## Rheology of emulsions, foams and gels

Rajendra P Borwankar\* and Suzanne E Case<sup>†</sup>

Recent advances in the area of food rheology include an increased interest in, and the successful application of, the more theoretical approaches to colloids and polymer rheology, first developed for and proven in colloid science and synthetic polymer science, to food systems. There have also been recent developments of novel equipment to study physical behavior of semi-solid foods, including surface forces apparatus and fiber spinning techniques.

#### Addresses

Kraft Foods, 801 Waukegan Road, Glenview, IL 60025, USA \*e-mail: rborwankar@kraft.com \*e-mail: scase@kraft.com

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#### Abbreviations

DSC	differential scanning calorimetry
КМ	konjac glucomannan
o/w	oil in water
WPI	whey protein isolate

#### Introduction

As half of this issue of Current Opinion in Colloid and Interface Science is devoted to foods, it is appropriate to begin this article with a brief description of the job of a food rheologist, especially in industry. As I described [1], the food rheologist in an industrial setting must interact with many other scientists and technologists. First, rheology is an important tool in the understanding of food interactions and food structure. Food rheologists can guide a product or process developer to manipulate and manage ingredient interactions to ones advantage. Second, because the texture of food is one of its key consumer attributes, understanding the inter-relationships between rheology and food texture are very important to the food developer. Finally, rheological properties are important engineering parameters in the design of manufacturing processes. As such, rheology is an important tool for a food technologist. Foods are structurally very complex. It is often possible to have dispersions, emulsions, foams and gels, all in a single food. As such, their rheological measurements are often not straightforward.

Recently, there have been significant strides made in the refinement of rheological measurement methods for foods, as well as in the development of models of their rheology. This article reviews the most recent papers in this area.

#### **Rheology of emulsions**

Traditionally, the rheology of emulsions has been studied by first investigating the rheology of solid dispersions (suspensions) and then examining how emulsions depart from their solid counterparts. One reason for this approach is that, compared to emulsions, dispersions are easier to prepare reproducibly and study in detail, and are also somewhat easier to model. We will use the same approach here. For superb reviews of the area of suspension rheology, the reader is referred to  $[2-4,5^{\bullet\bullet}]$ . A general functional form of the rheology of dispersions can be expressed as:

$$Rhp = f(Rhp^0, \phi, psd, I, A) \tag{1}$$

where Rhp is the rheological property of the dispersion, Rhp<sup>0</sup> is the rheological property of the dispersion medium,  $\phi$  is the volume fraction of the dispersed phase, psd refers to the particle size distribution, I captures all interparticle interactions and A denotes the state of aggregation. A will be a function of I. In the simplest case, the rheological property in question can be the viscosity. Then the equation can be simplified to:

$$\eta/\eta_0 = f(\phi, psd, I, A). \tag{2}$$

The dependence of the viscosity on phase volume fraction has been studied in the most detail and many functional forms are proposed [2-4,5..]. The viscosity increases with  $\phi$ , initially linearly, and then exponentially at large values of  $\phi$  and asymptotically approaches infinity at the close packing limit of the dispersion. The dependence on psd arises as a result of the dependence of interactions I or aggregation A on psd. This is true for monodispersed, or at least unimodal, dispersions. The phase volume fraction,  $\phi$ , here is the effective phase volume fraction and is affected by swelling of the dispersed phase by absorption of the solvent, adsorbed layer effects, nonspherical particles, among other things [3,5\*\*]. In fact, one way of looking at the effect of aggregation, A, is by how it affects the effective volume fraction due to entrapment of the dispersion medium in the aggregates.

Although polydispersity itself affects the rheology in only a minor way [3], using multimodal dispersions in specific ways can significantly reduce viscosities. Greenwood *et al.* [6<sup>••</sup>] recently published a key paper identifying diameter ratios when viscosities of a bimodal dispersion are minimized. These correspond to when the small particles are effectively able to pass through the voids between the large particles, effectively increasing the close packing limit. Verduin *et al.* [7<sup>••</sup>] used a gel-forming suspension and studied the shear thinning behavior above and below the gel temperature. They found a sharp decrease in the power law index as one passes from above the gel temperature to below it. Small angle light scattering was used to monitor the structural changes, which revealed that, both above and below the gel temperature, a high degree of anisotropy was induced by shear which can relax above the gel-forming temperature but not below it.

Emulsions behave not too dissimilarly from dispersions with regard to viscosity dependence on the above parameters. Typically, the viscosity of an emulsion would be somewhat lower than the viscosity of the suspension with the all parameters in the above equation being equal. Thus, whereas the Einstein equation predicts a slope of 2.5 for ideal suspensions at very low  $\phi$ , the Taylor equation for emulsions predicts that the slope would depend on the ratio of viscosities of the two phases and can be as low as one (when dispersed phase viscosity is negligible compared to the continuous phase viscosity). This is due to the interfacial mobility or fluidity. Particularly at small mean droplet size and with strong adsorption layers of emulsifiers, however, the effects of droplet distortion and interfacial mobility are small and the above equation for dispersions would apply quite well to emulsions.

Emulsion rheology often does not get as much attention from researchers as suspension rheology has traditionally received. For a review (albeit a dated one) of emulsion rheology literature see Sherman [8]. Dickinson and Golding [9\*\*] recently published a paper for which they carried out a detailed study of sodium-caseinate-stabilized emulsions at varying levels of the caseinate. They delineated the regions of bridging flocculation at low caseinate concentration when the emulsifier was not in sufficient quantity to saturate the interface; a Newtonian behavior was present at intermediate concentrations where the emulsifier provided a full surface coverage but very little excess emulsifier. Finally, a depletion flocculation region appeared at high caseinate concentration, that is, when it was present in a large excess beyond what was needed to saturate the interface. This paper describes the rheological differences arising out of the two different modes of flocculation commonly encountered in food emulsions.

In another paper, Dickinson and Yamamoto [10] have used rheological measurements to investigate the effect of added lecithin on heat set whey-protein-stabilized emulsion gels. The order of addition of the ingredients (whey protein and lecithin) was shown to be crucial and the effects were explained through the interactions of lecithin and whey proteins at the interface.

One particularly striking way that the emulsions do differ from dispersions is in not having a well defined close packing limit. Emulsions can pack beyond the close packing limit of solid dispersions because the droplets can and do distort. Thus, for example, mayonnaise is an oil in water (o/w) emulsion with a dispersed phase volume fraction well in excess of 0.8, and the droplets are distorted from being a spherical shape in the product [11•]. A concentrated emulsion, is one in which droplets are distorted because the volume fraction is above the closed packing limit. As this resembles a foam more than it does an ordinary emulsion, we will treat it in the next section. Another important way that the emulsions can differ from the solid suspensions is in the fact that the droplets can coalesce to form larger droplets. This does not pose any problem because coalescence basically results in a change in the psd.

# Rheology of foams and concentrated emulsions

Typically, foams are dispersions of gas in a liquid where the volume fraction of the gas is very high. As such, similar to the concentrated emulsions, the foam bubbles are highly deformed. In fact phase volume fractions of 0.95 or even 0.99 are not uncommon in foams. Concentrated emulsions do not go nearly as high because the emulsion usually inverts before that. Nevertheless, concentrated emulsions with distorted droplets are not uncommon. When flow is induced in such a system, the droplets or bubbles must deform further and slide past one another. During the course of the deformation the droplets store energy and, hence, such systems exhibit elasticity. Such systems also possess a yield stress which must be exceeded before flow can occur. The scale of the shear modulus as well as the yield stress in this situation is set by the Laplace pressure of the droplets  $(2\gamma/R)$  where  $\gamma$  is the interfacial tension and R is the radius of the bubble or the drop.

Microrheological models of foams have been receiving increased attention recently, especially with the availability of tremendous computer power. The pioneering work in the area was begun by Princen [12-14] who was interested in both foams and concentrated emulsions. Princen [12,13] and Princen and Kiss [14] used two dimensional models combined with experimental study to extend their work to three dimensional foams, and determined expressions for the yield stress and static shear modulus for foams and concentrated emulsions. Subsequently, many attempts have been made to extend these models to real three dimensional foams with their three dimensional structure. Very recently, Kraynik and Reinelt [15•] published a paper in which they computed bulk modulus and shear moduli for Kelvin, Williams and Weaire-Phelan foams using the Surface Evolver software. This software is fast becoming key in such computations. This software was also used by Lacasse et al. [16•], who studied deformation of small compressed droplets.

Although such works are theoretically challenging and intellectually stimulating, in our opinion they have limited practical value, especially for food foams. This is especially so because many food foams are not dry enough for these models to apply. For example, ice creams contain only about 50% air (or less) and whipped toppings contain about 75% air. Also, in the food system, polydispersity plays a very important role. None of these models have been aplied to a food system, with the exception of Chakrabarti et al. (S Chakrabanti, J Guth, A Thomas, Society of Rheology Annual Meeting, 1991, unpublished data) who presented an application of the functional form of the Princen and Kiss model to mayonnaise, but fitted a new set of constants. Recently, Coughlin et al. [17•] published an experimental procedure called the punch indentation test to measure the static shear modulus of foams. They used shaving foams in their measurement and their results are in good general agreement with the above theories.

### Rheology of gels

Physical testing methods have been used to characterize gels for decades. The original methods were empirical, including, texture profile analysis [18], the cone penetrometer [19] and the Bloom Gelometer [20], to name only a few. The values obtained from such empirical tests are useful only in reference to the specific method. As equipment has become more affordable with user friendly software, the empirical type testing has been replaced almost entirely by rheological methods as a means to study gel properties, resulting in a more fundamental understanding of the mechanisms of gelation.

The area of gel rheology incorporates both small strain type tests, as well as large strain fracture tests. The former have been used primarily to follow sol to gel transformation and structural properties of the final gel, whereas the latter describes the breakdown of the gel structure. The results of these two types of measures are not necessarily related. Mitchell [21] has noted that gel strength identified by a single point rupture test will not always rank a series of gels in the same order as tests which involve small deformations without rupture. This paper includes recent examples of research of both small and large strain tests.

With new food formulation requirements, due to the increased interests in fat-like properties in fat-free systems, the need to maintain specific functionality in light of continual advances in processing and simply the desire for new unique food properties, probing and understanding of mixed gel systems have been quite popular in the literature over the past several decades. Some of the more recent work includes the paper by Nishinari et al. [22•]. This work identified synergistic interactions between purified gellan and purified k-carrageenan gums as a result of loose interchain interaction or a phase separation in the double helical structure. This was identified by following the impact of mixture ratios and temperature on rheological properties. These were compared with predicted values based on additivity of separated viscosity of both polymer solutions. Also from Nishinari's laboratory are two recent papers studying the interaction of konjac glucomannan (KM) with corn starch [23•] and with gellan gum with and without salts [24]. The former paper used a combination of differential scanning calorimetry (DSC) with both small strain and large strain tests to determine the effect of KM on the gelatinization and retrogradation of corn starch. The authors anticipated that the addition of KM would stabilize retrogradation of the starch over time. They actually found an increase in modulus and fracture strength with the addition of KM over short times. Retrogradation, however, was retarded slightly over longer storage times (>5 days). In the latter paper, gellan gum and a medium molecular weight fraction of KM in the presence of sodium chloride showed synergy (the suggested mechanism was interaction of KM molecules on the surface of large aggregates of gellan helices) resulting in increased G' and G" with increased salt concentration.

The list of recently published rheological studies of mixed gel systems is long, including systems of malto-dextrin and caseinate [25<sup>•</sup>], casein-micelle-carrageenan mixtures [26<sup>•</sup>] and high pressure treated gelatin-whey-protein gels [27<sup>•</sup>]. Finally, a paper by Zasypkin *et al.* [28<sup>••</sup>] begins with a thorough overview of the different classifications of multicomponent gels. This is followed by a review of work using gelatin mixed with a variety of other components to demonstrate the various types of mixed gel behavior. Although the rheology is not described in great detail in this review article, it gives a feel for the impact rheology has on understanding the mixed gel systems.

Although this work is necessary and useful, many of the mixed gel papers written are rather uninteresting in that it is the 'same method different gum' syndrome. There have been, however, a few particularly interesting studies describing gelation behavior of single component gel systems using newer perspectives in identifying sol-gel transitions. Rather than identifying the gel point as the G' and G" crossover, as has been used traditionally, these researchers have adopted a more definitive analysis from the polymer science area developed by Winter and Chambon [29]. This procedure suggests that at the gel point G' and G" exhibit a power law dependence on the oscillation frequency. At the gel point, G' and G" have the same power law exponent. The power law exponent of the critical gel can lead to an understanding of the physical mechanism of the gelation process. The paper by Peyrelasse et al. [30..] is a nice treatment of the sol-gel transition of gelatin (a very well defined gelling system). Rigorous determinations of G' and G" conducted at the gel point found the power law exponent, n, to be equal to 0.62 at concentrations between 0.17 and 0.40 g/cm<sup>3</sup>. They also noted that in plotting  $\eta''$  versus  $\omega$ , in the vicinity of the gel point, the Martin-Adolf-Wilcoxon relaxation function of G(t) provides a better fit with experimental results than other models. They stated that above the gelation temperature, the molecules of gelatin in solution do not appear to be entangled as they lack a rubbery region on the time-temperature superposition master curve. In a similar paper, Labropoulos and Hsu [31.] determined that whey protein isolate (WPI) gels had a characteristic power law dependence of storage and loss moduli on frequency at the gel point. Different

treatments of the WPI dispersion resulted in the same value of the power law exponents (n=0.62-0.69) even though each WPI solution had a different gel time, ranging from 12 min to 164 min. Although there has always been a core group of food and biopolymer scientists whose approach has been more from this perspective, interest has been increasing and the gap which has existed between the more theoretical approach of the synthetic polymer scientists and the work being done on biopolymer rheology is ever shrinking. The greatest hurdle to overcome in this area is in working with the polydispersity and complexity of the chemical and structural aspects of the biological dispersions.

A noted limitation in the traditional uniaxial compression test method has been the effect of the changing strain rate as the test progressed. Rohm *et al.* [32] compared the standard method of testing under a constant speed to a relatively new method of decreasing the crosshead speed in order to maintain constant strain rate conditions. These researchers, studying the impact of methodology on elastic food gels, identified significantly lower values for apparent fracture strain under constant strain rate testing for gels with failure Hencky deformation >0.5-0.6. A decrease in fracture stress was also noted under constant strain rate. The impact of these findings on sensory correlations was discussed.

In a series of three papers, Keetals *et al.* [33-35] reported on the gelation and retrogradation of potato and wheat starch. This work used the standard small strain oscillatory testing to follow gelation. Large strain uniaxial compression tests, applying a constant crosshead speed, were utilized to determine fracture properties. Combining small and large strain testing, a hypothesis of the morphology of these gelled systems was developed.

# Advances in rheological measurement techniques

Chakrabarti [36] has recently reviewed some of the new developments in the area of food rheology measurement. Particularly interesting in the context of emulsions and foams are the extensional viscosity measurements and measurements of wall slip. A novel rheometer, based on the concept of filament stretching, has been developed by Plucinski *et al.* (J Pluckinski, RK Gupta, S Chakarbarti, personal communication) and was applied to measure extensional properties of mayonnaise. The extensional viscosity of mayonnaise was found to be higher than its shear viscosity.

Slip is an ever present complication in dispersed systems and occurs through an apparent slip mechanism because of the formation of a thin stratum of a lower viscosity liquid near the wall, which can be considered to be the suspending liquid free of the dispersed phase. Indeed, at wall stresses below the yield stress of the dispersion, flow can occur because of the formation of the slip layer. Heller and Kuntamukkula [37] have reviewed the foam rheology literature and concluded that in much, if not most, of this literature, the results are influenced by the thickness of the film of liquid formed along the walls, and do not truly represent the rheological character of foams. Ignoring these slip layer effects leads to geometric dependence of rheological properties, and the authors warn that whereas apparent viscosities can give order of magnitude estimates, their dependence on uncontrolled variables can lead to inconsistent results in practice. Food rheologists must become aware of the slip phenomena in dispersed systems and make sure that their procedures eliminate or factor out the effects of slip.

In a yet novel and evolving area of research, Israelachvili and co-workers [38••] are investigating rheology of emulsions and dispersions in thin layers using their surface forces apparatus and relating it to the structure and wetting characteristics of the products.

### Conclusions

Rheology represents a field that has tremendous potential in furthering our understanding of dispersions, emulsions, foams and gels, and potentially impacting the technological advances in the food industry. Rheological measurements can provide increased understanding of food component interactions, and can lead to a control of such interactions and food microstructure for our benefit. This can be achieved especially when we fully understand the relationships between rheology and food texture. Interpretation of rheology data in terms of ingredient interactions and/or structural changes necessitates the use of other physicochemical measurements in conjunction with rheological measurements, and also the use of more than one rheological measurement. Indeed, interpretation of rheological data in structural or interaction terms is merely a speculation unless backed by other physicochemical measurements. In this regard, [7\*\*,9\*\*,10] are noteworthy because the authors all used other physicochemical measurements in conjunction with rheological measurements, and often they used more than one rheological measurement.

New rheological measurement techniques are evolving and being refined, yet considerable work needs to be done in establishing the practical significance of rheological properties in the context of food texture, engineering process design and product structure and ingredient interactions to leverage these in developing food products.

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This chapter presents an excellent overview of the rheology of dispersions, with particular emphasis on the effect of interactions. Electroviscous effects are presented in special detail. The effect of phase volume fraction, solvation and shapes is also discussed. This chapter, together with reviews listed above [2,3] provide an excellent starting point for the study of rheology of dispersions and emulsions.

Greenwood R, Luckham PF, Gregory T: The effect of diameter 6.

ratio and volume ratio on the viscosity of bimodal suspensions of polymer lattices. J Colloid Interface Sci 1997, 191:11-21.

A study of the rheology of bimodal suspensions. Experiments revealed that minimum viscosities are attained for a volume ratio of 0.25 of small particles to large particles. This is close to the theoretically predicted value of 0.27. Diameter ratios where viscosity of bimodal suspensions is minimized are determined. Data are interpreted in terms of an increase in the effective close packing limit of the suspension.

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- Dickinson E, Golding M: Rheology of sodium caseinate stabilized oil-in-water emulsions. J Colloid Interface Sci 1997, 191:166-176.

This paper is an excellent study of emulsion rheology where three situations corresponding to bridging flocculation, no flocculation and depletion flocculation are examined. Stress sweeps, small deformation dynamic (oscillatory) measurements and time-dependent apparent viscosity are presented. Data are interpreted in combination with the authors earlier data on interfacial coverage and creaming stability for the same systems. Time-dependent rheological measurements have been shown to be useful in characterizing slow development and restructuring of floc networks in concentrated emulsions, and to give kinetic information concerning aggregation.

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The chapter reviews dressing and sauce emulsions and contains a section on rheology with several references. Viscoelastic measurements are presented for emulsion gels and for whey protein gels and data interpreted in terms of lecithin-whey protein interaction and whey protein surface cover-age. Order of addition effects are highlighted.

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 Shear moduli computed for Kelvin, Williams and Weaire-Phelan foams using the 'Surface Evolver' computer program. This paper is one of several papers published by the authors jointly and by Kraynik alone on foams. The main thrust of these papers has been the structure and rheology of foams. Reading these along with the papers by Princen (including the ones listed above) is a must for anyone interested in the field.

Lacasse M-D, Grest GS, Levine D: Deformation of small 16.

compressed droplets. Phys Rev E 1996, 54:5436-5446.

Another use of the Surface Evolver program, this paper begins with the response of individual droplet to deformation and builds it into a model for shear modulus.

Coughlin MF, Ingenito EP, Stamenovic D: Static shear modulus 17. of gas-liquid foam determined by the punch indentation test. J Colloid Interface Sci 1996, 181:661-666.

This paper provides direct measurements on shaving foams using the punch indentation test. The paper reports on several other measurements and ultimately reports good agreement with the static shear modulus relationships reported in references [13,14] above, which can be fortuitous considering errors in individual measurements. The authors, to their credit, provide a good review of the assumptions, experiments and artifacts and their implication on the conclusions. We noted some numerical discrepancies between the numerical constants from references [13,14] cited in this paper versus the actual values reported by the original authors.

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Yoshimura M, Tomohisa T, Nishinari K: Effects of konjac-23. glucomannan on the gelatinization and retrogradation of corn starch as determined by rheology and differential scanning calorimetry. J Agric Food Chem 1996, 44:2970-2976.

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- Manoj P, Kasapis S, Chronakis IS; Gelation and phase 25. separation in maltodextrin-caseinate systems. Food Hydrocolloids 1996, 10:407-420.

A thorough examination of the phase separation phenomena in polymer mixtures using maltodextrin and caseinate as the subject of study. This work combines calorimetry with a wide range of rheological tests on the single component and the mixed biopolymer gel.

Langendorff V, Cuvelier G, Launay B, Parker A: Gelation and 26. flocculation of casein micelle/carrageenan mixtures. Food Hydrocolloids 1997, 11:35-40.

This research was designed to better understand the observed (undesirable) phenomena of sedimentation of casein micelles in the presence of carrageenan above the gelation temperature. A combination of phase diagrams and theological testing was used to elucidate the mechanism involved in interactions between casein micelles and carrageenan chains.

#### 27. Walkenstrom P, Hermansson AM: High-pressure treated mixed gels of gelatin and whey proteins. Food Hydrocolloids 1997, 11:195-208.

This paper begins with a thorough review of previous research on high pressure studies of various gelling systems. Small and large strain deformation neological tests, in combination with light and transmission electron mi-croscopy, were used to develop a complete understanding of structure-function relationships of gels set via heat treatment or high pressure treatment.

Zasypkin DV, Braudo EE, Tolstoguzov VB: Mutlicomponent 28.

biopolymer gels. Food Hydrocolloids 1997, 11:159-170.

A nice tutorial from the research lab of one of the leaders in this area. The paper reviews the classifications of multicomponent gels systems containing two biopolymers. This is then followed by examples of each type using gelatin mixed with a variety of other components. Rheology is used throughout the paper to describe the interactions. Although the rheology is not described in great detail in this review, the original papers can be accessed to obtain a more complete understanding of the rheology used to study these mixed systems.

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samples. J Food Sci 1997, in press. This paper hypothesizes that tribological properties of foods are important in the perception of food texture, and presents measurements of such properties using the surface forces apparatus equipped with optical interferometric technique. The tribological properties are explained in terms of thin film morphology and other interfacial properties. It represents an emerging area of research in food texture.