

THE RHEOLOGY OF OIL-IN-WATER EMULSIONS

PART II. THE MICROSCOPICAL APPEARANCE OF EMULSIONS IN LAMINAR FLOW

BY ARNOLD AXON

From the Wellcome Chemical Works, Dartford, Kent

Received June 19, 1957

A microscope cell has been made to examine semi-solid oil-in-water emulsions while flowing in a thin layer. Unautoclaved and autoclaved emulsions from cetyl alcohol, sodium lauryl sulphate, liquid paraffin with and without bentonite show differences in the dispersion of the globules. Those in the unautoclaved emulsions are mainly associated, "flocculated", into loose clusters in which each globule is free to move independently of its neighbours. Composite masses of disperse phase, called agglomerates, are formed in emulsions prepared from a pre-formed "emulsifying wax" which are absent from emulsions formed by adding sodium lauryl sulphate to the aqueous phase. A compact type of floccule occurs in the autoclaved emulsions containing bentonite. The globules are surrounded by a hydrated layer of bentonite which restricts their movement and provides a mechanical barrier against deformation. Rigidity is supplied by association of the hydrated bentonite particles in a network to form a gel. The globules in the autoclaved emulsions without bentonite are readily deformed and show complete freedom of movement with no association into clusters.

In a previous paper¹ semi-solid emulsions were shown to have anomalous viscous behaviours which can be distinguished in a complete consistency curve determined on a rotational viscometer. Subsequently it was suggested² that the change from shear-rate thinning to uniform plastic flow, occurring when an emulsion containing bentonite was autoclaved, depended on the state of dispersion of the globules.

This paper records the differences in microscopical appearance of semi-solid oil-in-water emulsions prepared from liquid paraffin, sodium lauryl sulphate, cetyl alcohol with and without bentonite. The emulsions in motion are examined as a thin film and the observed microscopical differences are related qualitatively with the different anomalous viscous behaviours and the general macroscopic appearances. The presence of groups of globules (floccules or clusters) and of complex structures (agglomerates) which affect the appearance and consistency of the emulsions, and which are unrecognised in a static preparation, can be readily detected while the preparation is in motion. A microscope cell unit has proved valuable for the examination of emulsions of high disperse phase concentration. It is suitable for both direct and dark ground illumination.

The State of Dispersion of the Disperse Phase

The dispersion of fine particulate matter in a fluid may lead to the formation of small groups of particles. Pryce-Jones³ found that the state of flocculation of the particles determined the macroscopical flow properties of paint and the ease of re-distribution of the sediment after storage.

The degree of dispersion of the globules in concentrated emulsions has received little study, although clustering of the globules has been postulated to explain variation in the gravity creaming of milk⁴ and rubber latex^{5,6}, and the increase in viscosity of dairy cream which occurs in the re-bodying process⁷, and also the increase in consistency caused by homogenisation of oil-in-water emulsions⁸. Clustering of globules is known to occur in fluid emulsions^{9,10}, and its extent assessed in a roughly quantitative way by measuring both the rate of creaming and the volume of the cream formed with time¹¹.

The Emulsion Globule Membrane

The existence of an emulsion membrane in milk was postulated by Ascherson in 1840 in the "Haptogen" membrane theory¹², and confirmed microscopically after staining^{13,14} and also by electron photomicrographs^{17,18}. This membrane has been isolated and shown to be a combination of protein and phospholipid^{19,20}. Elkes and others²¹ have demonstrated the orientation of protein and phospholipid at the oil:water interface. A similar membrane is postulated for rubber latex²². Emulsions with casein²³ and saponin²⁴ as emulsifying agents also show membranes. Many finely divided powders will orientate at the oil:water interface and act as emulsifying agents by forming a mechanical barrier which inhibits the coalescence of globules²⁵⁻²⁷. Levich²⁸ has suggested that the adsorbed stabiliser forms an envelope of structured liquid around each globule of the disperse phase. Bancroft in 1913 put forward the first definite theory of the role of the "soluble" emulsifying agent at the oil:water interface as a film of two surface tensions, one on the water side and one on the oil side²⁹.

The Microscopy of Disperse Systems

Green³⁰ has pointed out some of the difficulties, such as distortion of the globules and the apparently regular polyhedral appearance of stiff concentrated emulsions when examined under the microscope and has suggested a method for obtaining a satisfactory preparation. Emulsions and suspensions have been examined microscopically to obtain size distribution data^{31,32} and to show the effect of freezing³³, electrical currents^{34,35} and flocculating agents³⁶. The flocculation of globules seem to have been confined to fluid preparations and dilutions of concentrated preparations in static mounts.

The rate of flow of disperse systems in capillary tubes has been used to determine the rheological properties of the dispersions^{37,38}. Green and Haslam³⁹ have used a narrow capillary tube for the direct microscopical measurement of the plastic viscosity and yield value of paints, but it gives little indication of the finer details of interparticle relations.

APPARATUS

A paper describing the use of an ultramicroscope for the detection of particles in smoke suggested a possible method for examining flowing emulsions⁴⁰.

THE RHEOLOGY OF OIL-IN-WATER EMULSIONS. PART II

The apparatus devised consists of a microscope cell which fits on the stage. A hypodermic syringe is attached to the perspex disc of the cell and provides a reservoir for the emulsion and a convenient means for propelling it. The cell unit (Fig. 1) consists of four parts: a pressure plate, a microscope cover glass, and a tunnelled perspex disc clamped together in a suitable metal fitment.

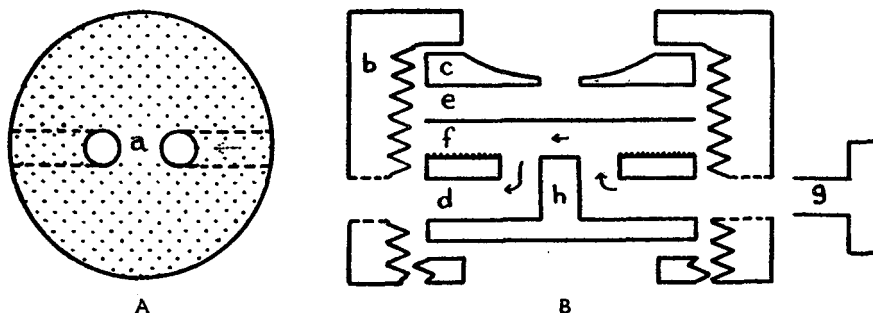


FIG. 1. Microscope cell unit. A. Plan of tunnelled gold-leafed perspex disc. B. Side elevation of cell unit.

- a*, Channel for laminar flow of the emulsion, from above, stippled area—gold leaf.
- b*, Brass union.
- c*, Pressure plate.
- d*, Channel.
- e*, Cover glass.
- f*, Gold leafed area.
- g*, Hypodermic syringe.
- h*, Perspex disc.

The tunnelled gold-leafed perspex disc. Two holes $\frac{1}{8}$ in. in diameter were bored diametrically opposite each other to within $\frac{1}{16}$ in. of the centre of a circular piece of perspex $\frac{1}{4}$ in. thick. Two similar holes approximately $\frac{1}{8}$ in. apart were bored from the upper surface of the perspex disc to complete the tunnelling. Gold leaf was applied to the upper surface of the disc except for the area between the two orifices. A microscope cover glass was placed on top of the gold-leafed perspex disc to enclose a small channel for the passage of the emulsion.

A metal pressure plate was placed over the cover glass to prevent bowing under the pressure of the flowing emulsion, which was viewed through a small central hole $\frac{1}{8}$ in. in diameter. The cell was encased in a brass union provided with holes contiguous with those in the perspex disc. The apparatus is compact, easily made, assembled, used, dismantled and cleaned.

EXPERIMENTAL

Oil-in-water emulsions were prepared by two methods to the following formulae: 1, 2, 3, and 4 and to the same formulae: I, II, III, and IV with the addition of 2 per cent of bentonite. An 8 per cent w/w suspension of bentonite was prepared in distilled water before the preparation of the emulsions. The characteristics are shown in Table I.

	1	2	3	4
Sodium Lauryl Sulphate B.P.	0.2	0.4	0.6	0.8 g.
Cetyl alcohol	1.8	3.6	5.4	7.2 g.
Liquid Paraffin B.P.	25.0	25.0	25.0	25.0 g.
Distilled Water B.P. to	100.0	100.0	100.0	100.0 g.

Method A. The cetyl alcohol was heated to 115°, to reproduce the conditions used in method B for the preparation of "emulsifying wax". Liquid paraffin was added to the hot cetyl alcohol and re-heated to 70°. The sodium lauryl sulphate was dissolved in hot water at about 70° and made up to weight at 70°, with water or bentonite suspension. The aqueous phase was added to the oil phase and the mixture homogenised with an immersion type homogeniser for one minute. The emulsion was poured immediately into a previously warmed screw capped jar which was sealed with a white rubber closure and a metal screw cap. The jar was slowly rotated on rollers while cooling to 25°, when the emulsion was distributed in several jars and each was sealed as before.

Method B. An "emulsifying wax" was prepared as described for the B.P. preparation from cetyl alcohol and sodium lauryl sulphate. The sodium lauryl sulphate was dispersed in the melted cetyl alcohol at 95° and 4 g. of water added for each 100 g. of "emulsifying wax". The mixture was heated to 115°, with vigorous agitation until a translucent mass resulted. Liquid paraffin was added and the mixture heated to 70°. Sufficient distilled water or bentonite suspension at 70° was added to the mixture. The two phases were mixed and the emulsions prepared and packed as in Method A.

Subsequent Treatment of Emulsions

Heating to 115°. The jars of emulsion were autoclaved at 115° for 30 minutes and gently rotated on rollers while cooling to 25°. They were kept in a room maintained at 25° ± 1°.

MICROSCOPICAL APPEARANCE OF EMULSIONS

Unautoclaved emulsions prepared without bentonite. The unautoclaved emulsions prepared by methods A and B flowed steadily through the microscope cell on application of slight pressure. The main body of globules moved in several streams or lamellae in which they have freedom of movement. The emulsion continued to flow after withdrawal of the applied pressure and gradually slowed and ceased. While most of the globules were stationary, some movement of the smaller globules in minute channels was often then seen. This system is described as being "loosely flocculated".

Emulsions prepared by methods A and B were readily distinguished. Those from an "emulsifying wax" (method B) showed numerous large masses (Figs. 2b, 3a) which were absent in emulsions prepared by method A (Fig. 2a). Each mass moved as a unit and was sufficiently compact to resist disruption, although its form was modified by pressures from the

TABLE I
THE CHARACTERISTICS OF SEMI-SOLID OIL-IN-WATER EMULSIONS
Without bentonite (white coloured)

	Macroscopical characters			Rotational viscometer consistency		Microscopical characters	
	General appearance	Feel	Consistency	U_{300}^*	f_{300}^\dagger	Flow behaviour	Globules
<i>Unautoclaved</i> 4.A ..	Granular Glossy Wet	Wet and cold Glove-like Easily absorbed	Soft	4.20	853	Smooth lamellae Loosely flocculated	Readily distorted
4.B ..	Granular Grainy	Warm and greasy Not readily absorbed	Soft	4.20	1110	Smooth lamellae Loosely flocculated Presence of agglom- erates	Readily distorted
<i>Autoclaved</i> 4.A ..	Smooth	Wet and cold Glove-like Easily absorbed	Soft	4.13	744	Globules completely free Deflocculated	Readily distorted
4.B ..	Smooth Slightly grainy	Warm and greasy Not readily absorbed	Soft	4.79	866	Globules completely free Deflocculated	Readily distorted
With bentonite (cream coloured)							
<i>Unautoclaved</i> IV.A ..	Granular Glossy Wet	Wet and cold Glove-like Easily absorbed	Soft	5.09	1205	Smooth lamellae Loosely flocculated	Readily distorted
IV.B ..	Granular Grainy	Warm and greasy Not readily absorbed	Firm	4.27	1706	Smooth lamellae Loosely flocculated Presence of agglom- erates	Readily distorted
<i>Autoclaved</i> IV.A ..	Smooth	Wet and cold Glove-like Easily absorbed	Medium- firm	5.75	1462	Smooth lamellae Flocculated	Sheathed in a hydrated layer
IV.B ..	Smooth Slightly grainy	Wet Not readily absorbed	Medium- firm	5.60	1421	Smooth lamellae Flocculated	Sheathed in a hydrated layer

* U_{300} : The "plastic viscosity" for emulsions showing plastic flow, and the limiting viscosity for emulsions showing shear-rate thinning, determined at 200 r.p.m. (91.4⁻¹ sec.).
 † f_{300} : The yield value in dynes per sq. cm. for emulsions showing plastic flow, and the torque intercept for emulsions showing shear-rate thinning (determined at 200 r.p.m. (91.4⁻¹ sec.).

flowing globules. This mass did not appear to correspond to that of a cluster or of a multiple globule described by Seifriz⁴¹. It is described as "an agglomerate" and consisted of an apparently unorganised granular matrix in which individual globules and small groups of globules were present (Fig. 3b).

Unautoclaved emulsions containing bentonite. The unautoclaved emulsions containing bentonite were indistinguishable from those without bentonite. The bentonite particles presented no distinguishing characteristics.

Autoclaved emulsions without bentonite. The autoclaved emulsions prepared by methods A and B flowed readily through the microscope cell on application of a slight pressure. The globules moved with complete

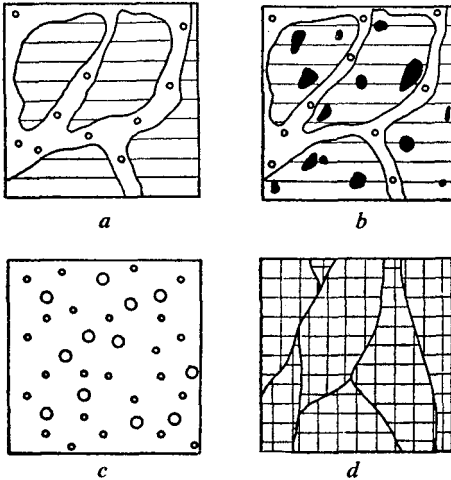


FIG. 2. Diagrammatic representation of the disperse phase in autoclaved and unautoclaved oil-in-water emulsions.

- a, Unautoclaved emulsion prepared either with or without bentonite by Method A.
- b, Unautoclaved emulsion prepared either with or without bentonite by Method B.
- c, Autoclaved emulsion prepared without bentonite by both methods.
- d, Autoclaved emulsion prepared with bentonite by both methods.

Horizontal lines, loose floccule of many hundred globules; squares, compact floccule of many hundred globules; open circles, globule of disperse phase; black areas, agglomerate.

an Agla micrometer syringe. When in flow, the globules moved in several streams or lamellae, and in each stream moved with the same relative velocity. The globules appeared to have little individual freedom of movement, the larger globules showing a readily observable boundary around them which prevented distortion and seemed to link the globules together. This system is described as "flocculated" (Figs. 2d, 3d).

DISCUSSION

Some of the macroscopical differences seen in the appearance and consistency of emulsions can be related qualitatively to the observed microscopical differences. The flow of oil-in-water emulsions in a thin

freedom without forming the distinct streams seen in the unautoclaved emulsions. The globules are fairly easily deformed. The flow continued after withdrawal of the applied pressure and gradually slowed and ceased. No agglomerates were present in either of the autoclaved emulsions. This system is described as "deflocculated" (Figs. 2c, 3c).

Autoclaved emulsions containing bentonite. The autoclaved emulsions containing 2 per cent bentonite prepared either by methods A or B presented a marked resistance to flow and pressure was needed to begin flow. The flow ceased immediately on withdrawal of the applied pressure and a pulsating flow followed the intermittent application of pressure. A controllable rate of flow was satisfactorily achieved by using

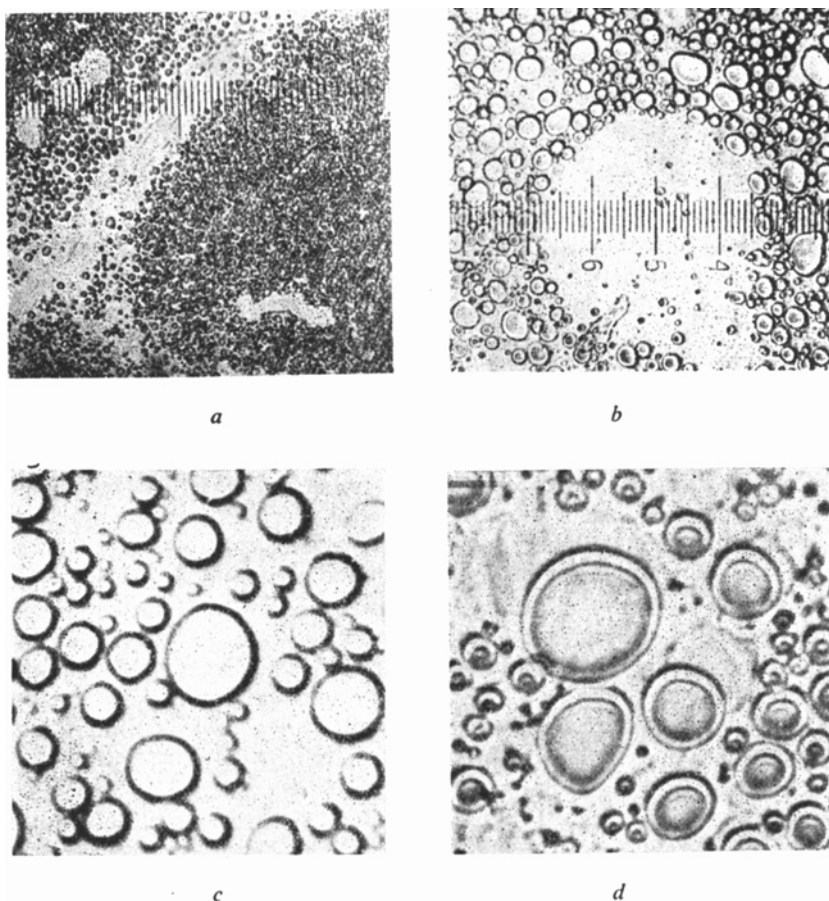


FIG. 3. Photomicrographs of:—

- a.* An unautoclaved emulsion (Method B). The presence of channels between the clusters in which isolated globules are freely moving and the presence of agglomerates within the clusters illustrate the characteristics typical of an unautoclaved emulsion prepared from an "emulsifying wax." (Compare Fig. 2*b*.) Magnification $\times 75$.
- b.* An agglomerate (Method B). Magnification $\times 300$.
- c.* An autoclaved emulsion prepared without bentonite (Compare Fig. 2*c*.) Magnification $\times 1000$.
- d.* An autoclaved emulsion prepared containing bentonite. Three of the globules show the hydrated layer of bentonite typical of autoclaved emulsions. Magnification $\times 1000$.

film shows differences in the disperse phase. The manner of movement of the globules and the relative pressure necessary to produce flow readily distinguishes different forms of globule association. Globules are associated into clusters in two different ways; the globules are free to move within the cluster (unautoclaved emulsions prepared with and without bentonite); the globules have little or no freedom within the cluster

(autoclaved emulsions prepared containing bentonite). The disperse phase can be obtained as globules which have complete freedom of movement (autoclaved emulsions prepared without bentonite); or dispersed as a composite mass, an agglomerate (unautoclaved emulsions prepared from an "emulsifying wax").

The "flocculation" of the globules in the unautoclaved emulsions is loose. Each globule is readily deformed and is free to move independently of its neighbours. Most of the globules are flocculated in this way, but other globules in the emulsion are completely free to move. This emulsion appears therefore as a heterogeneous dispersion of fairly dense floccules separated by a more fluid region in which the globules are free to move. On visual examination, these differences are recognised and described as "granularity".

A more compact type of flocculation of globules occurs in the autoclaved emulsions containing bentonite. The globules are restricted in their movement by a distinct zone which provides a mechanical buffer against deformation and a barrier to the approach of other globules. The floccules move as distinct streams or lamellae which are not separated by a more fluid region. This homogeneous dispersion produces an even scattering of light and is seen as a smooth and glossy emulsion.

The globules in the autoclaved emulsions prepared without bentonite are completely dispersed. All the globules show complete freedom of movement and are readily deformed. Since the globules are too small to be recognised by the eye as discrete units, the dispersion appears homogeneous and this produces an even scattering of light; it is a smooth and glossy emulsion.

The use of an "emulsifying wax" gives emulsions which show a striking microscopical appearance readily distinguished from unautoclaved emulsions prepared by method A. Numerous masses described as agglomerates are present. In the freshly prepared emulsions the matrix of the agglomerate appears to be of an unorganised granular nature, which in the aged becomes crystalline. The agglomerates are thought to be mainly composed of higher fatty alcohol. The presence of a large number of agglomerates increases the heterogeneity of the emulsion and gives a fine "grainy" appearance and a matt surface to the emulsion.

The consistency of an emulsion is a measure of the internal friction or its resistance to flow. Three factors concerned are, the resistance offered by the individual globules, the resistance offered by the clusters of globules, and the resistance offered by the agglomerates. The unautoclaved emulsions show an easy deformation of both the globules and floccules during flow; this would be expected therefore to produce a decrease in consistency with increasing rate of shear, namely shear-rate thinning. The presence of agglomerates and of the fine granular or colloidal dispersion of the cetyl alcohol in the emulsions prepared from an "emulsifying wax", would be expected to produce a noticeably thicker consistency. The emulsions would be expected to be slightly stiffer when prepared containing bentonite.

THE RHEOLOGY OF OIL-IN-WATER EMULSIONS. PART II

From the microscopical appearance of the autoclaved emulsions, those containing bentonite would be expected to offer the greatest resistance to flow. Deformation of the individual globules is prevented by a mechanical barrier and the free flow of the clusters is hindered by their compactness. The flow behaviour will show a distinct break when sufficient pressure has been applied to cause the rigidly held clusters to flow. Determination of a flow curve with the rotational viscometer, previously reported¹, does show a distinct yield value, before true flow begins. The bentonite particles appear to have become orientated at the oil: water interface during autoclaving to form a hydrated matrix around the oil globules. This matrix behaves as a bentonite gel and therefore imparts rigidity to the emulsion, changing the flow properties from shear-rate thinning to plastic flow.

The autoclaved emulsions prepared without bentonite would be expected to show the least resistance to flow because all the globules are readily deformed and completely free to move.

Acknowledgement. The author thanks Mr. M. H. Bennie of The Wellcome Chemical Works for his craftsmanship.

REFERENCES

1. Axon, *J. Pharm. Pharmacol.*, 1954, **6**, 830.
2. Axon, *ibid.*, 1956, **8**, 762.
3. Pryce-Jones, *J. Oil Col. Chem. Ass.*, 1934, **17**, 305.
4. Dunkley and Sommer, *Wis. Agr. Exptl. Sta. Res. Bull. No. 151*, 1944.
5. Schmidt and Kelsey, *Industr. Engng Chem.*, 1951, **43**, 406.
6. Twiss and Carpenter, *Proc. Rubber Tech. Conf.*, Paper No. 60, 81, 1938.
7. Bergmann and Svedberg, U.S. Patent 1,944,541 (1934).
8. Buglia, *Kolloid Z.*, 1907, **3**, 355.
9. Reid, *Missouri Agr. Exptl. Sta. Res. Bull. No. 100*, 1927.
10. Powis, *Z. physik Chem.*, 1914, **89**, 186.
11. Cockbain, *Trans. Far. Soc.*, 1952, **48**, 185.
12. Ascherson, *Arch. Anatomie, Physiologie*, p. 44, 1840; see translation *The Foundations of Colloid Chemistry*, Hatschek, p. 13, Benn, 1925.
13. Storch, *Analyst*, 1897, **22**, 197.
14. Voltz, *Pflug. Arch ges Physiol.*, 1904, **102**, 373, through ref. 42.
15. Bauer, *Biochem. Z.*, 1911, **32**, 362, through ref. 42.
16. Schmid, *Schweiz Milchztg.*, 1924, **50**, 17, through ref. 42.
17. Schwarz, *Neue Molkereiztg.*, 1947, **2**, 33, through ref. 42.
18. Hansson, *XII Int. Dairy Congress*, 1949, **2**, 27, through ref. 42.
19. Palmer and Weise, *J. Dairy Sci.*, 1933, **16**, 41.
20. Brunner, Duncan and Trout, *Food Res.*, 1953, **18**, 454.
21. Elkes, Frazer, Schulman and Stewart, *Proc. Roy. Soc.*, 1945, **184A**, 102.
22. Stamm and Kraemer, *J. Phys. Chem.*, 1926, **30**, 992.
23. Seifriz, *ibid.*, 1925, **29**, 746.
24. Ramsden, *Proc. Roy. Soc.*, 1903, **72**, 156.
25. Pickering, *J. chem. Soc.*, 1907, 2001.
26. Scarlett, Morgan and Hildebrand, *J. Phys. Chem.*, 1927, **31**, 1566.
27. Whipp and Bernhardt, Symposium on particle size analysis, *Trans. Inst. Chem. Eng. Supplement*, 1947, **25**, 104.
28. Levich, *Doklady Akad Nauk SSSR.*, 1955, **103**, 453, through *C.A.*, 1956, **30**, 3842i.
29. Bancroft, *J. Phys. Chem.*, 1913, **17**, 514.
30. Green, *Industrial Rheology and Rheological Structures*, 1st Ed., Chapman and Hall, London, 1949.
31. Cooper, *J. Soc. chem. Ind.*, 1937, **56**, 447T.
32. Lucas, *Industr. Engng Chem.*, 1938, **30**, 146.
33. Rochow and Mason, *ibid.*, 1936, **28**, 1296.

34. Lucas, *ibid.*, 1942, **34**, 1371.
35. Howe and Pearce, *Brit. J. Appl. Phys.*, 1955, **6**, 68.
36. Martin and Hermann, *Trans. Far. Soc.*, 1941, **37**, 30.
37. Scott Blair, Hening and Wagstaff, *J. Phys. Chem.*, 1939, **43**, 853.
38. Scott Blair, *A Survey of General and Applied Rheology*, 2nd Ed., Pitman, London, 1949.
39. Green and Haslam, *Industr. Engng Chem.*, 1925, **17**, 726.
40. Blacktin, *J. Soc. chem. Ind.*, 1938, **57**, 361.
41. Seifriz, *J. Phys. Chem.*, 1925, **29**, 744.
42. King, *The Milk Fat Globule Membrane*, 1st Ed., Commonwealth Agricultural Bureau, Farnham Royal, England, 1955.

DISCUSSION

The paper was presented by the AUTHOR.

MR. N. J. VAN ABBÉ (Loughborough). Modification due to reheating of emulsions not containing bentonite was often seen; this he thought was a matter of orientation of the hydrophilic-hydrophobic relation of the emulsifying agent. Was this taking part in the reported results? How much of the effect reported was due to the method of homogenisation or to ageing?

MR. HOBBS (Bristol). Had the behaviour of the emulsions in relation to changes in pH likely to be due to high temperature and time of heating been investigated?

MR. S. G. E. STEVENS (London). Were the effects the same for all kinds of bentonite?

MR. C. L. J. COLES (Greenford). Was not the granularity associated with solubility of cetostearyl alcohol in liquid paraffin at room temperature?

MR. A. E. DAVIS (Nottingham). Was there some special technique of applying gold leaf?

MR. H. D. RAPSON (Dorking). If a non-polar solvent were present, such as paraffin, there was inside the paraffin globule a system analogous to grease. This showed three or four phases which were temperature dependent and had hysteresis effect. If the solid phase was at equilibrium at the storage temperature, then it was probably a stable emulsion. If, however, it had been prepared at higher temperature, changes might take place at lower temperatures and the emulsion was probably unstable.

MR. N. J. VAN ABBÉ (Loughborough). Clarity might be obtained on some of the points by comparing the effect of sodium laurylsulphate emulsifying wax with non-ionic waxes.

MR. A. W. BULL (Nottingham) thought temperature was most important, particularly in relation to the lyophilic barriers. Had the jars been rotated on rollers at 45°, and then the emulsions allowed to cool without further agitation probably material differences would have been found.

MR. AXON replied he had not listed all changes in consistency. The point of interest was the concentration of the agglomerates which would

THE RHEOLOGY OF OIL-IN-WATER EMULSIONS. PART II

appear to increase with the concentration of the emulsifying wax. A change did take place in emulsions not containing bentonite, but it was not known whether this was due to change of orientation in the emulsifying agent at the interface. He had used only one homogeniser and had passed homogenised emulsions through a colloid mill where globule size had been reduced from $10\ \mu$ to 1 or $2\ \mu$ and the agglomerates had also been reduced in size. He had prepared emulsions by homogenisation only at 70° . The only difference after ageing of the emulsions was an observed increase in the crystalline nature of some of the disperse phase. He agreed that this was an important feature in maintaining stability. With a cetyl alcohol content of 10 per cent, globules with a marked crystalline nature were present but they were not observed in concentrations lower than 8 per cent. He did not know what happened to cetyl alcohol on homogenisation. There was little change in pH of emulsions containing bentonite which had been autoclaved. Only one bentonite had been used throughout and Mr. Coles was probably correct about the granularity. In making the cell units the Perspex was rubbed on paper and then applied to the gold leaf, which adhered for one operation. If gold size was applied to the Perspex the gold leaf adhered satisfactorily and could be used twenty times. The main point of the work was to be able to investigate a semi-solid emulsion a little better than was possible at the present time.