282. *Factors in the Stabilisation of Emulsions.*

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The influence of electrolytes on the electrokinetic potential of stabilised oil-in-water emulsions has been studied. No relation has been observed between electrical charge and emulsion stability in the absence of chemical reactions. The rôle of lyotropic phenomena has been investigated, with special reference to concentrated emulsions, and the results have been discussed and explained. The relation of other fundamental properties of emulsions to stability has been considered, and some general principles have been developed for a rational explanation of the behaviour of certain groups of emulsions.

Most of the early, critical studies of emulsions, especially those concerned with the rôle of the electrical charge, were based on work with oil hydrosols (unstabilised emulsions); it was therefore inevitable that the phenomena observed in this type of dilute emulsion should have provided the basis for discussion of the electrical properties of emulsions in general and the relationship of these properties to stability. This explains the outstanding importance assigned to electrical charge in emulsion stability by many workers.

Ellis (*Z. physikal. Chem.*, 1912, **78**, 321; **80**, 597; 1915, **89**, 145) studied the influence of electrolytes on the stability and cataphoretic velocity of two-phase emulsions, and Powis (*ibid.*, pp. 91, 179, 186) investigated the rôle of the interfacial potential in these systems. Ellis showed that stability and potential were closely related, whereas Powis introduced the notion of a critical potential, an idea which has been proved meaningless by Eilers and Korff (*Trans. Faraday Soc.*, 1940, **36**, 229).

Clowes (*J. Physical Chem.*, 1916, **20**, 407) extended Ellis's conclusions to emulsions of all kinds and types. He considered that, in stabilised emulsions, stability phenomena could be explained by a consideration of the electrical charge on the dispersed globules, which, in turn, was due to preferential adsorption of anions and cations. Bhatnagar (J., 1920, **117**, 552; 1921, **119**, 61, 1760) elaborated this theory, finding that emulsions are normally stabilised by the charge conferred on the droplets by the adsorbed hydroxyl or hydrogen ions. The phase inversion from one emulsion type to another, brought about by the addition of electrolytes, is considered to be due to neutralisation and reversal of charge following adsorption of an active ion of charge opposite to that stabilising the initial emulsion. We have already found (*Trans. Faraday Soc.*, 1939, **35**, 741) that Bhatnagar's hypothesis held only in systems where the interfacial film was chemically decomposed by the added electrolyte. Using chemically inert emulsifying agents, we were unable to detect any appreciable influence of even considerable concentrations of acids, bases, and salts on emulsion stability.

Quantitative, electrokinetic research on stabilised emulsions was carried out by Limburg (*Rec. Trav. chim.*, 1926, **45**, 772, 854), who found that, with oil hydrosols, stability was smallest at the isoelectric point. This point was reached only when salts with tervalent or quadrivalent cations (*i.e.*, salts which are hydrolysed in aqueous solution) were added to the system. When the emulsion was stabilised with a saponin, the shape of the curve obtained by plotting cataphoretic velocity against a function of the concentration of the electrolyte was the same as in the experiments with oil hydrosols. There was, however, no relationship between cataphoretic velocity and stability, nor was any decrease in stability noticed at the isoelectric point. In the absence of relationship between electrokinetic potential and stability, Limburg concluded that it was the protective film of emulsifying agent which conferred stability on the droplet.

Verwey (*Chem. Weekblad*, 1939, **36**, 800), who does not distinguish between stabilised emulsions and oil hydrosols, believes that in all kinds of emulsions the electrical conditions play a dominant rôle. The function of the emulsifying agent is said to be to shift the potential drop of the electrical double layer towards the outer phase, besides imparting to the droplets properties resembling those of solid surfaces. Schulman and Cockbain (*Trans. Faraday Soc.*, 1940, **36**, 651) postulate similarly that electrical charge on the oil droplets is a condition for the stability of oil-in-water emulsions. They report separation of internal phase incident on the neutralisation of the electrical charge. The emulsifying agents used by these authors, however, were liable to react with many of the electrolytes employed. It is clear that the occurrence of chemical interaction makes any subsequent phenomena observed unsuitable for generalisations concerning emulsion stability.

From the foregoing it is clear that the only quantitative investigation into the relationship between electrical charge and the stability of stabilised emulsions was that carried out by Limburg. We therefore studied the influence of electrolytes on the cataphoretic velocity of oil-in-water emulsions stabilised by a variety of agents, and determined whether changes in the potential were accompanied by similar changes in stability.

Results.—The agents selected were a saponin, a Daxad reagent, sodium oleate, and alumina. The Daxad reagents are described as polymerised sulphonic acids of the alkyl-aryl type, and we had confirmed (*loc. cit.*) that they are unaffected by substantial concentrations of acids, bases, and salts. The alumina also showed a high resistance to electrolytes. In contrast, sodium oleate reacts chemically with salts with multivalent cations, and the emulsions which it stabilises are, consequently, rendered unstable by the addition of salts of, *e.g.*, magnesium or aluminium.

1. *Saponin* (Fig. 1). The mobilities are relative values and were obtained by multiplying the reciprocals of the time periods taken by the globules to cover the standard distance by 1000.

No quantitative data could be obtained when sulphuric acid was used. The charge was, however, negative throughout.

In cases where the electrophoretic velocity was reduced to small values by electrolyte concentrations of less than 0·0001N, as with magnesium sulphate and potassium iodide, measurements were repeated with much higher concentrations of electrolyte. In no case, however, was a reversal of sign of charge effected; at higher electrolyte concentrations, cataphoretic flow will come to a standstill, owing to disturbances caused by electrolysis, and, occasionally, by creaming and clumping. The disturbing influence of electrolysis is, however, a general, not a specific property of electrolytes and is not noticeable except at relatively very high concentrations—much higher than the maximum concentration used in the present studies, *i.e.*, 0·0002N, but usually lower than 0·1N. It became clear that the absence of flow at high concentrations of electrolytes, when electrolysis could no longer be neglected, or whenever clumping or rapid creaming took place, was due to mechanical

Fig. 1.

The influence of electrolytes on the cataphoretic velocity of an emulsion stabilised by saponin.

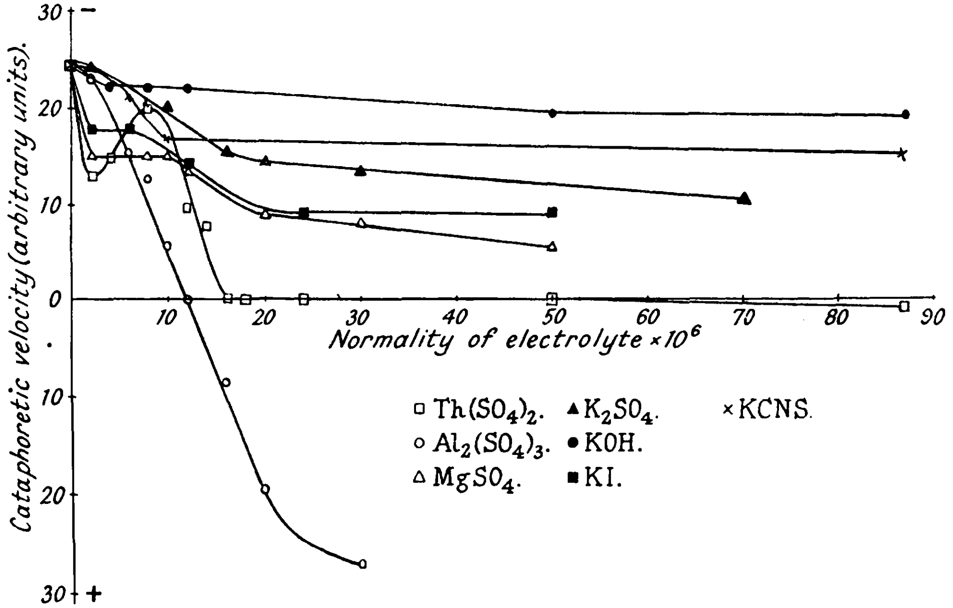
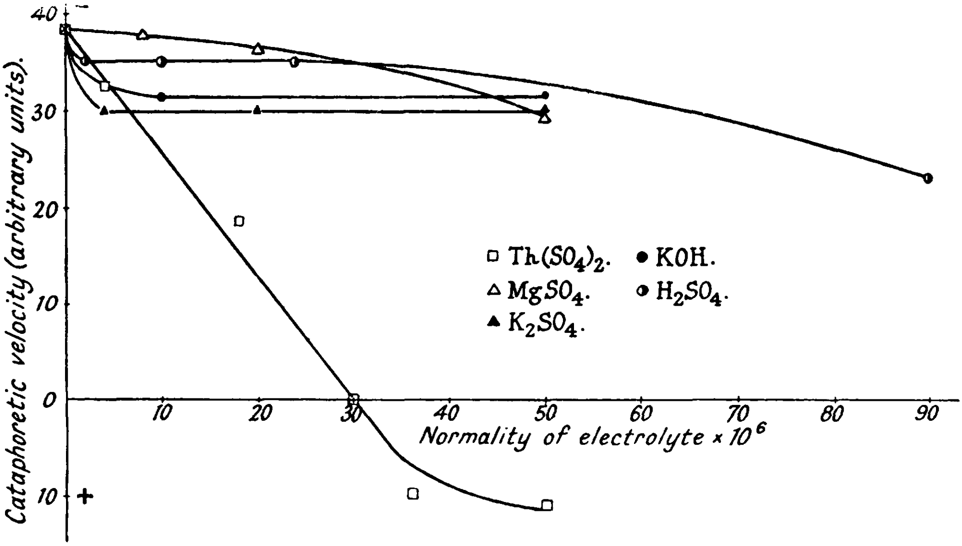


Fig. 2.

The influence of electrolytes on the cataphoretic velocity of an emulsion stabilised by Daxad 23.



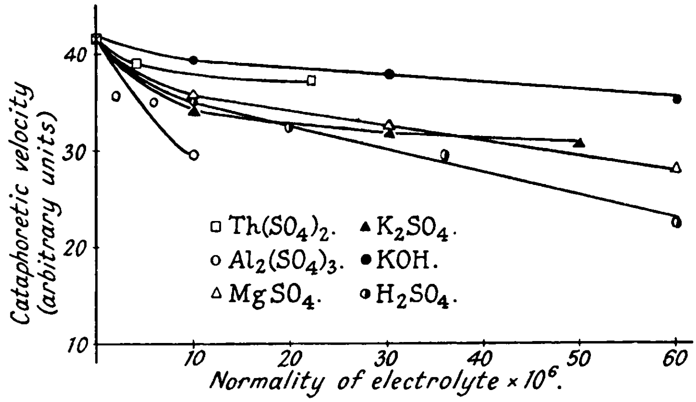
interference with flow and not to the absence of electric charge; the experiments with aluminium sulphate clearly illustrate the difference in character and meaning between the stoppage of flow at the isoelectric point and at high salt concentrations. (In this case, the disturbances at high electrolyte concentrations were caused by creaming and clumping.) Schulman and Cockbain (*loc. cit.*) did not specify the concentrations of their electrolytes, so it is possible that they were sufficiently high to cause disturbances of the kind just mentioned.

2. *Daxad* (Fig. 2). With aluminium sulphate, only qualitative measurements were possible, constant values being unobtainable. The position of the isoelectric point could, however, be estimated with considerable precision : it corresponded to a concentration of electrolyte of $24{\cdot}5 \times 10^{-6}$N.

3. *Sodium oleate* (Fig. 3). With thorium and aluminium sulphates, quantitative measurements became impossible as soon as the salt concentrations reached *ca.* 3×10^{-5}N and *ca.* $1{\cdot}5$—$2{\cdot}0 \times 10^{-5}$N respectively, since the flow came to a standstill a few seconds after the current had been turned on. It might be inferred that this disturbance was due to chemical decomposition, but it is noteworthy that these effects were absent when either magnesium sulphate or sulphuric acid was the electrolyte.

FIG. 3.

The influence of electrolytes on the cataphoretic velocity of an emulsion stabilised by sodium oleate.



4. *Alumina.* These measurements were very difficult, as there was a strong tendency for the particles to aggregate in clumps. Observations could only be made over very short periods of time, and quantitative determinations were impossible. It was, therefore, decided to limit the experiments with emulsions stabilised by alumina to a qualitative (or semi-quantitative) investigation of the effect of a salt with a multivalent cation. Aluminium sulphate was selected, as it had been shown, by the experiments described above, to have the most pronounced effect on the cataphoretic mobility of the oil droplets. The results are tabulated below, *t* being the time in seconds, and the relative mobility being expressed as 1000/*t*. Although they are little more than qualitative, they indicate that the changes brought about in the cataphoretic mobility by the addition of aluminium sulphate were relatively insignificant. Furthermore, it was found that the sign of charge of the globules remained unchanged, even at a concentration of aluminium sulphate as high as 10^{-4}N.

Influence of aluminium sulphate on the cataphoretic mobility of an emulsion stabilised by alumina.

$Al_2(SO_4)_3$, N × 10^6	0	2	4	6	8
t	20·9	23·1	17·6	16·5	21·8
1000/*t*	−47·9	−43·3	−56·8	−60·6	−45·9

Comparative Stability Tests.—In order to obtain an idea of the significance of the isoelectric point as witnessed after the addition of certain electrolytes, three samples of the saponin-stabilised emulsion used in the above experiments were observed in test-tubes. The first sample was left untreated, to the second was added such a concentration of aluminium sulphate that the isoelectric point was reached, and the normality of the salt in the third sample was substantially higher, *viz.*, 6×10^{-5}. All three samples were stable for many months, and all showed a tendency to creaming and clumping, the effect being equal in the first and the second sample, and much more marked in the third.

For comparison, an emulsion of the same oil concentration, *i.e.*, 0·091%, stabilised by sodium oleate, was similarly treated with aluminium sulphate, and all the oil soon separated. Even at a concentration of aluminium sulphate as high as 5×10^{-5}N, no reversal of sign of charge had been noticeable.

From these results certain regularities in the changes observed in the electrokinetic properties of stabilised emulsions on the addition of electrolytes become manifest and may be summarised thus: (1) Aluminium and thorium sulphates at certain concentrations reverse the sign of charge on the droplets of emulsions stabilised by the saponin and the Daxad agent, but not of emulsions prepared with either aged alumina or sodium oleate. (2) The effect of a given concentration of the same electrolyte on the ζ-potential of the dispersed globule is greater in emulsions prepared with the saponin than in those with Daxad, and smallest in the soap-stabilised emulsion. (3) There is a constant decreasing order of potency of equivalent amounts of the electrolytes, *viz.*: $Al_2(SO_4)_3 > Th(SO_4)_2 > MgSO_4$ and $H_2SO_4 > K_2SO_4 > KOH$. (4) The position of the anion of the electrolyte in the lyotropic series seems to be of little importance for its effect on the electrokinetic potential of emulsion globules.

(1) Since aluminium and thorium ions are respectively ter- and quadri-valent, a casual inspection of the results might suggest that the Schulze–Hardy rule could be extended to this field of study, but the difficulty arises that the effect of the tervalent is much larger than that of the quadrivalent ions. Limburg (*loc. cit.*), who found the same effect with saponin emulsions, ascribed it to the action of the hydrolysis products of the salts, whereas Powis, Kruyt, and Freundlich believe that the change in the sign of charge on the droplets is caused by the cation of the added salt. In support of his view, Limburg cites the inconsiderable change observed in the charge on the addition of barium chloride. The influence of magnesium sulphate, another salt with a multivalent cation which does not hydrolyse in aqueous solution, was similarly observed in our work to be comparatively insignificant. Furthermore, Limburg found that, by adding aluminium chloride together with sufficient hydrochloric acid to repress its hydrolysis, the negative sign of charge on the droplets was left unchanged. This evidence appears irrefutable.

It is not easy to explain the dissimilar effect of aluminium and thorium sulphates on emulsions prepared with the saponin and the Daxad agent on the one hand, and with alumina and sodium oleate on the other. The first group of emulsions must be assumed to be stabilised by a film which is capable of adsorbing the hydrolysis products of these salts. It is readily understood why this adsorption cannot take place at a sodium oleate film, for chemical interaction makes this impossible. Aged alumina is extremely inert in its behaviour towards electrolytes, but this is a less clear case.

The experiments with saponin emulsions and aluminium sulphate show that there is no relation in these systems between the electrokinetic potential and stability.

(2), (3) These phenomena can only be interpreted in terms of varying adsorption. In the light of the results obtained in the present investigation, any inference as to the relative stabilities of the emulsions could not be justified.

(4) No conclusion should be drawn from these results as to the importance of hydration in emulsion stability. In the systems under examination hydration would not appear to be an important factor. It is impossible, however, to generalise as to the rôle of lyotropic phenomena in the stability of oil-in-water emulsions, in view of the high dilution of the systems studied here.

Hydration and Emulsion Stability.—The above results make it clear that the electrical charge on the droplets is not a fundamental condition of emulsion stability. We are therefore led to ascribe stability to the properties of the film of emulsifying agent which surrounds each particle. In emulsions of the oil-in-water type, these films are generally strongly hydrophilic and, owing to hydration, are stable even at the isoelectric point. The influence of hydration in emulsions prepared with the strongly hydrophilic colloids is obvious. It led Fischer (" Fats and Fatty Degeneration," 1917, 5) to formulate his theory, according to which emulsions are stabilised, quite generally, by the formation of a complex between the agent and the dispersion medium. This theory has been disproved by experiment, but the importance of hydration and water content has been stressed from time to time as, *e.g.*, in the case of bituminous emulsions (Korzhuev and Gridchina, *Zavod. Lab.*, 1939, **8**, 739). Neither in the extremely dilute systems used for measuring cataphoretic velocities, nor in the moderately concentrated emulsions (*i.e.*, with an oil content up to 40%), were we able to detect any striking influence of lyotropic properties. It seemed desirable, therefore, to study emulsions of high internal phase concentration in which the globules are tightly packed and which might be expected to give a clearer idea of the part played by lyotropic phenomena in emulsion stability.

A number of concentrated oil-in-water emulsions stabilised by different agents were therefore prepared and saturated with salts whose anions are representative of those at either end of the lyotropic series. The emulsions stabilised by sodium oleate contained 90% of dispersed oil. Such a high concentration could not be attained with the other emulsifying agents, but it may be assumed that in all cases the globules were closely packed. Particulars of the emulsions are given on p. 1520, and the observed effects of the salt additions are shown below.

Influence of electrolyte addition on oil-in-water emulsions stabilised by various emulsifying agents.

Emulsifying agent.	Salt added.				
	(None.)	Na_2SO_4.	$Al_2(SO_4)_3$.	NaI.	KCNS.
Sodium oleate	(1) Viscous. Oil separation negligible for months	(2) Very viscous and gelatinous. Oil separation slow, free oil being *ca.* 15% after 3 days [*]		(3) Much more mobile than (1). Breaks within a few mins.	(4) Much more mobile than (1). Breaks instantaneously
Saponin	(5) Very viscous. Oil separation very slow	(6) Very viscous, somewhat more so than (5), but less than (7). Oil separation negligible	(7) Extremely viscous. No oil separation	(8) Fluidity increased. Much less stable than (5); *ca.* 10% of total oil separated within 24 hours	(9) Relatively very mobile. Breaks rapidly
Aged alumina	(10) Rather viscous. Slight amount of free oil observed	(11) Breaks instantaneously	(12) Breaks instantaneously	(13) Viscosity enormously increased. No free oil	(14) Like (13) in every respect
Bentonite	(15) Little oil separation	(16) Pronounced oil separation	(17) Pronounced oil separation	(18) Oil separation negligible	(19) Oil separation negligible

[*] K_2SO_4 produced about the same viscosity as in (1), but the oil separation was somewhat larger.

These experiments show that, for the action of salts on concentrated oil-in-water emulsions, the nature of the anion is determinative, that of the cation being unimportant. The effect of the anion is determined by its position in the lyotropic series. When subjected to the action of the various salts, those emulsions stabilised by colloidal electrolytes and lyophilic colloids behave in a diametrically opposite manner from those prepared with finely divided solids. The former are largely unaffected by the addition of sulphates (sodium oleate) or may be rendered even more stable (saponin); they break rapidly, however, in the presence of iodides and thiocyanates. The reverse is true for emulsions stabilised by finely divided solids such as alumina and bentonite. In all cases, however, a rise (or fall) in stability is accompanied by a rise (or fall) in viscosity. From these experiments it was difficult to decide whether the changes in stability were due to the corresponding changes in viscosity or whether both were symptoms of the same, as yet undefined, cause. In order to elucidate this a study was made of the action of the various electrolytes used as above on solutions or dispersions of the emulsifying agents.

For this purpose, separate samples of solutions (or dispersions) of the agents were treated in exactly the same manner as the emulsions stabilised by them. The effects were immediately noticeable with the soap solution, and with the saponin solution within 24 hours. The influence of the salts on the suspensions of alumina became clear after about a week. The results are summarised below.

Influence of salts on the solutions or dispersions of the emulsifying agents previously employed.

	Effect of salts.			
Agent.	$Al_2(SO_4)_3$.	Na_2SO_4.	NaI.	KCNS.
Sodium oleate	—	Solution not much changed in either turbidity or foam	Quite transparent. Volume of foam reduced	Quite transparent. Foaming reduced
Saponin	As turbid as untreated solution	Somewhat less turbid	Quite clear and transparent	Quite clear and transparent
Alumina	Coarsening and tendency towards flocculation	As with $Al_2(SO_4)_3$	Uniform distribution of particles preserved	Sedimentation only slight after prolonged time
Bentonite	Pronounced flocculation	Pronounced flocculation	No flocculation	Little flocculation

From the foregoing table it is clear that the action of any of the salts on the solution or dispersion of a given agent corresponds exactly to its effect on an emulsion stabilised by that agent. This proves that the destabilising effect of a given salt on a particular emulsion is the disruption of the adsorbed film of agent.

A consideration of the changes in the particle size of the emulsifying agents, caused by the addition of a given salt, affords an explanation of the diametrically opposed influences of that salt on the stability of the solutions of hydrophilic colloids on the one hand, and solid dispersions on the other, and, consequently, on the respective stabilities of emulsions prepared with either group of agents.

The tendency of thiocyanates and iodides, *i.e.*, salts whose anions lie at the hydrophobic end of the Hofmeister series, is to decrease the size of the sol particles (peptisation). Their influence on solid agents, like alumina and bentonite, is, therefore, beneficial, since the average particle size is relatively large even in the finest suspensions obtainable. Owing to its increased surface, a given quantity of agent will then be able to protect a larger amount of oil. Salts with highly hydrophilic anions, *e.g.*, sulphates, tend, by contrast, to increase the size of the sol particles (flocculation). Accordingly, they decrease the stability of suspensions and of emulsions prepared with solid agents.

The particle size in colloidal solutions is of a much lower order. Their stability and that of emulsions prepared with them is, therefore, inappreciably affected by the flocculating action of sulphates. The effect of thiocyanates and iodides, however, is to reduce the particle size of these colloids, which now approach the range of molecular size and lose their efficiency as emulsifying agents.

The hypothesis that there is a lower as well as an upper limit of particle size for the efficiency of emulsifying agents was tentatively advanced by Bechhold, Dede, and Reiner (*Kolloid-Z.*, 1921, **28**, 6) to explain the variations observed in the emulsifying properties of clays of different grain size. The above explanation links up the rôle of particle size and emulsifying efficiency with the influence of salts on the stability of colloidal systems, including stabilised emulsions.

From this it becomes apparent that the changes in viscosity, which accompany parallel changes in emulsion stability, are themselves secondary, not primary phenomena. The fundamental cause is the variation in the particle size.

General Discussion.—The results of the present research, together with those of other recent workers, all point to the strength of the adsorbed interfacial film as the chief factor favouring emulsion stability. Indeed—and this is demonstrated by those of the present studies which deal with highly concentrated emulsions—there is nothing to differentiate between the stability of the film and of the emulsion. The conclusions reached by Schulman and Cockbain (*loc. cit.*) point in the same direction. Other influences, however, also play a part, although it may be a subsidiary one; *e.g.*, the stabilising effect of high viscosity must be recognised as functioning by the retardation of coalescence of the individual droplets, but it has never been observed to be the main source of stability, and its function, like that of other secondary factors, is conditioned by the existence and nature of a protective film.

It should be realised that the systems whose behaviour under the action of electrolytes was studied were those in which no chemical interaction between agent and electrolyte took place. Whenever this occurs, the emulsion is rendered unstable immediately, irrespective of the agent concentration. As to the other (not chemical) influences of electrolytes on emulsion stability, these become important only when the concentration of emulsifying agent is too low for the formation of a condensed unimolecular film. With a sufficiently high concentration of agent, on the other hand, the electrical behaviour of an emulsion will bear a comparatively insignificant relation to its stability.

No less important than the concentration of emulsifying agent is that of internal phase. With emulsions containing low and medium concentrations of dispersed oil, electrolytes have little destructive influence, but concentrated emulsions, as we have seen, may be stabilised or broken according to the position in the lyotropic series of the ion of the added electrolyte and the degree of dispersion of the emulsifying agent. The physical and chemical nature of the internal phase liquid also helps to determine both ease of formation and stability of emulsions; work on this topic will be reported later.

In general, we may conclude that, besides the main source of stability, *viz.*, a coherent protective film, several other factors influence emulsion stability, their relative importance depending on the conditions under which the emulsion was prepared.

EXPERIMENTAL.

Preparation of Emulsions.—The emulsions were prepared by shaking the requisite amount of the oil phase with the aqueous medium in which the appropriate amount of agent had been dissolved. For the more concentrated emulsions, the oil was added in stages. The resulting emulsion was subsequently homogenised three times by means of a domestic cream machine. Emulsions for use in cataphoretic experiments were obtained by dispersing 10 g. of the mixture ($d = 1$) of kerosene and carbon tetrachloride in 100 g. of distilled water, containing 0·100 g. of the agent, in the manner described above, homogenising it three times, and diluting the resulting emulsion 100-fold. Such emulsions could be kept for several weeks without any alteration in the cataphoretic mobility being noticeable.

When the influence of electrolytes on the stability and other properties of the emulsion had to be studied, the electrolyte (in aqueous solution) was added to and shaken with the emulsion. In the electrokinetic experiments, measurements were taken 1—2 mins. after the electrolyte had been added.

Concentrated emulsions. The emulsion stabilised by sodium oleate contained 0·76% (calculated on the mass of disperse phase) of emulsifying agent and 90% of the oil phase; the saponin emulsion contained 83% of oil and 1% of a saponin; 1·8% of aged alumina stabilised an emulsion containing 75% of oil, and with 2·0% of bentonite 60% of oil was emulsified. With larger amounts of oil, the bentonite emulsions tended to suffer phase inversion. Separate samples of all these emulsions were saturated with anhydrous sodium sulphate, potassium sulphate, sodium iodide, and potassium thiocyanate severally. The results are in the table on p. 1518.

Measurement of Cataphoretic Velocity.—*Method.* The main choice lies between microscopic and macroscopic methods. For our purposes there was little to choose between the two methods. Limburg's chief objections (*loc. cit.*) against microscopic methods were (*a*) the disturbing effect of electro-osmosis, (*b*) the difficulties in controlling temperature. He therefore used a macroscopic method, a modified moving-boundary method, with thermostatic control. Mooney (*J. Physical Chem.*, 1931, **35**, 331) found the microscopic method satisfactory for measuring the cataphoretic velocity of oil drops in water; like Limburg, he used thin-walled cylindrical glass **U**-tubes as cells, and, without temperature regulation, was still able to restrict the mean error to 1%. Bondy (*Trans. Faraday Soc.*, 1939, **35**, 1093) similarly used a microscopic method in studying the cataphoresis of rubber latex. This type of method is convenient because, in a comparatively small apparatus, a large number of readings can be quickly taken with considerable precision. Bennister and King (J., 1938, 991) also used a microscopic method for investigating electrokinetic properties of activated charcoal, and we used this method with some modifications.

Apparatus. The cell, constructed entirely of Pyrex glass tubing (Bennister and King, *loc. cit.*), was carefully cleaned and steamed out between subsequent sets of measurements; steaming was necessary to expel any traces of electrolytes adsorbed on the glass. The cell was clipped to a wooden block which, in turn, was securely fixed to the microscope stage.

The dispersed oil droplets in the cataphoresis tube were illuminated by means of an intense low-voltage (6 v.) microscope lamp. The light was collected by a medium-power microscope objective and focused inside the tube directly below the microscope. The microscope objective was fixed to a movable metal arm attached to a Vernier scale. With such an arrangement cataphoretic velocities could be observed at any desired depth in the tube.

The eyepiece of the microscope was fitted with two parallel cross wires. The time taken by individual globules to travel from one wire to the other was measured. The current, 110 v., D.C., was then reversed, and the observations repeated in the opposite direction. Brownian movement was hardly ever observed, and by taking ten readings in each direction, a satisfactory mean value was obtainable.

The theoretical difficulties in finding that level in the cataphoresis tube at which the end-osmotic flow is zero and where, therefore, the true cataphoretic velocity of the globules can be observed, have been discussed by Bennister and King (*loc. cit.*). For the present studies, it was considered that the only way was to determine the required height as closely as possible by experiment. This was done, and even if this height was not exactly reached, certainly a near approach was made that sufficed for comparative measurements.

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