THE RHEOLOGY OF OIL-IN-WATER EMULSIONS

I. The Effect of Concentration of Constituents on Emulsion Consistency

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Received June 21, 1956

In a previous paper on the consistency of oil-in-water emulsions[1] it was stressed that semi-solid preparations show different kinds of anomalous viscous behaviour, and that to distinguish between them a complete consistency curve must be determined. Suitable consistency curves could be obtained with the variable speed rotational viscometer originally described by Green[2]. Oil-in-water emulsions of liquid paraffin and Emulsifying Wax B.P., both with and without bentonite were examined. Most exhibited shear-rate thinning, as might have been expected from the observations of many workers since Hatschek[3] first showed in 1911 that the viscosity of an emulsion varied with the rate of shear. The emulsions containing bentonite when autoclaved exhibited uniform plastic flow, an observation not previously reported. This has provided a basis for the comparison of this kind of emulsion, since uniform plastic flow can be characterised by two values, "U", the plastic viscosity in poises, and "f", the yield value, in dynes per sq. cm.

This paper records the preparation of oil-in-water emulsions of liquid paraffin, cetyl alcohol, sodium lauryl sulphate and bentonite and describes the effect on the consistency of changes in the concentration of each constituent of the emulsion. The work is part of a larger project which aims to show in quantitative units the significance of such expressive terms as "body", "podgy", "sloppy", and "stiff" which are incapable of conveying an accurate description of these physical characteristics. From a physico-chemical viewpoint, emulsions are complex systems and many variables are known to affect their ultimate viscous properties. A review of the literature on the consistency of emulsions gives an indistinct picture of the effect of the concentration of the constituents. This appears to be due to the difficulty of comparing emulsions which show shear-rate thinning[4] and the inadequacy of empirical methods of measurement for comparison purposes[5].

The Effect of the Concentration of the Emulsifying Agent

Several workers[6–10] have shown that different emulsifying agents will yield emulsions of markedly different consistencies for the same phase concentrations, but the effect of varying the concentration of the emulsifying agent has had few reports. Wilson and Parke[11] reported that the viscosity in a U tube viscometer, of mobile oil-in-water emulsions containing 70 per cent. of disperse phase increased with the concentration of the emulsifying agent. Toms[6] also showed, in a series of oil-in-water emulsions of equal phase ratio prepared from a variety of organic liquids as internal phase and a number of univalent soaps as emulsifiers, that an increase in the soap concentration produced in most cases an increase in viscosity, the magnitude of which varied with the nature of both the internal phase and the soap.

The Effect of the Volume Concentration of the Disperse Phase

In a variety of emulsions, an increase in the concentration of disperse phase up to a critical value causes only a slight increase in the viscosity[8,12–15]. Above this critical concentration there is a sudden rise to a maximum viscosity above which instability and inversion of the emulsion occurs. Leighton, Leviton and Williams[16], showed a maximum value followed by a minimum in the curve relating viscosity and fat concentration in ice-creams when measured by the sagging beam method.

The Effect of the Globule Size Distribution of the Disperse Phase

There are conflicting reports on the effect of the globule size distribution on the consistency of oil-in-water emulsions. Sibree[17] reported that the limiting viscosities of fine oil-in-water emulsions of liquid paraffin emulsified with 1 per cent. sodium oleate solution were "not very different from that of the coarse emulsions". Leviton and Leighton[18] found no change in viscosity when the globule diameter was reduced from 3μ to $0{\cdot}7\mu$ in emulsions of 10 and 30 per cent. milk fat. Lyttleton and Traxler[19] and Terry, Gabriel and Blott[20], however, working on asphaltic bitumen emulsions, showed that an emulsion of equal sized globules was more viscous than an emulsion having a large variation in globule size.

EXPERIMENTAL

Preparation of Emulsions

Emulsions were prepared to the following formulæ.

Liquid paraffin B.P.	15 to 40 g.
Sodium lauryl sulphate B.P.	0·2 to 1·0 ,,
Cetyl alcohol	2 to 10 ,,
Bentonite B.P.	0 to 3 ,,
Distilled water B.P.	100 ,,

The cetyl alcohol was melted in the liquid paraffin by heating in a hot air oven at 70° C. The sodium lauryl sulphate was dispersed in this oil phase. Sufficient water at 70° C. was added to make the total weight 70 g. The emulsion was then formed by homogenisation with an immersion type homogeniser[21] for one minute. The emulsion after cooling to room temperature, was distributed in several pots and each was diluted with water or a bentonite suspension to give concentrations of bentonite from 0 to 3 per cent. in the finished product and mixed intimately. These final emulsions were then transferred to screw-capped jars and sealed with efficient white rubber wad closures.

TABLE I

THE EFFECT OF CONCENTRATION OF CONSTITUENTS ON THE CONSISTENCY OF AUTOCLAVED OIL-IN-WATER EMULSIONS

Constituent held constant Percentage w/w	Principal constituent varied Percentage w/w	Subsidiary constituents varied Percentage w/w	Effect on the limiting value of the viscosity at 25° C. Determined at $91 \cdot 4^{-1}$ sec. (200 r.p.m.)	Effect on the value of the torque intercept or yield value at 25° C. Determined at $91 \cdot 4^{-1}$ sec. (200 r.p.m.)
Liquid paraffin 25	Bentonite 0 to 3	Sodium lauryl sulphate 0·2 to 1·0 Cetyl alcohol 2 to 10	Directly proportional to the bentonite content Figure 1. Table II	Directly proportional up to 2 per cent. bentonite, but tends to increase exponentially above 2 per cent. bentonite at the higher concentrations of sodium lauryl sulphate and cetyl alcohol Figure 2. Table II
Liquid paraffin 25	Sodium lauryl sulphate 0·2 to 1·0	Bentonite 0 to 3 Cetyl alcohol 2 to 10	Directly proportional to the sodium lauryl sulphate content Table II	Directly proportional to the sodium lauryl sulphate content at 2, 4 and 6 per cent. cetyl alcohol. Directly proportional above 0·4 per cent. sodium lauryl sulphate at 8 and 10 per cent. cetyl alcohol Table II
Liquid paraffin 25	Cetyl alcohol 2 to 10	Sodium lauryl sulphate 0·2 to 1·0 Bentonite 0 to 3	Increases exponentially with the cetyl alcohol content Figure 3. Table II	Increases exponentially with the cetyl alcohol content Figure 4. Table II
Cetyl alcohol 6	Bentonite 0 to 3	Sodium lauryl sulphate 0·2 to 0·8 Liquid paraffin 15 to 40	Directly proportional to the bentonite content Table III	Directly proportional to the bentonite content Table III
Cetyl alcohol 6	Sodium lauryl sulphate 0·2 to 0·8	Bentonite 0 to 3 Liquid paraffin 15 to 40	Directly proportional to the sodium lauryl sulphate content Table III	Directly proportional to the sodium lauryl sulphate content Table III
Cetyl alcohol 6	Liquid paraffin 15 to 40	Sodium lauryl sulphate 0·2 to 0·8 Bentonite 0 to 3	Directly proportional to the liquid paraffin content Figure 5. Table III	Increases exponentially with the liquid paraffin content Figure 6. Table III

TABLE II

AUTOCLAVED EMULSIONS CONTAINING 25 PER CENT. LIQUID PARAFFIN AND VARYING AMOUNTS OF SODIUM LAURYL SULPHATE AND BENTONITE

Sodium lauryl sulphate per cent.	0·2		0·4		0·6		0·8		1·0	
	U_{200}*	f_{200}†	U_{200}	f_{200}	U_{200}	f_{200}	U_{200}	f_{200}	U_{200}	f_{200}
Bentonite per cent.	Emulsions containing 10 per cent. cetyl alcohol									
0	5·86	1036	5·97	1665	6·36	1717	6·56	1834	6·67	1912
1	6·26	1178	6·49	1706	7·10	2185	7·41	2263	7·73	2361
2	6·93	1110	8·05	1990	8·47	2419	8·90	2458	9·53	2692
3	8·05	1581	9·06	2153	9·22	2848	9·32	3278	9·96	3844
	Emulsions containing 8 per cent. cetyl alcohol									
0	4·05	799	4·05	975	4·72	1218	4·42	1205	4·79	1259
1	4·17	812	4·94	1178	5·09	1286	5·09	1354	5·16	1571
2	5·01	907	5·60	1367	5·75	1598	6·34	1706	6·04	1923
3	5·68	1137	6·19	1760	7·00	1841	7·00	2029	7·20	2966
	Emulsions containing 6 per cent. cetyl alcohol									
0	2·96	415	3·11	493	3·01	549	3·01	618	3·29	793
1	3·01	337	3·04	484	3·46	636	3·46	751	3·76	948
2	3·54	542	4·05	812	4·13	894	4·27	1083	4·42	1462
3	4·13	691	4·42	1029	4·72	1164	4·57	1340	4·72	1503
	Emulsions containing 4 per cent. cetyl alcohol									
0	1·49	184	1·52	228	1·61	240	1·68	249	1·61	256
1	1·78	203	1·78	256	1·76	260	1·91	300	2·16	350
2	2·08	268	2·11	309	2·31	355	2·61	429	2·56	438
3	2·58	387	2·58	415	2·84	558	2·78	544	2·96	609
	Emulsions containing 2 per cent. cetyl alcohol									
0	0·83	86	0·85	88	1·05	124	1·11	129	1·14	171
1	1·04	92	1·14	118	1·21	131	1·28	157	1·19	191
2	1·18	129	1·28	157	1·46	187	1·62	194	1·76	230
3	1·35	157	1·66	212	1·77	233	1·87	235	1·88	237

* U_{200} .. The plastic viscosity "U" in poises determined at 200 r.p.m. ($91{\cdot}4^{-1}$ sec.)
† f_{200} .. The yield value "f" in dynes per sq. cm., determined at 200 r.p.m. ($91{\cdot}4^{-1}$ sec.)

Subsequent Treatment of Emulsions

The jars of the different emulsions were subjected to two different heat treatments in order to make uniform the air content and the rate of cooling, and to study the changes in consistency due to autoclaving.

Heating at 70° *C.* The jars of emulsion were immersed for 30 minutes in a water bath thermostatically controlled at 70° C.

Heating at 115° *C.* The jars of emulsion were autoclaved at 115° C. for 30 minutes.

Narayanaswamy and Watson[22] and Sibree[17] have stated that the incorporation of small amounts of air can give misleading values for the consistency of an emulsion. Between 2 and 10 ml. of air per 100 g. was found to be incorporated as minute air bubbles in the more viscous emulsions during their preparation. All, however, were sufficiently fluid at 70° C. to enable the air bubbles to rise, yielding emulsions of negligible air content, shown by tests on some by the method described by Blagg[23].

TABLE III

AUTOCLAVED EMULSIONS CONTAINING 6 PER CENT. CETYL ALCOHOL AND VARYING AMOUNTS OF LIQUID PARAFFIN AND BENTONITE

Liquid paraffin per cent.	15		20		25		30		40	
	U_{200}	f_{200}	U_{200}	f_{200}	U_{200}	f_{200}	U_{200}	f_{200}	U_{200}	f_{200}
Bentonite per cent.	Emulsions containing 0·8 per cent. sodium lauryl sulphate									
0	2·10	481	2·65	541	2·84	711	3·68	894	5·05	1422
1	2·55	596	2·80	731	3·68	880	4·20	1124	5·23	2153
2	2·80	704	3·58	968	4·05	1083	4·72	1632	7·52	3142
3	3·35	907	3·79	1090	4·90	1435	5·85	2004	8·05	3941
	Emulsions containing 0·4 per cent. sodium lauryl sulphate									
0	1·92	372	2·29	508	2·84	697	3·58	927	4·72	1313
1	2·28	433	2·80	596	3·68	860	4·05	934	5·16	1557
2	2·61	487	3·28	738	3·96	887	4·64	1124	5·45	1652
3	3·09	623	3·68	853	4·57	1151	5·23	1415	*	*
	Emulsions containing 0·2 per cent. sodium lauryl sulphate									
0	2·32	345	2·73	399	3·09	528	3·58	718	*	*
1	2·28	311	2·65	345	3·32	487	3·91	609	*	*
2	2·51	359	3·05	521	3·54	542	4·05	718	*	*
3	2·76	474	3·54	596	3·91	704	4·72	927	*	*

* These emulsions could not be prepared by the dilution method due to instability of the primary emulsion.

Storage. The emulsions were kept at room temperature before the viscosity determinations which were carried out within five days of their preparation.

Determination of the Viscosity Curve

Two viscosity curves were drawn from measurements made at 25° C., on a rotational viscometer in which the rate of shear and torque, expressed in basic fundamental units, are calculated from the dimensions of the instrument.

1. Emulsions containing 25 per cent. liquid paraffin, from 2 to 10 per cent. cetyl alcohol, from 0·2 to 1·0 per cent. sodium lauryl sulphate, and from 0 to 3 per cent. bentonite.
2. Emulsions containing from 15 to 40 per cent. liquid paraffin, 6 per cent. cetyl alcohol, from 0·2 to 0·8 per cent. sodium lauryl sulphate, and from 0 to 3 per cent. bentonite.

RESULTS

The results reported in this paper have been confined to those obtained from autoclaved emulsions. Autoclaved emulsions containing 25 per cent. of liquid paraffin, from 2 to 10 per cent. of cetyl alcohol, from 0·2 to 1·0 per cent. of sodium lauryl sulphate, and from 1 to 3 per cent. of bentonite, showed thixotropic uniform plastic flow, as did the autoclaved emulsions containing 6 per cent. cetyl alcohol, from 15 to 40 per cent. liquid paraffin, from 0·2 to 0·8 per cent. sodium lauryl sulphate and from 1 to 3 per cent. of bentonite. All the autoclaved emulsions containing 0 and 0·5 per cent. of bentonite showed thixotropic shear-rate thinning. The principal results of the variation of concentration of constituents on

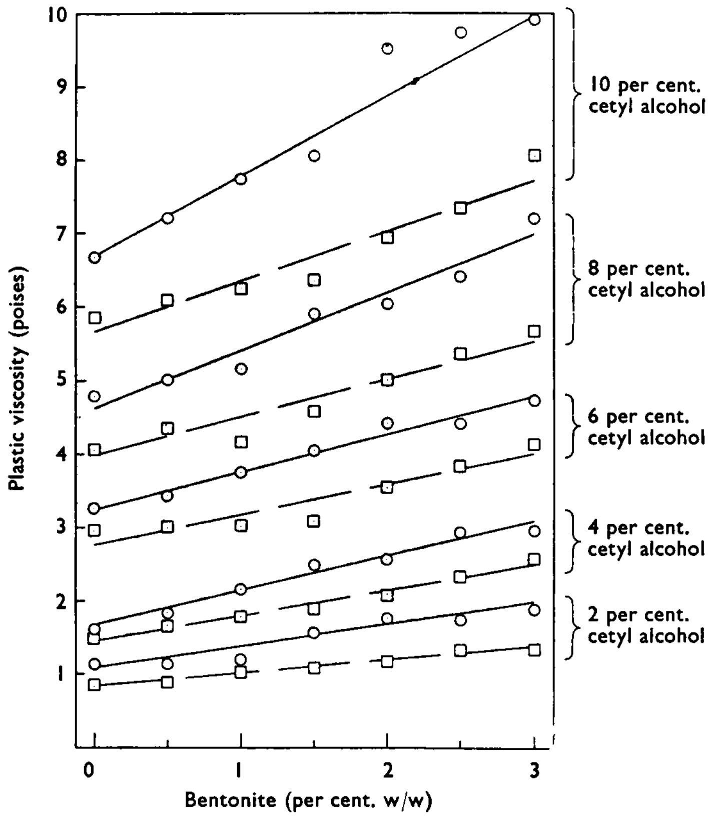

FIG. 1. The effect of the concentration of bentonite on the plastic viscosity of autoclaved oil-in-water emulsions containing 25 per cent. liquid paraffin and varying concentrations of cetyl alcohol and sodium lauryl sulphate.

○——○ 1·0 per cent. Na lauryl sulphate.
—□— 0·2 ,, ,, ,, ,,

emulsion consistency are summarised in Table I and in Figures 1 and 2. The quantitative values obtained for the plastic viscosity and yield value are shown in Tables II and III and Figures 1 to 6.

All the emulsions heated to 70° C., showed thixotropic shear-rate thinning. It was found that the quantitative values obtained for the limiting value of the viscosity, and the value of the torque intercept, followed an essentially similar but less distinct pattern to those shown by the autoclaved emulsions.

DISCUSSION

The values of the plastic viscosity and yield value for autoclaved emulsions confirm the generally observed "rule of thumb".

1. It is easier to increase the consistency of an oil-in-water emulsion by increasing the amount of the oil than by increasing the primary emulsifying agent sodium lauryl sulphate. Thus in Figures 1, 2, 5 and 6

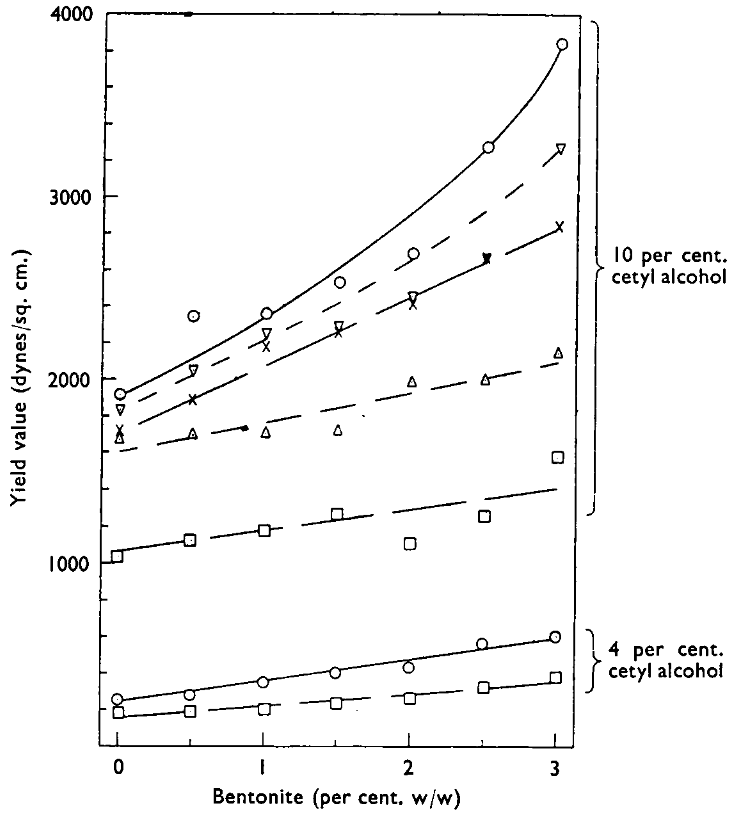

FIG. 2. The effect of the concentration of bentonite on the yield value of autoclaved oil-in-water emulsions containing 25 per cent. liquid paraffin and varying concentrations of cetyl alcohol and sodium lauryl sulphate.

○——○ 1·0 per cent. Na lauryl sulphate
-▽- — 0·8 ,, ,, ,, ,,
——× 0·6 ,, ,, ,, ,,
— —△ 0·4 ,, ,, ,, ,,
—□— 0·2 ,, ,, ,, ,,

only small increases in the plastic viscosity and yield value are obtained with a several fold increase in the sodium lauryl sulphate content, while large increases are obtained for increases in cetyl alcohol or liquid paraffin.

2. A greater increase in consistency can be obtained from small increments in the cetyl alcohol content than from similar increments in the liquid paraffin content. It is seen from Figures 3 and 4 that a marked increase in viscosity is coupled with a marked increase in yield value for small increases in the cetyl alcohol content, whereas in Figures 5 and 6, similar increases in the liquid paraffin content show noticeably smaller increases in both the viscosity and yield value.

Some encouraging signs suggest that some of the qualitative terms which are in current use to describe the consistency of an emulsion can be accurately interpreted in quantitative terms. The most important general impression which the author observed during this study was the relation of yield value to the qualitative description "body". This is particularly noticeable in the two series of emulsions containing increasing concentrations of (*a*) sodium lauryl sulphate and (*b*) bentonite, where the increase in consistency is mainly due to the increase in yield value. The yield

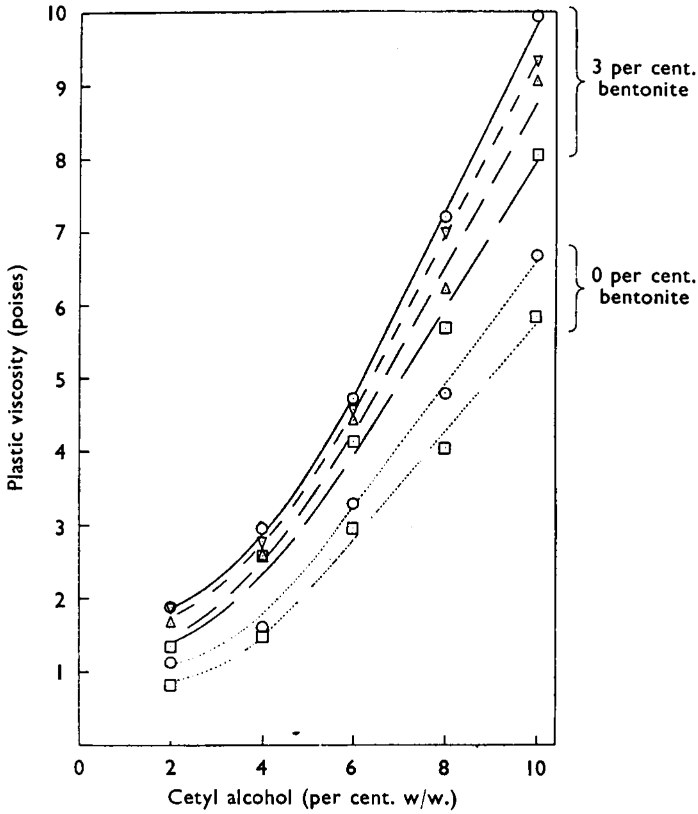

FIG. 3. The effect of the concentration of cetyl alcohol on the plastic viscosity of autoclaved oil-in-water emulsions containing 25 per cent. liquid paraffin and varying concentrations of bentonite and sodium lauryl sulphate.

3 per cent. bentonite	*0 per cent. bentonite*
⊙——⊙ 1·0 per cent. Na lauryl sulphate	⊙....⊙ 1·0 per cent. Na lauryl sulphate
▽——▽ 0·8 „ „ „ „	⊡....⊡ 0·2 „ „ „ „
△——△ 0·4 „ „ „ „	
⊡——⊡ 0·2 „ „ „ „	

value is that force in dynes per sq. cm. which must be applied before streamline flow commences. As the yield value increases, the resistance to deformation increases, and it is this *rigidity at rest* namely yield value which appears to be comparable to the description "body" of the preparation. The qualitative expressions used may however infer differing phenomena to different investigators. Consideration of the effect of

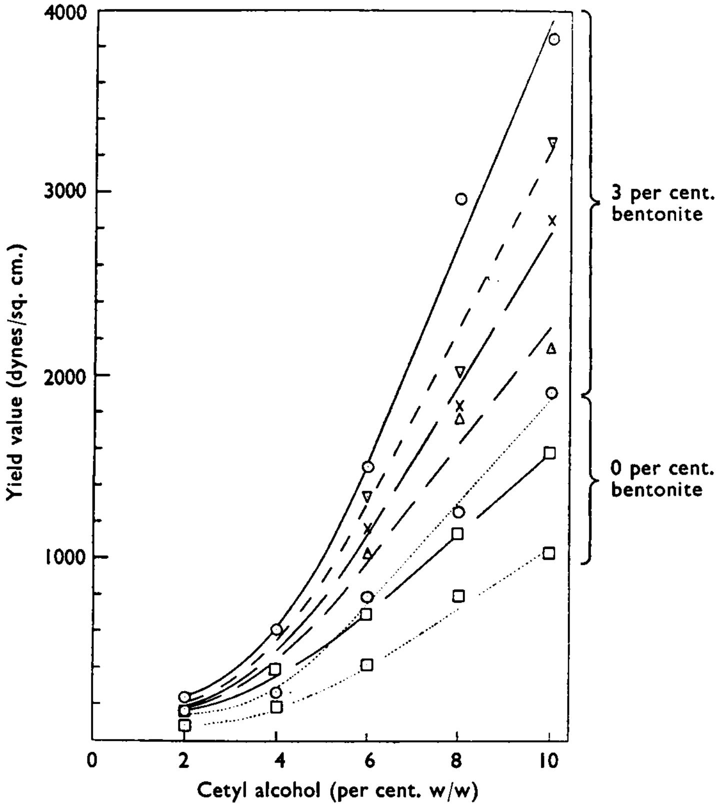

FIG. 4. The effect of the concentration of cetyl alcohol on the yield value of autoclaved oil-in-water emulsions containing 25 per cent. liquid paraffin at varying concentrations of bentonite and sodium lauryl sulphate.

3 per cent. bentonite

⊙——⊙ 1·0 per cent. Na lauryl sulphate
▽——▽ 0·8 " " " "
×——× 0·6 " " " "
△——△ 0·4 " " " "
⊡——⊡ 0·2 " " " "

0 per cent. bentonite

⊙ . . . . ⊙ 1·0 per cent. Na lauryl sulphate
⊡ . . . . ⊡ 0·2 " " " "

other phenomena such as thixotropic breakdown with shear and with time, and the regain of the original consistency with time, will have to be investigated before the qualitative expressions are adequately characterised.

The literature review indicates that the question of globule size distribution cannot be ignored, nevertheless, this factor has not been studied in the present work. Great care has been taken to ensure a constant technique for the preparation of the emulsions studied and in view of this it has been assumed that any differences in the globule size distribution or the flocculation of the globules are a direct result of changes in the concentration of the constituents of the emulsion. When it is considered that the autoclaved emulsions with and without bentonite were prepared from the same primary emulsion to give two different types of flow behaviour, viz., shear-rate thinning and uniform plastic flow, it is suggested that a flocculation of the globules occurs and the manner and extent of flocculation is probably more important than the globule size distribution. It is suggested as a reasonable hypothesis that plastic flow can be explained by the formation of an interlinked structure of flocculated globules within a gel network and that in emulsions which exhibit shear-rate thinning, the globules have remained dispersed or have flocculated into loose but unconnected aggregates.

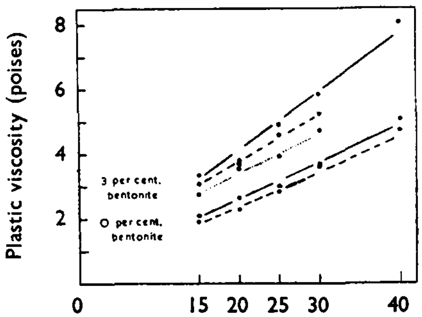

FIG. 5. The effect of the concentration of liquid paraffin on the plastic viscosity of autoclaved oil-in-water emulsions containing 6 per cent. cetyl alcohol, 0 and 3 per cent. bentonite and varying concentrations of sodium lauryl sulphate.

.... 0·2 per cent. Na lauryl sulphate
--- 0·4 ,, ,, ,, ,,
—— 0·8 ,, ,, ,, ,,

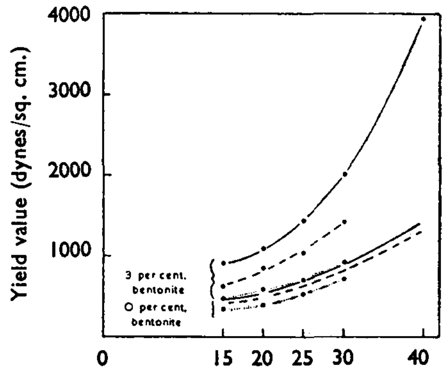

FIG. 6. The effect of the concentration of liquid paraffin on the yield value of autoclaved oil-in-water emulsions containing 6 per cent. cetyl alcohol, 0 and 3 per cent. bentonite and varying concentrations of sodium lauryl sulphate.

.... 0·2 per cent. Na lauryl sulphate
--- 0·4 ,, ,, ,, ,,
—— 0·8 ,, ,, ,, ,,

CONCLUSIONS

1. All the unautoclaved emulsions show thixotropic shear-rate thinning.

2. Over a wide range of concentration of each constituent, the autoclaved emulsions containing from 1 to 3 per cent. of bentonite show thixotropic plastic flow.

3. The autoclaved emulsions give values for plastic viscosity and yield value which conform to a definite pattern.

4. The plastic viscosity is directly proportional to the bentonite, sodium lauryl sulphate, and liquid paraffin content of the emulsion but increases exponentially with increase in the cetyl alcohol content.

5. The yield value is directly proportional to the bentonite content up to 2 per cent., but tends to increase exponentially above 2 per cent., at the higher concentrations of sodium lauryl sulphate and cetyl alcohol. The yield value is directly proportional to the sodium lauryl sulphate content for 2, 4, and 6 per cent. cetyl alcohol and directly proportional

to the sodium lauryl sulphate content above 0·4 per cent. for emulsions containing 8 and 10 per cent. cetyl alcohol.

The yield value increases exponentially with increase in the cetyl alcohol and liquid paraffin content.

SUMMARY

1. A variable speed rotational viscometer was used to determine the consistency of a series of oil-in-water emulsions containing varying concentrations of liquid paraffin, sodium lauryl sulphate, cetyl alcohol, and bentonite. The results amplify the present knowledge of the effect of the concentration of constituents on the consistency of an oil-in-water emulsion system.

2. The autoclaved emulsions containing from 1 to 3 per cent. bentonite exhibited thixotropic plastic flow over a wide range of concentrations of the other constituents.

3. All the unautoclaved emulsions exhibited thixotropic shear-rate thinning.

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DISCUSSION

The paper was presented by the AUTHOR.

MR. E. W. RICHARD (Upminster) referred to the statement on page 763 of the paper "There are conflicting reports on the effect of the globule size distribution on the consistency of oil-in-water emulsions" and said he took it that the author meant that in some cases, if the emulsions were homogenised and the globule size reduced and made more uniform, there was a thickening and in other cases a thinning. He confirmed that this happened from his own experience with different emulsions and emulsifying agents. He had found that the penetrometer, an instrument not commonly used in pharmacy but described in the Institute of Petroleum's handbook on standard methods, was a useful empirical tool for testing semi-solid emulsions.

DR. D. TRAIN (London) suggested that there was a discontinuity in the curves shown in Figures 1 and 2 at 1½ per cent. bentonite concentrations. Again, in Figure 5 he suggested the uppermost curve should be a shallow exponential and not a straight line.

MR. W. P. HUTCHINSON (Oxford) said that under the influence of irradiation the viscosity of certain oils changed.

MR. A. AXON, in reply, pointed out that he had quoted two authors who had stated that the effect of the emulsifying agent was most marked. He had kept to one primary emulsifying agent in the paper. He maintained that most of the effect of the emulsifying agent was to cause a difference in the aggregated state of the globules. The penetrometer was a one point instrument and had been considered previously. It gave an arbitary value which was very limited in its application for anything other than a routine control method. It would serve little purpose to attempt to distinguish between Newtonian flow, uniform plastic flow, sheer rate thinning, shear rate thickening, yield value and plastic viscosity. He had endeavoured to draw the best continuous line through the recorded points. If there is in fact a discontinuity at 1·5 per cent. bentonite concentration in Figures 1 and 2 then he was unable to give an explanation.