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Food Chemistry 90 (2005) 627–634

Food Chemistry

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Stabilization of olive oil – lemon juice emulsion with polysaccharides

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Received 2 February 2004; received in revised form 6 April 2004; accepted 6 April 2004

Abstract

An olive oil – lemon juice Greek salad dressing was developed employing xanthan gum as stabilizer and gum arabic or propylene glycol alginate as emulsifier in various combinations. In general, samples containing xanthan gum arabic were more stable against oil droplet coalescence and less stable against creaming. The use of propylene glycol alginate in the place of gum arabic, on the other hand, resulted in emulsions of higher creaming stability. Application of steady shear rheology and determination of rheological parameter values from the shearing stress-rate of shear curves, indicated that the rheological properties of the dressings were decreased with storage. Dressing texture assessment preference tests indicated that potential consumers of the product may opt for a medium viscosity product and this has to be taken into consideration when designing polysaccharide – stabilized dressings exhibiting a decrease in their textural characteristics with storage time.

Keywords: Salad-dressing; Stability; Rheology; Polysaccharides; Oxidation

1. Introduction

Olive oil is an important component of the diet of the countries surrounding the Mediterranean Sea. It has always been the principal source of fat in their cuisine. It is rich in monounsaturates and has a resistance to oxidative changes because of its moderate unsaturation and the presence of nutrient and non-nutrient antioxidants. When substituted for saturated fat in the diet, olive oil is associated with lower low-density lipoprotein cholesterol. There is also increasing evidence that some of its constituents, mainly phenolic antioxidants, inhibit or modulate oxygen-related reactions and have a substantial favorable effect against oxidative injury (Boskou & Visioli, 2003).

Recently olive oil is gaining interest among consumers of northern Europe, USA, Canada, Australia and other countries, mainly due to the belief that there is a positive role of the Mediterranean diet in the prevention of certain diseases (Boskou & Visioli, 2003). A large body of epidemiological and laboratory studies have showed that the incidence of coronary heart disease and certain cancers is lowest in the Mediterranean basin (Keys, 1995; Trichopoulou, 1995). This growing enthusiasm is a stimulus to further propagation of its use and the finding of new applications.

Olive oil has been used for centuries for a variety of culinary purposes, including frying because of its remarkable thermal oxidation resistance (Garcia Mesa, Jimenez-Marquez, Beltran-Maza, & Friaz-Ruiz, 1998; Romero, Guesta, & Sanchez-Muniz, 1998) but mainly in salads either as it is or in the form of "Greek salad dressing". The latter is by definition a mixture of virgin olive oil and lemon juice instantly prepared before use. It is a rich source of biophenols, lipid-soluble and water-soluble vitamins (tocopherols, β-carotene, ascorbic acid). It also contains flavonoids and lignans (Boskou & Visioli, 2003). The recommended ratio of ingredients is: ∼100 g olive oil to the juice of a large lemon.

Greek salad dressing is, as already mentioned, instantly prepared and this has certain drawbacks, e.g., it can not be used in catering meals, dishes served in airplanes, convenience foods, etc. It has to be stressed that similar products, based on olive or other vegetable oils, vinaigrettes or other instant emulsions can be found in the retail market.

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doi:10.1016/j.foodchem.2004.04.023

Salad dressings comprise a broad range of oil-in-water emulsion products, which vary in fat content (20–65%) and viscosity (Dickinson & Stainsby, 1982). Various emulsifiers and stabilizers (e.g., proteins, polysaccharides) are used for obtaining a stable emulsion with a good shelf life. Emulsifiers act by one or two mechanisms, including reduction of interfacial tension between oil and water phase, or covering oil droplets with a charged layer to create a physical barrier preventing flocculation (Walstra, 1986). Stabilizers usually stabilize emulsions by increasing the viscosity of the aqueous phase.

Polysaccharides are among the most widely used in the food industry to stabilize oil in water emulsions and control their rheological properties (Paraskevopoulou et al., 2003; Paraskevopoulou, Kiosseoglou, Alevisopoulos, & Kasapis, 1997; Phillips & Williams, 2000). They are generally odorless, colourless, and tasteless, have low energy value and digestibility. The majority of them show little surface activity and their incorporation into oil-in-water emulsions is aimed inhibiting droplet creaming by increasing the viscosity of the aqueous phase thus preserving the desired textural properties of the emulsion. The main stabilizers used are xanthan gum, galactomannans, intact or modified starches, propylene glycol alginate, pectin and carboxymethyl cellulose. By using amphiphilic polysaccharides such as gum arabic or propylene glycol alginate, which adsorb onto the surface of the droplets and prevent aggregation by steric and/or electrostatic forces, enhancement of stability against flocculation and coalescence can be achieved.

The objective of the present work was to develop a "Greek salad dressing" containing two basic ingredients of the Mediterranean diet, olive oil and lemon juice that would exhibit reasonable stability over prolonged storage. Xanthan gum in combination with gum arabic or propylene glycol alginate were evaluated for their effect on creaming behaviour, rate of coalescence, and both rheological and sensory properties of the salad dressings. The effect of polysaccharide addition level and olive oil oxidative stability (measured as peroxide value) was taken into consideration.

2. Materials and methods

2.1. Materials

Gum arabic and xanthan gum were obtained from Sigma Chemical Co., (USA), and propylene glycol alginate (referred to as PGA), from Nutra Sweet Kelco Co (USA). Extra virgin olive oil was provided by an Oils and Fats Plant located in the Athens area. Lemon juice was a commercial sample purchased in a local supermarket. Benzoic acid, obtained from Riedel de Haen (Germany), was used as preservative.

2.2. Preparation of salad dressings

50% v/v oil-in-water emulsions were prepared as follows: Gum arabic was first dispersed in lemon juice to form a gum solution of 0.5% or 1.0% w/v and the oil was slowly added whilst stirring using a propeller-type mechanical stirrer. The resulting crude emulsion was then homogenized for 1 min at 9500 rpm using an Ultra-Turrax T25 homogenizer (IKA Instruments, Germany) equipped with an S25KG-25F dispersing tool. To this emulsion a xanthan gum in lemon juice solution was progressively added so that the xanthan gum would reached a predetermined concentration level (0.30%, 0.40% and 0.45% xanthan in the continuous phase). The addition was conducted under agitation followed by a second stage of homogenization at 9500 rpm for 1 min. PGA – stabilized emulsions were made in a similar way, but homogenization was carried out at 13,500 rpm. Benzoic acid was added at a concentration of 1% (w/v) in the continuous phase. The pH values of the final samples ranged between 3.4 and 3.6.

A total of 12 salad dressings were produced. After the emulsions had been formed they were stored at room temperature before any tests were carried out. For each type of dressing three samples were prepared. The results given are the means of three measurements.

2.3. Determination of droplet size distribution

The droplet size distribution of the salad dressings was determined using the microscope method (Mita, Iguchi, Yamada, Matsumoto, & Yonezawa, 1974). The sizes of more than 1000 droplets were determined per sample and the mean volume diameter (D_v) of the droplets was derived using the equation

$$D_v = \sqrt[3]{\frac{\sum n_i \cdot D_i^3}{\sum n_i}}, \tag{1}$$

where n_i is the number of droplets with diameter D_i.

Their stability against oil coalescence was determined by following the change with time of the oil droplet distribution patterns. The rate of droplet coalescence (K) was calculated from the slope of the graph of $\ln N$ versus time, according to the following mathematical equation (Sherman, 1983)

$$\ln N_t = \ln N_0 - K \cdot t, \tag{2}$$

where, N_t and N_0 are the numbers of droplets per unit volume of emulsion at times t and $t = 0$, respectively, and

$$N_t = \frac{6 \cdot \varphi}{\pi \cdot D_v^3} \cdot 10^{12}, \tag{3}$$

where, φ is the oil phase volume fraction.

2.4. Creaming studies

The creaming behaviour of the salad dressings was followed by monitoring visually the development with time of the serum layer at the bottom of a glass volumetric cylinder containing 20 ml of the emulsion and stored at room temperature for a period of 3 months.

2.5. Rheological studies

Shearing stress-rate of shear data were obtained at 25 °C with the aid of a Brookfield DV-II, LV viscometer, equipped with the SC4-18/13R, SC4-31/13R or SC4-16/8R small adapters depending on sample viscosity. The shear data were then analyzed according to the power law equation $\tau = K \cdot \gamma^{\eta}$ to obtain the consistency index (K) and the flow behaviour index (η) of the salad dressing. The yield stress values, τ_0, were estimated from the Casson equation

$$\tau^{0.5} = \tau_0^{0.5} + K_1 \cdot \gamma^{0.5}. \quad (4)$$

The power law parameters were calculated from the regression coefficients of $\log \gamma$ and $\log \tau$ data, while the magnitude of τ_0 was determined from linear regression analysis of the square roots of shear rate and shear stress data.

2.6. Sensory evaluation

Three salad dressings containing polysaccharides at various concentrations and combinations were evaluated by applying the preference test method (Jellinek, 1984). The panelists were asked to make their evaluation on the basis of viscosity. The samples selected varied significantly in their viscosity. The analyses were performed, immediately after their preparation, by an untrained panel consisting of 30 judges. The panelists were students and members of staff of the Laboratory and were asked to rank the emulsions in order of preference by pouring them. The dressings were served in plastic cups (~15 ml each cup) at 20 °C.

2.7. Oxidative stability

Oxidative stability of olive oil or emulsions was assessed by determining the increase in the peroxide value with storage time. In the case of emulsions the oil phase was first separated by repeated freeze-thaw cycles followed centrifugation.

3. Results and discussion

Gum arabic is a hydrocolloid well-known for its surface activity and is widely used as emulsifier/stabilizer of flavour oils in the soft drink industry. In these systems the polysaccharide acts as a genuine emulsifier that adsorbs at the oil droplet surface, forming a film of high surface shear viscosity. The film-forming ability of the gum is strongly connected with its protein (2%) component, covalently linked to the carbohydrate molecule. The oil droplets are stabilized against flocculation and coalescence due to a strong steric barrier effect arising from polysaccharide blocks protruding towards the emulsion continuous phase from neighbouring droplet surfaces, where they are anchored through the more hydrophobic protein moiety of the gum (Dickinson, 2003).

Unlike other polysaccharides which stabilize colloidal systems by enhancing continuous phase viscosity, gum arabic solutions exhibit very low viscosity and are essentially Newtonian below 40% concentration (Tan, 1990). In salad dressings, due to density differences

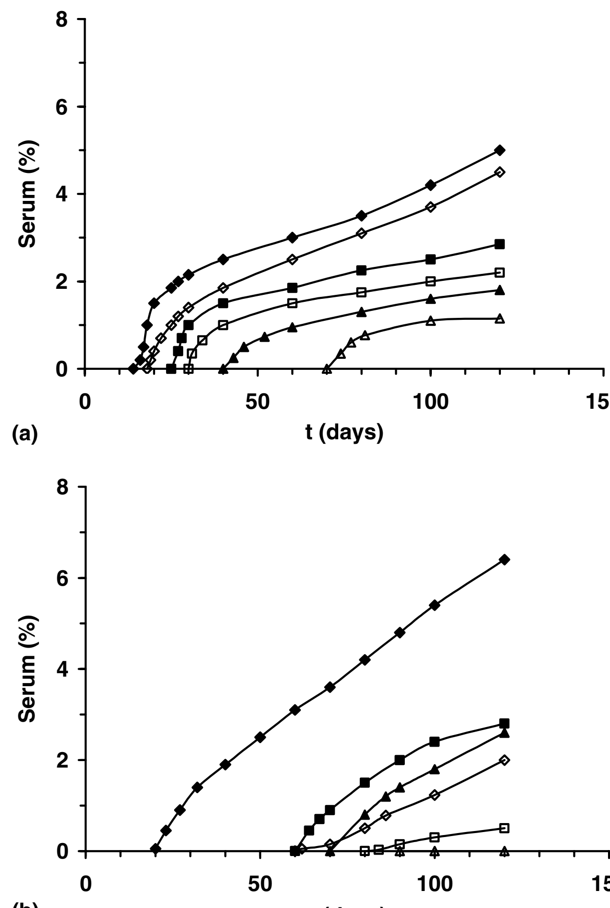

Fig. 1. (a) Influence of xanthan gum content on creaming behaviour of olive oil – lemon juice dressings containing gum arabic 0.5% (w/v) (◆■▲) and 1% (w/v) (◇□△) as emulsifier. *Key*: ◆◇ 0.30% (w/v) xanthan; ■□ 0.40% (w/v) xanthan; ▲△ 0.45% (w/v) xanthan. (b). Influence of xanthan gum content on creaming behaviour of olive oil – lemon juice dressings containing PGA 0.5% (w/v) (◆■▲) and 1% (w/v) (◇□△) as emulsifier. *Key*: ◆◇ 0.30% (w/v) xanthan; ■□ 0.40% (w/v) xanthan; ▲△ 0.45% (w/v) xanthan.

between the continuous and the oil phase, creaming of oil droplets will become apparent during storage for relatively short time periods if gum arabic is used on its own. A thickener such as xanthan gum has, therefore, to be incorporated in the system if long term stability against creaming is required. The effect of xanthan addition on creaming behaviour of salad dressing emulsions prepared with gum arabic is shown in Fig. 1(a). All the samples, irrespective of gum content, exhibited a sharply defined "delay period" typical of concentrated emulsions stabilized with polysaccharides (Parker, Gunning, Ng, & Robins, 1995). During this time period the emulsions remained stable against creaming, as no serum separation was observed and they creamed rapidly thereafter. The length of "delay period" depended on gum concentration, with the most stable sample containing 1% gum arabic and 0.45% xanthan. Since, however, gum arabic content does not affect the continuous phase viscosity (Table 1), the higher stability of 1% gum arabic emulsions may be attributed to the lower droplet size of the samples. The oil droplets of the 0.5% gum arabic emulsions are expected to rise at a higher rate according to Stokes law. However, the presence of xanthan in the systems is expected to result in extensive flocculation of oil droplets due to the depletion effect caused by the non-adsorbing xanthan gum molecules (Dickinson, 1993). According to Parker et al. (1995), addition of xanthan to pourable salad dressings induces depletion flocculation of the droplets and formation of a three-dimensional weak gel network structure that retards the process of droplet creaming. The "delay period" of the samples studied by these workers increased with xanthan concentration between 0.05% and 0.7%.

The sharp boundary lines from the start of the creaming process and the rapid creaming, leaving a clear serum phase observed for our samples, is typical of emulsions with a high oil volume fraction and high polysaccharide concentration that are heavily flocculated. The "delay period", according to Manoj, Fillery-Travis, Watson, Hibberd, and Robins (1998), should represent the time needed for channels to form within the droplet network, which allow continuous phase flow and phase separation. Velez, Fernandez, Munoz, Williams, and English (2003) suggested that the "delay period" in the creaming process of emulsions stabilized by non-adsorbing polysaccharides should be connected with the rheological properties of the continuous phase and the contribution of the oil droplet flocculation to creaming behaviour should be expected to be marginal. The oil phase fraction (10%) of the emulsions they studied may justify such an assumption. For more concentrated emulsions, however, the rheological properties of the system as a whole are expected to determine the length of the delay period (Robins, Manoj, Hibberd, Watson, & Fillery-Travis, 2001). The higher stability against creaming observed for emulsions containing 1% gum arabic compared to those of 0.5%, for equal xanthan concentration, should be attributed to the formation of a droplet network exhibiting a higher resistance to buoyancy forces as a result of a greater number of interdroplet contacts within the network. This assumption is supported by the results of Table 2 which presents rheological parameter values extracted from the application of the Casson model to shearing

Table 1
Shear viscosity of the continues phases at $\gamma = 2\ s^{-1}$ as a function of gum concentration

		η (cps)		
		Xanthan		
		0.30%	0.40%	0.50%
Gum arabic (%)				
	0.5%	325	700	925
	1.0%	325	810	925
PGA (%)				
	0.5%	750	1200	1500
	1.0%	875	1600	2000

Table 2
Rheological parameters from power and Casson equations for gum arabic/xanthan gum stabilized Greek-type salad dressings

Time (days)		Gum arabic					
		0.5% w/v			1.0% w/v		
		Xanthan gum (% w/v)					
		0.30%	0.40%	0.45%	0.30%	0.40%	0.45%
t = *0*	*K*	41.4^{a}	60.9^{b}	77.6^{c}	56.8^{b}	87.4^{c}	110.9^{d}
	n	0.19^{a}	0.18^{a}	0.17^{a}	0.16^{a}	0.16^{a}	0.16^{a}
	T_0	27.6^{a}	36.7^{b}	46.5^{c}	39.9^{b}	53.9^{d}	62.7^{e}
t = *20*	*K*	29.9	44.1	51.1	26.5	49.8	67.3
	n	0.26	0.29	0.29	0.28	0.28	0.29
	T_0	17.1	21.5	22.1	15.1	22.4	30.0
t = *100*	*K*	24.8	34.5	42.6	28.9	37.3	46.9
	n	0.28	0.32	0.36	0.33	0.34	0.35
	T_0	13.3	15.6	17.6	14.3	16.2	17.2

a–e: Different letter superscripts for each rheological parameter indicate significant difference at $P < 0.05$.

stress-rate of shear data. Fig. 2 exhibits the fitting of the Casson curve and the power law model (R^2: 0.960–0.990) in a set of shearing stress-rate of shear data of emulsions prepared with 0.5% (w/v) gum arabic. On an equal xanthan content basis, all the samples exhibited higher consistency index and yield stress values. Additionally, the parameters decreased with storage time, which was expected on the basis of the results of Table 4, where an increase of droplet size with ageing can be observed. Table 4 also incorporates rate of coalescence values obtained by analysis of the droplet size data against time according to Eq. 2. This is shown in Fig. 3, which exhibits optimum fitted curves (R^2: 0.930–0.990) to the data for emulsion droplet size increase with time. The increase of droplet size with time, however, may complicate the picture regarding emulsion creaming, since this process is expected to be enhanced as a result of droplet coalescence leading to a weakening of the flocculated droplet network structure.

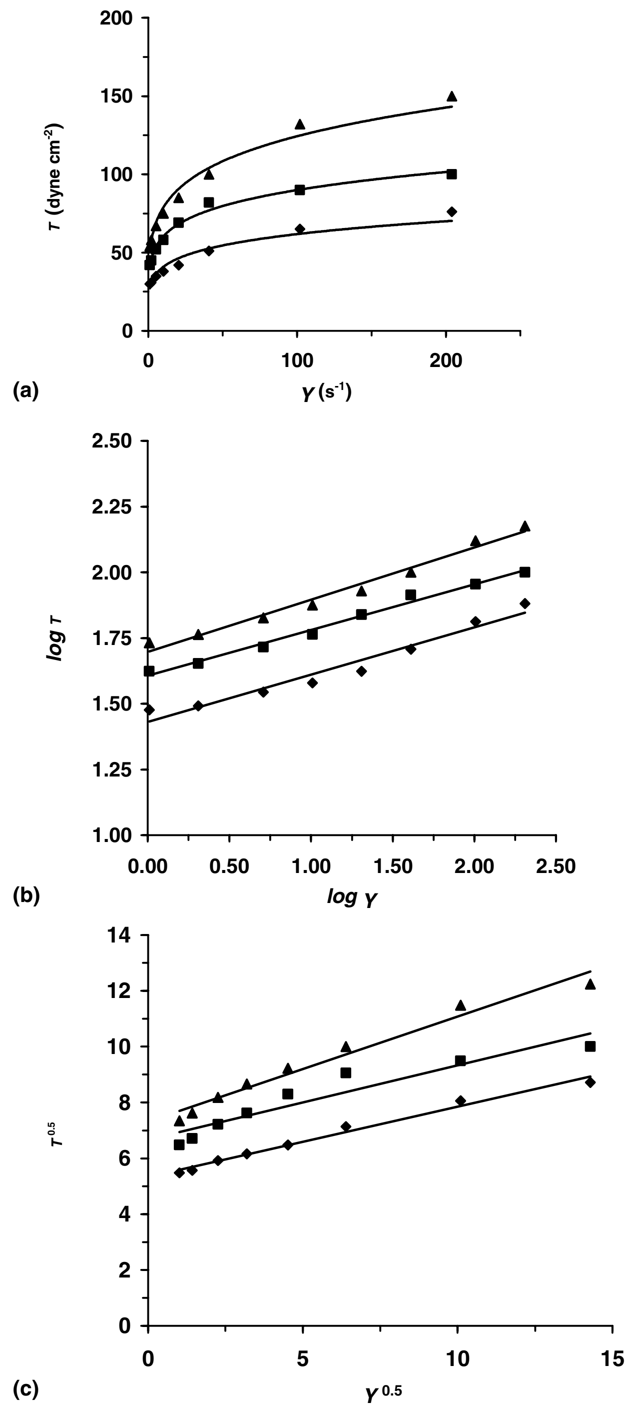

Fig. 2. Analysis of shearing stress-rate of shear: (a) experimental data of olive oil – lemon juice dressings prepared with gum arabic 0.5% (w/v) according to the Casson (b), and the power law (c) model. *Key*: ◆ 0.30% (w/v) xanthan; ■ 0.40% (w/v) xanthan; ▲ 0.45% (w/v) xanthan.

When PGA is used in place of gum arabic (Table 5), emulsions of a larger droplet size, but exhibiting higher creaming stability and having increased rheological parameter values, are obtained (Table 3). PGA is the propylene glycol ester of alginic acid. The molecule, having both lipophilic and hydrophilic parts, can adsorb at o/w interfaces and acts as emulsifier while at the same time the gum provides a smooth, creamy texture to products such as salad dressings (King, 1983).

The emulsions were also less stable against coalescence. In general, their stability against creaming was much higher and, in fact, the sample containing 1% PGA and 0.45% xanthan exhibited no serum separation over a storage time of 120 days (Fig. 1(b)). Still its rate of coalescence was much higher compared to the emulsion based on the addition of gum arabic.

The continuous phases of xanthan – PGA emulsions are about twice as viscous as those of xanthan gum arabic (Table 1). This was expected in view of the fact that alginate, due to its highly extensive molecular structure and mechanical inflexibility, produces solutions exhibiting high viscosity (Harding, 1998). Therefore, in spite of the fact the oil droplet size is much larger compared to gum arabic emulsions, the PGA emulsions exhibited more pronounced consistency index and yield stress values (Table 3). Another interesting observation regarding PGA – stabilized emulsions was that, although the oil droplet size appeared to increase with ageing time faster compared to gum arabic ones, the diminution of the rheological parameter values with time was less pronounced. This emphasizes the importance of continuous phase rheology in determining, along with droplet interactions, the rheological behaviour of the emulsions when the oil droplet size is relatively large (Sherman, 1983). Additionally, the emulsion continuous phase is expected to influence the creaming behaviour. Due to a significantly reduced number of oil droplet interactions following droplet coalescence, a weakening of the flocculated droplet network will result leading to low yield stress values. Overall, however, even after an ageing period of 100 days the yield stress values of PGA emulsions remained higher than those of gum arabic ones, a result that emphasizes the dominant role of the

Table 3
Rheological parameters from power and Casson equations for PGA/xanthan gum stabilized Greek-type salad dressings

Time (days)		PGA					
		0.5% w/v			1.0% w/v		
		Xanthan gum (% w/v)					
		0.30%	0.40%	0.45%	0.30%	0.40%	0.45%
t = 0	K	64.5[a]	82.4[b]	111.4[c]	92.6[b]	105.4[c]	154[d]
	n	0.28[a]	0.28[a]	0.25[a]	0.34[b]	0.33[b]	0.28[a]
	T_0	35.1[a]	41.2[b]	53.9[c]	33.1[a]	38.8[a]	54[c]
t = 20	K	47.8	61.5	77.7	91.7	102	134
	n	0.28	0.30	0.30	0.30	0.30	0.28
	T_0	25.2	31.1	36.5	36.5	41.4	53.4
t = 100	K	42.8	52.7	64.4	60.8	66	86.9
	n	0.25	0.28	0.31	0.32	0.33	0.33
	T_0	24.6	28.9	33.5	26.0	28.1	28.5

a–d: Different letter superscripts for each rheological parameter indicate significant difference at $P < 0.05$.

Table 4
Effect of gum arabic and xanthan gum concentration on the mean volume diameter increase with time of Greek-type salad dressings

Ageing time (days)	D_v (μm)					
	0.50% gum arabic			1% gum arabic		
	Xanthan			Xanthan		
	0.30%	0.40%	0.45%	0.30%	0.40%	0.45%
0	22.9	22.3	15.1	9.7	10.0	9.7
6	23.7	23.5	17.9	10.9	11.0	10.7
18	23.8	24.0	19.2	11.2	12.6	12.4
33	24.5	25.8	21.6	12.0	15.0	14.3
69	25.0	30.4	25.7	12.4	17.0	16.6
107	28.0	36.7	29.6	16.4	18.5	18.9
$K \times 10^7$	0.54[a]	1.55[b]	1.96[b]	1.60[b]	2.11[c]	2.20[c]

a–c: Different letter superscripts indicate significant difference at $P < 0.05$.

Table 5
Effect of PGA and xanthan gum concentration on the mean volume diameter increase with time of Greek-type salad dressings

Ageing time (days)	D_v (μm)					
	0.50% gum arabic			1% gum arabic		
	Xanthan			Xanthan		
	0.30%	0.40%	0.45%	0.30%	0.40%	0.45%
0	23.4	31.0	38.0	16.9	21.4	21.5
7	36.9	38.6	39.5	20.6	21.7	23.6
14	42.1	40.6	44.9	22.5	25.0	26.7
21	–	–	–	24.2	26.2	29.7
36	–	–	–	28.2	29.7	32.5
$K \times 10^7$	–	–	–	4.62[a]	3.32[b]	4.10[a]

a,b: Different letter superscripts indicate significant difference at $P < 0.05$.

continuous phase viscosity in the stabilization of the former.

Sensory evaluation of three selected samples of low, medium and high viscosity indicated that the panelists prefer a dressing that is not too viscous but at the same time is not very thin (Fig. 4) and spreads over a surface not very rapidly. Concerning the evaluation of dressing spreadability, and possibly of other textural characteristics, one point has to be stressed. The emulsions may suffer significant alterations of their rheological properties as a result of oil droplet coalescence during prolonged storage. Thus, following ageing a fresh product with acceptable consistency and spreadability may become less acceptable or a relatively thick sample, which

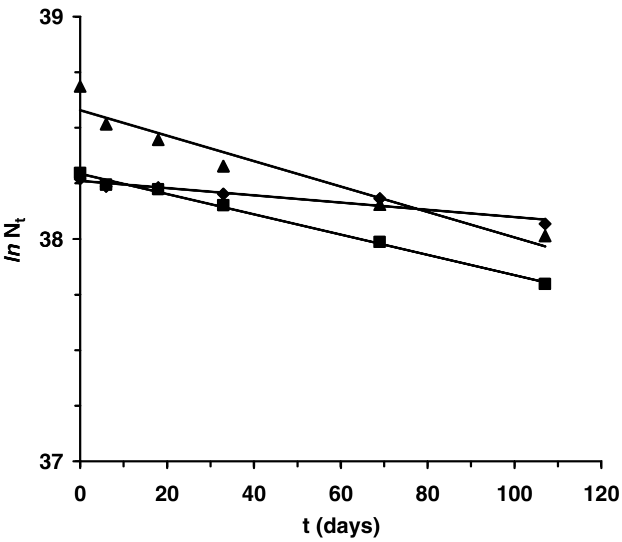

Fig. 3. ln N_t – time plots of experimental data obtained for olive oil – lemon juice dressings prepared with gum arabic 0.5% (w/v). *Key*: ◆ 0.30% (w/v) xanthan; ■ 0.40% (w/v) xanthan; ▲ 0.45% (w/v) xanthan.

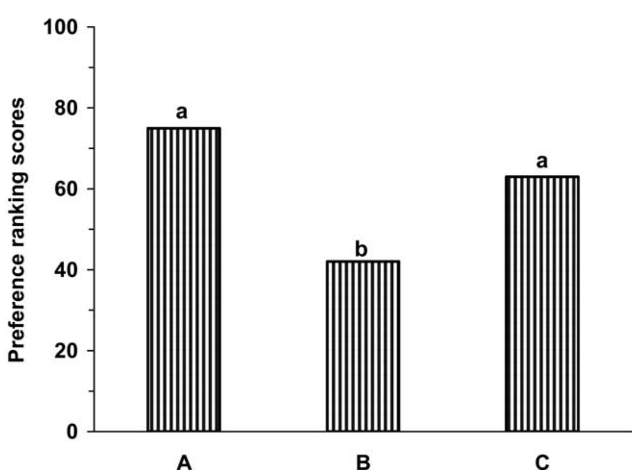

Fig. 4. Effect of viscosity on preference ranking scores of Greek–type salad dressings containing 0.30% xanthan–0.5% gum arabic: (A) low viscosity, 0.30% xanthan–1% PGA, (B) medium viscosity, 0.45% xanthan–1% PGA (C) high viscosity. a,b: Different letter superscripts indicate significant difference at $P < 0.05$.

the panelists rate low with regard to acceptability, may become thinner and more preferable.

The emulsified mixtures had a remarkable stability to oxidation, which was indicated by periodical measurements of peroxide values. Only a small increase in PVs was observed from the initial value (9.410 mequiv/kg), during the period of emulsion stability tests.

4. Conclusions

All design parameters affected salad dressings, mainly their creaming behaviour and rheological parameters (consistency index and yield stress values). The delay time before creaming begins depended on the viscosity of continuous phase, the droplet size and the polysaccharide type. In general, any combination of 0.30–0.45% xanthan with 0.5–1% gum arabic or PGA seemed to exhibit satisfactory functionality which may be exploited for commercial preparations. Further research is needed in order to prepare stable salad dressings against droplet coalescence over long periods of storage.

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