Effect of Carboxymethyl Cellulose Concentration on Rheological Behavior of Milk and Aqueous Systems. A Creep and Recovery Study

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ABSTRACT: Carboxymethyl cellulose (CMC) is an anionic polysaccharide used mainly as stabilizer and thickener agent. The purpose of this study was to investigate the effect of CMC concentration on viscoelasticity of dairy and aqueous model systems through the analysis of creep and recovery tests. The viscoelastic properties of different concentrations of CMC (0.75, 1.00, 1.25, and 1.50% w/w) in two milk systems (skimmed milk and whole milk) were compared with those of the same concentration of biopolymer in aqueous solution. Creep curves were fitted to a six parameter mechanical model (Burger + Kelvin-Voigt), whereas an empirical equation was used for recovery. The creep and recovery properties of samples were clearly affected by both the type of dispersing media and the CMC concentration. The whole-milk system was more elastic than both the biopolymer (CMC) aqueous solution and the skimmed-milk sample, indicating that in whole-milk samples some new interactions could take place between macromolecules. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 1626–1632, 2009

Key words: carboxymethyl cellulose; milk; biopolymer; viscoelastic properties; creep

INTRODUCTION

Sodium carboxymethyl cellulose (CMC), known universally as CMC, is a linear, long chain, anionic polysaccharide. In foods, the basic properties of CMC that enhance its value are its ability to thicken solutions, act as a moisture binder, dissolve rapidly in both hot and cold aqueous systems, and improve the texture of a wide range of food products. It is tasteless, odourless, and forms clear solutions.[1] Cellulose gum is physiologically inert and noncaloric, because it is not metabolized by the human digestive system. The latter property makes it particularly useful to formulate dietetic foods. This gum is compatible with a wide range of other food ingredients, particularly proteins, sugars, and other hydrocolloids where synergistic interaction can occur.[2,3] Nowadays, CMC is being used as an alternative thickener to starch in semisolid dairy products.[4]

Several rheological studies on aqueous CMC systems have been reported in the literature.[5–7] Dapía et al.[8] reported higher shear thinning behavior for increased CMC concentrations. Edali et al.[9] studied the rheological properties of high concentrations of CMC solutions and observed shear thinning and thixotropic behavior. The results of the frequency sweep indicated that dynamic viscosity was dependent on CMC concentration for all the solutions. Kulicke et al.[10] observed an increase in the dynamic moduli (G' and G'') with CMC concentration due to an increase in the number of entanglements in aqueous solutions. As stated before previous rheological studies have been carried out in predominantly CMC aqueous solutions, but there are few studies of complex systems like dairy systems where CMC may interact with carbohydrates and milk proteins. When cellulose gum is added to systems containing protein, it can cause unexpected effects on its rheological properties, texture and stability.[2,11] In mixed protein-polysaccharide systems, associative electrostatic interactions can lead to coacervation or soluble complex formation depending on the nature of the biopolymers and the solution condition (pH and ionic strength).[12] Several authors have studied the whey protein-CMC interactions at the droplet surfaces in o/w emulsions.[11,13–15]

Creep and recovery test can be used to study the viscoelastic properties of aqueous and milk systems. They could help to understand the possible internal structure of the aqueous and milk systems and the

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structural variations associated with the introduction of changes in composition. In a creep test, an instantaneous stress is applied to the sample and the change in strain is observed over time. When the stress is released, some recovery may be observed as the material tends to return to the original shape. The response of the material is an indication of its liquid-like or solid-like behavior. Oscillatory measurements also provide this kind of information, but frequency dependence must be analyzed by empirical functions only suitable for the ranges studied,[16,17] or by equations with a large number of parameters (Maxwell generalized model). However, creep curves follow mechanical models with only four or six parameters,[18,19] which allows results to be more properly and easily compared. Chattong et al.[20] showed that the redistribution of fat in xanthan treated sausages produced only limited effects when examined by oscillatory rheological techniques whereas creep compliance testing showed major changes.

With regard to dairy products, creep and recovery tests have been used to study the interaction between biopolymers and milk[21] as well as the effect of fat reduction on the viscoelastic properties of cheese.[22–26] In general, creep measurements showed that fat reduction decreased viscoelasticity. However, at present, there exists no information about the effect of fat reduction together with CMC addition on the viscoelastic behavior of milk systems with a matrix close to dairy desserts using creep and recovery tests.

The purpose of this study was to investigate the effect of CMC concentration on viscoelasticity (rheological behavior) of dairy and aqueous model systems through the analysis of the parameters obtained from creep and recovery tests. The viscoelastic properties of different concentrations of CMC in two milk systems (skimmed milk and whole milk) were compared with those of the same concentration of biopolymer in aqueous solution.

MATERIALS AND METHODS

Sample preparation and composition

Samples were prepared from CMC (Akucell AF3265 Akzo Nobel, Amersfoort, The Netherlands), sucrose, commercial whole (25% w/w protein, 39% w/w carbohydrate, 26% w/w fat, and 1.2% w/w calcium) and skimmed (34% w/w protein, 52% w/w carbohydrate, 1% w/w fat, and 1.2% w/w calcium) milk powder (Central Lechera Asturiana, Siero, Spain), vanilla aroma (375 48A Lucta S.A., Barcelona, Spain) and yellow-orange colorant (Vegex NC 2c WS mct, CHR Hansen S.A., Barcelona, Spain). The CMC was dispersed in two media: water and milk. Aqueous solutions were prepared by using deionized water; whereas milk dispersions were prepared in both whole and skimmed milk. Twelve different formulations were prepared varying in concentration of CMC (0.75% w/w, 1.00% w/w, 1.25% w/w, and 1.50% w/w) and dispersing media (water, whole milk, and skimmed milk), whereas the following amounts remained fixed: sucrose (6% w/w), vanilla aroma (0.016%w/w), colorant (0.052% w/w), and the weight of rehydrated milk (80% w/w). Rehydrated whole and skimmed milk were prepared in advance by dissolving 13.5% (w/w) whole and skimmed milk powders, respectively, in deionized water to obtain a final fat content of 3.5% and 0.14%, respectively. Milk powder was dispersed in deionized water, at 250 rpm and 85°C for 10 min, with the help of a magnetic stirrer and a hot plate (Ared, Velp Scientifica) and stored at 4 ± 1°C overnight to ensure complete hydration of the milk proteins. To prepare samples, a dry blend of sugar with CMC was added to the dispersing media, with the added colorant, and stirred (Heidolph RZR 1, Germany) at room temperature for 35 min. Five min before the end vanilla aroma was added. The sample was transferred to a closed flask and stored (4 ± 1°C; 24 h) before rheological measurements. Two batches of each concentration combination were prepared and each batch was measure twice.

Rheological measurements

Creep and recovery measurements were performed at 10 ± 1°C in a controlled stress rheometer RS1 (ThermoHaake, Karslruhe, Germany) with temperature controlled by a Haake circulating water bath. A parallel plates geometry (6-cm diameter; 1-mm gap) was used. Before being tested, samples were placed in the measuring system and allowed to rest for 10 min for structure recovery and temperature equilibration. After putting carefully the sample between the plates, the excess material was wiped off with spatula. A fresh sample was loaded for each measurement.

Stress sweeps were made between 0.02 and 300 Pa, at a frequency of 1 Hz, in all the systems studied to determine the linear viscoelasticity zone. Creep and recovery tests were carried out by applying instantly a constant stress within the linear viscoelastic region, σ_0, which was maintained for 300 s, then finally removed, allowing the sample to relax for 300 s. The system deformation per unit stress, called compliance: $J(t) = \gamma(t)/\sigma_0$, was measured.

Experimental design and data analysis

The effects of CMC concentration and of the type of dispersing media and their interaction on creep and recovery data were analyzed by a two-way analysis of variance (ANOVA). Experimental designs included 2 factors: CMC concentration (four levels: 0.75% w/w, 1.0% w/w, 1.25% w/w, and 1.5% w/w), and type of dispersing media (three levels: water, whole milk, and skimmed milk). Significant differences between individual samples were determined by the Fisher's test ($\alpha = 0.05$). Analysis of variance were carried out with XLSTAT-Pro software v.2007 (Adinsoft, Paris, France).

RESULTS AND DISCUSSION

Figure 1 shows the effect of CMC concentration on the values of compliance as a function of time, $J(t) = \gamma(t)/\sigma_0$, obtained for aqueous solution, skimmed-milk and whole-milk systems. As expected, the viscoelastic characteristics of both water and milk systems mainly depended on the CMC concentration and on the nature of the dispersing medium.

In all three dispersing media studied, one may observe a decrease of the compliance values with the increase in CMC concentration, meaning a greater resistance to deformation. This is in agreement with the findings of Ghannam and Esmail[7] and Edali et al.,[9] who studied the rheological properties of CMC aqueous solutions. These authors reported a decrease of the compliance values with the increase in CMC concentration. Among dispersing media, the elasticity and resistance to deformation of samples was higher for the whole-milk system than for both the aqueous solution and the skimmed-milk system. The compliance values of whole-milk systems were about 100 times lower than in the other dispersing media, what is indicative of a stronger structured matrix. It is interesting to point out that the 0.75% CMC skimmed-milk sample [Fig. 1(b)] showed a typical curve for liquid-like systems, i.e., a straight line from zero and practically no recovery, whereas the rest of systems showed both viscous and elastic characteristics (some instantaneous deformation and a certain percentage of recovery). This different behavior of 0.75% CMC skimmed-milk sample may suggest some interaction between skimmed milk ingredients with this low concentration of CMC, destabilizing the developed network when dispersed in the aqueous system. This negative effect might be compensated by the higher presence of fat in the whole-milk medium.

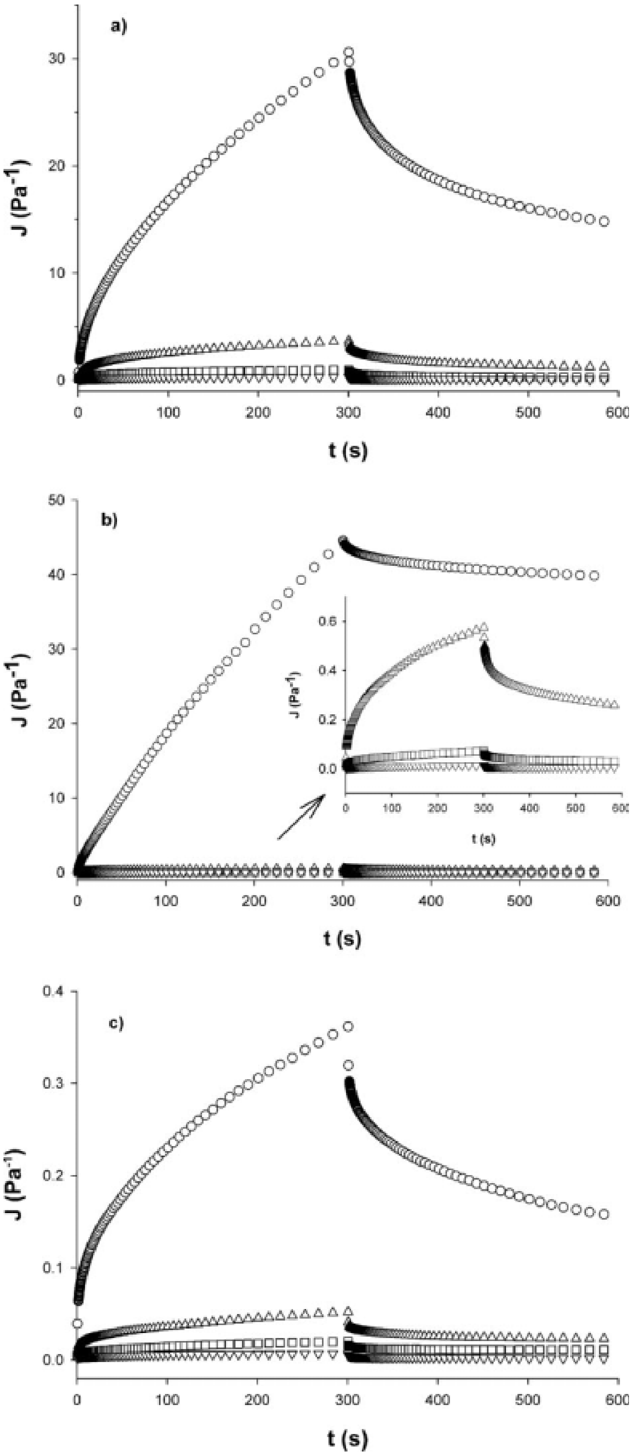

Figure 1 Compliance versus time in creep and recovery tests for aqueous dispersions (a), skimmed-milk (b) and whole-milk (c) systems at different CMC concentrations (○ = 0.75%; △ = 1%; □ = 1.25%; ▽ = 1.5%).

Creep test

The mechanical models that reflect the deformation of a system in this type of tests range from the simplest approaches (spring or dashpot) to generalized approaches including the association of a large number of components. To characterize the viscoelastic behavior of both aqueous and milk systems during the creep test, the J values versus time in the interval $0 \leq t \leq 300$ was fitted to a six parameter model [eq. (1)], formulated by adding an additional Kelvin-Voigt element to the four-parameter Burgers model, which consists of a Kelvin-Voigt and a Maxwell model placed in series[27] (Fig. 2):

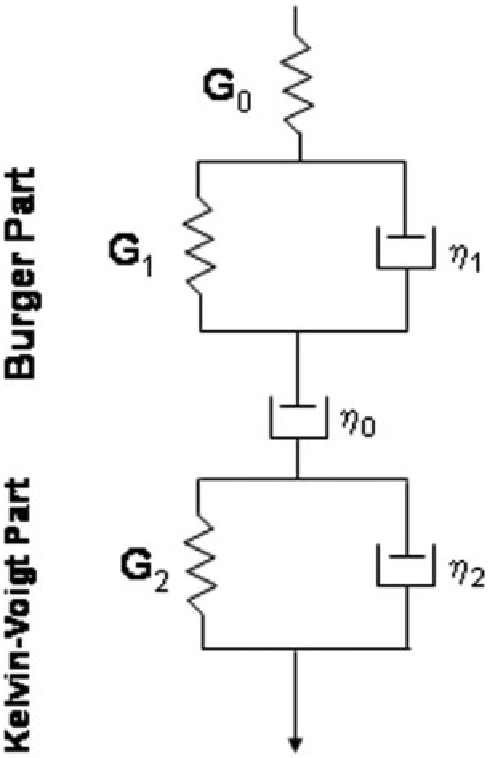

Figure 2 Six parameter model for creep compliance.

$$J(t) = \frac{1}{G_0} + \frac{1}{G_1}\left[1 - \exp\left(\frac{-tG_1}{\eta_1}\right)\right] + \frac{1}{G_2}\left[1 - \exp\left(\frac{-tG_2}{\eta_2}\right)\right] + \frac{t}{\eta_0} \quad (1)$$

where $J(t)$ represents the overall compliance at any time t, G_0 is the instantaneous elastic modulus of the Maxwell unit, the dashpot of the Maxwell element represents the residual viscosity (η_0), and G_1 and G_2 are the elastic moduli and η_1 and η_2 are the internal viscosities associated with the first and second Kelvin-Voigt elements respectively.[28] This model was used successfully to model the viscoelastic behavior of skim milk curd[29] and cheese.[25,26,30]

Table I shows the values of the parameters obtained: G_0, η_0, G_1, η_1, G_2, and η_2. Considering the correlation coefficient values (r), compliance values versus time fitted well to the model for the three systems studied ($0.998 < r < 0.999$).

An ANOVA of two factors with interactions was done to analyse how the type of dispersing media and concentration of CMC influenced G_0, η_0, G_1, η_1, G_2, and η_2 values. Results showed that both dispersing media and CMC concentration effects were significant ($P < 0.001$), and that there was a significant interaction effect ($P < 0.02$) between them for all six variables considered. This interaction indicated that the effect of CMC concentration on the variables considered differed depending on the type of dispersing media.

In all three dispersing media studied, the addition of CMC reinforced the structure incrementing the values of all the parameters present in the six-element model. The increase observed in viscosity parameters was greater than the increase produced in elastic parameters. Table I shows higher values of the instantaneous elastic modulus of the Maxwell unit (G_0) and the elastic modulus of the two Kelvin elements (G_1 and G_2) with increasing concentration of CMC, indicating the harder and more rigid nature of the system. A higher G_0 indicates a more elastic nature and higher recovery ability, as it corresponds to the initial instantaneous deformation. In addition, an increase in η_0 was observed with an increase in CMC concentration, indicating a higher resistance to flow. Moreover, a large variation on creep parameters was found among the three different systems. Whole-milk samples presented the highest creep

TABLE I
Mean Values of the Creep Parameters Obtained from Fitting the Variation of Compliance with Time to eq. (1) and Correlation Coefficients

CMC concentration (% w/w)	Dispersing media	G_0 (Pa)	η_0 (Pa s)	G_1 (Pa)	η_1 (Pa s)	G_2 (Pa)	η_2 (Pa s)	r
0.75	Aqueous dispersion	8.5^{a}	164.0^{a}	1.1^{a}	72.8^{a}	3.6^{a}	18.4^{a}	0.999
	Skimmed milk	2.4^{a}	9.2^{a}	0.1^{a}	8.5^{a}	0.8^{a}	17.4^{a}	0.999
	Whole milk	20.9^{a}	2110.6^{ab}	25.2^{ab}	77.7^{a}	8.3^{a}	536.3^{ab}	0.999
1	Aqueous dispersion	27.6^{a}	2063.1^{ab}	18.1^{a}	58.8^{a}	7.9^{a}	362.0^{ab}	0.999
	Skimmed milk	19.5^{a}	1151.7^{ab}	5.0^{a}	249.6^{ab}	13.7^{a}	34.0^{a}	0.999
	Whole milk	96.8^{bc}	14037.0^{b}	169.5^{b}	265.2^{ab}	87.1^{c}	3347.4^{cd}	0.999
1.25	Aqueous dispersion	59.1^{ab}	9403.7^{ab}	56.8^{ab}	168.6^{a}	31.6^{ab}	1229.1^{ab}	0.999
	Skimmed milk	111.4^{bc}	9519.4^{ab}	113.7^{ab}	195.9^{a}	53.2^{b}	1836.4^{bc}	0.999
	Whole milk	269.3^{d}	49107.5^{d}	423.7^{c}	657.6^{bc}	238.8^{d}	8506.3^{e}	0.998
1.5	Aqueous dispersion	127.6^{c}	32695.0^{c}	142.8^{ab}	347.4^{ab}	103.8^{c}	4528.5^{d}	0.999
	Skimmed milk	322.6^{d}	58068.5^{d}	534.1^{c}	959.5^{c}	235.4^{d}	10697.7^{f}	0.999
	Whole milk	411.3^{e}	118865.0^{e}	951.0^{d}	1655.6^{d}	704.7^{e}	22134.0^{g}	0.999

G_0 and η_0 are the elastic modulus and the corresponding residual viscosity of the Maxwell unit, respectively. G_1 and G_2 are the elastic moduli of Kelvin-Voigt springs and η_1 and η_2 are the corresponding dashpot viscosities.

Different superscript letters denote significant differences between samples ($\alpha = 0.05$).

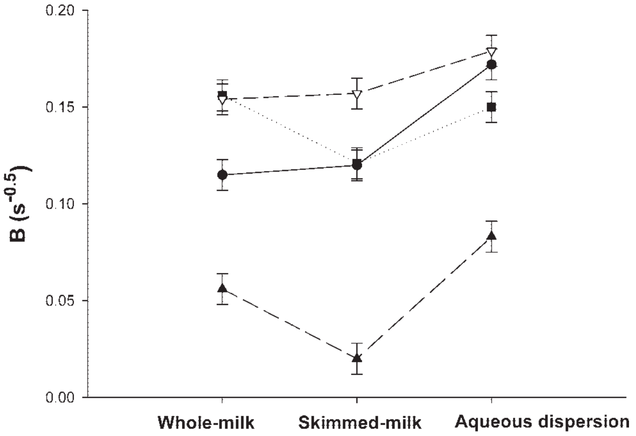

Figure 3 Average values of *B* parameter obtained from the fits by eq. (2) for whole-milk, skimmed-milk, and aqueous dispersions at different CMC concentrations (0.75% = ▲, black dashed line; 1% = ■, dotted line; 1.25% = ●, solid line; 1.5% = ▽, gray dashed line).

parameters values compared with aqueous solutions and skimmed-milk systems, as a result of which whole-milk systems deform less easily. The 0.75% CMC skimmed-milk sample showed the lowest creep parameters values, which is indicative of its viscous nature. The values of the creep parameters of 1.5% CMC whole-milk sample exceed those of all the rest, thus defining it as the sample with the greatest opposition to deformation (Table I).

At 0.75% CMC concentration, there were no statistical significant differences ($\alpha = 0.05$) in the creep parameter values of the three systems studied (aqueous solution, skimmed milk, and whole milk) (Table I). As the CMC content was relatively low, the presence of polysaccharide was not expected to have any appreciable effect on sample rheology. Differences between systems became apparent when CMC concentration was increased. In general, at 1% and 1.25% CMC concentration creep parameter values of aqueous solutions and skimmed-milk samples were similar to each other and both were lower, than those of whole-milk samples. Finally, for the highest CMC concentration (1.5%) creep parameter values were observed to be significantly ($\alpha = 0.05$) higher for whole-milk samples than for skimmed-milk samples, which in turn were higher than for aqueous solutions. These results are in accordance with those obtained from oscillatory tests[31]: for the highest CMC concentration (1.5%) storage modulus (G') and complex dynamic viscosity (η^*) values were observed to be significantly ($\alpha = 0.05$) higher for whole-milk samples than for skimmed-milk samples, which in turn were higher than for aqueous solutions.

Recovery test

The recovery part of the test is a measure of the decline of the material deformation when the stress is removed. The compliance during the recovery does not follow the creep model, although both tests would expected to be symmetrical, so an empirical equation[32] was used to fit the values.

$$J(t) = J_\infty + J_{\mathrm{KV}} \exp\left(-Bt^{0.5}\right) \qquad (2)$$

where J_∞ is the residual compliance corresponding to the permanent deformation of the Maxwell dashpot, J_{KV} is the recovery due to Kelvin-Voigt elements, t is the time, and B is a parameter related to the recovery rate of the system.

Figure 3 shows the *B* values obtained from the fits by eq. (2) of both aqueous and milk systems. In all three systems, *B* values were significantly ($\alpha = 0.05$) lower at the lowest CMC concentration (0.75%) indicating that the recovery rate of these systems was much lower than the rest of samples formulated with higher CMC concentrations (1%, 1.25%, and 1.5%). In addition, at the lowest CMC concentrations (0.75% and 1%) skimmed-milk samples showed significant lower *B* values than whole-milk and aqueous dispersions.

In addition, the initial shear compliance J_{SM} (i.e., the deformation suffered by the Maxwell spring) was obtained by using eq. (3), where J_{MAX} is the compliance value for the longest time (300 s) in the creep transient analysis, which corresponds to the maximum deformation.

$$J_{\mathrm{SM}} = J_{\mathrm{MAX}} - (J_\infty + J_{\mathrm{KV}}) \qquad (3)$$

Compliance values versus time fitted well to the eq. (2) for all three systems studied ($0.996 < r < 0.999$). As in the previous section, analysis of variance showed that recovery parameters were significantly affected by both dispersing media and CMC concentration ($P < 0.001$) and that there was a significant interaction ($P < 0.01$) between them for all variables considered.

To compare more properly the capability of recovery of each sample, it is necessary to observe relative amounts, better than absolute values, as they are in different ranges. The percentage deformation of each element of the model was calculated by

$$\%J = \left[\frac{J_{\mathrm{element}}}{J_{\mathrm{MAX}}}\right] \times 100 \qquad (4)$$

where J_{element} is the corresponding compliance: J_{SM}, J_{KV}, or J_∞. Therefore, mechanical characterization of the system was established by calculating the contribution of each of the elements of the model, at the maximum deformation to which the system is subjected. The values obtained are reported in Figure 4. When comparing the three systems studied, the contribution of the Maxwell spring to the total deformation ($\%J_{\mathrm{SM}}$) of the aqueous solutions and

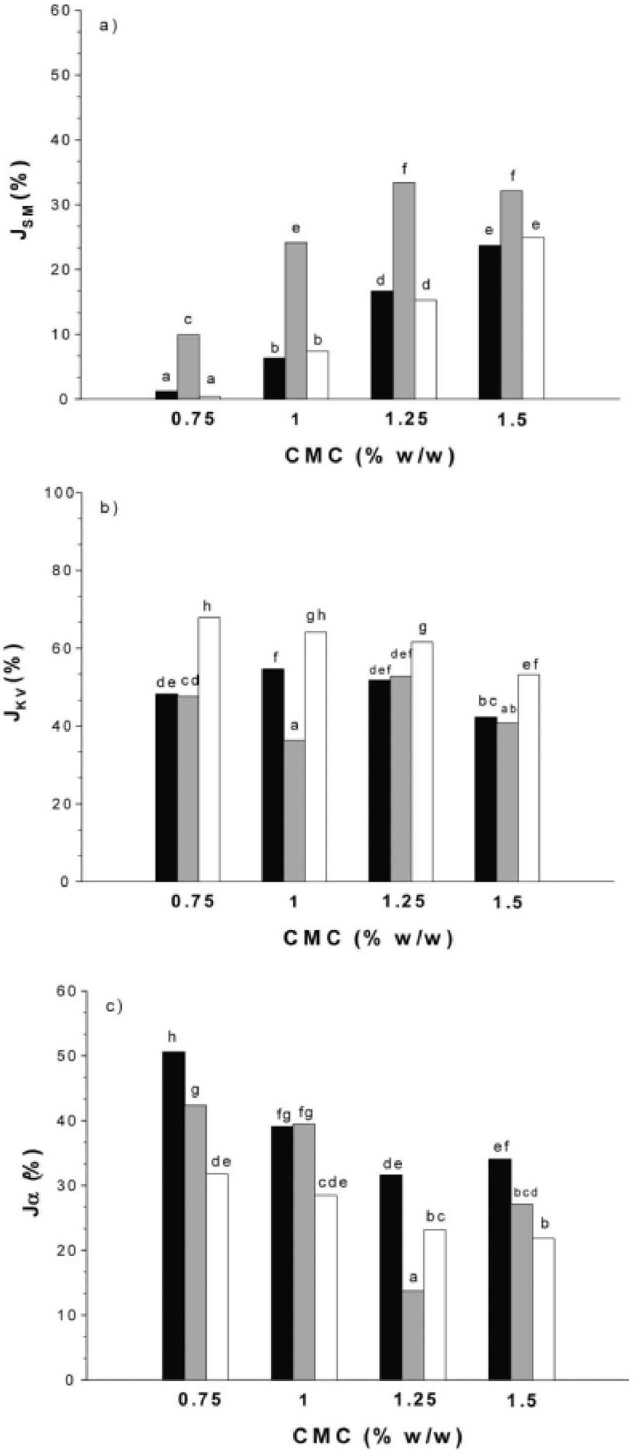

Figure 4 Average values of $\%J_{SM}$ (a), $\%J_{KV}$ (b), and $\%J_{\infty}$ (c) of skimmed-milk (■), whole-milk (▨) and aqueous dispersions (□). In each graph different superscript letters denote significant differences between samples ($\alpha = 0.05$).

skimmed-milk samples were similar and both were significantly ($\alpha = 0.05$) lower than for whole-milk samples [Fig. 4(a)]. Taking into account that whole-milk systems have the highest fat content, a higher resistance to deformation because of the oil droplets together with a molecular interaction could be the reason of the fact that whole-milk systems showed the highest elastic compound. In general, within each dispersing medium, $\%J_{SM}$ values increased with CMC concentration. The recovery ability of the systems, i.e., their elastic nature, was greater for higher CMC concentrations, as indicated in creep results. Although $\%J_{KV}$ values were slightly higher for aqueous solutions than for milk systems, the Kelvin-Voigt element contributed in a similar proportion for the three systems and for all the CMC concentrations studied [Fig. 4(b)]. Moreover, $\%J_{\infty}$ values showed a decrease with the increase of CMC concentration similar to the observed increase of $\%J_{SM}$ values with CMC concentration. In addition, it should be noted that the 0.75% skimmed-milk sample was the formulation with the greatest contribution to the Maxwell dashpot to deformation ($\%J_{\infty}$), in coincidence with the fact that is the sample with the most liquid behavior [Fig. 4(c)].

Changes in the characteristics of the dispersing medium clearly altered the viscoelastic properties of samples. In general, whole-milk samples showed increased elastic properties, the higher fat content seemed to strengthen the system when they were compared with both skimmed-milk systems and aqueous solutions. Moreover, a remarkably enhancement in elastic properties was observed in whole-milk systems with a high CMC content (>1% w/w). This may suggest some substantial interaction between the protein adsorbed in oil droplets and the CMC. A similar trend was observed by Dickinson and Pawlowsky[33] in o/w emulsions with similar medium conditions, i.e., almost neutral pH and an anionic polysaccharide (dextran sulphate). Dickinson and Galazka[34] observed also an enhanced stability of the bovine serum albumin-containing emulsion due to the presence of dextran sulphate during emulsification. This behavior was attributed to the formation of a bovine serum albumin-dextran sulphate complex on the emulsion droplet surface, which produced a thicker, stronger, steric stabilization layer.

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