LIGHT SCATTERING FROM GEL-FORMING SYSTEMS DURING AND AFTER SETTING.

PART II. SODIUM STEARATE IN OCTYL ALCOHOL AND SODIUM STEARATE IN DECYL ALCOHOL: DEPOLARIZATION MEASUREMENTS

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In two recent communications, Sundaram (1953*a*, 1953*b*) has measured the depolarization factors of the light scattered in different directions from gel-forming systems of sodium oleate in xylene and has observed that the particles in a gel-forming system of sodium oleate in a non-polar solvent like xylene increase in size during gelation, thereby confirming the inferences drawn by previous investigators on the cooling curves (Prasad and Hattiangdi, 1945; Adarkar and Hattiangdi, 1945), viscosity (Prasad, Hattiangdi and Vishvanath, 1945; Hattiangdi and Adarkar, 1946) and syneresis (Prasad, Hattiangdi and Mathur, 1945; Prasad and Sundaram, 1951) of these systems. In the present investigation, a systematic study has been made of the depolarization factors of the light scattered in different directions during the cooling of gel-forming systems of sodium stearate in octyl and in decyl alcohols.

EXPERIMENTAL

A. Preparation of the gel-forming systems:—

Gel-forming solutions containing 0·6%, 1·0% and 1·5% of sodium stearate in octyl and in decyl alcohols were prepared in Pyrex glass test-tubes of internal diameter 1·5″, adopting the method described by Sundaram (1953*a*). When the temperature of the solution reached 130°C., the test-tube was removed from the oil-bath and transferred to the observation bath for the depolarization measurements.

B. Experimental arrangement and procedure:—

The experimental arrangement and procedure adopted for the depolarization measurements were exactly the same as the one used by Sundaram (1953*b*). All necessary precautions were taken to minimise errors, and the depolarization factors of the light scattered at 45° (forward), 67°, 90°, 112° and 135° to the direction of the incident beam were measured.

C. Measurement of the gelation temperatures:—

The gelation temperature of these gel-forming systems were measured in the usual manner (Sundaram, 1953*a*), using Fleming's method for noting the gelation point.

RESULTS

The values of the depolarization factors of the light scattered in different directions are given in Tables I to VI in which T represents the temperature at which measurements were made, t_g represents the temperature of gelation, C represents the percentage concentration of soap in the system, and ρ_u, ρ_v and ρ_h have their usual significance.

TABLE I

Sodium Stearate in Octyl Alcohol

$C = 0{\cdot}6\%$ $t_g = 40°C.$

T °C.	Angle of scattering 45°			Angle of scattering 67°			Angle of scattering 90°				Angle of scattering 112°			Angle of scattering 135°		
	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	$\Delta\rho_u$	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u
120	13·2	10·9	70·4	24·9	13·2	61·0	39·0	11·8	37·5	16·3	37·5	14·7	49·0	18·0	..	70·4
110	13·2	10·9	70·4	24·9	13·2	61·0	39·0	11·8	37·5	16·3	37·5	14·7	49·0	18·0	11·2	70·4
100	13·2	10·9	70·4	24·9	13·2	61·0	39·0	11·8	37·5	16·3	37·5	14·7	49·0	18·0	11·2	70·4
90	13·2	..	70·4	24·9	..	61·0	39·0	11·8	37·5	16·3	37·5	14·7	49·0	18·0	11·2	70·4
80	13·2	10·9	70·4	24·9	13·2	61·0	39·0	11·8	37·5	16·3	37·5	14·7	49·0	18·0	11·2	70·4
70	13·2	10·9	70·4	..	13·2	61·0	39·0	11·8	37·5	16·3	37·5	14·7	49·0	18·0	11·2	70·4
65	..	..	..	24·9	13·2	61·0	39·0	11·8	37·5	16·3	37·5	14·7	49·0	18·0	11·2	70·4
60	13·2	10·9	70·4	24·9	13·2	61·0	..	11·8	..	..	37·5	14·7	49·0	18·0	11·2	70·4
55	11·8	7·7	63·3	20·8	10·6	50·8	..	..	37·5	..	33·3	13·2	47·2	11·8	7·2	58·9
50	11·8	..	63·3	..	6·7	39·0	39·0	11·8	37·5	16·3	30·7	10·6	42·2	..	6·7	..
45	9·9	..	58·9	17·2	6·7	...	49·0	11·2	32·3	12·2	24·9	6·7	33·3	9·9	5·8	52·8
40	9·9	6·2	58·9	17·2	..	39·0	52·8	9·9	27·3	9·2	24·9	6·7	..	9·9	..	52·8
35	9·9	6·2	58·9	17·2	6·7	39·0	52·8	9·9	27·3	9·2	24·9	6·7	37·7	9·9	5·8	52·8

TABLE II

Sodium Stearate in Octyl Alcohol

$C = 1{\cdot}0\%$ $t_g = 40°C.$

T °C.	Angle of scattering 45°			Angle of scattering 67°			Angle of scattering 90°				Angle of scattering 112°			Angle of scattering 135°		
	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	$\Delta\rho_u$	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u
120	..	14·0	75·6	27·3	15·5	65·6	40·6	12·5	36·1	13·6	28·3	13·2	56·8	18·9	13·2	75·6
110	19·8	14·0	75·6	27·3	..	65·6	40·6	12·5	36·1	13·6	28·3	13·2	56·8	18·9	13·2	75·6
100	19·8	14·0	75·6	27·3	15·5	65·6	40·6	12·5	36·1	13·6	28·3	..	56·8	18·9	13·2	75·6
90	19·8	14·0	75·6	27·3	15·5	65·6	..	..	36·1	..	..	13·2	56·8	18·9	13·2	75·6
80	19·8	14·0	75·6	27·3	15·5	65·6	40·6	12·5	36·1	13·6	28·3	13·2	56·8	18·9	13·2	75·6
70	16·3	9·9	67·9	..	15·5	65·6	40·6	12·5	..	13·6	28·3	..	..	10·6	7·2	72·1
65	14·0	..	63·3	..	..	..	..	12·5	36·1	..	..	13·2	56·8	..	7·2	..
60	14·0	8·2	63·3	17·2	7·2	42·2	40·6	12·5	36·1	13·6	23·8	8·2	39·0	9·9	6·2	68·0
55	..	..	..	15·5	..	..	45·5	11·8	34·7	13·5	21·7	7·2	34·7	..	6·2	68·0
50	14·0	8·2	63·3	15·5	5·3	33·3	49·0	9·3	27·3	10·2	21·7	7·2	34·7	9·9	6·2	68·0
45	..	..	..	15·5	5·3	33·3	..	7·7	20·8	6·7	..	..	..	..	..	..
40	14·0	8·2	63·3	..	5·3	33·3	56·8	7·7	20·8	6·7	21·7	..	34·7	..	6·2	68·0
35	14·0	8·2	63·3	15·5	5·3	33·3	56·8	7·7	20·8	6·7	21·7	7·2	34·7	9·9	6·2	68·0

TABLE III

Sodium Stearate in Octyl Alcohol

$C = 1{\cdot}5\%$ $t_g = 63{\cdot}5°C.$

T °C.	Angle of scattering 45°			Angle of scattering 67°			Angle of scattering 90°				Angle of scattering 112°			Angle of scattering 135°		
	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	$\Delta\rho_u$	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u
120	11·8	7·2	65·6	21·7	11·2	49·0	43·8	13·2	37·5	14·1	27·3	13·2	54·8	18·0	14·0	81·0
110	11·8	7·2	65·6	..	11·2	49·0	43·8	13·2	37·5	14·1	..	13·2	54·8	..	14·0	81·0
100	11·8	7·2	65·6	21·7	..	49·0	43·8	13·2	37·5	14·1	27·3	13·2	54·8	18·0	14·0	81·0
90	11·8	7·2	65·6	21·7	11·2	49·0	43·8	13·2	37·5	14·1	27·3	13·2	54·8	18·0	14·0	81·0
80	11·8	7·2	65·6	21·7	11·2	49·0	45·5	11·8	32·3	11·1	27·3	13·2	54·8	18·0	..	81·0
75	11·8	7·2	..	18·9	8·8	45·5	..	..	..	..	21·7	8·2	39·0	..	14·0	..
70	..	4·5	..	14·0	4·9	34·7	50·9	8·8	23·8	7·7	20·8	7·2	36·1	15·5	11·2	72·9
65	7·7	4·5	58·9	..	..	34·7	54·8	7·2	..	..	..	..	..	15·5	11·2	72·9
60	7·2	3·8	52·8	..	4·1	29·5	..	..	23·8	..	..	4·9	30·7	..	6·2	66·5
55	7·2	3·8	52·8	13·2	4·1	29·5	58·9	6·2	15·5	3·8	..	..	..	11·2	6·2	63·3
50	..	..	..	..	..	..	58·9	6·2	15·5	3·8	17·2	4·9	30·7	11·2	6·2	63·3
40	7·2	3·8	52·8	13·2	4·1	29·5	58·9	6·2	15·5	3·8	17·2	4·9	30·7	11·2	6·2	..
35	7·2	3·8	52·8	13·2	4·1	29·5	58·9	6·2	15·5	3·8	17·2	4·9	30·7	11·2	6·2	63·3

TABLE IV

Sodium Stearate in Decyl Alcohol

$C = 0{\cdot}6\%$ $t_g = 53{\cdot}0°C.$

T °C.	Angle of scattering 45°			Angle of scattering 67°			Angle of scattering 90°				Angle of scattering 112°			Angle of scattering 135°		
	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	$\Delta\rho_u$	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u
120	11·2	6·7	58·9	20·8	9·3	50·9	47·2	11·8	32·3	11·1	22·7	..	52·8	22·7	..	52·8
110	11·2	6·7	58·9	20·8	9·3	50·9	47·2	11·8	32·3	11·1	22·7	10·6	52·8	22·7	10·6	52·8
100	11·2	6·7	58·9	20·8	9·3	50·9	47·2	11·8	32·3	11·1	22·7	10·6	52·8	22·7	10·6	52·8
90	11·2	6·7	58·9	20·8	9·3	50·9	47·2	11·8	32·3	11·1	22·7	10·6	52·8	22·7	10·6	52·8
80	11·2	6·7	58·9	20·8	9·3	50·9	47·2	11·8	32·3	11·1	..	10·6	52·8	..	10·6	52·8
70	11·2	6·7	58·9	20·8	9·3	50·9	47·2	10·6	..	11·1	22·7	10·6	..	22·7	10·6	..
65	11·2	6·7	58·9	..	..	..	..	10·6	28·3	9·1	..	..	..	..	..	..
60	..	4·9	..	16·3	6·2	42·2	47·2	..	28·3	..	18·0	6·2	39·0	18·0	6·2	39·0
55	8·2	4·1	50·9	15·5	4·5	32·3	58·9	8·8	24·9	8·7	..	6·2	39·0	..	6·2	39·0
50	6·2	3·1	49·0	15·5	4·5	32·3	61·0	6·2	19·8	8·1	15·5	4·9	32·3	15·5	4·9	32·3
45	6·2	3·1	49·0	..	..	..	65·6	4·9	17·2	7·8	15·5	4·9	32·3	15·5	4·9	32·3
40	..	..	..	15·5	4·5	32·3	65·6	4·9	17·2	7·8	15·5	4·9	32·3	15·5	4·9	32·3
35	6·2	3·1	49·0	15·5	4·5	32·3	65·6	4·9	17·2	7·8	..	4·9	32·3	..	4·9	32·3

TABLE V

Sodium Stearate in Decyl Alcohol

$C = 1{\cdot}0\%$ $t_g = 60°C.$

T °C.	Angle of scattering 45°			Angle of scattering 67°			Angle of scattering 90°				Angle of scattering 112°			Angle of scattering 135°		
	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	$\Delta\rho_u$	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u
120	13·2	10·6	78·3	29·5	8·8	36·1	34·7	12·5	34·7	12·2	..	13·2	58·9	15·5	..	78·3
110	13·2	..	78·3	29·5	8·8	36·1	34·7	12·5	34·7	12·2	29·5	13·2	58·9	..	11·2	78·3
100	..	10·6	..	29·5	8·8	36·1	34·7	12·5	34·7	12·2	29·5	13·2	58·9	15·5	11·2	..
90	13·2	10·6	78·3	29·5	8·8	36·1	34·7	12·5	34·7	12·2	29·5	13·2	58·9	15·5	11·2	78·3
80	13·2	10·6	78·3	27·3	7·2	30·7	34·7	12·5	34·7	12·2	29·5	13·2	58·9	..	..	78·3
75	..	..	78·3	..	..	..	34·7	12·5	34·7	12·2	20·8	..	..	15·5	11·2	78·3
70	..	10·6	78·3	18·9	3·8	21·7	34·7	12·5	34·7	12·2	20·8	6·7	39·0	..	11·2	78·3
65	13·2	10·6	78·3	..	2·8	19·8	34·7	..	34·7	..	14·7	4·5	30·7	15·5	11·2	..
60	9·9	6·2	58·9	15·5	2·8	19·8	36·1	7·7	26·0	11·7	14·7	4·5	30·7	11·8	8·2	72·9
55	9·9	..	..	..	..	19·8	39·0	7·7	22·7	10·2	..	..	30·7	8·2	..	67·9
50	8·8	4·5	49·0	..	..	..	42·2	6·2	20·8	9·0	14·7	4·5	30·7	8·2	5·3	..
45	..	..	..	15·5	2·8	19·8	45·5	5·8	18·0	7·1	14·7	..	30·7	..	..	..
35	8·8	4·5	49·0	15·5	2·8	19·8	45·5	5·8	18·0	7·1	14·7	4·5	30·7	8·2	5·3	67·9

TABLE VI

Sodium Stearate in Decyl Alcohol

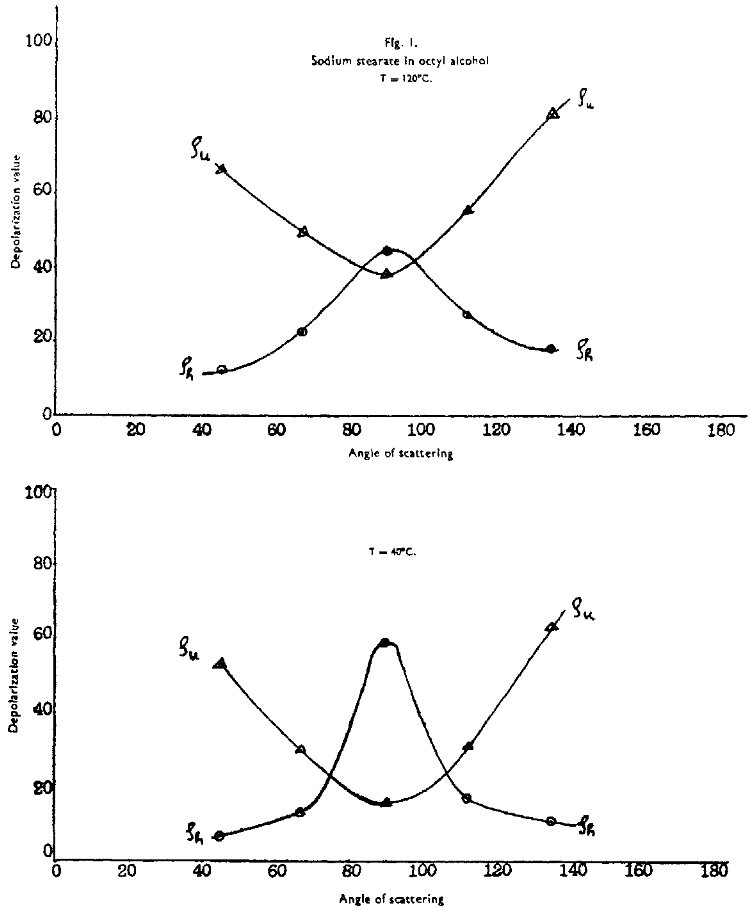
$C = 1{\cdot}5\%$ $t_g = 75°C.$

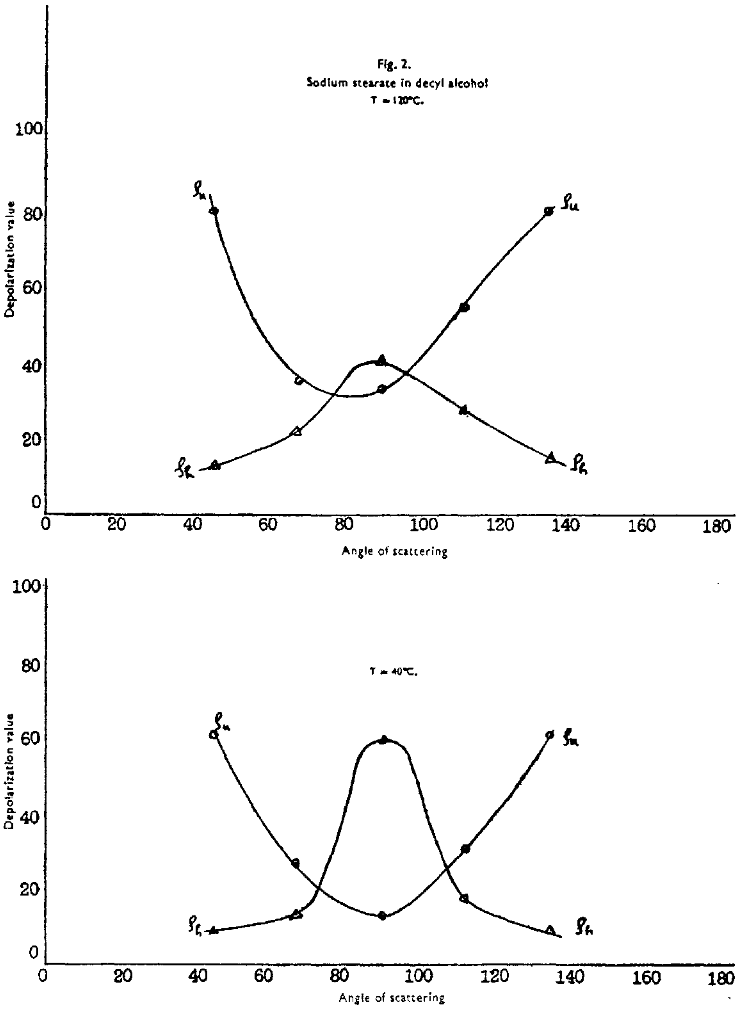
T °C.	Angle of scattering 45°			Angle of scattering 67°			Angle of scattering 90°				Angle of scattering 112°			Angle of scattering 135°		
	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u	$\Delta\rho_u$	ρ_h	ρ_v	ρ_u	ρ_h	ρ_v	ρ_u
120	13·2	9·9	81·0	21·7	7·2	34·7	40·6	13·2	33·3	10·0	27·3	13·2	54·8	14·7	10·6	81·0
110	13·2	9·9	..	21·7	7·2	34·7	40·6	13·2	33·3	10·0	..	13·2	54·8	14·7	10·6	81·0
100	13·2	9·9	81·0	21·7	7·2	34·7	40·6	13·2	33·3	10·0	27·3	13·2	54·8	14·7	10·6	81·0
90	13·2	9·9	81·0	14·7	7·2	..	47·2	9·9	26·0	8·6	27·3	13·2	54·8	..	10·6	81·0
80	10·6	7·2	67·9	13·2	3·8	32·3	52·8	8·2	21·7	6·6	27·3	9·9	54·8	14·7	10·6	..
75	9·9	7·2	..	..	3·1	27·3	54·8	7·2	19·8	6·4	21·7	8·2	39·0	11·8	7·2	67·9
70	9·3	5·8	61·0	13·2	3·1	27·3	58·9	5·8	14·7	3·7	20·8	7·2	36·1	..	5·8	..
60	9·3	..	61·0	13·2	..	27·3	60·3	4·9	12·5	3·2	..	4·9	30·7	9·3	5·8	61·0
50	..	5·8	61·0	..	3·1	..	..	4·9	..	..	17·2	..	30·7	9·3	5·8	61·0
40	9·3	5·8	..	13·2	..	27·3	60·3	..	12·5	..	17·2	4·9	..	..	5·8	..
35	9·3	5·8	61·0	13·2	3·1	27·3	60·3	4·9	12·5	3·2	17·2	4·9	30·7	9·3	5·8	61·0

Discussion of Results

It is seen from Tables I to VI that the values of ρ_u, ρ_v and ρ_h at any two angles of scattering situated symmetrically about the transverse direction (90°) are not the same, that is to say, the polarization of scattering is unsymmetrical with respect to the transverse direction.

The relations put forward by Ganesan (1924) and by Guinand and Tonnelat (1947) connecting the depolarization value with the angle of scattering are not found to hold good in the present investigation. The values of ρ_u and ρ_h have been plotted against the angle of scattering at two temperatures for one concentration of each system and the graphs obtained, shown in Figs. 1 and 2, clearly bring out the unsymmetrical nature of the polarization of scattering. It is seen that the graphs combine the features exhibited in the theoretical graphs of Krishnan (1938) for small anisotropic particles and for large spherical particles. This indicates that the particles in these systems are anisotropic and of appreciable size. Since the unsymmetrical nature of the graphs is more prominent at a higher temperature than at a lower one, it seems probable that the particles decrease in size during the cooling and gelation of these systems.





It is also seen from Tables I to VI that the values of ρ_h of the transversely scattered light are initially (at 130°C.) low and remain constant on cooling the systems up to a temperature a few degrees higher than the gelation temperature; on further cooling, however, the values increase over a short range of temperature and again become constant. The values of ρ_v of the transversely scattered light are initially low and remain constant till the systems are cooled to about the gelation temperature while the values of ρ_u are initially high and remain constant during this initial stage of cooling. On further cooling both the values of ρ_u and of ρ_v decrease for a while and again become constant. The increase in the value of ρ_h and the decrease in the value of ρ_v indicate that the particles decrease in size and in anisotropy during the cooling and gelation of these systems. The changes in the values of ρ_u are due to the changes in the values of ρ_v and ρ_h since the three depolarization factors are connected by the relation, $\rho_u = \frac{1+1/\rho_h}{1+1/\rho_v}$ (cf. Krishnan, 1935*a*, 1935*b*, 1938). The value of ρ_u can be split up into two parts, one due to the anisotropy of the particles and the other due to their size. The anisotropic part of ρ_u value is given, to a first approximation, by the factor, $\frac{2\rho_v}{1+\rho_v}$ and that due to size is given by $\Delta\rho_u$, where $\Delta\rho_u = \rho_u - \frac{2\rho_v}{1+\rho_v}$ (cf. Sivarajan, 1951). The values of $\Delta\rho_u$ have been calculated in the present case and are given in column 11 of Tables I to VI. The values of $\Delta\rho_u$ decrease during the cooling of the systems indicating that the size of the particles decreases during gelation. This supports the inference made on the basis of the changes in the values of ρ_h.

The observed results may be explained on the following considerations. It has been observed that in soap-water systems the soap does not go into molecular solution but forms clusters which are colloidal in nature (colloidal micelles). A similar phenomenon may be taking place in the polar solvents used in the present investigation and the soap may be forming colloidal clusters when dispersed in the polar solvents at the higher temperature. During the cooling of the soap-solvent systems, the forces holding the soap molecules in the form of clusters may be weakening and thereby causing the breakdown of the clusters. This will cause a reduction in the size of the particles. The broken-up particles may be getting solvated at the same time and, due to the decrease in the thermal energy of the system, the system sets to a gel.

The study of X-ray diffraction of soap solutions in polar solvents has shown that the soap particles are oriented with the hydrocarbon chains end to end and the polar group turned towards the solvent molecules. On cooling, the decrease in the thermal energy of the system probably causes an alteration in the orientation of the particles in such a manner that the 'effective size' of the particles decreases. This decrease in 'effective size' continues till the particles take up the solvent and the system sets to a gel. Once the systems have set to gels at their gelation temperatures, very little changes can take place in the orientation of the particles and hence the values of the depolarization factors attain constancy on further cooling.

A comparison of the Tables I to VI shows that the trend of the changes in the values of the depolarization factors is the same in systems of different concentrations. Further, in the case of transverse scattering, the values of $\Delta\rho_u$ at lower temperatures are distinctly higher in systems containing lower amount of soap, indicating that the 'effective size' of the particles is bigger in systems of lower concentrations than in those of higher ones. This may be due to the fact that the particles in systems of lower concentrations may be undergoing lesser changes in orientation than those in systems of higher concentrations. This view is supported by the fact that the final particles in systems of lower concentration show a higher value for ρ_v, that is, are more anisotropic, than the final particles in systems of higher concentration.

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Summary

The measurement of the depolarization factors, ρ_u, ρ_v and ρ_h, of the light scattered at different directions from gel-forming systems of sodium stearate in octyl and in decyl alcohols has shown that the particles in these systems are anisotropic and of appreciable size. The

changes taking place in the values of the depolarization factors of the transversely scattered light during the cooling of these systems have shown that the particles decrease in size and in anisotropy during gelation. The size of the final particles seems to be bigger in gels of lower concentrations than in those of higher ones. An attempt has been made to explain the observed results on the consideration of the behaviour of soap in polar solvents.

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