

Linear and non-linear viscoelasticity of low-in-cholesterol mayonnaise

Viscoelasticidad lineal y no lineal de mayonesas con bajo contenido en colesterol

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Five different mayonnaise products were prepared with constant composition except for the type of egg product used. The linear viscoelastic functions were superposed using a time-temperature superposition method and the shift-factor showed an Arrhenius-like temperature dependence. The thermal susceptibility was found to be higher for processed-egg-containing mayonnaise and when egg yolk was used instead of whole egg as the emulsifier. Transient flow curves always showed a stress overshoot, but a stress undershoot was found for whole-egg-containing emulsions at high shear rates. Processed yolk gave rise to higher storage modulus values than did native yolk. This effect lost significance when yolk was diluted by native egg white. Mayonnaise made from egg yolk was always more viscous and did not show a stress undershoot in transient flow. The non-linear relaxation modulus was factorized as the product of the linear relaxation modulus and the damping function. Hence, the Wagner model was able to predict the transient flow of these emulsions fairly well. However, this model failed at low shear rates. This fact may be explained on the basis of wall-slip phenomena.

Keywords: mayonnaise, viscoelasticity, viscosity, cholesterol removal, emulsifier

Se han evaluado las propiedades viscoelásticas de cinco mayonesas de idéntica composición a excepción del tipo del derivado de huevo con el que se prepararon. Las funciones viscoelásticas lineales de estos productos se superpusieron mediante un método de superposición tiempo-temperatura, los factores de superposición utilizados mostraron una dependencia con la temperatura de tipo Arrhenius. Esta dependencia fue mayor en las mayonesas que contenían huevo manufacturado o yema de huevo, en vez de huevo entero, como emulsionante. El flujo transitorio mostró siempre un máximo del esfuerzo, aunque también se observó un mínimo del mismo a altas velocidades de cizalla en emulsiones que contenían huevo completo. El huevo manufacturado dio lugar a mayores valores del módulo de almacenamiento que el huevo sin tratar. Este efecto pierde significación cuando la yema se diluye con clara de huevo nativo. La mayonesa preparada con yema de huevo fue siempre más viscosa y no mostró un mínimo del esfuerzo en flujo transitorio. El módulo de relajación no lineal se factorizó como el producto del módulo de relajación lineal y la función de amortiguación. Como resultado, se pudo usar el modelo de Wagner para simular el flujo transitorio de estas emulsiones. Sin embargo, este modelo no pudo ser utilizado a baja velocidad de cizalla. Este hecho se puede explicar por fenómenos de deslizamiento de la pared del sensor.

Palabras clave: mayonesa, viscoelasticidad, viscosidad, extracción de colesterol, emulsionante

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INTRODUCTION

The primary industrial use of egg yolk is the manufacture and stabilization of emulsions such as mayonnaise, salad dressing and sauces. Egg proteins and phospholipids act as emulsifiers which allow the formation of stable oil/water emulsions, giving the desired appear-

ance, mouthfeel and texture to mayonnaise. Other components of egg yolk contribute to the flavor and color, which should not be intensified by any other ingredient of mayonnaise. Egg yolk also contributes to the cholesterol in a proportion of about 5% of the yolk (Shenstone, 1968).

There is considerable consumer interest in traditional foods, such as mayonnaise, with low cholesterol content. The consumer would prefer that the low or cholesterol-free food have all of the sensory qualities of the traditional food. Knowledge of viscous and viscoelastic properties of food emulsions is essential for the design of flow processes and unit operations, providing useful information for quality control, storage stability and sensory evaluation of textural properties concerning the consumer's acceptance of the commercial product (Rahalkar, 1992).

Several authors have conducted research into the emulsion stability and rheological properties of mayonnaise at steady and transient flow (Tiu and Boger, 1974, Bistany and Kokini, 1983, Fighi and Shoemaker, 1983, Goshawk *et al.*, 1998), oscillatory shear (Elliot and Ganz, 1977, Berjano *et al.*, 1990, Muñoz and Sherman, 1991, Gallegos *et al.*, 1992a, Franco *et al.*, 1995) and creep tests (Kiosseoglou and Sherman, 1983). Linear and non-linear viscoelasticity data have been used to describe the transient flow of commercial mayonnaise by a non-linear viscoelasticity model (Wagner's model) (Campanella and Peleg, 1987; Gallegos *et al.*, 1992b).

However, the processing of egg yolk may produce some unwanted effects on the egg ingredients such as lipid oxidation, protein denaturation or disruption of the phospholipid-protein complexes which provide the maximum functional performance of egg yolk (Burley and Vadehra, 1989). Consequently, the rheological characteristics and stability of their emulsions may be altered.

Based on these considerations, a study of the linear and non-linear viscoelastic properties of emulsions stabilized with native and processed egg products (spray-dried and cholesterol-extracted) was carried out. The main goal of this study was to contribute to the development of a low-cholesterol mayonnaise with suitable rheological properties compared with those found in commercially accepted mayonnaise by studying the influence that the use of processed egg yolks exerts on the linear and non-linear viscoelasticity of mayonnaise.

MATERIALS AND METHODS

Five different mayonnaise products were prepared with constant composition (73% soybean oil; 16% egg product; 10% white vinegar and 1% salt) but varying the type of egg product used as the emulsifier (Table 1).

Control of the mayonnaise preparation procedure was found to be very important. Thus, special care was taken to prepare every batch following exactly the same

procedure. Each mayonnaise was prepared by adding two thirds of the total oil content to a mixture of egg, vinegar, salt and the remaining oil which had been placed in a Warring Blendor jar. The addition was carried out while blending at about 5000 rpm. All the products were stored at 4 °C after the preparation process.

Dynamic and transient flow measurements (three replicates) were performed in a Bohlin rheometer, Viscosity-Oscillation-Relaxation model, using a Mooney-Ewart-type sensor system, C14, ($R_b/R_c = 1.1$). Six temperatures (from 5 to 30 °C) were tested for each mayonnaise in oscillatory tests at frequencies (ω) ranging from 0.031 to 31.4 s⁻¹. Transient flow measurements were carried out at a constant shear rate which ranged from 0.06 to 5.83 s⁻¹. Temperature was kept constant at 20 °C. Stress relaxation tests were made for shear strains ranging from 0.02–1.00, at 20 °C.

The effects of ageing as well as a statistical study (for a significance level of 95%) were discussed in a previous paper (Guerrero and Ball, 1994). Since these mayonnaise products presented a decrease in the viscoelastic functions with ageing followed by a plateau region, it was necessary to wait for 15 days after the preparation of the mayonnaise before taking any measurements.

RESULTS AND DISCUSSION

Linear dynamic viscoelasticity

A strain sweep test was previously conducted at 20 °C and 1 Hz for each mayonnaise in order to find the viscoelastic linear range. The linear range depended on the type of mayonnaise evaluated. Thus the products prepared from yolk showed a wider linear range than those prepared from whole egg. A small value for maximum strain amplitude, 0.006, which assures permanence in the linear range, was selected to perform oscillatory tests.

Table 1. Egg products used in the preparation of mayonnaise.

Tabla 1. Derivados de huevo utilizados en la preparación de las mayonesas.

Mayonnaise	Egg derivate		
	Yolk (50% solids)	White (12% solids)	Solids Content (%)
NW	Native	Native	24
DW	Dried ^a /water	Native	24
LW	Low-in-cholesterol ^b / water	Native	24
NY	Native	Native	43
DY	Dried/water	Native	43

^aSolids content: 96–97%.

^bSolids content: 97–98%.

The storage modulus, G' , was always higher than the loss modulus, G'' , within the experimental frequency range (Figure 1). Hence, the emulsions presented a predominantly elastic response. Moreover, there is a tendency for G' to exhibit a plateau (Figure 1a), accompanied by a minimum in loss tangent. Although emulsion NY did not present that minimum in the experimental frequency window, it would be expected at lower frequencies, corresponding to a flocculated emulsion (Franco *et al.*, 1995). This zone, at intermediate frequencies, is known as the plateau region. In emulsion rheology, this region is related to the formation of an elastic structural network due to interactions among the emulsifier molecules located at the oil–water interface of adjacent droplets (Dickinson, 1989, Franco *et al.*, 1995, Guerrero *et al.*, 1998). When the emulsion is stabilized by proteins, the three-dimensional network is favored by entanglements among protein segments adsorbed at the oil–water interface. The same behavior was reported when a sucrose stearate was used in combination with egg yolk (Franco *et al.*, 1995).

The dynamic linear viscoelasticity behavior of these systems may be described by means of a generalized Maxwell model with a high number of relaxation times, which leads to a discrete relaxation spectrum of the

material. Nevertheless, a continuous linear relaxation spectrum, $H(\lambda)$, was obtained from G' and G'' by inverting the following equations:

$$G'(\omega) = G_c + \int_{-\infty}^{\infty} H(\lambda) \frac{\omega^2 \lambda^2}{1 + \omega^2 \lambda^2} d(\ln \lambda) \quad (1)$$

$$G''(\omega) = \int_{-\infty}^{\infty} H(\lambda) \frac{\omega \lambda}{1 + \omega^2 \lambda^2} d(\ln \lambda) \quad (2)$$

using regularization techniques (Weese and Friedrich, 1994). Its calculation was performed with the commercial software RheoSpec (Guerrero *et al.*, 1998). There was good agreement between the experimental values of the storage, $G'(\omega)$, and loss, $G''(\omega)$, moduli and those values predicted from the relaxation spectrum (Figure 1a). A well pronounced plateau region was observed (Figure 1b), which indicated the occurrence of extensive flocculation and consequently a fully developed elastic network. The same trend was observed for all the other emulsions. Moreover, an increase in the emulsion resting time at the sensor system leads to a more structured system, as can be deduced from the development of its plateau region.

The values of the storage modulus, G' , and the dynamic viscosity, $\eta' = G''/\omega$, as a function of frequency and temperature may be superposed using a time–temperature superposition method (Ferry, 1980). As can be observed (Figures 2, 3 and 4) both master curves presented a power law variation with frequency and could be used to predict the linear viscoelasticity of these systems at another temperature.

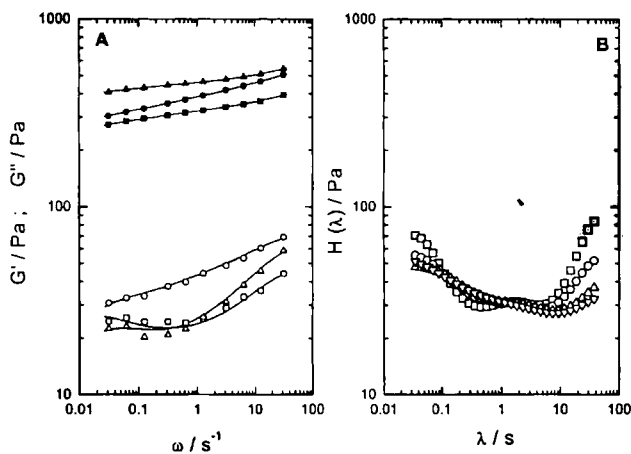


Figure 1. Dynamic linear viscoelasticity at a temperature of 20 °C. (a) Storage modulus, G' , for mayonnaise NW (■), NY (●), and DY (▲); loss modulus, G'' , for mayonnaise NW (□), NY (○) and DY (△); (—) values from $H(\lambda)$. (b) Influence of resting time on the relaxation modulus of mayonnaise NW (□, 10 min; ○, 20 min; △, 30 min; ▽, 40 min).

Figura 1. Viscoelasticidad lineal a 20 °C: (a) módulo de almacenamiento, G' , para las emulsiones NW (■); NY (●); and DY (▲) y módulo de pérdidas, G'' , para las mayonesas NW (□); NY (○) and DY (△), (—) valores calculados de $H(\lambda)$; (b) influencia del tiempo de reposo sobre el espectro de relajación de la mayonesa NW (□, 10 min; ○, 20 min; △, 30 min; ▽, 40 min).

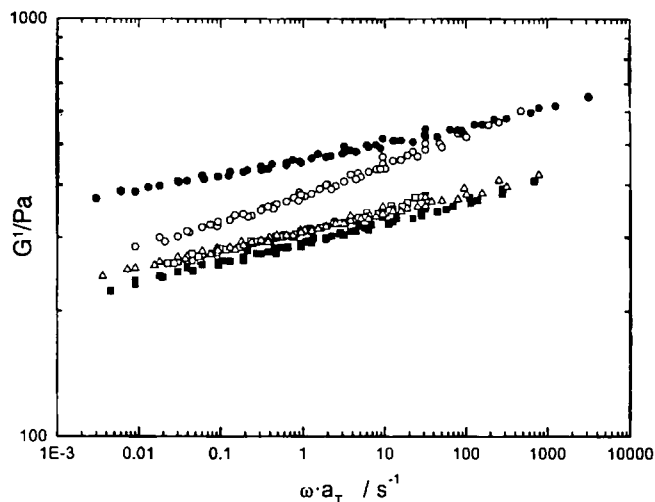


Figure 2. Master curve of storage modulus for different emulsions studied. (□) NW; (■) DW; (△) LW; (○) NY; (●) DY. The reference temperature is 20 °C.

Figura 2. Curva maestra del módulo de almacenamiento para diferentes emulsiones. (□) NW; (■) DW; (△) LW; (○) NY; (●) DY. La temperatura de referencia es 20 °C.

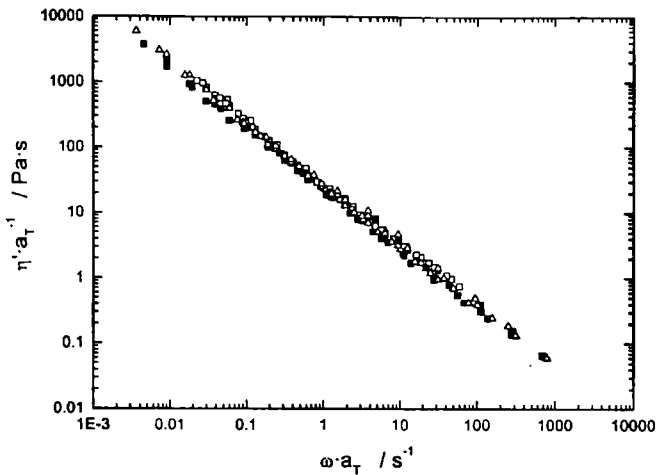


Figure 3. Master curve of η' for whole-egg-containing mayonnaises. (□) NW; (■) DW; (△) LW. The reference temperature is 20 °C.

Figura 3. Curva maestra de η' para mayonesas que contienen huevo completo. (□) NW; (■) DW; (△) LW. La temperatura de referencia es 20°C.

There were no important differences between whole-egg containing mayonnaises. However, these were markedly different from egg-yolk-containing emulsions. Thus, mayonnaise NY and DY showed higher values of G' than whole-egg-containing mayonnaises. Mayonnaise DY showed the maximum value of G' and the minimum slope in G' .

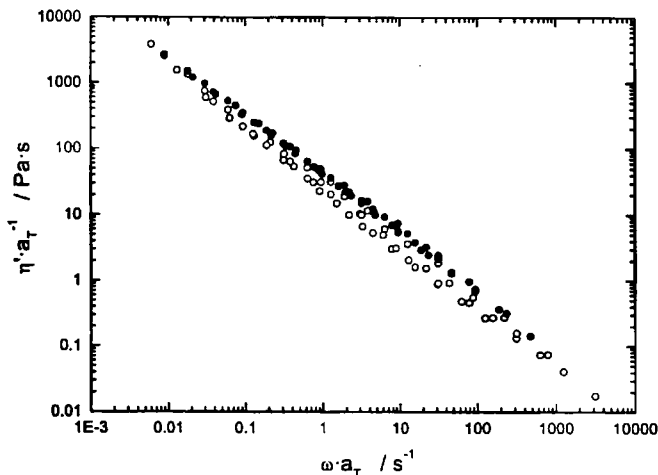


Figure 4. Master curve of η' for egg-yolk-containing mayonnaises. (○) NY; (●) DY. The reference temperature is 20 °C.

Figura 4. Curva maestra de η' para mayonesas que contienen yema de huevo. (○) NY; (●) DY. La temperatura de referencia es 20°C.

The shift-factor values were obtained empirically for the five mayonnaise products studied (Figure 5). These shift-factors superposed all the viscoelastic functions (Figures 2, 3, and 4) for G' and η' . An Arrhenius-like equation described the dependence between the shift-factor and temperature fairly well:

$$a_T = A \exp(E_a/RT) \quad (3)$$

where T is the absolute temperature (K), A is a pre-exponential factor and R is the gas constant.

The slope of the relationship between both variables (Figure 5) is the activation energy (E_a) which gives information about thermal susceptibility of the emulsion. Thus, this activation energy is higher for mayonnaise containing processed egg and in the cases in which egg yolk is used instead of whole egg as the emulsifier. There was no significant difference between mayonnaise DW and mayonnaise LW.

Stress relaxation measurements

The linear relaxation modulus, $G(t-t')$, can be obtained from the linear relaxation spectrum of the material, as follows:

$$G(t-t') = G_c + \int_{-\infty}^{\infty} H(\lambda) \exp\left(-\frac{t-t'}{\lambda}\right) d(\ln \lambda) \quad (4)$$

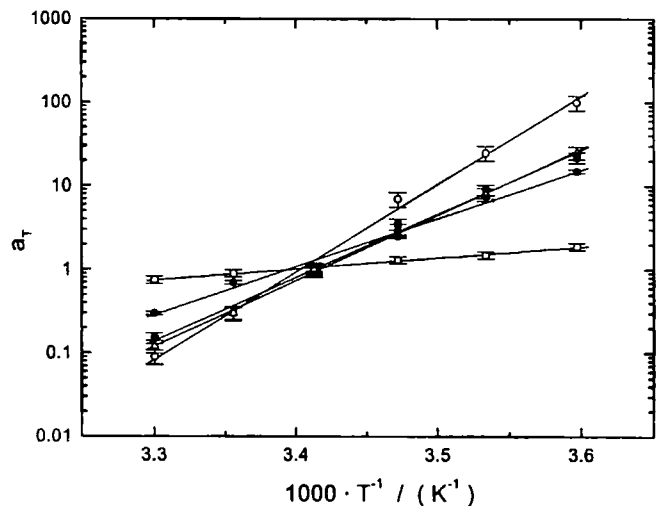


Figure 5. Temperature dependence of the shift factor for the different emulsions studied. (—) values from Arrhenius-type equation; (□) NW $E_a = 25.8$ J/mol; (■) DW $E_a = 146$ J/mol; (△) LW $E_a = 151$ J/mol; (●) NY $E_a = 111$ J/mol; (○) DY $E_a = 201$ J/mol.

Figura 5. Variación con la temperatura de factor de superposición para las diferentes emulsiones estudiadas. (—) Ecuación tipo Arrhenius; (□) NW $E_a = 25.8$ J/mol; (■) DW $E_a = 146$ J/mol; (△) LW $E_a = 151$ J/mol; (●) NY $E_a = 111$ J/mol; (○) DY $E_a = 201$ J/mol.

where $(t - t')$ is the time following the deformation.

On the other hand, the linear viscoelastic relaxation modulus may also be obtained using different approximated methods such as the Ninomiya–Ferry method (Ferry, 1980):

$$G(t - t') = G'(\omega) - 0.40G''(0.4\omega) + 0.014G''(10\omega) \Big|_{\omega=1/t} \quad (5)$$

The values of this modulus, calculated from both methods, for mayonnaise NW (Figure 6) coincided with those obtained from stress relaxation tests when a very low shear strain ($\gamma = 0.02$) was applied to the sample. According to the dynamic measurements, this strain corresponded to the linear range of viscoelasticity for these mayonnaise products. Figure 6 also shows the values of the non-linear relaxation modulus for different shear strains, obtained by applying to the sample a shear strain outside the linear viscoelastic range.

A change in the shape of the relaxation curves took place as the applied strain went outside the linear range (Figure 6). Thus, at low values of γ the relaxation modulus did not fit a power law equation as obtained for some commercial mayonnaise (Gallegos *et al.*, 1992a). However, a power law decay was eventually reached at long relaxation times, where all the curves seemed to have the same slope. As a result, the relaxation modulus could be factorized at long times (higher than λ_k) as:

$$G(t - t', \gamma) = G(t - t')h(\gamma) \quad (t > \lambda_k) \quad (6)$$

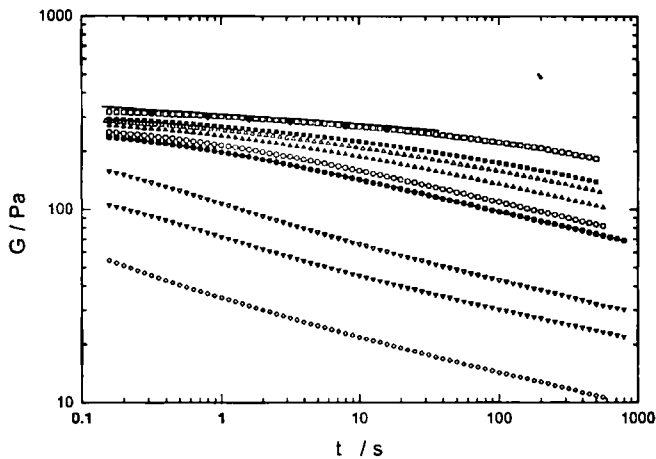


Figure 6. Linear and non-linear relaxation modulus versus elapsed time after different strain values, γ (\square) 0.02; (\blacksquare) 0.07; (\triangle) 0.10; (\blacktriangle) 0.13; (\circ) 0.17; (\bullet) 0.20; (∇) 0.29; (\blacktriangledown) 0.46; (\diamond) 0.92; (\blacklozenge) values from Ninomiya-Ferry equation; (—) values from $H(\lambda)$.

Figura 6. Modulos de relajación lineal y no lineal obtenidos a diferentes valores de deformación, γ (\square) 0.02; (\blacksquare) 0.07; (\triangle) 0.10; (\blacktriangle) 0.13; (\circ) 0.17; (\bullet) 0.20; (∇) 0.29; (\blacktriangledown) 0.46; (\diamond) 0.92; (\blacklozenge) valores calculados de la ecuación Ninomiya-Ferry; (—) valores calculados de $H(\lambda)$.

where λ_k is a material time constant and is defined as the time at which the ratio $G(t - t', \gamma)/G(t - t')$ levels off; for emulsion NW, λ_k is about 100 s. Hence, the non linear relaxation modulus is factorable in two terms, one depending on time, $G(t - t')$, and the other depending on strain, the so-called ‘damping function’ $h(\gamma)$. The damping function provides information about the material structural breakdown due to shear.

A decrease of the damping function with increasing strain was observed (Figure 7). This behavior can be described by the Soskey–Winter damping function (Soskey and Winter, 1984) fairly well:

$$h(\gamma) = \frac{1}{1 + a\gamma^b} \quad (7)$$

where parameters $a = 21$ and $b = 1.64$ are material parameters.

Transient flow tests

A stress overshoot was always observed, but a stress undershoot may appear as shear rate rises (Figure 8). This behavior was always found with whole-egg-containing mayonnaise.

Emulsions made from egg yolk (NY and DY) were always more viscous than the others (Figure 9). Moreover, their transient flow curves did not show a stress undershoot. Stress decay was fitted to a generalized sum of first-order kinetic functions:

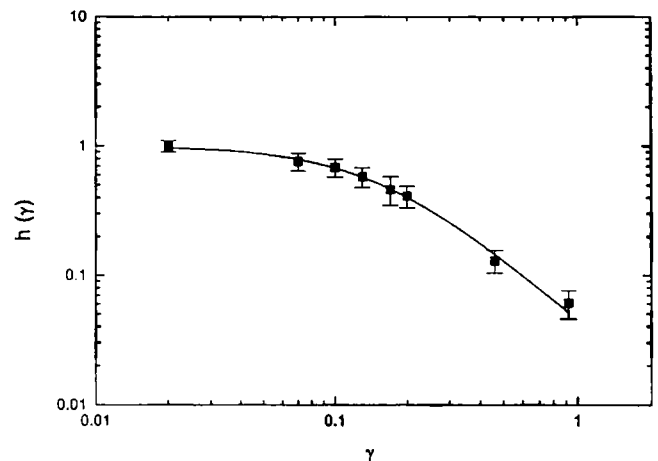


Figure 7. Evolution of the damping function with the applied strain for mayonnaise NW at 20 °C. (\blacksquare) experimental values; (—) values from Soskey-Winter equation.

Figura 7. Evolución de la función amortiguación con la deformación para la mayonesa NW, a 20 °C. (\blacksquare) valores experimentales; (—) ajuste a la ecuación de Soskey-Winter.

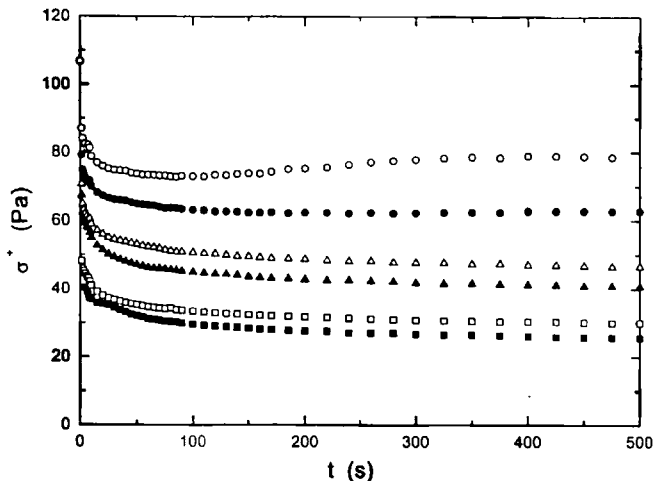


Figure 8. Transient flow curves for mayonnaise NW at different shear rates and 20 °C. (■) 0.06 s⁻¹; (□) 0.29 s⁻¹; (▲) 0.58 s⁻¹; (△) 1.16 s⁻¹; (●) 2.93 s⁻¹; (○) 5.83 s⁻¹.

Figura 8. Flujo transitorio de la mayonesa NW a diferentes velocidades de cizalla y 20 °C. (■) 0,06 s⁻¹; (□) 0,29 s⁻¹; (▲) 0,58 s⁻¹; (△) 1,16 s⁻¹; (●) 2,93 s⁻¹; (○) 5,83 s⁻¹.

$$\sigma^* - \sigma_c^* = \sum_{j=1}^n (\sigma_{o,j}^* - \sigma_{e,j}^*) \exp(-k_j t) \quad (8)$$

where $n = 2$, σ_c^* is the equilibrium stress, and k_j the kinetic parameters (Figoni and Shoemaker, 1983). The parameter k_1 (Figure 10) increased with shear rate and should be related to a reversible structural breakdown

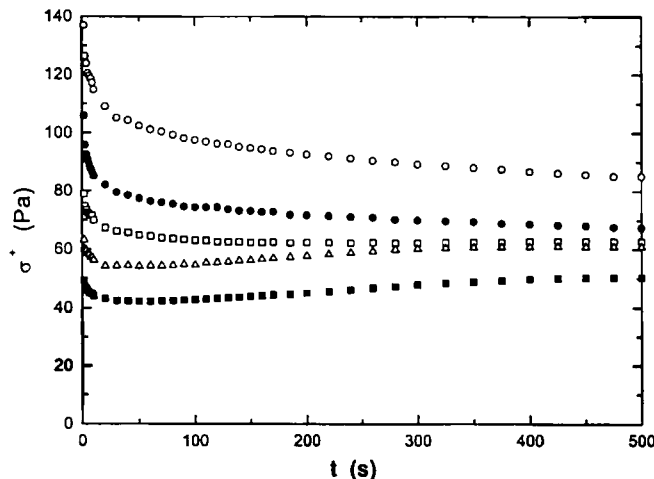


Figure 9. Transient flow curves for the different emulsions studied at 2.93 s⁻¹ and 20 °C. (■) DW; (□) NW; (△) LW; (●) NY; (○) DY.

Figura 9. Flujo transitorio de las diferentes emulsiones estudiadas a 2,93 s⁻¹ y 20 °C. (■) DW; (□) NW; (△) LW; (●) NY; (○) DY.

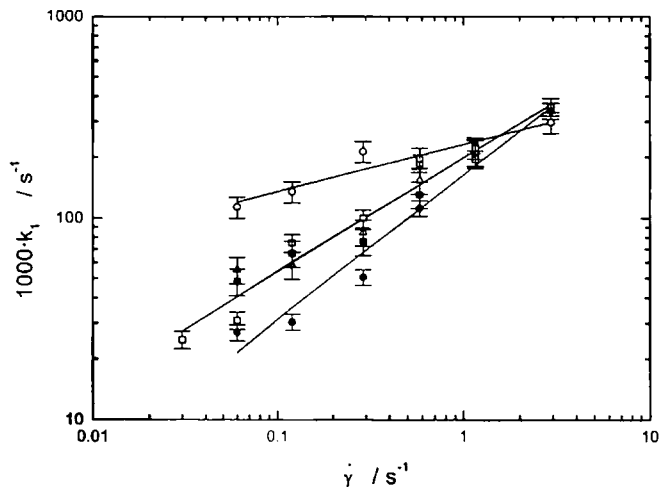


Figure 10. Stress decay kinetic parameters versus shear rate for the different emulsions studied. (■) DW; (□) NW; (△) LW; (●) NY; (○) DY.

Figura 10. Parámetros de la cinética de descenso del esfuerzo para las diferentes emulsiones estudiadas. (■) DW; (□) NW; (△) LW; (●) NY; (○) DY.

process of the emulsion (shear-induced deflocculation of droplets). The parameter k_2 , which was experimentally set to a value of $k_1/30$, may be related to both reversible and irreversible processes, i.e. shear induced coalescence (Partal *et al.*, 1997a).

There were no significant differences among the kinetic constants for whole-egg-containing mayonnaise. Mayonnaise DY showed the highest kinetic values, while mayonnaise NY had the lowest ones. However, an increase in shear rate caused the above mentioned differences to disappear.

Transient flow of these systems may be predicted from stress relaxation measurements using the Wagner non-linear viscoelastic model (Wagner, 1976). In the reduced stress, σ^*/σ_c^* , for emulsion NW (Figure 11), σ_c^* was calculated from Eqn (8). Figure 11 also shows the reduced stress predicted by the Wagner model:

$$\frac{\sigma(t-t', \gamma)}{\sigma(\infty, \gamma)} = \frac{\sigma^*}{\sigma_c^*} = \frac{\int_{-\infty}^t m(t-t') \cdot h(\gamma) \cdot \gamma(t-t') dt'}{\lim_{t \rightarrow \infty} \int_{-\infty}^t m(t-t') \cdot h(\gamma) \cdot \gamma(t-t') dt'} \quad (9)$$

where the damping function was obtained from Eqn (7) and the memory function, $m(t-t')$, is calculated by deriving Eqn (4), as follows:

$$m(t-t') = \frac{dG(t-t')}{dt'} \quad (10)$$

Experimental transient stress values and model predictions matched fairly well at the highest shear rates (Fig-

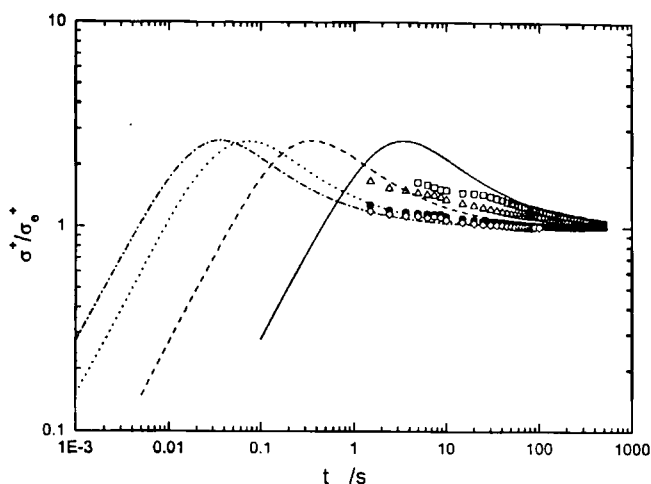


Figure 11. Experimental and predicted values of the transient shear stress for mayonnaise NW. (□) 0.06 s^{-1} ; (Δ) 0.58 s^{-1} ; (●) 2.93 s^{-1} ; (\diamond) 5.83 s^{-1} ; (—) model.

Figura 11. Comparación entre los valores experimentales del esfuerzo transitorio y la predicción para la mayonesa NW. (□) $0,06 \text{ s}^{-1}$; (Δ) $0,58 \text{ s}^{-1}$; (●) $2,93 \text{ s}^{-1}$; (\diamond) $5,83 \text{ s}^{-1}$; (—) modelo.

ure 11), but the model failed at low shear rate. Mayonnaises usually show wall-slip phenomena during steady flow (Franco *et al.*, 1998), especially in the range $0.001\text{--}1 \text{ s}^{-1}$ (Plucinski *et al.*, 1998). This fact could explain the lack of concordance observed at shear rates below 1 s^{-1} .

Furthermore, the time necessary to reach the overshoot predicted by the model, which became shorter with increasing shear rate, was much lower than the value obtained by experimental observation (Figure 11). As a consequence, the overshoot values predicted by the model were higher than the experimental values, which depend on the technical limitations of the rheometer. Moreover, the Wagner model is not able to predict the undershoot that takes place in these systems at high shear rates.

FINAL REMARKS

The increase in the storage modulus, as well as in the non-linear viscoelasticity values, produced by the replacement of the native egg yolk by a processed egg yolk in an emulsion may be associated with the higher viscoelasticity of the processed egg yolk. This, in turn, is a consequence of the thermal treatment to which the yolk was subjected during the drying process, since a certain degree of denaturation of protein may take place.

When egg yolk is replaced by whole egg this effect vanishes because of the dilution of yolk proteins. Moreover, the lower viscoelasticity of mayonnaise made from

whole egg is due to the fact that the replacement of egg yolk by egg white decreases the amount of ingredients involved in emulsification and stabilization, since egg white contains about 25% of the total solids present in egg yolk.

Furthermore, from the values of the shift factor we may conclude that the processed-egg-containing mayonnaise samples (DW, LW, DY) are considerably more temperature dependent than mayonnaise made from native egg (NW, NY). This dependence is most remarkable for mayonnaise DY.

The transient flow response showed a stress overshoot followed by a stress decay which tends to a steady-state value. The shear-induced structural destruction of mayonnaise DY showed a low shear rate dependence of its kinetic functions (Figure 10). A stress undershoot may also be found at high shear rate in whole-egg-containing mayonnaise. A decrease in the mean droplet size at high shear rate would explain these results, as has been found for model emulsions (Partal *et al.*, 1997b).

A factorable non-linear viscoelastic constitutive equation has been used to predict the above-mentioned transient flow behavior. However, a lack of concordance is found as shear rate decreases. This fact has been related to wall-slip phenomena.

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