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Rheology, particle-size distribution, and stability of low-fat mayonnaise produced via double emulsions

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Abstract In this study, the effects of the double emulsification method on the rheological properties, particle size, and stability of low-fat mayonnaise were studied. Different water-phase-to-oil ratios (2:8 and 4:6) of primary emulsions and different stabilizer types (sodium caseinate, xanthan gum, and lecithin-whey protein concentrate) were used to produce double-emulsified mayonnaise. As a control sample, mayonnaise was prepared conventionally. Sodium caseinate was found to be the most efficient stabilizer. In the presence of sodium caseinate, the stability and apparent viscosity of double-emulsified mayonnaise increased but their particle sizes decreased. It was found that flow behavior of double-emulsified and conventionally prepared mayonnaise could be described by the power law model. The double-emulsified mayonnaise samples were not different from the control samples in terms of stability and particle size. In addition, using the double emulsion method, it was possible to reduce the oil content of mayonnaise to 36.6%.

Keywords: mayonnaise, double emulsion, fat reduction, stability

Introduction

Adverse health problems such as cancer, coronary heart disease, obesity, and diabetes are related to excess consumption of fat (1-4). Consequently, costumers demand reduced-fat food products as healthier food choices. Therefore, food producers and scientists attempt to find novel ways to produce low-fat content and low-calorie foods without quality loss (5). Polysaccharides, gums, carboxymethylcellulose, pectins, fiber, and maltodextrose are used as fat replacers as well as thickeners and stabilizers in low-fat food systems. However, the inclusion of fat analogs may result in loss of texture and sensory properties. Since double emulsion enables to encapsulate water particles inside the oil phase, it has the potential to reduce the oil content in food systems while maintaining a similar quality and sensory features as those of whole-fat food.

Double emulsions facilitate controlled release of active agents, encapsulation of substances, and targeted delivery; therefore, they are utilized in various industries such as agriculture, fuel energy, and chemical engineering (6). Milk, mayonnaise, salad dressing, and cake batters can be considered as examples of food emulsion systems. Double emulsions can be added to these food products to improve their functionality and reduce their salt and fat content (7,8).

Double emulsions can be categorized into two types: water in oil in water (W/O/W) and oil in water in oil (O/W/O) (9-11). In this study, since the mayonnaise system is an oil-in-water emulsion, the prepared double-emulsion type for fat reduction in this system is W/O/W.

Mayonnaise is a widely used thick creamy sauce, which produced using vegetable oil, acidic components (maleic acid, acetic acid, and citric acid), an emulsifier (naturally occurring egg lecithin), flavoring agents (sweetener, salt, garlic, or mustard), an inhibitor for unwanted crystals, texture enhancers, and stabilizers (12). The most significant characteristic of mayonnaise is its high oil content (65-75%) (13).

There are limited studies on the use of double emulsions for fat reduction in various food products. According to the study of Cofrades *et al.* (17), it was possible to reduce the fat content in pork meat when W/O/W double emulsions were utilized. Lobato-Calleros *et al.* used a W/O/W emulsion to produce low-fat fresh cheese without a significant quality loss (14). Lobato-Calleros *et al.* (15) also applied this technique to produce low-fat yogurt. However, no study has investigated the reduction of fat content in mayonnaise using the double emulsification method. Moreover, the physical properties of low-fat mayonnaise produced via the double emulsion method have not yet been studied. Apart from being a novel way of using double emulsions for low-fat mayonnaise production, this method can be cost-effective because water droplets are encapsulated into oil droplets in this method. Thus, the objective of the study is to investigate the rheological properties, particle size, and stability of low-fat mayonnaise produced using double emulsions. The effects of different stabilizers on the physical properties of mayonnaise were also investigated.

Materials and Methods

Sunflower oil, the main ingredient in the formulation of double-emulsified mayonnaise, was purchased from Komili Temizlik Ürünleri Pazarlama Inc. (Topkapı, Istanbul, Turkey). Sodium chloride (PubChem CID: 5234), xanthan gum (XG) (PubChem CID: 7107), casein sodium salt from bovine milk, sodium caseinate (SC) (PubChem SID: 176259489), and sodium azide (PubChem CID: 33557) were supplied by Sigma-Aldrich Chemical Co. (St. Louis, MO, USA). Polyglycerol polyricinoleate (PGPR) (PubChem CID: 92041135), an efficient lipophilic emulsifier used in the oil phase during the first emulsification process, was obtained from ETİ Inc. (Eskisehir, Turkey). Soy lecithin (L) (PubChem SID: 274953548) was obtained from LIPOID GmbH (Ludwigshafen, Germany). Whey protein concentrate (WPC) (80% protein content) was supplied by Tunçkaya Chemical Materials (Istanbul, Turkey). Grape vinegar and lemon juice were supplied by Kavaklıdere Wine Inc. (Ankara, Turkey). Table sugar (PubChem CID: 5988) was supplied by Torku, Konya Sugar Corp. (Konya, Turkey).

Method Herein, double-emulsified mayonnaise samples were fabricated according to the two-step emulsification method (17). Dissolved 1 g/100 g sodium chloride (NaCl) in distilled water is used as the inner water phase (W1). Dissolved polyglycerol polyricinoleate (PGPR) 3 g/100 g by weight in sunflower oil was used as the oil phase (O) for the double-emulsified mayonnaise samples. The water-phase-to-oil (W1/O) ratio for the primary emulsion was chosen as 2:8 and 4:6. The initial step for primary-emulsion fabrication was heating both the oil phase (O) and water phase (W1) to 50°C for 15 min. For the primary emulsion with a W1/O ratio of 2:8, 20 g water phase (W1) was added to 80 g oil phase (O). Initial homogenization was performed using a high-speed homogenizer (IKA T25 Digital Ultra-Turrax; IKA®-Werke GmbH & CO. KG, Staufen, Germany) at 16,000 rpm for 10 min. The prepared primary emulsions were stored at 4°C for further use.

For secondary emulsification, the required water phase (W2) was prepared via dissolution of different stabilizers. These stabilizers were chosen as 15 g/100 g by weight sodium caseinate (SC), 1 g/100 g xanthan gum (XG), or the combination of 4 g/100 g lecithin (L) and 11 g/100 g whey protein concentrate (WPC). The concentrations of the stabilizers and emulsifiers were determined via preliminary experiments. In addition, 0.05 g/100 g sodium azide (SA), 3 g/100 g lemon juice, 2 g/100 g vinegar, 2 g/100 g sugar, and 1 g/100 g salt by weight were dissolved in the W2 phase using a magnetic stirrer (Heidolph MR 3001 K; Heidolph Instruments GmbH & Co, Schwabach, Germany) at 300 rpm for 60 min for antimicrobial concerns and flavor. For the control emulsion, the conventional method of emulsification was applied, i.e., oil was homogenized into the W2 phase instead of the primary emulsion.

For the double-emulsified mayonnaise, the secondary water phase (W2) prepared using different stabilizers was poured into the chopping–mixing bowl of a food processor (Arçelik K-1190 Robolio, 700 W; Arçelik Inc., Istanbul, Turkey); further, water-in-oil (W1/O) emulsions were added to the W2 solution, drop wise. The ratio of the primary emulsion (W1/O) to the W2 phase was set via preliminary experiments. The prepared mayonnaise was poured into a beaker for further analysis and stored in the refrigerator.

Particle size of mayonnaise The particle-size distribution and mean particle sizes of the double-emulsified mayonnaise samples were determined using a laser-diffraction particle size analyzer (Mastersizer 3000; Malvern Instruments, Worcestershire, UK). Refractive indices of 1.464 and a globule absorbance of 0.01 were used in all measurements. The limit of the obscuration range was set as 8-20%. Measurements were performed in duplicate for each sample. The Sauter mean diameter, *D* (3, 2), was calculated using the following equation (13):

$$D(3,2)=\frac{\Sigma n_i d_i^3}{\Sigma n_i d_i^2} \quad (1)$$

where, d_i represents the diameter of particles (m) and n_i represents the number of related particles per unit volume of the total particles.

The span, i.e., polydispersity of size distribution, was enumerated using the following formulation (13):

$$\text{Span}=\frac{d_{0.9}-d_{0.1}}{d_{0.5}} \quad (2)$$

where, $d_{0.9}$, $d_{0.5}$, and $d_{0.1}$ represent the diameter of particles (m) that are inside the range of 90, 50, and 10%, respectively, of the cumulative sample particles.

Stability To determine the stability of the mayonnaise samples, centrifugation was used. The double-emulsified mayonnaise sample (M_0) was centrifuged at 3,913x *g* for 15 min using a centrifuge (Hettich Mikro 200/200R; Sigma Laborzentrifugen GmbH, Osterode, Germany). After separation of the supernatant part (M_1) from double-emulsified mayonnaise, the stability of the emulsion was determined using the following equation:

$$\text{Emulsion stability (\%)}=\frac{M_1}{M_0}\times 100 \quad (3)$$

where M_1 denotes the ratio of the separated part and M_0 denotes the initial weight of the emulsion.

Rheological measurements In rheological measurements, a cone and plate viscometer (Kinexus, Malvern Instruments) was used. In each measurement, a cone angle of 4°, diameter of 40 mm, and gap of 0.001 mm were set. During measurements, the temperature was kept at 25°C.

Statistical analysis To determine whether there is a significant difference between the samples, analysis of variance (ANOVA) was used ($p \leq 0.05$). Tukey's test was utilized using Minitab with a significance level of 5% (Version 16.2.0.0, Minitab Inc., Coventry, UK).

Results and Discussion

The observations made in this study reveal that there is a critical volume fraction of the dispersed phase up to which viscosity of emulsion increases. When the internal droplet fraction of a double emulsion is more than the critical value, it turns into a simple emulsion (16). In other words, an increase in the amount of the dispersed phase elevates the viscosity of a double-emulsified mayonnaise sample. This elevation in the viscosity of emulsions is attributed to the formation of a closely packed thick system as the internal phase ratio increases (13). Each hydrophilic emulsifier type has a critical concentration in the dispersed phase that can encapsulate the included oil phase without disruption. Herein, for the SC solution, XG solution, and combination of lecithin and whey protein concentrate (L-WPC) solution, the critical fraction of dispersed phase was determined as 61, 64, and 76%, respectively, via preliminary experiments.

Particle size of the mayonnaise samples Particle size is a substantial parameter for emulsion systems as it affects rheology, stability, storage life, texture, and taste of emulsions (13). The characteristics of mayonnaise samples and their stabilities are associated with the mean particle size and particle-size distribution of the oil droplets.

Two different particle-size distributions for double emulsion systems have been reported: monomodal (17,18) and bimodal (8,19). In this study, both monomodal and bimodal distributions were observed depending on the stabilizer type used in the W2 phase. As can be seen in Fig. 1, each hydrophilic emulsifier type resulted in a different pattern of size distribution in the double-emulsified mayonnaise samples. Samples with SC had a monomodal size distribution, whereas XG and L-WPC led to a bimodal size distribution within the mayonnaise samples. According to the study performed by Hemar *et al*. (20) and Su *et al*. (21), double-emulsion systems containing PGPR followed a bimodal particle-size distribution. However, Cofrades *et al.* (17) observed monomodal size distribution in emulsions containing PGPR, SC, and L-WPC. The differences in particle-size distribution were dependent on the homogenization qualifications, composition of the double emulsion, and viscosity of ingredients as well as the concentration and type of emulsion.

Table 1 shows the Sauter mean diameter (D(3,2)), volume mean diameter (D(4,3)), and span-value results. The Sauter mean diameter and volume particle-size results were found to be correlated. The Sauter mean diameters of the samples containing XG were significantly higher than that of samples containing SC or L-WPC (Table 1) ($p\leq0.05$). The Sauter mean diameter of the double-emulsified mayonnaise samples containing SC was the smallest, followed by the samples containing L-WPC and XG, respectively (Table 1). XG, an anionic thickener, significantly increases the viscosity of the second aqueous phase (W2) (22). Throughout the mixing and homogenization processes, particles of the primary emulsion were distributed in the viscous structure of the continuous phase, and stabilization was achieved by the viscous network of XG. Nevertheless, the highly viscous structure of the continuous phase resulting from the thickening characteristics of XG restrained the formation of fine, evenly distributed droplets of the primary emulsion. The development of a thick layer around the emulsion droplets could also explain why the mayonnaise sample containing XG had larger particle size than the other samples. A high concentration of biopolymers and high molecular weight might generate a thick gel around the particles regardless of their size.

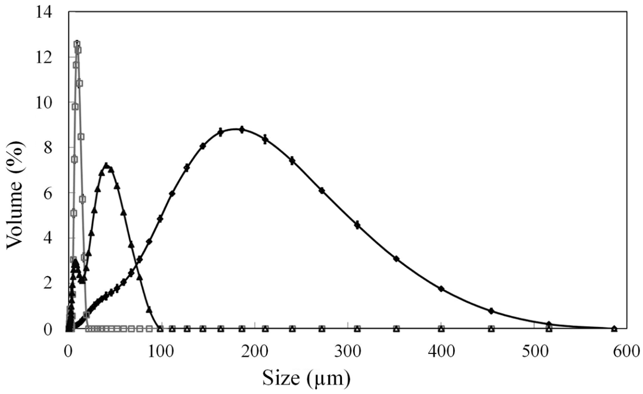

Fig. 1. Particle-size distribution graph of the double-emulsified mayonnaise samples with a water-to-oil ratio of 4:6 containing sodium caseinate (□), xanthan gum (◇), and lecithin-whey protein concentrate (△).

Table 1. Particle-size distribution for the double-emulsified and conventionally prepared mayonnaise samples

Mayonnaise Sample	Sauter mean diameter (μm) D (3,2)	Volume mean diameter (μm) D (4,3)	Span
SC-Conventional	5.29±0.049[d1)]	8.29±0.039[d]	1.120±0.012[d]
SC-2.8	3.49±0.035[d]	7.22±0.045[d]	1.033±0.016[d]
SC-4.6	3.76±0.056[d]	7.73±0.068[d]	1.110±0.057[d]
XG-Conventional	73.84±2.397[c]	85.72±3.412[c]	1.733±0.037[ab]
XG-2.8	205.42±7.516[b]	228.51±8.635[b]	1.850±0.026[ab]
XG-4.6	226.96±4.872[a]	249.68±7.913[a]	1.920±0.021[a]
L-WPC-Conventional	12.58±0.601[d]	26.98±2.520[d]	1.384±0.027[c]
L-WPC-2.8	13.29±0.240[d]	29.19±3.224[d]	1.533±0.111[c]
L-WPC-4.6	13.72±0.098[d]	29.79±0.088[d]	1.733±0.013[b]

[1)]Means containing different letters within the same column are significantly different ($p\leq0.05$).

For double-emulsified mayonnaise samples containing XG, the primary emulsion with a ratio of 2:8 had smaller particle size than that with a ratio of 4:6. In the mayonnaise samples containing L-WPC and SC, the Sauter mean diameters of the mayonnaise samples were not affected by different W1/O ratios of the primary emulsion. Moreover, there was no significant difference between the conventionally prepared mayonnaise and double-emulsified mayonnaise

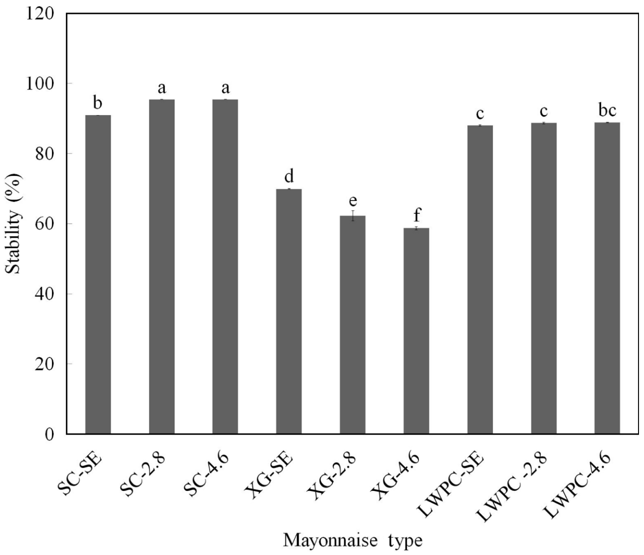

Fig. 2. Stability of the conventionally prepared mayonnaise (SE) and double-emulsified mayonnaise with water-to-oil ratios of 2:8 and 4:6 containing (SC): sodium caseinate, (XG): xanthan gum, and (L-WPC): lecithin-whey protein concentrate. Bars with different letters represent the significant difference ($p \leq 0.05$).

samples in terms of particle size when L-WPC or SC was used ($p \leq 0.05$).

Rheological properties of the mayonnaise samples The rheological properties are affected by formulation and process conditions (23). Their effect on consumer choice makes the rheological properties of mayonnaise crucial.

The effects of different hydrophilic emulsifiers and the water-to-oil ratio in the primary emulsion on the rheological properties of mayonnaise were evaluated. All the mayonnaise samples behaved as non-Newtonian fluids. Moreover, an increase in shear stress decreased the apparent viscosity of the double-emulsified mayonnaise samples; this can be concluded as a shear thinning behavior. The same behavior of double emulsion systems has been reported in the literature (24-26). The shear thinning behavior resulted from structural deformation of the network structure. The shear stress applied caused deformation of the droplets of the secondary emulsion and production of the primary emulsion (27).

In the study, investigations of the rheological properties of mayonnaise showed that flow behavior could be described by the power law model (Table 2). In the literature, there are studies describing the flow behavior of mayonnaise on the basis of the power law, Herschel–Bulkley model, and Carreau model (5,28,29). Different types of emulsifiers affected the rheological properties differently. The highest consistency coefficient (K) was achieved by the samples containing SC. This could be attributed to the decreased particle size of the mayonnaise samples produced using SC because a reduced particle size is known to improve the rheological properties of double emulsions. Conversely, the samples prepared using XG, characterized by a larger particle size, and had the lowest consistency index.

Table 2. Power law model, $\eta = K\gamma^{n-1}$, coefficients of the mayonnaise samples prepared using different emulsifier types and ratios of the primary emulsion

Mayonnaise Sample	K (Pa.s^n)	n	R^2
SC-Conventional	31.72±5.939$^{c1)}$	0.3747±0.016^{bcd}	0.9995
SC-2.8	321.2±5.303^{a}	0.3545±0.017^{cd}	0.9990
SC-4.6	295.3±3.464^{b}	0.3290±0.001^{d}	0.9972
XG-Conventional	9.472±0.221^{d}	0.1759±0.017^{e}	0.9758
XG-2.8	2.892±0.071^{d}	0.6153±0.074^{a}	0.9980
XG-4.6	1.525±0.160^{d}	0.4869±0.012^{b}	0.9922
L-WPC-Conventional	9.940±0.063^{d}	0.4051±0.017^{bcd}	0.9950
L-WPC-2.8	9.526±0.166^{d}	0. 4341±0.336^{bc}	0.9971
L-WPC-4.6	8.155±0.067^{d}	0.3699±0.006^{bcd}	0.9638

[1)]Means containing different letters within the same column are significantly different ($p \leq 0.05$).
K, consistency coefficient; n, flow behavior index

Since particle size and apparent viscosity were inversely related, an increase in particle size led to weaker rheological properties. In other words, mayonnaise samples with SC had a minimum average particle size of 4.18 μm and the highest consistency coefficient values. It was observed that as the particle size of mayonnaise decreased, the apparent viscosity increased (Table 1).

Stability of mayonnaise Stability is a crucial quality parameter of mayonnaise. The stability of the mayonnaise samples can be predicted on the basis of their particle size and rheological properties. It was found that as the particle size of oil droplets dispersed in the water phase decreased, the stability of mayonnaise increased (30).

As the viscosity of a system increases, according to the well-known Stoke's Law, velocity of a particle decreases and the velocity of movement is proportional to the square of the radius (31). Thus, small particles are separated at lower velocities owing to the gravitational forces, and this behavior has been observed in the literature (7,17,32). The behavior that reduced the particle size and increased the stability of the double-emulsified mayonnaise samples was detectable in the particle-size and stability results of the samples containing XG. In other words, samples containing XG had the largest particle size and lowest consistency index as they had the lowest stability values. Gravitational separation is one of the pronounced problems regarding the instability of double emulsions, which can be explained as separation of the disintegrated phases of an emulsion system owing to the densities of the phases. Additionally, it was reported that emulsions' small particle size increased the viscosity of double-emulsified mayonnaise (33). Increased viscosities of emulsion systems decreased the movement of droplets and inhibited coalescence, sedimentation, and other instabilities within the emulsion. The combined effect of increased viscosity of an emulsion and reduced particle size increased the stability of the mayonnaise samples (Fig. 2). This effect was observed in various emulsion studies related to cellulose nanofibrils (25), gum Arabic, and XG (34). The highest stability values were obtained with the minimum particle size and highest viscosity values in samples containing SC (Table 1).

The particle size of the double-emulsified mayonnaise samples prepared using L-WPC was smaller than that of the samples containing XG (Table 1). Moreover, the consistency index of the samples containing L-WPC was higher than that of the samples containing XG. Therefore, as expected, the stability of the mayonnaise samples containing L-WPC was found to be higher than that of the samples containing XG (Fig. 2). The double-emulsified mayonnaise samples with high consistency coefficients of the power model had high stability. Increased consistency of the emulsion accompanied by a small particle size increased the stability of the emulsion (25).

Oil content reduction in mayonnaise The main objective of this study was to observe the characteristics of fat-reduced mayonnaise samples prepared using double emulsions by evaluating their physical properties and stability. The inclusion of a primary phase into the mayonnaise samples decreased the total oil content. Table 3 shows the oil contents of the conventionally prepared mayonnaise samples and double-emulsified mayonnaise samples, with primary emulsions comprising a water-to-oil ratio of 2:8 and 4:6. The incorporation of the primary emulsion with a ratio of 2:8 decreased the oil content of the conventionally prepared mayonnaise samples by 20%. This decrease was 40% in the case of the primary emulsion with a ratio of 4:6. The conventionally prepared mayonnaise samples containing SC along with the primary emulsion with a ratio of 4:6 had similar stability, particle size, and viscosity values. In addition, this formulation had an oil content of 36.6%. Thus, it was possible to reduce the oil content of mayonnaise without losing the quality using double emulsions.

The double-emulsions samples containing SC had the smallest particle size whereas the ones containing XG had the largest. The stability of the mayonnaise samples was found to be correlated with their particle size and apparent viscosity. In terms of stability, particle size, and rheological properties, the double-emulsified mayonnaise samples containing SC may be used as an alternative to commercially available mayonnaise with lower oil content. In addition, the oil content of mayonnaise was reduced to 36% using double emulsions, which was 40% less than the oil content of conventionally prepared mayonnaise. Thus, the double emulsion method can be recommended for producing stable, low-fat mayonnaise without adversely affecting its physical properties.

Disclosure The authors declare no conflict of interest.

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Table 3. Oil content of the mayonnaise samples

W2 formulation type	Conventionally prepared mayonnaise (g/100 g)	Double-emulsified mayonnaise with ratio of 2:8 (g/100 g)	Double-emulsified mayonnaise with ratio of 4:6 (g/100 g)
SC	61.0	48.8	36.6
XG	64.0	51.2	38.4
L-WPC	76.0	60.8	45.6

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