

Article

Rheological Properties of Low Oil Mayonnaise by Replacing Oil Droplets with Agar Micro-Gels

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We constructed a low-calorie mayonnaise prototype in which part of the oil was replaced with agar micro-gels. The viscosity of a full oil mayonnaise (F-mayo) at the shear rate of 5 s^{-1} was $36.0\text{ Pa}\cdot\text{s}$, while the value of a half oil mayonnaise (H-mayo) sharply decreased to $1.75\text{ Pa}\cdot\text{s}$. However, the value of an oil reduced mayonnaise containing agar micro-gels (A-mayo) recovered to $38.2\text{ Pa}\cdot\text{s}$. Furthermore, we obtained structural information in mayonnaise by analyzing dynamic modulus with a weak-gel model. The characteristic parameter, the coordination number (z), was 13.9 for F-mayo. However, the value of H-mayo was 6.54, which was almost half that of F-mayo. The z value of A-mayo was significantly improved to 11.7. From these results, it was suggested that the low oil mayonnaise blended with the agar microgel showed the similar rheological properties of the normal mayonnaise, that is, it is expected that a texture of A-mayo is similar to the normal mayonnaise.

Key Words: Mayonnaise / Micro-gels / Weak-gel model / Agar / Low calorie

1. INTRODUCTION

Mayonnaise is a dressings that is commonly used on salads and in other foods to enhance their desirability. The market for dressings is growing and healthier versions of mayonnaise are being developed¹⁾. In particular, low-calorie mayonnaise is strongly needed as obesity caused by surplus calorie intake has become a serious social problem.

Mayonnaise is an O/W emulsion containing large amount of oil. The typical characteristic texture of mayonnaise is “spoonful-ness”, which is a paste like flow property. The high consistency of mayonnaise is due to its high oil content. When the inner phase volume fraction exceeds a critical value, the emulsion shows elasticity according to Eq. 1²⁾.

$$G \approx 1.77 \frac{\Gamma}{r} \phi^{1/3} (\phi - \phi_0) \quad (1)$$

where G is the modulus, Γ is the interfacial tension, r is the inner phase droplets radius, ϕ is the volume fraction of the inner phase, and ϕ_0 is the critical volume fraction when the modulus collapses to zero. The type of elasticity underlies the typical texture and mouthfeel of mayonnaise, namely its “spoonful-ness”. In other words, mayonnaise manufacturing requires the inner oil phase to exceed a critical condition

($\phi > 0.7$, at least). The majority of calories in mayonnaise come from its oil. Therefore, to reduce the amount of calories in mayonnaise, the oil content must also be reduced. However, as mentioned above, reducing the oil content of mayonnaise will transform it into a liquid-like emulsion, which is the greatest obstacle for the development of low calorie mayonnaise. As mayonnaise is O/W emulsion, the aqueous phase must be thickened for oil reduced mayonnaise. There are several methods that can be used to achieve this purpose. The most easy and popular way is adding a viscosity thickener to the aqueous phase. The ingredients of common oil reduced mayonnaise contain food additive such as polysaccharides. These water dispersed polysaccharides thicken the emulsion but their textures are far from that of standard mayonnaise³⁾. Since the dietary habits and preferences of consumers are very “conservative”, the development of a novel type of the oil-reduced mayonnaise with a more natural texture is necessary.

Agar micro-gels, which are prepared in W/O emulsion systems have been extensively studied^{4,5)}. Their use in low-calorie mayonnaise was inspired from the studies on water swellable micro-gels synthesized in W/O micro-emulsions⁶⁻⁸⁾. It has been confirmed that the micro-gels are suitable rheology control agents for cosmetics⁹⁾. On the other hand, studies on the polysaccharide based micro-gels using several procedures have been reported, namely sheared gelation¹⁰⁾, phase separation of mixed polysaccharides¹¹⁾, shearing bulk

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gels^{12,13}, and using W/O emulsion system¹⁴). In particular, the procedure using W/O emulsion is applicable to low-calorie mayonnaise development.

Previous studies have demonstrated the use of micro-gels as rheology modifiers¹⁵⁻¹⁹). In terms of food texture control, the size and shape of the micro-gel is important. The advantage of the micro-gels preparation used in the W/O emulsion system is that we have succeeded in preparing tiny particles, with a diameter of less than 10 μm ^{4,14}). The advantage of these micro-gels is the size and shape, which is completely spherical due to the interfacial tension between the water and oil phases. This controlled shape is advantageous for quality control, particularly in terms of rheological characteristics, for the processed foods.

It is quite difficult to quantitatively analyze the rheological properties of condensed colloidal foods such as mayonnaise. Condensed colloidal foods generally exhibit weak structure consisting of colloidal particles which can be easily destroyed under shear flow, and it is important to study this weak-structure to further understand the properties of condensed colloidal foods. To obtain information concerning the weak structure, we employed a weak-gel model²⁰) for analyzing their dynamic modulus under the small deformation.

For the development of a novel low-calorie mayonnaise, we prepared a model mayonnaise using an agar micro-gel oil suspension as its oil phase. To evaluate the rheological properties of the model mayonnaises, the steady state viscosities and dynamic moduli under linear viscoelastic ranges were measured, and the dynamic modulus was analyzed using the weak-gel model. We also investigated the rheological properties of the model mayonnaise containing agar micro-gels with various rigidities to determine the influence of the micro-gel on the flow behavior of the model mayonnaise containing micro-gels with various hardness.

2. EXPERIMENTAL

2.1 Materials

Agar (Wako Pure Chemical Corp, Osaka, Japan), xanthan (Sigma-Aldrich Co., US-MO) and Soy beans oil (The Nissin Oillio Group, Ltd., Tokyo, Japan) were used without further purification. Sucrose erucic fatty acid esters ER-190 (Mitsubishi Chemical Foods Corp., Tokyo, Japan) were used as emulsifiers. For preparing model mayonnaise, frozen egg yolk (Kewpie egg Corp., Tokyo, Japan) and grain vinegar (Mizkan Holding Co., Ltd., Aichi, Japan) were used. Distilled water was used as the aqueous solvent.

2.2 Preparation of agar micro-gels

The agar micro-gel oil suspensions (AMOS) were prepared according to a method reported previously⁴). The preparing procedure is briefly described herein. First, a heated aqueous solution of agar (150 mL; 1-3 wt% agar containing based on the aqueous phase) was added to the soybeans oil (150 mL), which dissolved erucic sucrose ester (ER-190: Mitsubishi Food Chemicals, Tokyo) as an emulsifier (3 wt% based on the oil phase) while stirring with a homo-mixer. The W/O emulsion was cooled to 10 °C to create agar micro-gel particles in the oil phase. AMOS density was measured using a density meter (DMA 600; Anton Paar) at 25 °C. The volume fraction of the micro-gels in the suspension was 0.5 according to the following equation.

$$\rho_{em} = \phi \rho_w + (1 - \phi) \rho_{oil} \quad (2)$$

where ϕ , ρ_{em} , ρ_w and ρ_{oil} are the volume fraction of the aqueous phase, the density of the emulsion, the density of the aqueous phase (agar gel) and the density of the soybeans oil, respectively. The sample code, AMOS1, AMOS2, and AMOS3 indicate that contains 1, 2 and 3 wt% agar in the micro-gels, respectively. AMOSs were used to prepare a model mayonnaise, A-mayo series (see Table I).

The agar microgels were successfully prepared by the

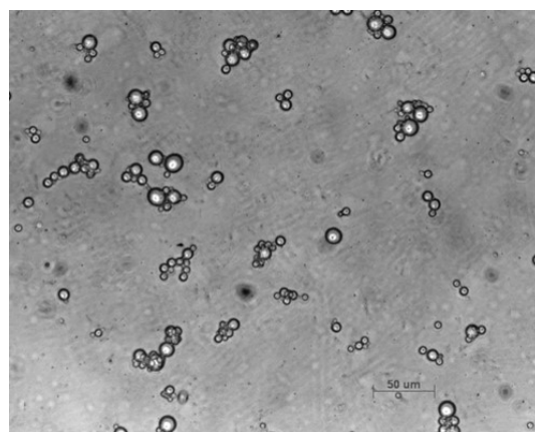


Fig. 1 Optical micrograph of agar microgel particles (AMOS2).

Table I Recipes of the model mayonnaise.

Ingredients	F-mayo	H-mayo	A-mayoi*	X-mayo
Soy beans oil	66	33	-	33
Egg yolk	15	15	15	15
vinegar	15	15	15	15
salt	4	4	4	4
D.W.	-	33	-	-
AMOSi*	-	-	66	-
Xanthan 2% sol	-	-	-	33

The values indicate wt %

* “i” indicates the agar content (wt%) in the micro-gels

method described above. An optical microscope image of a typical example (AMOS2) is shown in Fig. 1. As you can see, it can be seen that spherical microgel particles having a diameter of several micrometers were obtained.

2.3 Model Mayonnaise

The recipes for the model mayonnaises are listed in the Table I. The model mayonnaises were prepared by common procedure as illustrated in Fig. 2. Standard samples, F-mayo (full oil mayonnaise) and H-mayo (half oil mayonnaise) were prepared according to the procedure described in Fig. 2a, namely, vinegar, salt, and egg yolk were mixed and the oil phase was subsequently added under agitation using a cooking mixer. The A-mayo series was prepared according to the procedure described in Fig. 2b, namely the AMOSs were used as the oil phase. As described in Section 2-2, since the oil volume fraction of AMOSs was 0.5, the total oil content of the A-mayo series were reduced by half compared to that of F-mayo. On the other hand, X-mayo, the model mayonnaise where a part of the oil phase was substituted for xanthan 2 wt% aq. sol, was prepared according to the procedure described in Fig. 2c. This model mayonnaise also has its oil content reduced to half that of F-mayo.

Although we did not systematically examine the stability of these model mayonnaises, no macroscopic destabilization was observed for the prepared model mayonnaise for at least a few weeks after preparation. The rheological measurements were performed within one week after the model mayonnaise preparation.

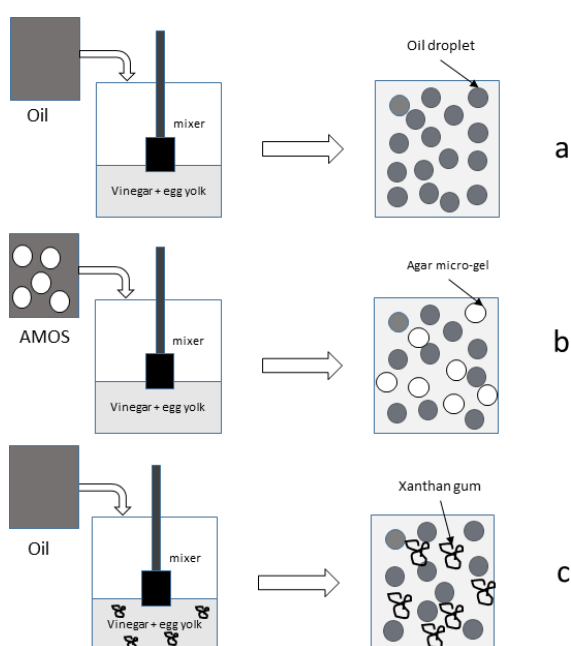


Fig. 2 Schematic of the preparation of the model mayonnaise. a: standard mayonnaise, b: using the agar micro-gel oil suspension (AMOS), c: adding xanthan to the aqueous phase.

2.4 Rheology measurements

A strain-controlled rheometer (ARES; TA instruments) was used for rheological measurement. A parallel-plate fixture ($d = 25$ mm, $gap = 1.5$ mm) was used, and the steady state viscosities were measured at shear rates of 1 to 100 s^{-1} . The frequency dependence of the dynamic modulus was measured at angular frequencies ranging from 0.5 to 50 rad/s. The applied strain was within the linear viscoelastic region ($\gamma = 0.01$). All measurements were performed at 25°C .

2.5 Data analysis using the weak-gel model

The dynamic moduli for the model mayonnaises were analyzed using the weak-gel model²⁰⁾ through the following equation.

$$G^*(\omega) = A_f \omega^{1/z} \quad (3)$$

where A_f and z are the gel strength and coordination number, respectively. The dynamic modulus results were fitted using Eq. 3 to obtain two characteristic parameters. The weak-gel model is based on the cooperative flow theory developed by Bohlin²¹⁾ and Winter' gel theory²²⁾. In particular, z is an important parameter for considering the structure in the system. We assumed an ideal "flow unit" in the system, and the real structure of the material was substituted by a cooperative arrangement of the "flow unit" forming 3D "weak" networks. The physical meaning of z refers to the number of "flow units" interacting each other that can be used to determine the deformation response of the system. We assumed that oil droplets in the mayonnaise can be considered as a "flow unit". The A_f value indicates the mechanical strength of the structure consisting of "flow units". Therefore, these two parameters can be used to estimate the mechanical properties of complex soft-matter.

3. RESULTS AND DISCUSSION

3.1 Steady state viscosity

The steady state viscosity results for the model mayonnaises are shown in Fig. 3 (a and b). The apparent viscosity of H-mayo greatly decreased compared to that of F-mayo over the entire shear rate ranges. The apparent viscosities at 5 s^{-1} (η_5) for F-mayo and H-mayo were 36.0 and 1.75 Pa·s, respectively, whereas the A-mayo2 and X-mayo samples showed η_5 values of 38.2 and 30.0 Pa·s, respectively. The low-calorie model mayonnaise's η_5 values were similar to the value of F-mayo. However, the apparent viscosity at 50 s^{-1} (η_{50}) for the low-calorie model mayonnaise, A-mayo2 and X-mayo

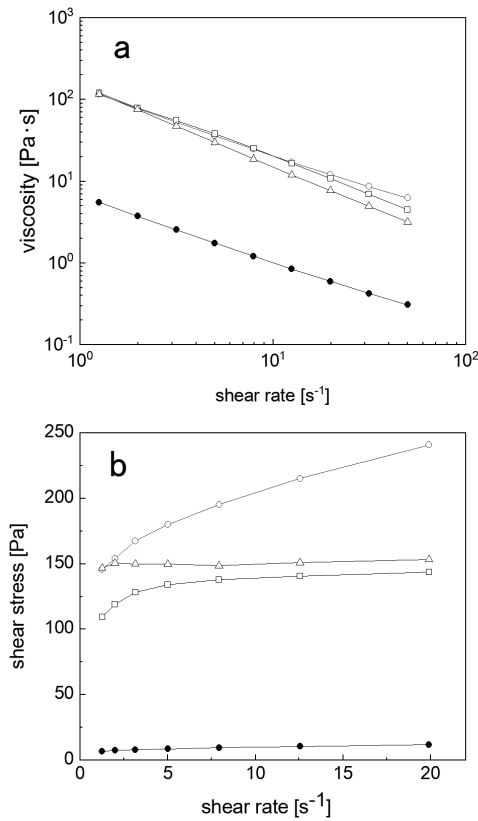


Fig. 3 The shear rate dependence on the apparent viscosity (a) and flow curves (b). The open circles, closed circles, triangles, and squares denote the results of the F-mayo, H-mayo, A-mayo2, and X-mayo samples, respectively.

were lower than that of F-mayo.

Figure 3b shows the relationship between the shear stress and shear rate (flow curves). Although the viscosity behavior of the F-mayo, A-mayo2, and X-mayo were similar each other at low shear rates, their flow curves differed significantly. The flow curve of the F-mayo showed a typical pseudo-plastic flow with an apparent yield stress of approximately 150 Pa. A-mayo2 and X-mayo showed similar behavior in low shear rate range, but the flow curves for A-mayo2 and X-mayo look were relatively flat above 5 s⁻¹. This indicates that the shear stress never developed at moderate and high shear rates. It is possible that shear banding occurred the model mayonnaise, but the details of the phenomenon remain unclear.

The most important texture of mayonnaise is its “mouth-feel” or “spoonful-ness”. These characteristics are strongly related to the rheological properties under small deformation or slow flow conditions. Both A-mayo2 and X-mayo are good candidates for low-calorie mayonnaise as they exhibit similar flow properties at low shear rates. To confirm this expectation, further oscillation studies were performed.

3.2 Dynamic modulus

To understand the rheological properties of the model

mayonnaises under small deformation condition, the dynamic modulus of the samples were measured, and the results analyzed using the weak-gel model.

The frequency dependence of the dynamic moduli for F-mayo and H-mayo are shown in Fig. 4a.

The dynamic modulus of H-mayo was greatly decreased compared to that of F-mayo. However, the storage modulus exceeded the loss modulus at all frequencies. This indicated that H-mayo contained an inner structure which propagated stress, although its strength was weak. The storage and loss moduli, are obeying the scaling law, that we cannot determine a characteristic parameter integrally. In Fig. 4b, the mechanical properties of A-mayo2 and X-mayo are shown. They obeyed the scaling law in a similar manner to those of F-mayo and H-mayo. To compare the mechanical properties of the samples, weak-gel model analysis was applied. If the complex modulus and frequency dependency obey the scaling law, we can obtain characteristic parameters using Eq. 3.

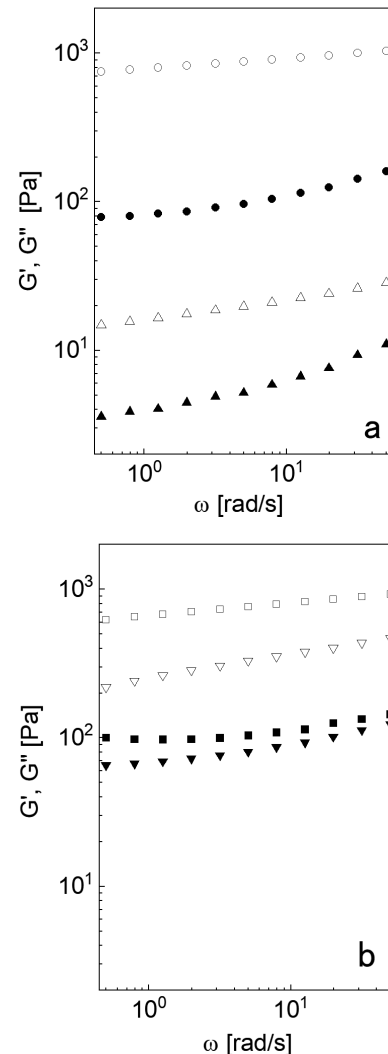


Fig. 4 The frequency dependence on the dynamic moduli of the F-mayo (circles) and H-mayo (triangles) samples (a); and A-mayo2 (squares) and X-mayo (inverted triangles) samples (b). The open and closed symbols denote the storage and loss moduli, respectively.

The complex modulus and frequency dependency for the model mayonnaises are shown in Fig. 5. The plots and lines indicate the experimental results and calculated values using Eq. 3, respectively. It is clear that the fitting using Eq. 3 accurately described the experimental results of the samples.

The weak-gel model's characteristic parameters were obtained after analysis, namely the coordination number (z) and gel strength (A_f). As mentioned above, the coordination number is important parameter for describing colloidal food systems. According to the theoretical background of the weak-gel model, the coordination number reflects the spatial information of the inner structure, which propagates the applied stress. Bohlin considered an ideal "flow unit" to calculate the total energy of the system²¹). In the model mayonnaise, the emulsification particles (oil droplets in mayonnaise) were considered to be the "flow unit". Therefore, the z value reflects the inner oil phase fraction. Another parameter, the gel strength (A_f), indicates the "consistency" of the sample, which is related to the mechanical strength of the micro structure in the model mayonnaises.

To easily compare the weak-gel model parameters of the samples, histograms are shown in Fig. 6 (a and, b). From Fig. 6 a, the z results for F-mayo and H-mayo are 13.9 and 6.54, respectively, indicating the number of "flow units" next to a "certain flow unit" with mechanical interaction. If the "flow unit" is a uniform hard core particle, the theoretical value of z should be 16 in 3D system. Therefore, the result for F-mayo (13.9) indicates that the oil droplets were almost close packed with mechanical interactions. On the other hand, the value for H-mayo was almost half that of F-mayo

and is exactly proportional with the inner oil fraction ratio. These results indicate that this theoretical model is ideal for analyzing mayonnaise rheology.

The results of the low calorie model mayonnaises, A-mayo2 and X-mayo, are also quite interesting. The z value for A-mayo2 was 11.7 even though its oil phase fraction was equal to H-mayo. This result indicates that the micro-gel mimic the oil droplets (the right illustration in Fig. 2b). The agar micro-gel particles adopt weak structure with oil droplets, which can propagate the stress in the model mayonnaise. On the other hand, the z value for X-mayo was 6.56, which is almost equal to that of H-mayo. This indicates that xanthan molecules, which are dissolved in aqueous phase, never acted as a "flow unit" to propagate the stress.

The A_f values for the model mayonnaises corresponded to the z values. When the oil fraction was reduced by half, the A_f value drastically reduced, from 784 (F-mayo) to 15.8 (H-mayo). This result corresponds with the z value, namely, the reduction of the oil droplets with coincident with the reduction of stress propagation capability. The A_f value for A-mayo2 was quite improved compared to that of H-mayo even though both samples contained the same oil ratio. This supports our above speculation. On the other hand, the A_f value of X-mayo was lower than that of A-mayo2, indicating that the xanthan molecules did not work well as a "flow unit" for stress propagation in the system.

Recently, the weak-gel model has been used for studies

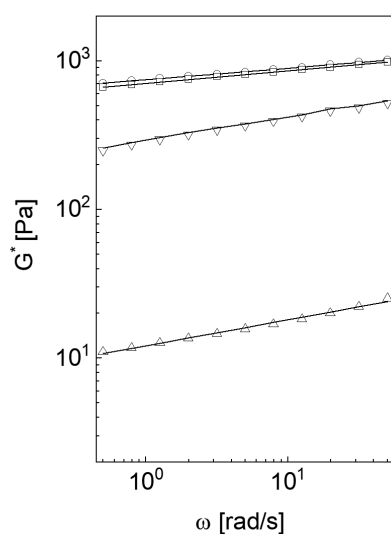


Fig. 5 The frequency dependence on the complex modulus of the model mayonnaises. The circles, triangles, squares, and inverse triangles denote the measured values for the F-mayo, H-mayo, A-mayo2, and X-mayo samples, respectively. The lines denote the best fit results of the analysis using Eqs. 3 for each sample.

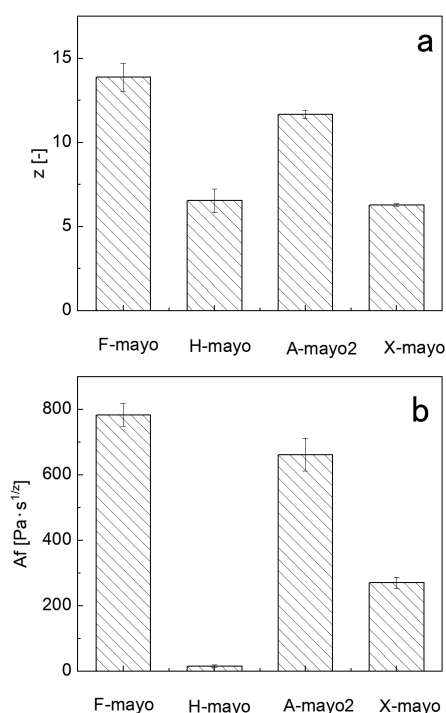


Fig. 6 The weak-gel model parameters for the model mayonnaises. a: coordination number (z) and b: gel strength (A_f). The values are the average and standard deviations ($n = 5$).

on complex soft-matter, such as cake butters²³⁾, cheese²⁴⁾, apple tissue²⁵⁾, tofu gels²⁶⁾, and bitumen²⁷⁾. The flow behaviors of the agar micro-gel oil suspensions were also investigated using this model⁵⁾, and it was demonstrated that the model worked well. In this study, the rheological properties of the model mayonnaises can be successfully analyzed using the theory. The most important advantage of the weak-gel model is that it gives a physical meaning to the scaling index, which is related the number density and spatial distribution of “flow unit”. Namely, we can obtain structural information of food from rheological data.

3.3 The effect of the micro-gel particles hardness

The low oil model mayonnaise containing agar micro-gels particles provided a good imitation of standard mayonnaise with respect to its rheological properties. We previously demonstrated that the weak-gel model parameters vary depending on the hardness of the micro-gel particles⁵⁾. Therefore, it is expected that changing the hardness of the agar micro-gel particles can lead to change the mayonnaise texture.

We compared the rheological properties of the model mayonnaises containing the agar micro-gels in various agar concentrations, namely A-mayo1, A-mayo2, and A-mayo3 (Table I). The complex modulus dependence on the frequency for the A-mayos are shown in Fig. 7. It is clear that the modulus increased with agar concentration in the agar micro-gels, and plots obeyed the scaling law. Therefore, the weak-gel model analyses were performed. The weak-gel model parameters which are plotted in Fig. 8. It is clear that the z values are independent of the agar concentration in the micro-gel particles, but the A_f value were dependent on the

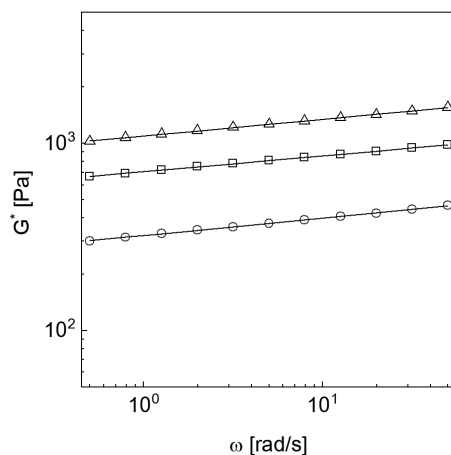


Fig. 7 The frequency dependence on the complex modulus of the model mayonnaises containing agar micro-gels with various rigidities. The circles, squares, and triangles denote the measured values for A-mayo1, A-mayo2, and A-mayo3 samples, respectively. The lines denote the best fit results of the analysis using Eq. 3 for each sample.

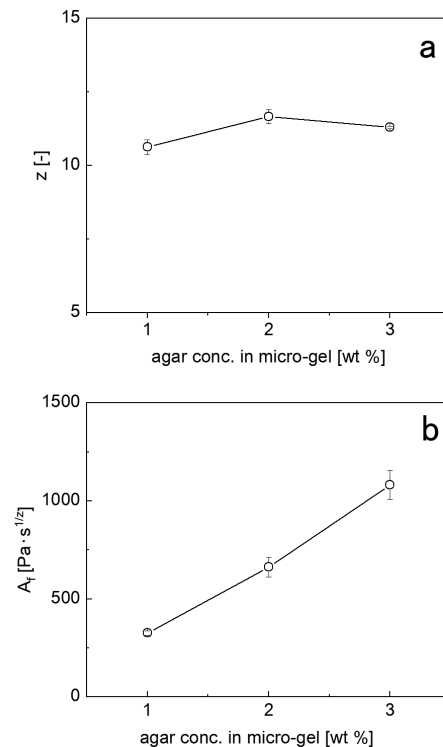


Fig. 8 The agar concentration dependence of the weak-gel model parameters. a: coordination number (z) and b: gel strength (A_f). The values are average and standard deviations ($n = 5$).

agar concentrations. Although we did not measure the hardness of these micro-gel particles directly, it is clear that the rigidity of the micro-gel particles depend on agar concentration. This result is quite important with respect to texture modification for low-calorie mayonnaise.

3.4 Advantages of applying agar micro-gels for preparation of low oil mayonnaise

As described above, mayonnaise is a high internal aqueous phase O/W emulsion, in which emulsified particles are closely packed. Therefore, the friction between the emulsified particles in the system is an important parameter that must be considered. In particular, “spoonful-ness”, which is an important textural feature of mayonnaise reflects the elasticity of the food, which originates crowding the interfaces between the aqueous and oil phases. Therefore, it is extremely important to understand the structure of the interface within the system. It is thought that phospholipids (lecithin) in egg yolk contribute to the emulsion stability of mayonnaise, however, yolk granules are also considered to play an important role. Recently, it has been revealed that the structure of the egg yolk granules are spherical with a diameter of 1 to 10 nm by X-ray scattering²⁸⁾. It is considered that the emulsified oil droplets of mayonnaise may be also stabilized by the yolk granules, *i.e.*, as like Pickering emulsion. We tried to label the agar microgel with fluorescence and observe it with

a fluorescence microscope to visualize the dispersion state of oil droplets and microgel particles of the model mayonnaise system, but we could not achieve the fluorescent label of agar molecules (it is generally difficult to chemically modify a neutral polysaccharides). Therefore, microstructure analysis of the interface of the mayonnaise is considered to be a major issue in the future. One possible way to study such micro-structure of the interfaces is a scattering method using quantum beams. In particular, small angle X-ray scattering (SAXS) method is considered to be effective as one of the methods for analyzing such an interface nano-structure. In fact, we have already started a study on the model mayonnaise using SAXS method as described below.

As well known, xanthan is a polyelectrolyte having a glucuronic acid in its molecule and swells largely in water to exert a thickening effect. Although it is expected to exhibit a thickening effect with weak alkalinity due to having a glucuronic acid, in fact, the thickening effect is exhibited even under acidic conditions, and also has a thickening effect even under high salt concentration conditions²⁹⁾. It is considered that the hydrated xanthan exerts the thickening effect by the excluded volume effect at the concentration above the overlapping concentration. Agar micro-gels are also believed to fill the space of the low oil mayonnaise continuous phase and improve consistency through its excluded volume effect. However, strangely, although it is expected that the low HLB (hydrophilic-hydrophobic balance) emulsifiers (sucrose erucic fatty acid ester) were absorbed to the surfaces of the agar micro-gels, the micro-gels were dispersed in the aqueous phase in the model mayonnaise. It is expected that egg yolk granules would also be absorbed to the surfaces of the micro-gels as mentioned above.

We succeeded in quantitatively comparing and evaluating the combination effect of agar micro-gels and xanthan using the weak-gel model, and it became clear that there is a big advantage in agar micro-gels as a result. In particular, the coordination number (z) was found to be extremely useful as a parameter quantitatively indicating the spatial arrangement of emulsified particles in mayonnaise. The “spoonful-ness” is related to the feeling under the low shear rate in the mouth. Therefore, the weak-gel model parameters are useful for considering the relation between the texture and rheological properties of the food. The shape of the agar micro-gel particles is another important aspect. As is well known, a critical particle size exists for feeling a sense of incongruity, which is approximately 10 μm . The size of the agar micro-gel particles, prepared using the W/O emulsion system, was few μm , sufficiently small to obtain the proper mouth feeling.

Moreover, the micro-gel particles exhibited perfectly spherical shapes due to the interfacial tension in the emulsion system. This is important in term of turning the rheological properties, particularly for quality control during food manufacturing. From these findings, the agar micro-gels were considered to be a promising candidate as a textural control agent low-calorie mayonnaise.

Further research is required as there are some remaining issues. First of all, in order to industrial produce low-calorie mayonnaise using agar micro-gels, systematic stability evaluation is necessary. Another unsolved problem is the differences in the flow curves shown in Fig. 3. The differences was likely caused by the nano-structure of the interfaces as mentioned above. To resolve these issues, we will perform a structural study employing SAXS and SANS. Further studies regarding the nano-structure of the interfaces of the agar micro-gel particles will soon be reported.

4. CONCLUSIONS

We successfully prepared a prototype of novel low oil mayonnaise where some of the oil was replaced with agar micro-gels prepared in W/O emulsion system. Both xanthan gum and agar micro-gel suppressed the decrease in apparent viscosity of model mayonnaise in which the oil content was halved. However, analysis of the dynamic modulus behavior of both model mayonnaises with the weak-gel model has proved that model mayonnaise formulated with agar micro-gels is more similar to the rheological properties of standard mayonnaise. These results show that agar micro-gels are promising candidates as texture modifiers for condensed colloidal foods such as mayonnaise.

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