udl.cat,vfalguera@tecal.udl.cat, *garvin@tecal.udl.cat

INTRODUCCTION

Plant food fluids represent a very extensive and important sector of the food industry. Most of them are manufactured under industrial processes that change their original structure, generating a fluid in which the dispersed material contributes significantly to its rheological behavior. Determination of rheological properties of food, from the simplest to the most complex, is necessary at different stages of the food process: preserving, transformation, storage stability and consumption (Falguera *et al*., 2010).

Food fluid products manufacturing includes different food process unit operations, from transportation from the ground or farm to the final product, pumping through pipelines, pasteurization, filtration, evaporation, sterilization, among others, and finally some processes that preserve their shelf life, such as refrigeration and freezing, among others.

Rheological properties of food fluids are necessary to predict engineering parameters to develop the process and design the equipment. A correct design of unit operations is essential to optimize the process and prevent over-dimension of means and consequently avoid an economic waste.

Some fruit fluids show thixotropy, which is identified by a continuous decreasing of the apparent viscosity with shearing time and a recovering of this viscosity once that shear rate is stopped (Lozano and Ibarz, 1994). The thixotropic response of a fluid may be determined by measuring shear stress as a function of time at a given shear rate that remains constant during the instrumental test. There are different rheological models that may fit the thixotropic behavior of fluid food. Three of the most used relationships are the following ones (Weltman, 1943; Hahn *et al.*, 1959; Figoni and Shoemaker, 1983):

Weltman (1943) $$\sigma = A - B \ln t \quad (1)$$

Hahn et al. (1959) $$\log(\sigma - \sigma_e) = p - at \quad (2)$$

Figoni and Shoemaker (1983)
$$\sigma = \sigma_e + (\sigma_i - \sigma_e)\exp(-kt) \quad (3)$$

Where: σ is the shear stress (Pa), σ_i and σ_e are the initial and equilibrium stresses (Pa), which are related with initial and long times, respectively, and *A*, *B*, *p*, *a* and *k* are model parameters for each equation.

Rheological behavior of food fluids may be described by different constitutive equations that correlate the two basic measures of shear stress and shear rate. In spite of the fact that there are a large number of rheological models, several researchers (Rha, 1978; Rao *et al.*, 1984; Kokini, 1992; Canet *et al.*, 2005) have mentioned that one of the most useful and applicable equations to fit the flow response of experimental data for many fluids, is the Herschel and Bulkley model:

$$\sigma = \sigma_0 + K \cdot \gamma^n \quad (4)$$

Where: σ is the shear stress (Pa), σ_0 is the yield stress (Pa), is the shear rate (s^{-1}), *K* is the consistency coefficient ($Pa \cdot s^n$), and *n* is the flow behavior index (dimensionless).

The flow behavior of fruit juices and their fluid derivates is strongly affected by the juice characteristics as well as the fruit properties. The presence of pulp solids in the dispersed phase in the fruit juice contributes to the non-Newtonian nature and particularly to developing the yield stress parameter. It was observed that many of the fruit juices and purees are well fitted by the Herschel and Bulkley model (Maceiras *et al.*, 2007; Vélez-Ruiz, 2009).

The main purpose of this work is to analyze the rheological behavior of banana puree at different temperatures (5, 15, 25, 35, 45, 55, 65 and 75ºC) and soluble solids content (12, 15, 18 and 22ºBrix), studying its flow response and its thixotropic nature.

MATERIAL AND METHODS

Samples were provided by NUFRI, a juice industry located in Mollerussa (Lleida, Spain). These samples were obtained from *Musa Cavendishii,* from Costa Rica.

The analysis of the physicochemical characteristics of the samples was carried out according to the IFFJP (1991) methods.

Soluble solids were obtained using a digital refractometer Atago RX-100.

Sugar content was determined with Fehling reagent.

pH was measured at 20ºC using a Crison MicropH 2001 pH-meter.

Pulp content was measured using a Selecta centrifuge.

Formol index shows the amino acid content. The method consists of a titration with NaOH 0.1M of the acidity of the compounds formed by the reaction of formaldehyde and amino acids contained initially in the puree.

Pectin content was determined using a Philips PU 8700 spectrophotometer.

The obtained values for physicochemical characteristics are listed in table 1.

Table 1.- *Physicochemical characteristics*

Soluble solids (ºBrix)	22
Sugar content (g/L)	81
pH	4.5
Pulp content (%)	40
Formol index (mL/mL)	23
Pectin content (mg G.A./kg)	56

Rheological measurements

Rheological measurements were carried out using a Haake RS 80 rheometer provided with a cone-plate sensor. The temperature of the samples was kept constant at the desired value using a TC 501 equipment with a precision of ± 0,2ºC. Preliminary tests indicated that a resting time of 300 seconds after loading the sample was enough to get reproducible results. Startup experiments were performed measuring the shear stress versus time when a constant shear rate was applied to the sample. At least two replicas were made for every experiment.

Rheological behavior was studied at shear rates from 0 to 1.500 s^{-1}, and thixotropy was evaluated at two shear rates: 10 and 20 s^{-1}.

Statistical analysis

The experimental results obtained in this study were fitted to different kinetic and mathematical models using the Statgraphics Plus 5.1 statistic data processing software (STSC In. Rockville, Md, USA). All adjustments and estimates were calculated at a 95% significance level.

Table 2.- Values of rheological parameters of Weltmann equation as a function of temperature and shear rate

Weltman: $\sigma = A - B\ln t$						
	$\dot{\gamma}$ =10s^{-1}			$\dot{\gamma}$ =20s^{-1}		
T (ºC)	**A** (Pa)	**B** (Pa)	**r²**	**A** (Pa)	**B** (Pa)	**r²**
5	37.84±0.05	2.31±0.08	0.9242	40.93±0.03	1.98±0.05	0.7888
15	39.66±0.06	2.60±0.2	0.9392	39.53±0.03	1.88±0.07	0.8314
25	37.15±0.05	2.50±0.1	0.8802	36.53±0.03	1.69±0.05	0.7287
35	30.34±0.07	1.87±0.07	0.9312	31.19±0.03	1.30±0.09	0.7761
45	30.14±0.03	2.04±0.08	0.8895	26.65±0.03	0.68±0.06	0.4595
55	27.52±0.05	1.48±0.05	0.9282	27.60±0.03	0.87±0.07	0.4598
65	26.41±0.02	1.10±0.04	0.9387	24.93±0.03	0.58±0.08	0.6308
75	23.96±0.04	0.79±0.05	0.7829	26.68±0.03	0.74±0.07	0.6054

Table 3.- Values of rheological parameters of the Hahn equation as a function of temperature and shear rate

Hahn: $\log(\sigma - \sigma_e) = p - at$								
T	$\dot{\gamma}$ =10s^{-1}				$\dot{\gamma}$ =20s^{-1}			
ºC	σ_e (Pa)	P (Pa)	a (Pa·s^{-1})	r²	σ_e (Pa)	P (Pa)	a (Pa·s^{-1})	r²
5	26.09±0.05	2.302±0.002	0.0288±0.0004	0.9846	31.40±0.07	2.479±0.008	0.0636±0.0005	0.9739
15	25.85±0.07	2.434±0.003	0.0245±0.0005	0.9893	30.53±0.05	2.415±0.005	0.0625±0.0007	0.9826
25	24.26±0.06	2.350±0.002	0.0284±0.0003	0.9593	28.50±0.02	2.377±0.007	0.0663±0.0007	0.9823
35	20.82±0.02	2.101±0.005	0.0278±0.0007	0.9816	24.63±0.01	2.154±0.008	0.0700±0.0004	0.9525
45	19.95±0.07	2.151±0.001	0.0311±0.0005	0.9783	23.48±0.05	1.938±0.006	0.0979±0.0006	0.8618
55	20.12±0.03	1.901±0.008	0.0337±0.0005	0.9887	23.42±0.07	1.990±0.01	0.0915±0.0005	0.8627
65	20.79±0.06	1.493±0.005	0.0250±0.0008	0.9855	22.13±0.04	1.740±0.01	0.0895±0.0004	0.7717
75	20.11±0.08	1.425±0.007	0.0473±0.0004	0.8960	23.09±0.03	1.649±0.007	0.0722±0.0007	0.8742

Table 4.- Values of rheological parameters of Figoni-Shoemaker equation as a function of temperature and shear rate

Figoni-Shoemaker: $\sigma = \sigma_e + (\sigma_i - \sigma_e)\exp(-kt)$								
T	$\dot{\gamma}$=10s^{-1}				$\dot{\gamma}$=20s^{-1}			
ºC	σ_i (Pa)	σ_e (Pa)	k (s^{-1})	r²	σ_i (Pa)	σ_e (Pa)	k (s^{-1})	r²
5	36.09±0.02	26.10±0.05	0.0289±0.0005	0.9846	43.34±0.05	31.40±0.07	0.0630±0.003	0.9739
15	37.26±0.06	25.85±0.08	0.0245±0.0002	0.9893	41.73±0.05	30.53±0.05	0.0625±0.0008	0.9826
25	34.74±0.04	24.26±0.02	0.0284±0.0002	0.9593	39.28±0.03	28.50±0.06	0.0663±0.0005	0.9823
35	29.01±0.07	20.82±0.07	0.0278±0.0003	0.9815	33.25±0.08	24.63±0.04	0.0700±0.0003	0.9525
45	28.54±0.05	19.95±0.03	0.0311±0.0005	0.9783	30.43±0.04	23.48±0.04	0.0978±0.0004	0.8618
55	26.82±0.04	20.12±0.05	0.0337±0.0007	0.9887	30.81±0.05	23.43±0.05	0.0916±0.0003	0.8627
65	25.24±0.03	20.79±0.06	0.0250±0.0005	0.9855	27.84±0.06	22.13±0.06	0.0895±0.0008	0.7717
75	24.26±0.04	20.11±0.07	0.0473±0.0003	0.8959	28.30±0.03	23.09±0.02	0.0722±0.0007	0.8742

RESULTS AND DISCUSSION

Thixotropy

Figure 1 shows the evolution of shear stress as a function of time at a temperature of 5ºC. The quantification of thixotropy was conducted adjusting experimental data to equations (1), (2) and (3) for all analyzed shear rates and temperatures. Tables 2, 3 and 4 summarize all parameters of the three theoretical equations for thixotropy.

In all cases, the best fittings were obtained with Figoni-Shoemaker model.

This model describes the thixotropic behavior in a kinetic constant of breakdown of the internal structure of banana puree. Moreover, this model allows to quantify the structure breakdown by means of the parameter $(\sigma_i - \sigma_e)$ and the quantity of remaining structure with the parameter (σ_e). An increase in the shear rate $(\dot{\gamma})$ causes an increase in initial shear stress (σ_i), in equilibrium shear stress (σ_e) and in their difference (σ_i-σ_e), showing that an increase in the applied shear rate causes more internal structure breaking.

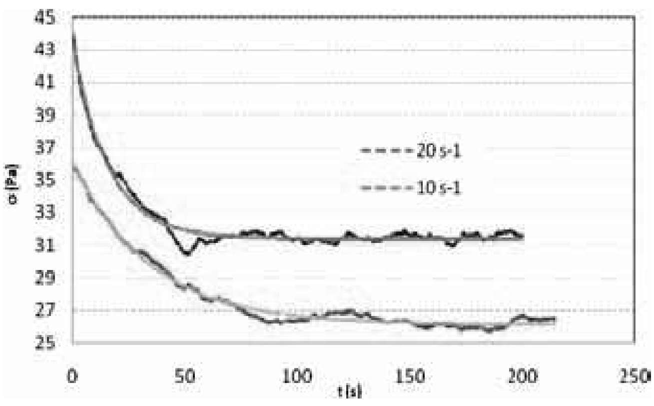

Figure 1.- Shear stress evolution at 5ºC Rheological behavior

Table 5.- Values of rheological parameters of the Herschel-Bulkley model at different concentrations and temperatures.

$$\sigma = \sigma_0 + K \cdot \dot{\gamma}^n$$

C(ºBrix)	T(ºC)	σ_0 (Pa)	K(Pa·s^n)	n	$\eta_{ap\ 100s^{-1}}$ (Pa·s)	r^2
22	5	4.19±0.06	20.8±0.8	0.203±0.04	0.57±0.1	0.9745
	15	2.29±0.04	22.8±0.6	0.179±0.03	0.54±0.09	0.9338
	25	1.90±0.3	17.2±0.8	0.209±0.02	0.47±0.06	0.9317
	35	1.14±0.08	20.5±0.7	0.187±0.08	0.49±0.2	0.8200
	45	1.64±0.05	13.7±0.5	0.214±0.07	0.38±0.1	0.9861
	55	1.73±0.06	14.0±0.5	0.216±0.01	0.39±0.03	0.9860
	65	1.31±0.07	11.4±0.7	0.251±0.05	0.37±0.1	0.9578
	75	1.90±0.5	11.5±0.8	0.225±0.06	0.34±0.1	0.9581
18	5	5.48±0.9	7.98±0.7	0.327±0.01	0.41±0.06	0.9725
	15	4.74±0.8	7.10±0.6	0.322±0.01	0.36±0.05	0.9717
	25	2.88±0.2	6.52±0.2	0.319±0.01	0.31±0.02	0.3695
	35	1.21±0.4	6.33±0.4	0.226±0.03	0.19±0.04	0.7181
	45	1.35±0.2	6.41±0.2	0.274±0.02	0.24±0.03	0.8989
	55	1.10±0.07	6.40±0.06	0.294±0.007	0.26±0.01	0.9908
	65	1.06±0.1	7.69±0.2	0.185±0.05	0.19±0.05	0.8874
	75	1.71±0.1	6.70±0.3	0.217±0.05	0.20±0.05	0.9049
15	5	2.45±0.5	2.46±0.3	0.419±0.01	0.19±0.03	0.9750
	15	2.29±0.5	2.07±0.2	0.422±0.02	0.17±0.03	0.9668
	25	2.28±0.5	2.59±0.2	0.386±0.04	0.18±0.05	0.8142
	35	2.20±0.4	2.67±0.3	0.361±0.01	0.16±0.03	0.9688
	45	1.87±0.4	2.46±0.3	0.359±0.01	0.15±0.02	0.9657
	55	1.93±0.3	2.11±0.2	0.367±0.01	0.13±0.02	0.9784
	65	1.80±0.2	1.90±0.1	0.375±0.01	0.12±0.01	0.9835
	75	2.00±0.2	1.41±0.1	0.401±0.01	0.11±0.01	0.9824
12	5	2.22±0.4	1.11±0.1	0.49±0.02	0.13±0.02	0.9717
	15	1.91±0.5	1.10±0.2	0.46±0.03	0.11±0.03	0.9443
	25	1.90±0.4	0.85±0.1	0.48±0.02	0.10±0.02	0.9601
	35	1.98±0.6	1.21±0.4	0.34±0.03	0.08±0.03	0.9809
	45	1.40±0.3	1.05±0.1	0.42±0.02	0.09±0.02	0.9636
	55	1.38±0.2	0.96±0.1	0.42±0.02	0.08±0.01	0.9679
	65	1.00±0.2	0.70±0.08	0.44±0.01	0.06±0.01	0.9761
	75	1.19±0.2	0.52±0.08	0.47±0.02	0.05±0.01	0.9644

Meanwhile, an increase in the temperature causes a decrease in the initial shear stress (σ_i), in equilibrium shear stress (σ_e) and in their difference (σ_i-σ_e), showing that an increase in the temperature causes more internal structure breaking. Breakdown kinetic constant (*k*) increases when both the shear rate and the temperature are increased, so the breakdown structure rate increases if the shear rates and the temperature increases.

Rheological behavior

Table 5 shows the parameters obtained with the Herschel-Bulkley model. The values of the apparent viscosity at a shear rate of 100 s^{-1} are included.

The yield stress (σ_0) decreases when temperature increases, although there is not a clear pattern. It also does not clearly relate to the concentration.

The consistency coefficient (*K*) decreases as the temperature increases. In Table 6 it can be seen that the consistency coefficient increases with the soluble solids content following a power pattern, and the parameters of these adjustments are showed.

Table 6.- Potential equation adjustments for the relation between Consistency index and soluble solid contents.

$$K = K_0 C^b$$

T (ºC)	$K_0 \cdot 10^6$	b	r^2
5	4,42	4,96	0,9941
15	2,66	5,12	0,9856
25	3,58	4,98	0,9999
35	10,00	4,66	0,9933
45	2,15	4,33	0,9980
55	1,07	4,56	0,9947
65	4,00	4,88	0,9795
75	7,94	5,39	0,9828

The flow behavior index (n) does not clearly follow a defined pattern with the temperature. In table 7 it can be seen that the flow behavior index decreases linearly with the soluble solids content, and the parameters of these adjustments are showed. In all cases, the flow behavior index is always clearly below the unit, thus the rheological behavior is pseudoplastic.

Table 7.- Linear equation adjustments for the relation between flow behavior index and soluble solid contents.

$n \quad n \; - mC$			
T (ºC)	n_0	$m \cdot 10^2$	r^2
5	0,84	2,86	0,9978
15	0,82	2,85	0,9805
25	0,79	2,65	0,9986
35	0,58	1,79	0,9014
45	0,68	2,14	0,9921
55	0,67	2,09	0,9987
65	0,67	2,16	0,8047
75	0,78	2,69	0,9093

The apparent viscosity decreases as the temperature increases. In table 8 it can be seen that the apparent viscosity increases linearly with the soluble solids content, and the parameters of these adjustments are showed.

Table 8.- Linear equation adjustments for the relation between apparent viscosity and soluble solid contents.

$\eta_{ap} = \eta_0 + xC$			
T (ºC)	η_0	$x \cdot 10^2$	r^2
5	0,46	4,73	0,9841
15	0,47	4,55	0,9856
25	0,38	3,86	0,9957
35	0,44	4,01	0,9357
45	0,29	3,02	0,9922
55	0,33	3,28	0,9906
65	0,33	3,12	0,9766
75	0,31	2,91	0,9890

The variation in apparent viscosity with temperature can be described by an Arrhenius-type equation (Saravacos 1970; Rao *et al*. 1984; Ibarz *et al*. 1992a; Ibarz *et al*. 1992b):

$$\eta_a = \eta_\infty \exp\left[\frac{E_a}{RT}\right] \quad (4)$$

where: η_a is the apparent viscosity (Pa·s), η_∞ is the apparent viscosity at infinite temperature (Pa·s), E_a is the activation energy of flow (kJ·mol^{-1}), R is the Gas constant, and T is the absolute temperature (K). The values obtained for different soluble solids content are shown in Table 9. Flow activation energy increases with the soluble solids content and the apparent viscosity at infinite temperature decreases.

Table 9.- Values of parameters of the Arrhenius equation at different concentrations.

C	E_a	η_∞
° Brix	kJ·mol^{-1}	Pa·s
12	3.72±0.07	5.68±0.09
15	4.02±0.08	4.22±0.1
18	4.41±0.07	4.59±0.08
22	5.88±0.05	3.09±0.07

CONCLUSIONS

The conclusions extracted from our study are:

- The banana puree of 22ºBrix shows thixotropy and the Figoni-Shoemaker model fits the best.
- An increase in the shear rate (γ) causes an increase in initial shear stress (σ_i), in equilibrium shear stress (σ_e), in their difference (σ_i-σ_e) and in the breakdown kinetic constant (*k*).
- An increase in the temperature causes a decrease in the initial shear stress (σ_i), in equilibrium shear stress (σ_e), in their difference (σ_i-σ_e) and an increase in the breakdown kinetic constant (*k*).
- Regarding flow behavior data, the Herschel-Bulkley model fits the best and the flow behavior index is always below the unit.
- The yield stress (σ_0) decreases with temperature and does not clearly relate with soluble solids content.
- The consistency index (*K*) decreases with temperature and increases potentially with soluble solids content.
- The flow behavior index (*n*) does not clearly relate with temperature and decreases linearly with the soluble solids content.
- The variation in apparent viscosity with temperature can be described by an Arrhenius-type equation. The activation energy (E_a) increases with soluble solids content and the apparent viscosity at infinite temperature decreases with soluble solids content.

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