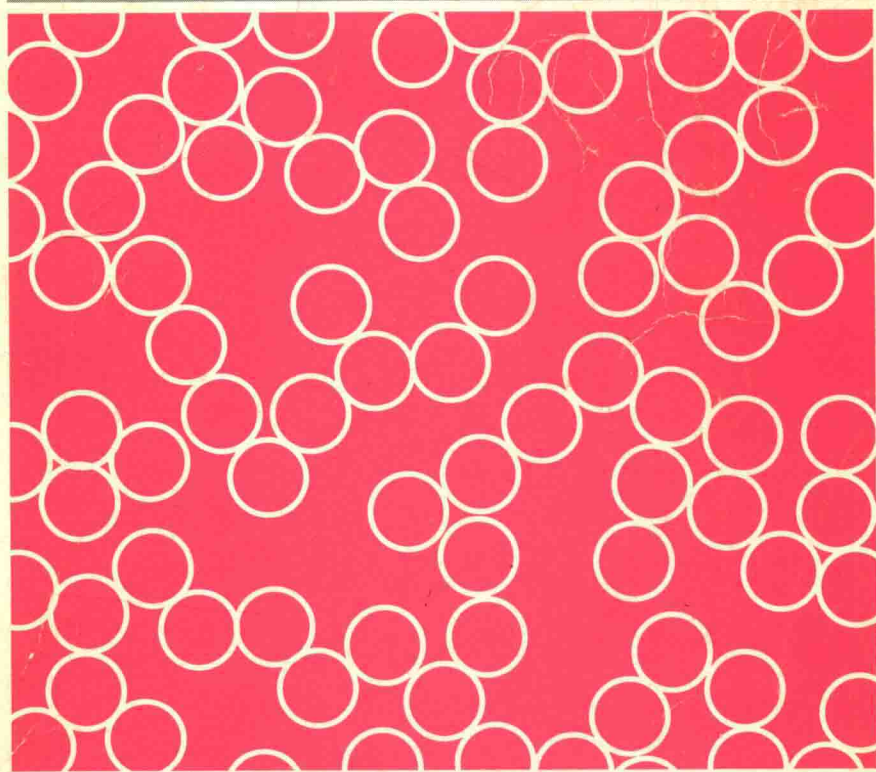


ERIC DICKINSON

An Introduction to Food Colloids



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An Introduction to Food Colloids

Preface

It is by studying little things that we attain the great art of having as little misery and as much happiness as possible.

Samuel Johnson

What seems to make us happy when we first come into this world is a regular supply of a warm white colloid called milk. As we grow up, we acquire a taste for other food colloids like butter, cheese, mayonnaise, ice-cream, and so on. Colloidal particles are the little things that give these foods their characteristic properties. It gives pleasure to the mind to learn why milk is a stable colloid, and how it can be transformed into cheese and yoghurt. In terms of the food we like to eat, the little things mean a lot.

This is a book about food seen through the eyes of a physical chemist. It is meant primarily to be read by students of food science following an undergraduate course at university level. It may, however, also be useful to students of chemistry, biology, or engineering taking a course in surface and colloid science, and to qualified food technologists who wish to learn more about the physical chemistry of food.

Even in an introductory text such as this, some background knowledge must be taken for granted. I have boldly assumed that the reader is familiar with the basic structural chemistry of food components and with some simple physical concepts. Some elementary mathematical knowledge is also required, though the number of equations has been kept to the absolute minimum needed for an adequate understanding of modern theoretical developments. An effort has been made to give some insight into what is special about the colloidal state of matter. The largest chapter of the book is devoted to emulsions—arguably the most important type of food colloid. In a little book like this, some large topics are inevitably left out or treated in a cursory manner. To any reader who fails to find a topic that should have been included, I can do little more than apologize, and then point him or her to the bibliographies at the end of each chapter.

I have tried to give the subject of colloids a historical perspective, whilst also emphasizing what I believe to be the most important recent developments. I shall feel that I have been successful if the book gives an impression to the newcomer that the subject of food colloids is a

genuinely modern and exciting field at the forefront of current scientific research.

I have obtained much happiness from the study of food colloids. I am grateful to my research students and research collaborators for what they have taught me about the subject over the past ten years or so. In particular, I should like to thank George Stainsby for introducing the subject to me, and for making valuable comments on the draft manuscript of this book.

Leeds

September 1991

E.D.

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Contents

1 The field of study	1
1.1 The colloidal state of matter	1
1.2 Colloid stability	5
1.3 Colloids in food	8
1.4 Emulsifiers and stabilizers	11
1.5 Potentials of mean force	14
1.6 Structure of colloids	18
1.7 Brownian motion and random walks	24
2 Surface activity	30
2.1 The liquid interface	30
2.2 Measurement of surface and interfacial tension	33
2.3 Adsorption from solution	37
2.4 Self-association of surfactants	40
2.5 Critical micelle concentration	44
2.6 Food emulsifiers	47
3 Rheology	51
3.1 What is rheology?	51
3.2 Rheological terminology	54
3.3 Experimental methods	56
3.4 Suspensions of spherical particles	61
3.5 Solutions of macromolecules	63
3.6 Viscoelasticity of solutions, dispersions, and gels	66
3.7 Rheology of emulsions	70
3.8 Interfacial rheology	73
3.9 Chocolate	76
4 Emulsions	79
4.1 Structural complexity	79
4.2 Creaming	82
4.3 Coalescence	88
4.4 Interactions between emulsion droplets	94
4.5 Flocculation	101

4.6 Ostwald ripening	109
4.7 Phase inversion	110
4.8 Emulsion formation	115
4.9 Testing emulsifier efficiency	119
5 Foams	123
5.1 Bubbles galore	123
5.2 Disproportionation	125
5.3 Stability of polyhedral foams	128
5.4 Foam formation	130
5.5 Beer foam	133
5.6 Egg-white foam	135
5.7 Ice-cream: a frozen foam	137
6 Proteins at liquid interfaces	140
6.1 Thermodynamics of protein adsorption	140
6.2 Kinetics of protein adsorption	144
6.3 Structure of adsorbed protein layers	147
6.4 Interfacial functionality and protein structure	151
6.5 Competitive adsorption of proteins	155
6.6 Competitive adsorption of proteins with surfactants	163
6.7 Interaction of polysaccharides with adsorbed proteins	170
7 Dispersions	174
7.1 Colloidal particles in food: casein micelles	174
7.2 The electrical double-layer	177
7.3 Electrostatic stabilization and DLVO theory	180
7.4 Steric stabilization	184
7.5 Concentrated dispersions	188
7.6 Structure of aggregates, gels, and sediments	194
Index	201

1

The field of study

1.1 The colloidal state of matter

Blood, cement, ink, milk, and paint—what do these important everyday materials have in common? They are all heterogeneous. They flow, and can also become solid. They scatter light. They contain structural entities with at least one linear dimension in the size range 1 nm – $c. 1\text{ }\mu\text{m}$. Surface chemistry and Brownian motion strongly affect their properties. With so much in common, it seems proper to regard these notionally disparate materials, and many others, as belonging to nothing less than a distinct class of their own. We call it the colloidal state of matter.

The simplest type of colloidal system consists of a single *dispersed phase*[†] of particles in a second continuous phase called the *dispersion medium*. By the word ‘phase’, we mean a region of the system, either spatially connected or discontinuous, in which there is constancy of measurable intensive variables like density, pressure, and dielectric constant. A system of solid particles smaller than $c. 1\text{ }\mu\text{m}$ suspended in a liquid medium is called a *colloidal dispersion* (or *sol*). If the particles are polymeric, the colloid is a *latex* (plural *latices*). If the dispersion medium is aqueous, the colloid is a *hydrosol*. While generally much larger than the molecules of the dispersion medium, the particles in a colloidal dispersion are small enough to execute vigorous Brownian motion. A system of particles larger than $c. 1\text{ }\mu\text{m}$ which are prone to settle under gravity is usually referred to as a *suspension*. A dispersion or suspension of liquid droplets in a liquid continuous phase is called an *emulsion*. A *foam* is a dispersion or froth of gas bubbles, and an *aerosol* is a dispersion of particles or droplets in a gas. When the continuous phase is solid, the terms ‘solid dispersion’, ‘solid emulsion’, and ‘solid foam’ are sometimes used. *Multiple colloids* contain two or more dispersed phases, so that a *multiple emulsion* may consist of finely divided droplets of one phase dispersed in larger droplets of a second phase which are themselves dispersed in a third phase. In *network colloids*, two or more phases exist as interpenetrating networks with elements of colloidal dimensions. A

[†]The elided form *disperse phase* is widely encountered.

colloid having a liquid dispersion medium, but whose overall properties are solid-like, is called a *gel*.

The making and manipulation of colloids goes back as far as civilization itself. The ink used by ancient Egyptians was a dispersion of carbon black particles in an aqueous continuous phase containing gum from the *Acacia* tree or albumin from egg-white. Gold suspensions were familiar to alchemists in the Middle Ages—the beverage ‘potable gold’ was supposedly the elixir of life. In fact, gold sols were a favourite with many of the early colloid investigators. The renowned Purple of Cassius was a gold sol made by reducing a soluble gold salt with stannous chloride.

Careful observations of colloidal phenomena began in the early nineteenth century. The particles which Robert Brown noticed under the microscope to be in vigorous thermal motion were pollen grains from the plant *Clarkia pulchella*. The first proper study of colloid stability was probably that of Francesco Selmi, who showed that salt addition to sols of silver chloride or Prussian Blue ($\text{Fe}_4(\text{Fe}(\text{CN})_6)_3$) had a destabilizing effect. About this time (the mid-nineteenth century), Thomas Graham demonstrated the slow diffusive behaviour of many colloidal systems, work which initiated much of the nomenclature of traditional colloid science. In a separate pioneering investigation, Michael Faraday observed that gold sols with a ruby-coloured appearance, made by reducing gold salts with phosphorus, changed their colour from red to blue upon heating or addition of salt. Faraday correctly attributed the colour change, and the associated enhancement of sedimentation, to an increase in effective particle size caused by aggregation, and he showed that the colloidal particles could be protected from aggregation by gelatin. The theory of light scattering was developed by Rayleigh towards the end of the nineteenth century. Extensive light-scattering experiments on colloids were carried out by Tyndall. The formal classification of colloidal systems came into being only at the beginning of the twentieth century through the work of Freundlich, Ostwald, and Zsigmondy.

Traditionally, a colloidal dispersion is classified as being either *lyophilic* or *lyophobic* depending on whether the surface of the particles has a strong or weak tendency to become wetted (solvated) by the dispersion medium. The equivalent terms *hydrophilic* and *hydrophobic* are commonly used for the same purpose with hydrosols. To these two traditional classes is sometimes added a third category of *association colloids* to describe non-macromolecular solutions containing surfactant structures such as micelles or microemulsion droplets, though in some quarters these association colloids are still grouped under the lyophilic heading.

Scientifically, a lot has happened since the terms ‘lyophilic’ and ‘lyophobic’ were first proposed by Perrin and Freundlich nearly a century ago. While the term ‘lyophilic’ was originally associated with solutions of

gums like gelatin which have a remarkably high affinity for water, modern convention would now regard most macromolecular lyophilic colloids (sometimes called *colloidal solutions*) as being true solutions—in the thermodynamic sense—of polymer molecules or macro-ions. The word ‘lyophilic’ has, for this reason, rather fallen into disuse. On the other hand, the word ‘lyophobic’ is still commonly used, though even here there is potential for ambiguity, since many ostensibly lyophobic colloids do have lyophilic polymeric material present at the surface of the particles. A dispersion of latex particles in water, with no solubilized polymer or surfactant present, constitutes a *bona fide* lyophobic sol. Such a system is destabilized by the addition of a moderate amount of salt: this is the hallmark of a traditional lyophobic sol (see Section 7.3).

In most colloidal systems, the particles are not all of the same size. In addition, there may also be a distribution of other particle attributes such as shape or electrical charge. We call such systems *polydisperse* to distinguish them from ideal *monodisperse* systems in which all the particles are exactly (or nearly) identical. Where just a few discrete sizes or types of particle occur, the term *paucidisperse* (from the Latin *paucus* meaning ‘few’) is appropriate. Closeness to pure monodispersity is a desirable attribute in colloids made for fundamental studies in the laboratory because many theories relating to colloids apply only to the ideal monodisperse situation.

Let us now try to relate the concept of the colloidal state to the other states of matter. With almost religious devotion, elementary textbooks of physics or chemistry limit themselves to the celebrated trinity of solid, liquid, and gas. We shall see below that there is nothing sacrosanct about this particular way of dividing up the natural world. Certainly, in everyday life, much of what we see around us seems to fit into one of these three states; but, talk to an astrophysicist and one learns that over 99 per cent of the known universe is in a (fourth) state of matter called ‘plasma’!† However, we do not have to look to the heavens to find materials that cannot simply be described as solid, liquid, or gas. For instance, what are we to make of the glassy (or amorphous) state? Is it another genuine state of matter to add to the list, or is it just a convenient way to distinguish a non-crystalline solid from a crystalline one? And what about mysterious states such as liquid crystals, helium at low temperatures, electrons in metals, and high-temperature superconductors? Does any one of these merit special classification as a state of matter in its own right, or is each to be fitted into the basic existing categories in the Procrustean manner?

†A hot ionized material consisting of nuclei and electrons is called a plasma. A fundamentalist might call it a gaseous system of charged particles—that is, an animated gas, but a gas none the less. A historical case for its status as a full state of matter can be traced back to the ancient Greeks: earth (solid), water (liquid), air (gas), and fire (plasma), in order of increasing entropy.

The more one attempts to wrestle with these semantic and philosophical questions, the more one realizes that there are really just two general conditions of matter: *ordered* and *disordered*. Some states are more or less ordered than other states. All materials are best described in terms of the degree and type of order present within them, as determined by the conflicting requirements of energetic and entropic considerations. On this basis, the colloidal state is more disordered than the bulk phases from which it is formed, but it may itself be ordered in so far as the dispersed particles may be characterized by uniform features and arranged within the system in a regular and organized manner. Each of the specified states of matter, including the colloidal one, is simply a convenient shorthand label for identifying the types of entities present (atoms, molecules, subatomic particles, colloidal particles) and the global degree and type of order present within the material. The colloidal state of matter exists, then, purely and simply because it is useful and convenient for us to define it to exist.

Colloid scientists are inherently practical people. They work with classes of materials as diverse in use as ceramics, cosmetics, detergents, dyestuffs, foodstuffs, pesticides, pharmaceuticals, and sewage. They are employed, usually in industry, to manipulate the colloidal state of matter so as to optimize the properties of some particular manufactured product, or some effluent or raw material. Formally, the scope of colloid science includes systems having solid as well as liquid dispersion media. In practice, though, most colloid scientists handle 'wet' materials, i.e. those having a liquid (or liquid-like) continuous phase. Colloids are clearly 'materials', but the fields of 'colloid science' and 'material science' remain distinct in the minds of both scientists and technologists. The division is partly due to historical accident, and partly arises because the experimental techniques used in the two fields are different. Having become a physical chemist by adoption or training, the colloid scientist is at home with liquids and solutions, but is less familiar with solid materials which seem still to be the province of the metallurgist, the solid-state physicist, or the mechanical engineer. This situation may change in the future since it is likely that the making of many of the advanced materials required for the twenty-first century (e.g. lightweight aerogels, high-quality ceramics) will require a detailed knowledge of colloid chemistry.[†] So we must be careful in trying to draw too fine a line of demarcation between wet colloids and solid materials, since many of the latter (opal, concrete, chocolate, etc.) are produced from the former by simple operations like

[†]In the words of an eminent industrial scientist: 'I have termed colloid science the "midwife of invention" because the intellectual approach peculiar to colloid science can generate such understanding that the delivery of a new product to the market is facilitated, even if colloid science (like the midwife) was not involved in its conception.' (Birchall, D. (1989). *Chem. Ind.*, 403.)

cooling or evaporation. In any case, even for a typical wet colloid the distinction between 'solid' and 'liquid' is not clear cut. Depending on the nature of the observation, many food colloids are characterized by behaviour that can be regarded as both solid-like and liquid-like (see Chapter 3). Which brings us back again to the problem of distinguishing between different states of matter. . . .

1.2 Colloid stability

A colloid is said to be *stable* if, over a certain period of time, there is little detectable aggregation or settling of particles.

In many colloidal systems, a stable *thermodynamic* state is notionally reached only when all particles or droplets have become united in a single homogeneous lump of dispersed phase, and therefore any apparent stability must be regarded as a purely *kinetic* phenomenon. In a lyophobic sol, a foam, or an emulsion, the massive reservoir of excess free energy in the interfacial region gives rise to an overall free energy for the system that is well above its global minimum. Such a colloid cannot be formed by spontaneous dispersion, and, when formed, it is thermodynamically unstable, or at best metastable. But, if its rate of change is negligibly slow over the time-scale of interest, we say that it is *colloidally stable*. Nevertheless, the meaning of the term in practice is sometimes rather diffuse, and it should always be accompanied by a description of the nature of the stability (see below).

There are some lyophilic colloids which do exist in a state of proper thermodynamic equilibrium. In this category we find macromolecular solutions and surfactant-containing association colloids (micelles, micro-emulsions, etc.). Lyophilic sols of this type are formed spontaneously (albeit incredibly slowly) when dry coherent material such as rubber or soap is brought into contact with a dispersion medium. They exist as multicomponent microheterogeneous, single-phase systems with no tendency to change their steady-state structures in the absence of chemical reaction. When these lyophilic sols become unstable, what occurs is not so much particle aggregation, but rather a global separation of the system into two liquid-like phases, a process that is commonly referred to as *coacervation*. One way to produce a *coacervate* is by mixing two incompatible polymer solutions.

Aggregation is the process whereby two or more colloidal particles become stuck together by forces of unspecified origin and magnitude. Where the structure of aggregates or the nature of the aggregation process is reasonably clear, the words *coagulation* (from the Latin *cogere* meaning 'to drive together') and *flocculation* (from the Latin *floccus* meaning 'tuft of wool') are used to distinguish between the formation of rigid aggregates and loose aggregates. In what is now known as the La Mer convention,

aggregates produced by coagulation have interparticle distances of the order of atomic dimensions, whereas flocculated particles are held relatively far apart. Forces between coagulated particles are strong, whereas those in flocculated aggregates (*flocs*) are weak. The process of flocculation is normally regarded as being reversible. Coagulation ultimately leads to the irreversible separation of a *coagulum*. The words 'reversible' and 'irreversible' are used here in their thermodynamic sense. In fact, under favourable circumstances, both flocculation and coagulation can be pushed in the opposite direction; these processes are known as *deflocculation* and *peptization* respectively. Adding salt to a lyophobic sol (e.g. polystyrene latex) may induce coagulation;† polymer addition may lead to flocculation. Despite La Mer's exhortation, some colloid scientists still continue to treat the two terms as if they were synonymous. The only justification for this is that in certain circumstances the aggregate structure may be unknown or difficult to guess. In such instances, we shall adopt the generic term 'aggregation'.

The most obvious visible manifestation of colloid instability is the settling of particles, droplets or aggregates under gravity—*sedimentation* or *creaming* depending on whether the dispersed phase moves down or up. The rate depends on the size of the settling unit and the density difference between the phases. In a *diuturnal* suspension, the settling speed is of the order of a few micrometres per second. A *sediment* is the concentrated suspension formed by sedimentation of a dilute suspension, and a *cream* is the concentrated emulsion formed by creaming of a dilute oil-in-water (O/W) emulsion. The settling of a water-in-oil (W/O) emulsion is called creaming by some authors and sedimentation by others; and some authors restrict use of the term 'creaming' to emulsions only. A cream may be flocculated or colloiddally stable, but the droplets should not have coalesced to any significant extent. If coalescence is extensive, it leads to the formation of a macrophase, and the emulsion is said to have been 'broken'. Thermodynamic stability is reached when all the droplets in an emulsion have coalesced into a single homogeneous region of oil or aqueous solution.

The very existence of a phenomenon such as flocculation implies the presence of long-range forces between colloidal particles. These net interparticle attractive forces arise from the combined effects of a very large number of separate forces between pairs of atoms or molecules on different particles. Except for highly polar materials, it is the sum of the London dispersion‡ forces that contributes nearly all the van der Waals attraction between colloidal particles. Although dispersion forces

†The salt concentration in sea water is high enough to induce such coagulation. When river water containing colloidal clay runs into the sea, the brine induces coagulation. This is a major cause of silting in estuaries.

‡The word 'dispersion' here comes from the phenomenon of *optical* dispersion.

between atoms or molecules are relatively short-ranged, extending only over a fraction of a nanometre, the net effect of adding together some 10^{10} – 10^{20} such interactions is to produce a long-range interparticle attraction of considerable strength and range (say 1 – 2 kT at surface-to-surface separation 5 – 10 nm, where k is Boltzmann's constant and T is the absolute temperature). Therefore, in order to confer colloid stability an additional long-range repulsive interaction, which is of comparable range to, and at least as strong as, the van der Waals attractive interaction, must exist between the particles.

There are two main ways of stabilizing a colloidal system: *electrostatic stabilization* and *polymeric stabilization*. In an electrostatically-stabilized (*electrocratic*) colloid, particles are kept apart by Coulombic repulsion between charged surfaces. In polymeric stabilization, particles are kept apart by macromolecules which may be attached to the surface (*steric stabilization*) or dissolved in the dispersion medium as an entangled solution or weak network. Many food colloids containing adsorbed protein are stabilized by a combination of electrostatic and steric mechanisms.

A gel is a special sort of colloid. It can be defined as a soft, elastic, deformable solid made from a connected assembly of colloidal particles or macromolecules. The dispersed or solubilized component forming the gel network is generally present in much smaller amount than the solvent making up most of the mass. On the scale of the freely diffusing small molecules or ions in the aqueous dispersion medium, the gel is 'liquid'. But, on the macroscopic scale, the gel is 'solid' because the three-dimensional network of particles or polymer molecules has the capacity to store mechanical energy during deformation. The range of food biopolymers that can form polymer gels is rather large; it includes many types of polysaccharides (agar, alginate, carrageenan, pectin, etc.) and some proteins (gelatin, myosin, etc.). Dairy products like cheese and yoghurt are examples of food gels consisting of a network of aggregated colloidal particles (casein micelles).

A biopolymer solution may be converted into a biopolymer gel by changing the temperature or by addition of a new component to the solution. Typically, the temperature jump leads to a change in biopolymer conformational state; this induces a biopolymer association process, and if the concentration is high enough gelation may occur. The likely conformational change on lowering the temperature is one of ordering, in which case the gelation process may be regarded as a sort of frustrated polymer crystallization (e.g. the cold-setting of gelatin). Where the temperature is raised, biopolymer disordering is usually involved, with subsequent network formation deriving from a complex set of newly induced intermolecular interactions (e.g. the heat-setting of globular proteins). In non-thermal gelation processes, a biopolymer network

formation may be induced by changing the pH, or by adding salt, alcohol, or an enzyme. In some instances the added species are directly involved in the biopolymer association (e.g. calcium ions in the gelation of alginate). In other cases, the added component merely drives the conformational change which induces the association process, rather as a temperature change does, by changing the balance between the intra- and inter-molecular interactions (e.g. by breaking hydrogen bonds, or screening ionic forces). Though the detailed chemistry of biopolymer gels is beyond the scope of this book,[†] it should be noted that many food colloids do contain biopolymer gels as contributory stabilizing elements. The rheological aspects of these systems will be discussed in Chapter 3.

It is interesting to record that much of the pioneering experimental work in colloid science was carried out with food biopolymers. One of the earliest measures of colloid stability was the 'gold number' of Zsigmondy, defined as the number of milligrams of 'protective agent' (*Schutzkolloide*) per 10 cm³ of gold sol which is just sufficient to prevent its changing from red to blue when 1.0 cm³ of 10 wt per cent NaCl solution is added. Table 1.1 lists the gold numbers for a few natural polymers. We see that gelatin is a very efficient stabilizer, whereas starch is quite ineffective. Understanding the reason for this sort of difference in terms of the modern concepts of physical chemistry is to a large extent what this book is all about.

Table 1.1 Gold numbers of natural polymers

Biopolymer	Gold number (mg per 10 cm ³)
Gelatin	0.005–0.01
Sodium caseinate	0.01
Albumin	0.1–0.2
Gum arabic	0.15–0.25
Dextrin	6–20
Potato starch	~ 25

Zsigmondy, R. (1901). *Z. Analyt. Chem.*, **40**, 697.

1.3 Colloids in food

A typical food is a complex, multi-component, multi-phase system. Figure 1.1 gives a schematic representation of some of the main structural elements which may be encountered. We can distinguish two distinct types of entities: particles and macromolecules. The particles may be

[†]For an excellent introduction to the topic, see: Clark, A. H. (1991). In *Food polymers, gels and colloids* (ed. E. Dickinson). Royal Society of Chemistry, Cambridge, p. 322. More comprehensive information on different kinds of protein and polysaccharide gels can be found in: Harris, P. (ed.) (1990). *Food gels*. Elsevier Applied Science, London.

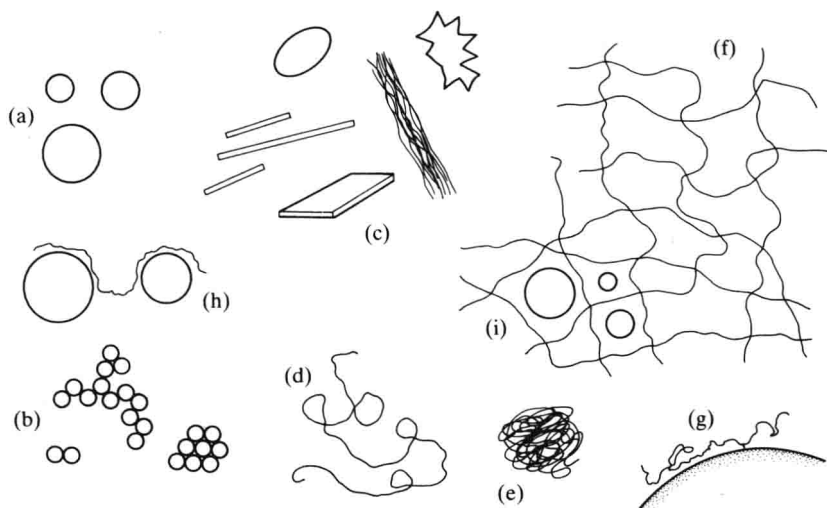


Fig. 1.1. Types of structural entities encountered in food colloids (highly schematic): (a) dispersed spherical particles; (b) aggregated spherical particles; (c) non-spherical particles; (d) disorganized macromolecule; (e) compact macromolecule; (f) macromolecules associated into a gel; (g) macromolecule adsorbed on particle surface; (h) macromolecule bridging between particles; and (i) particles trapped in macromolecular network.

exactly spherical (gas bubbles, oil or water droplets) or approximately spherical (fat globules, protein or starch granules). They may occur in a wide range of sizes—from nanometres (surfactant micelles) to micrometres (emulsion droplets) to millimetres (foam bubbles). Particles may exist as isolated dispersed entities, or they may be stuck together to form aggregates of various shapes, sizes, and structures. Non-spherical particles may be needle- or plate-like (ice, fat, or sugar crystals), fibrous or sheet-like (filaments, membranes), or deformed spheres (droplets, bubbles) distorted by mechanical forces or high density packing. Food macromolecules may be compact and highly organized (globular proteins) or chain-like and disorganized (many polysaccharides and denatured proteins). Molecular weights vary between tens of thousands of daltons (many proteins) to several millions of daltons (many polysaccharides). Aggregates of macromolecules or particles may be so large as to span across macroscopic dimensions, thereby producing a gel-like network. Macromolecules (especially proteins) may adsorb at the surface of solid particles, droplets and bubbles, or may bridge between such entities contributing to the state of particle aggregation. Particles may also become trapped within a network of macromolecules (a filled gel).

The study of food colloids, then, is the physico-chemical study of food viewed as a collection of dispersed particles and macromolecules in various states of microscopic and sub-microscopic organization. We are concerned with structure and stability on the colloidal scale, and with the