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Effect of Inulin Formulation on the Microstructure and Viscoelastic Properties of Low-Fat Mayonnaise Containing Modified Starch

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ABSTRACT: Combinations of three types of inulin differing in the degree of polymerization, that is, short, medium, and long chained (0–10%), and modified starch (0–3%) with different composition ratios were prepared according to the D-optimal design of experiments. The microstructural and rheological characteristics of the prepared samples were analyzed to study the effect of the inulin composition on the low-fat mayonnaise. Rheological characterizations, including oscillatory frequency sweep tests, transient creep, and stress relaxation analysis, were carried out on the samples. An optical microscope was used to observe the microstructure. According to the results, the effects of all types of inulin were precarious in the presence of starch ($\geq$1.5%). In fact, a relationship was found between the inulin type and concentration and also the starch content in all of the prepared samples; with increasing starch content ($\geq$1.5%), inulin chain length, and concentration of long-chain inulin ($\geq$5%), the elastic properties of the emulsion were improved and showed a higher resistivity against deformation. Furthermore, a more packed structure with a larger average particle diameter and dominant monodispersity were observed under such conditions. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 130: 801–809, 2013

KEYWORDS: morphology; polysaccharides; viscosity and viscoelasticity

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INTRODUCTION

Mayonnaise, prepared by the precise mixing of egg yolk, oil, vinegar, and spice (particularly mustard), is a semisolid oil-in-water emulsion typically containing 70–80% fat, in which the egg yolk acts as an emulsifier.[1] These emulsions, especially those with low fat, are thermodynamically unstable systems that turn into stable emulsions with the addition of emulsifiers; this decreases the surface tension by the introduction of hydrocolloids and increases the continuous-phase viscosity. Both procedures consequently led to the restriction of the oil disperse-phase mobility.[2,3]

In recent times, the utilization of inulin as a hydrocolloid, which can be a substitute of fat in functional foods, has grown because of its health-promoting and technological properties. Inulin is a polysaccharide consisting of GF_n (fructans which start with glucose) and F_m (fructans which start with fructose) compounds, in which m and n are the numbers of fructose units that are interconnected with β(2–1) bounds ending in a terminal glucose (G) molecule.[4] Inulin, in addition to its nutritional characteristics, can be used as a potential low-calorie gelling and texturizing agent.[5] In fact, it absorbs a considerable volume of water in aqueous solutions, such as a hydrocolloid media, and forms a crystalline gel–particle network within the continuous phase; this leads to the development of high viscosity under small stresses. It also intensifies the system's elastic characteristics and inhibits the creaming instability phenomenon in the emulsion.[6] Several research studies have been published recently on the rheological behavior of low-fat-emulsion- or mayonnaise-containing hydrocolloid constituents.[3,7–12] James[13] and Mantzouridou,[14] respectively, used medium-chain (8–12 units) and long-chain (23 units) inulins as substituents for fat and prebiotic compounds in salad dressing formulations. It is well known that the use of inulins with different chain lengths result in different technical properties; for example, increasing the inulin chain length decreases its solubility[16] and simultaneously improves the thermal stability and gelability.[17] To the best of our knowledge, there have been no reports on the introduction of an optimum combination of short-, medium-, and long-chain inulins into low-fat mayonnaise. In the

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latest research,[18] the physicochemical, sensory, and some rheological properties of low-fat mayonnaise containing different composition ratios of the three types of inulins and modified starch were investigated. In this article, we discuss the effect of the inulin composition ratio on the dynamic and transient viscoelastic characteristics of the final product in the formulation in which inulin was used instead of modified starch. With regard to the potential capability of rheology as a useful technique for observing the microstructural properties of materials, the relationship between the rheological behavior and microstructure of low-fat mayonnaise containing inulin was also studied.

EXPERIMENTAL

Materials

Three types of inulin, that is, short-chain (2–10 units, Frutafit CLR), native (8–13 units, Frutafit IQ), and long-chain (≥23 units, Frutafit TEX!) inulin, were provided by Sensus (Roosendaal, The Netherlands). Acetylated potato distarch phosphate (cold swelling, Emjel EP300), xanthan gum (GRINDSTED xanthan 200), guar gum (GRINDSTED GUAR 250), and milk protein (Nutrialc DR7015), from Emsland-Stärke GmbH (Emsland, Germany), Danisco (Copenhagen, Denmark), Danisco (Copenhagen, Denmark), and Arla (Arhus, Denmark), respectively, were also used in this research. All other ingredients used to prepare mayonnaise samples, such as soybean oil, egg yolk, vinegar, sugar, salt, and mustard, were supplied from the R&D Center of Behrouz Food Industries Co. (Tehran, Iran).

Preparation of Inulin Suspension

Different combinations of three types of inulins used in the mayonnaise samples were prepared according to the runs determined by D-optimal design as a type of combined mixture design with points selected according to a mathematical algorithm. Details of the candidate runs are reported in Table I. Each combination (totally fixed at 10%) was suspended in water and mixed by a mechanical overhead stirrer (EUROSTAR Power Control-Visc 6000, IKA, Staufen, Germany) at 1200 rpm. The suspension was then heated in an 80°C water bath for 15 min to form a gel and was stored at 5°C until it was formulated in the mayonnaise samples.

Table I. Combined Mixture Design of 28 Treatments Containing Frutafit CLR, Frutafit IQ, Frutafit TEX!, and Modified Starch

	Independent variables			
Run	Frutafit CLR (%)	Frutafit IQ (%)	Frutafit TEX! (%)	Modified starch (%)
Control	0	0	0	3
R1	6.67	1.67	1.67	0.75
R2	6.67	1.67	1.67	2.25
R3	0	5	5	1.5
R4	0	10	0	3
R5	10	0	0	3
R6	1.67	6.67	1.67	2.25
R7	1.67	6.67	1.67	0.75
R8	10	0	0	0
R9	10	0	0	1.5
R10	0	5	5	3
R11	5	0	5	1.5
R12	5	5	0	3
R13	0	10	0	1.5
R14	0	0	10	0
R15	10	0	0	3
R16	5	0	5	0
R17	1.67	1.67	6.67	0.75
R18	10	0	0	0
R19	0	10	0	3
R20	0	5	5	0
R21	5	5	0	0
R22	0	0	10	1.5
R23	5	0	5	3
R24	5	5	0	1.5
R25	0	0	10	3
R26	0	0	10	3
R27	0	10	0	0
R28	0	0	10	0

Mayonnaise Preparation

The mayonnaise samples were formulated as follows: fresh egg yolk = 8%, vinegar (11% acetic acid) = 4.5%, soybean oil = 30%, salt = 2.0%, mustard = 0.5%, sugar = 4.7%, xanthan = 0.02%, guar = 0.08%, milk protein = 0.5%, modified starch (0–3%), and different composition ratios of the three types of inulin. At the first stage, egg, inulin dispersion, and vinegar were mixed together, and then, all of the powder ingredients (including modified starch) were added and stirred homogeneously by a pilot turbo mixer (MHM-60K-I high-speed vane disk stirrer, Arkan Felez, Iran). Finally, oil was added very slowly with stirring at 6000 rpm for 6 min. The emulsions were stored in glass sealed jars, left for 24 h at room temperature, and then analyzed by rheological and microscopic techniques.

Rheological Characterization

Oscillatory Experiments. All rheological measurements were carried out with a stress-controlled rheometer (Paar Physica MCR 3000, Anton Paar GmbH, Austria) with a parallel-plate geometry (diameter = 25 mm, plate gap = 1 mm) under a nitrogen atmosphere at 25°C and in the frequency range 0.01–100 Hz. Before testing, strain sweep tests were carried out at fixed frequency of 1 rad/s. The linear viscoelastic zone was determined as 0.4%, accordingly.

Transient Experiments. The creep and creep recovery experiments were carried out under a fixed shear stress of 2.5 Pa, which was kept constant for 300 s and then recovered at the same period of time. The stress relaxation test was carried out at a constant strain of 0.4% (within the viscoelastic zone) for 300 s.

Optical Microscopy

A reflective optical microscope (Carl Zeiss, Jenaver, Germany) was used to investigate the microstructure of the prepared

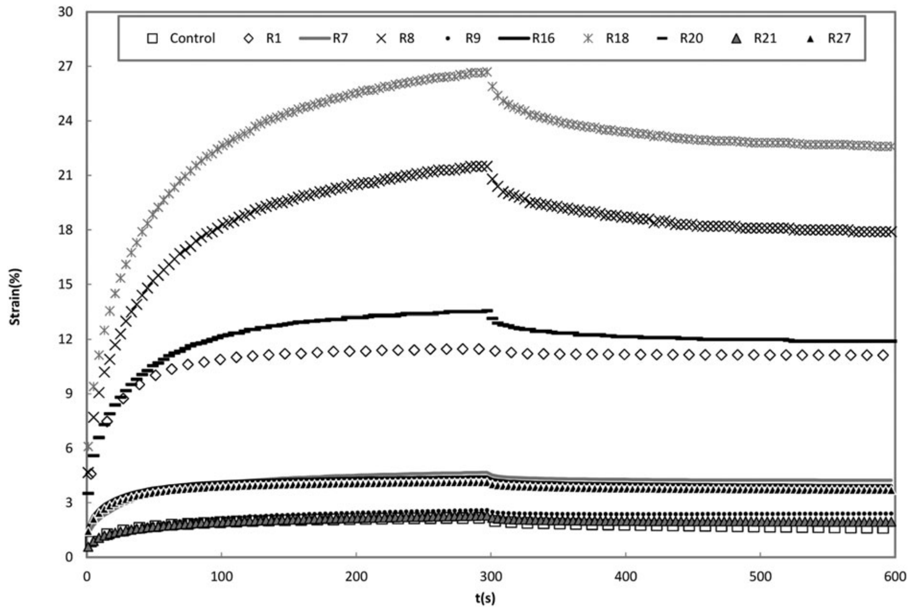

Figure 1. Strain variations versus time (t) in the creep experiments for the control and R1, R7, R8, R9, R16, R18, R20, R21, and R27.

samples. A microscope glass slide was covered with a droplet of each sample and observed at a magnification of 100×.

Statistical Analysis

Design Expert (release 7.0.0, Stat-Ease, Inc., Minneapolis, Minnesota, USA) was used to design the sample compositions (Table I) and analyze the experimental results because it is considered to be a powerful problem-solving technique for improving the process performance, yield, and productivity when a process is affected by a number of parameters. This method provides a simple, systematic, and efficient methodology for the optimization of control factors. A common problem in posttrial design of experiment (DOE) analysis is the lack of data. Before the planning of additional experimental trials to complete a design matrix, the attempt to estimate missing data by means of prediction models could be an interesting alternative. Accordingly, there have been several studies on the optimization of different features of processing parameters affecting the material properties using DOE methods reported in the literature.[19,20]

In this study, four factors, that is, the compositions of short-, medium-, and long-chain inulin and also modified starch, each at six levels were considered with Design Expert software. For four parameters each at six levels, the traditional full factorial design would require 4^6, that is, 4096, experiments; this would be difficult, time consuming, and costly. A combined design could consist of a combination of mixture components and process factors. There are two design types used to create combined designs: D-optimal and user defined. D-optimal design (used here) points are selected to minimize the variance associated with the estimates of the coefficients in the model that the user specifies. The design space is defined by the low- and high-level constraints on each factor and any multifactor constraints.

D-optimal point selection chooses points from the candidate point set that are spread throughout the design region. The points selected are model-dependent and are selected with only this criterion in mind from many possible combinations of factors and their levels. Therefore, for adequate design, the D-optimal points should be augmented to provide estimates of pure error by replication and to determine the lack of fit with excess design points. Given a candidate set, the D-optimal design process for mixture designs works exactly the same way as the response surface method designs. D-optimal point selection minimizes the determinant of the $(\mathbf{X'X})\ -\mathbf{1}$ information matrix.

Table I summarizes the compositions and nomenclatures of the control sample and 28 designed runs composed of 0–10% short-chain, medium-chain, and long-chain inulin and 0–3% modified starch (Frutafit CLR/Frutafit IQ/Frutafit TEX!/modified starch). The emulsion stability, color characteristics (L^*, a^*, b^*, and ΔE), flow behavior, and some viscoelastic parameters were considered the response variables.

RESULTS AND DISCUSSION

Creep Experiments

As shown in Figure 1, the structural deformation in the elastic region (elastic deformation β_{elastic} and maximum elastic deformation $\beta_{\max}$ values) under shear stress compared to the control sample was intensified in the case of shorter inulin chain lengths, long-chain inulin concentrations less than 5%, and starch contents below 0.75%. Accordingly, the deformation undergone by sample R18, which only contained short-chain inulin (10%), was maximized. This observation suggested that short-chain inulin did not contribute in the formation of the gel–particle network within the continuous-phase mayonnaise emulsion. The phenomenon could have been attributed to the high solubility of short-chain inulin, which inhibited the formation of a gel network, which was strong enough against the deformations under applied shear stress.[21]

Figure 1 shows that with increasing inulin chain length, the viscoelastic characteristics of the samples approached those of

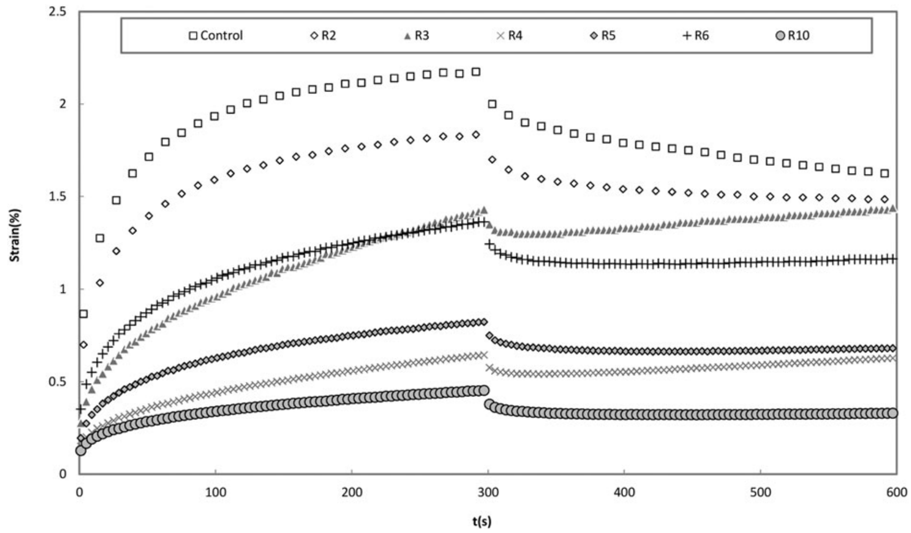

Figure 2. Strain variations versus time (t) in the creep experiments for the control and R2, R3, R4, R5, R6, and R10.

the control sample. In fact, the increased gel strength confirmed the interactions established between the starch and long-chain inulins.

In a comparison of Figures 2–4, the minimum elastic deformation (minimum $\beta_{elastic}$ and β_{max} values) was observed in R26, which contained the highest content of long-chain inulin and modified starch. This might have been due to the increased strength of the three-dimensional gel network established in R26 and the interactions between inulin chains and starch with increasing inulin and starch concentration and also inulin chain length. According to the creep experiment results, Dolz et al.[12] concluded that the addition of some types of hydrocolloids (e.g., modified starch, xanthan, locust bean gum, and xanthan/locust bean gum hybrid) as gelling agents to the mayonnaise emulsion (containing 34% oil) would strengthen the structure and increase the elastic modulus values (G_1 and G_0) and viscosity (η_0) of the emulsion. They ascribed the increase observed in the elastic modulus values to the deformation of spherical disperse-phase oil droplets under a constant shear stress undergone by the system during creep experiments and the increased strength of the three-dimensional gel networks within the continuous phase of the emulsion. In fact, the effective surface area of the oil droplets and, consequently, the surface tension between them increased because of the deformation. This might have justified the system resistance against the deformations.

There seemed to be a tight adaption between the strain variation trends of the control sample with R28; this indicated that under equal shear stresses, 10% of long-chain inulin showed an interstructural deformation similar to that of the control sample containing just 3% modified starch. In other words, because of the structural characteristics and deformation and/or destruction resistivity, 10% long-chain inulin might act as a proper substituent of 3% modified starch in low-fat products.

Oscillatory Tests

According to Figures 5 and 6, typically (illustrated in the Supporting Information, Figures S1 and S2), the hydration

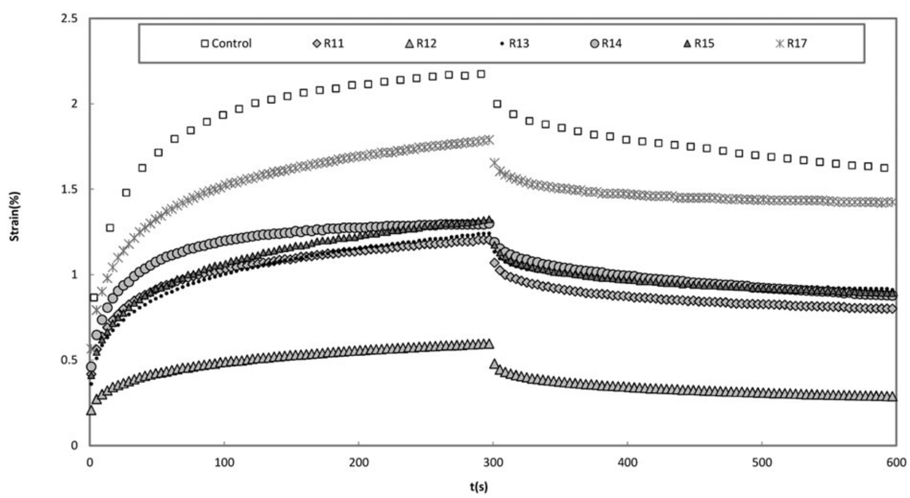

Figure 3. Strain variations versus time (t) in the creep experiments for the control and R11, R12, R13, R14, R15, and R17.

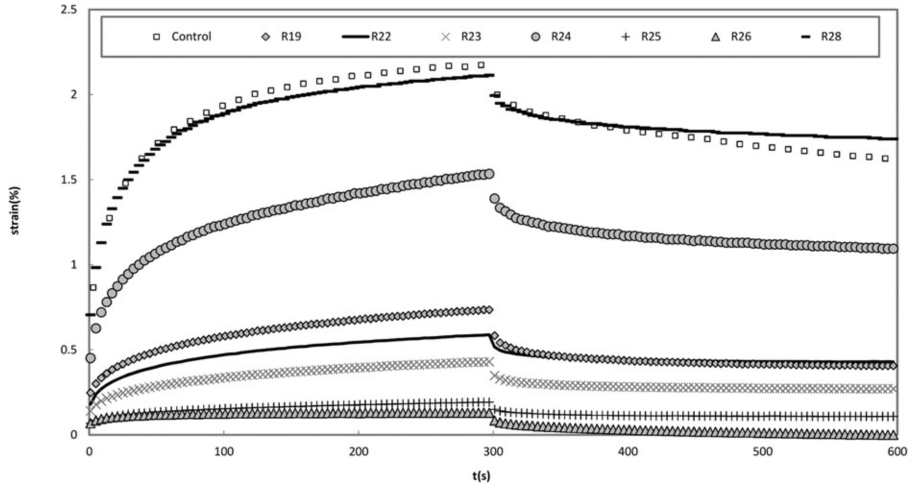

Figure 4. Strain variations versus time (t) in the creep experiments for the control and R19, R22, R23, R24, R25, R26, and R28.

phenomenon that occurred with increasing long-chain inulin and modified starch concentration resulted in the formation of a strong, three-dimensional gel network, and the viscosity increased in the emulsion continuous phase. With respect to the similarities between the characteristics of R28 and the control sample evidenced by both the creep and oscillatory experiments, the equal effect of 10% inulin and 3% starch on the rheological behavior of low-fat mayonnaise was confirmed. Furthermore, the simultaneous presence of concentrated long-chain inulin and modified starch within the samples improved the zero complex viscosity; for example, in R9, R13, and R22, increasing trends were perceptible. This trend was maximized in R25 because of the synergistic effect of interactions between starch and inulin. The increasing polymer chain length from R9 (10% short-chain inulin) and R13 (10% medium-chain inulin) to R22 (10% long-chain inulin), which all contained 1.5% modified starch, was accompanied by an expected increase in the viscosity; this confirmed the effect of the polymer chain length on the system viscosity, the major contribution of long-chain inulin in the crystallization, and the consequent formation of a stronger gel network.

In R25, with increasing concentration of starch up to 3%, a positive interaction was observed between the inulin particle gel network and modified starch polymer chains within the emulsion media containing disperse-phase oil droplets. Zimeri and Kokini[21] investigated the rheological characteristics of several aqueous systems containing inulin and waxy maize starch with different compositions and observed a decrease in the zero shear viscosity with increasing inulin content at polymer concentrations lower than 20% and the converse behavior above 30%. They believed that this would was due to the formation of a dominant continuous inulin network, which trapped the starch granules and resulted in the intensification of synergistic interactions between inulin and starch. According to Gonzalez et al.,[22] the low solubility and consequent affinity of long-chain inulin to form gel microcrystals in the presence of starch hydrated granules is representative of an increase in the viscoelastic properties.

With this formulation suggested for the low-fat mayonnaise, the final inulin and modified starch concentration, 10 and 0–3 %, respectively, provided a 30% concentration and led to the mentioned interactions and consequent viscosity and storage

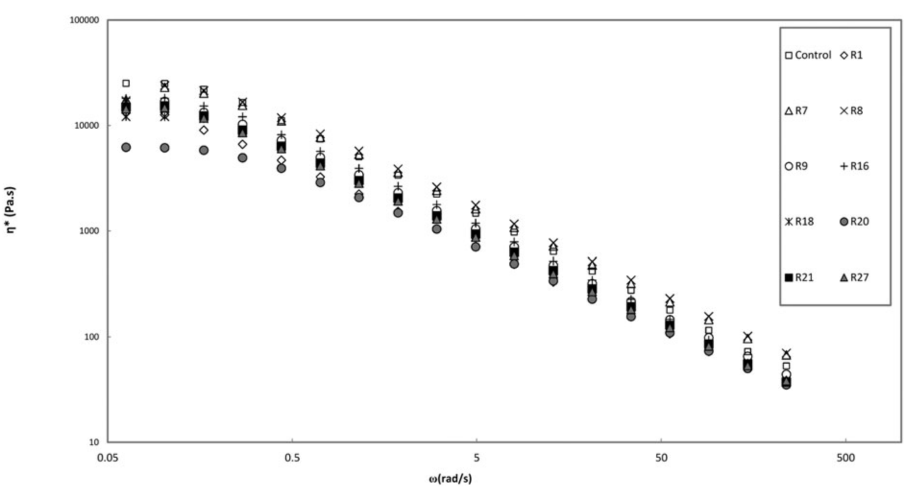

Figure 5. Complex viscosity (η^*) versus the angular frequency (ω) for the control and R1, R7, R8, R9, R16, R18, R20, R21, and R27.

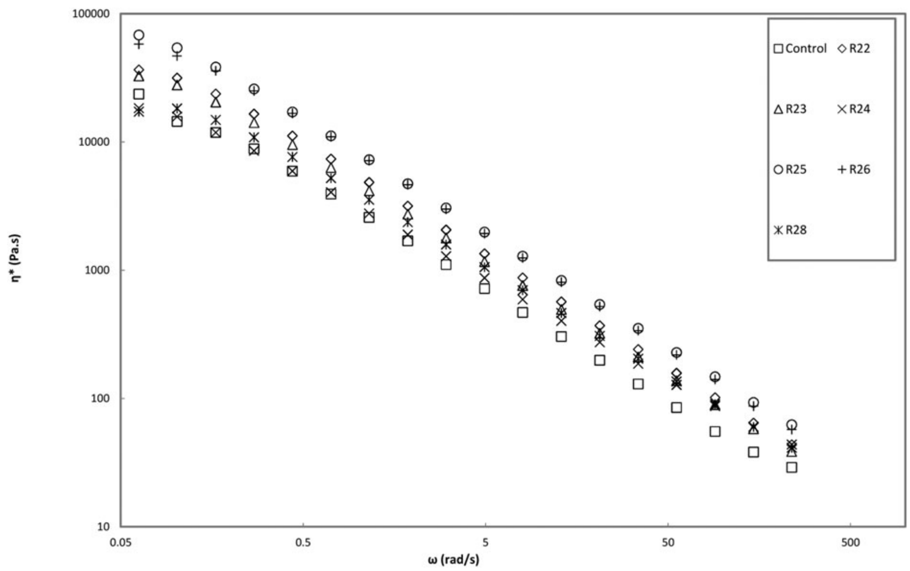

Figure 6. Complex viscosity (η^*) versus the angular frequency (ω) for the control and R19, R22, R23, R24, R25, R26, and R28.

modulus increases in the samples with higher inulin and starch concentrations. Gonzalez-Tomás et al.[22] also reported significant increases in the complex viscosity and storage and loss moduli of semisolid dietary products containing 7.5% long-chain inulin and 3.25 and/or 4% modified tapioca starch.

Stress Relaxation Tests

Figures 7 and 8 show the typical stress relaxation experiment data, which confirm confirming the results of creep tests; as with increasing inulin chain length, long-chain inulin concentration (≥5%) and modified starch content (≥1.5%) characteristics of the low-fat mayonnaise samples approaches to the viscoelastic solid state. The shear stress undergone by the samples also increased, and this resulted in a decrease in the strain. Accordingly, as evidenced by the creep test results, the maximum elastic deformation appeared in R18 (not shown here; see Figures S3 and S4 in the Supporting Information), which contained 10% short-chain inulin, and the minimum structural elastic deformation corresponded to R26, which had the highest content of long-chain inulin and modified starch. Considering $\tau_{elastic}$ and $t_{relaxation}$ respectively as elastic shear stress and relaxation time; R18 and R26, respectively, presented viscoelastic liquidlike (low $\tau_{elastic}$ and $t_{relaxation}$ values) and solidlike (high $\tau_{elastic}$ and $t_{relaxation}$ values) properties that corresponded to the highest and lowest structural deformations against the applied strain. Furthermore, the shear stress variation trend in R28, which contained 10% long-chain inulin, competed with the control sample; this indicated the affinity of the systems containing 10% long-chain inulin and 3% modified starch to form a stable gel–particle network within the emulsion continuous phase, which acted against intrastructural deformations in a way similar to that of the modified starch.

Figure 9 shows typical the microstructure of control sample, R18, and R26. Mayonnaise microstructure depends on the

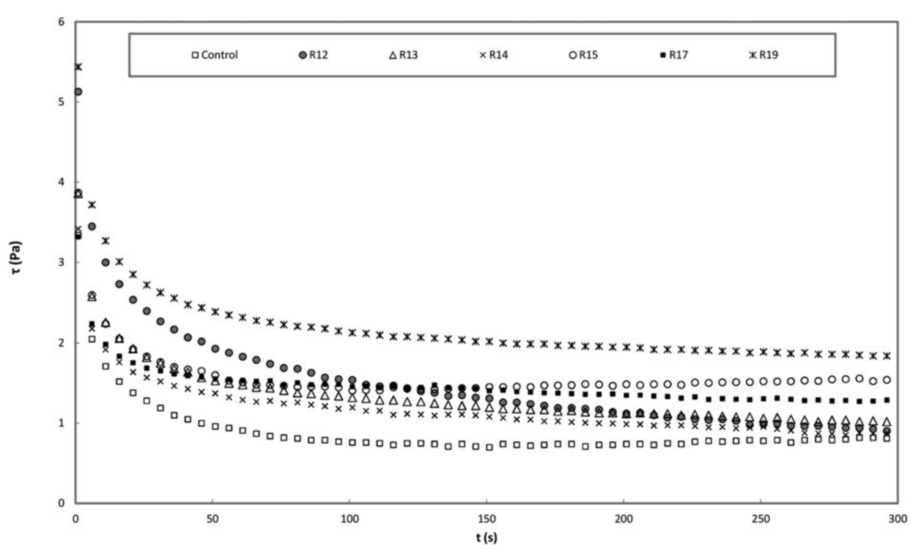

Figure 7. Shear stress (τ) variations versus time (t) in the stress relaxation experiment for the control and R12, R13, R14, R15, R17, and R19.

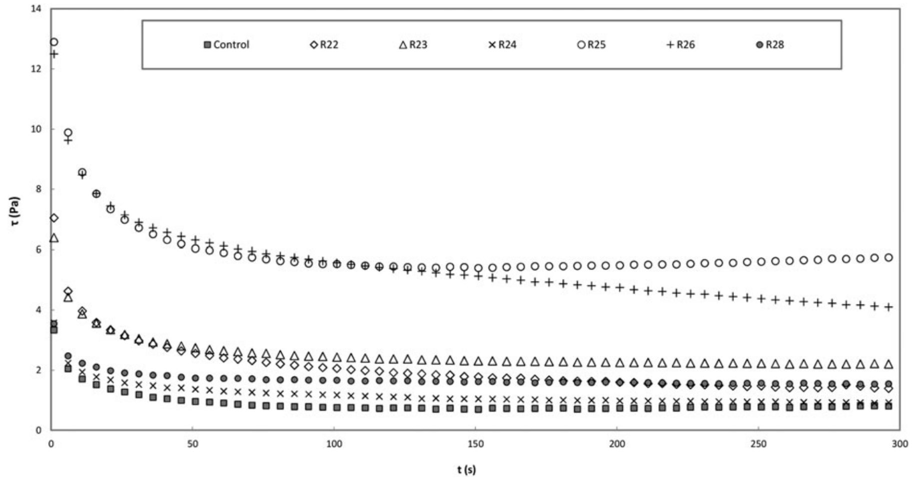

Figure 8. Shear stress (τ) variations versus time (t) in the stress relaxation experiment for the control and R22, R23, R24, R25, R26, and R28.

emulsion disperse-phase particle size, the amount and distribution of the emulsifying agent, and the aqueous phase viscosity.[23] Sample R18 had the smallest oil droplet diameter (<10 μm) and the lowest packing density, whereas in the case of R26, the packing of the droplets within the emulsion structure increased with increasing oil droplet diameter (ca. 30 μm). The increasing trend of particle diameter in the control sample continued, but the packing quality was depressed compared to that of R26. Mun et al.[23] and Nikzade et al.[24] found that low-fat mayonnaises with different formulations, with consideration of the hydrocolloid composition nature used and processing parameters applied, presented different types of microstructures. A comparison of the micrographs corresponding to R18 (3% starch) and R26 (10% starch) containing the highest contents of short-chain and long-chain inulin, respectively, showed that the disperse particle packing and average diameter increased with increasing inulin chain length, long-chain inulin concentration, and modified starch content. This confirmed the conclusions based on the rheological measurements. The disperse particle diameter enlargement in R26 compared to the control sample was ascribed to the formation of a stronger gel network within the aqueous media and to the more intense packing of the particles in the presence of long-chain inulin. In the three micrographs shown in Figure 9, that is, R18, the control sample, and R26, the polydispersity dominancy decreased, respectively. In R18, despite the smaller particle diameter, the highest polydispersity was observed. The monodispersity increased with increasing continuous-phase viscosity in the presence of inulin and starch. Similar results were reported for low-fat mayonnaise by Nikzade et al.[24]

According to the results discussed earlier, the emulsion structure got more packed with increasing inulin chain length, long-chain inulin concentration, and modified starch content. This behavior was more pronounced in R26. The packed structure of the oil droplets present in the gel network formed within the continuous phase led to the intensification of the elastic characteristics and resistivity against deformation.[12] Generally, the three-dimensional particle gel long-chain inulin network, especially in the presence of modified starch, plays a significant role in the reinforcement of the mayonnaise emulsion network structure.

Design Method Validation

Table II presents the values predicted by the combined mixture design method for the response values involved here. To validate the optimization results, optimum runs suggested by the DOE method were conducted. As shown, the characterization results were satisfactorily (with a maximum deviance of 14.7%) near the predicted values (see Table III).

CONCLUSIONS

The effects of different concentrations and composition ratios of the constituents of low-fat mayonnaise, including the three types of inulin (short-chain, medium-chain, and long-chain inulin) and modified starch, on the emulsion microstructural

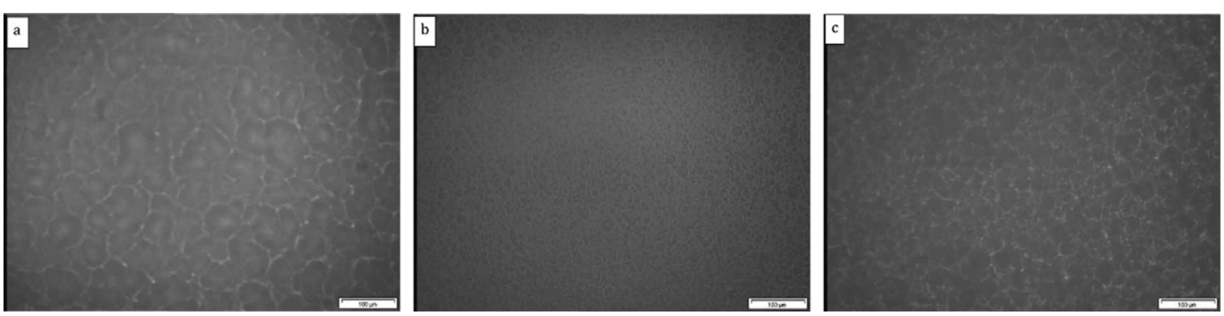

Figure 9. Optical microscope micrographs of the low-fat mayonnaise sauce samples: (a) control sample, (b) R18, and (c) R26.

Table II. Response Variables Involved in the Creep and Stress Relaxation Tests Predicted by the Combined Mixture Design Method

	Independent variables				Predicted response variables						
	Inulin				Creep					Stress relaxation	
Run	Short chain (%)	Native	Long chain (%)	Modified starch (%)	$\gamma_{elastic}$ (%)	γ_{max} (%)	G_0 (Pa)	G_1 (Pa)	η_0 (Pa s)	$\tau_{elastic}$ (Pa)	$t_{relaxation}$ (s)
1	5.71	0	4.29	2.2	0.36	0.54	6.94	4.62	1749	1.27	48
2	3.17	0.1	6.73	1.18	0.21	0.30	11.90	8.33	2217	1.92	73
3	3.46	0	6.54	1.22	0.19	0.27	13.15	9.25	2476	2.32	84
4	0	1.67	8.33	1.34	0.13	0.18	19.23	13.88	3172	3.37	112

Table III. Response Variables Involved in the Creep and Stress Relaxation Tests Measured for the Optimum Runs

	Independent variables				Measured response variables						
	Inulin				Creep					Stress relaxation	
Run	Short chain (%)	Native (%)	Long chain (%)	Modified starch (%)	$\gamma_{elastic}$ (%)	γ_{max} (%)	G_0 (Pa)	G_1 (Pa)	η_0 (Pa s)	$\tau_{elastic}$ (Pa)	$t_{relaxation}$ (s)
1	5.71	0	4.29	2.2	0.32	0.49	7.81	5.1	1962	1.44	54
2	3.17	0.1	6.73	1.18	0.23	0.31	10.86	8.06	2427	1.86	78
3	3.46	0	6.54	1.22	0.21	0.31	11.90	8.06	2207	2.1	76
4	0	1.67	8.33	1.34	0.12	0.16	20.83	15.62	3349	3.48	127

and rheological characteristics were investigated. The results show that increasing the inulin chain length, long-chain inulin concentration (≥5%), and modified starch content (≥1.5%) improved the structural deformation and elastic resistivity of the emulsion system. On the other hand, the increased packing of the emulsion disperse-phase particles, average particle diameter enlargement and monodispersity dominancy all indicated the major contribution of long-chain inulin in the crystallization phenomenon and the consequent strengthening of the three-dimensional gel network in the emulsion continuous phase, which increased the media phase viscosity of the oil droplets and improved the establishment of interactions between inulin and starch. According to the analysis based on the combined mixture design method, the response variables were significantly affected by the inulin chain length and concentration and also the modified starch content.

It seems that high concentrations of long-chain inulin (>5%) would be a proper substituent for prebiotic fat, stabilizing and strengthening the elastic characteristics of the emulsion. The presence of 10% long-chain inulin in the formulation of low-fat mayonnaise may present similar viscoelastic behavior compared to the same compound containing 3% modified starch instead of inulin. This confirms that the suggested concentration of long-chain inulin is comparable with modified starch.

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