PHASE INVERSION IN TWO-PHASE LIQUID SYSTEMS

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New data on critical holdups of dispersed phase were measured at which the phase inversion took place. The systems studied differed in the ratio of phase viscosities and interfacial tension. A weak dependence was found of critical holdups on the impeller revolutions and on the material of contactor; on the contrary, a considerable effect of viscosity was found out as far as the viscosity of continuous phase exceeded that of dispersed phase.

In liquid extraction, a great interfacial area is achieved by intensive agitation of two immiscible liquid phases by dispersing one of them in the form of small droplets into the second, continuous phase. To reach a high efficiency of extraction, it is suitable that the continuous phase should be that one in which there is a greater resistance to mass transfer because it is not possible to ensure sufficiently intensive stirring inside the droplets. The reason for the choice of a certain type of dispersion can consist in different rate of coalescence in a settler or some specific requirements for the process, e.g., the measurement of concentration, pH or dosing the reagent. The operating conditions must be simultaneously chosen in such a way that type of dispersion should not be changed spontaneously.

If the holdup of some of the phases is very low, this phase is always the dipersed phase, and on the contrary, at high holdup, the phase is always the continuous one. Between these limiting values lies the hysteresis region in which both the types of dispersion may exist. It was found out[4] that at the beginning of agitating in this region of holdups, as the continuous phase is established that one in which the impeller is immersed. Unless the holdup of dispersed phase reaches the critical value, the type of dispersion is stable. The phenomenon during which the interchange of continuous phase for the dispersed phase takes place, and vice versa, is called the phase inversion.

The mechanism of phase inversion and the factors influencing the critical value of holdups have not hitherto been sufficiently known. The up-to-now published data on critical values of holdups of dispersed phase are only partial, and moreover, often considerably differ[1,2,7] for the same systems. There exists mutual discrepancy even among

the data expressing the qualitative effect of some factors influencing the critical holdups, e.g., stirring intensity[4,5,7] and interfacial tension[2,3].

In most cases[1 – 4,7,8], the values of critical holdups were measured up to 70% dispersed phase. In other works[5,6], analogously to this paper, substantially higher values exceeding even 90% were established.

In this work, the critical holdups of dispersed phase were measured for 17 pairs of mutually immiscible liquids differing in viscosities and interfacial tensions. The impellers of three different diameters and the settlers made of two kinds of materials were employed in this study.

EXPERIMENTAL

The experiments were carried out in an equipment which consisted of mixer and settler. The mixer bottom had inside dimensions 125 × 125 mm, the settler bottom has inside dimensions 125 × 345 mm. The baffle height was 150 mm, the wall height was 180 mm. All the vessel was covered with a cover in which an impeller with adjustable revolutions from 10 to 30 s^{-1} was built in. The impeller was exchangeable and was made of Teflon. The impellers of diameters 30, 40, and 70 mm were employed. The impeller was located in the height of 66 mm from the mixer bottom. The impeller was on the interface if the 50% holdup of phases was in the mixer. Two versions of this equipment were used. The material of which they were made was, in the first case, perspex, in the second case, the combination of glass and ATLAC resin.

Both phases were intensively agitated in the mixer for at least two hours before the experiment for their mutual saturation to take place. Simultaneously both the phases were pumped from the settler to the mixer for the thorough mixing of phases also in the settler to occur. On stopping the mixing and settling the dispersion, the holdup in the mixer of this phase which should be continuous was increased about to 80% of the total volume. It was sufficient in all cases to establish the required type of dispersion after repeated mixing-up the content of mixer.

Immediately after the repeated switching the impeller on, the dispersed phase began to be pumped from the settler into the mixer. So its holdup in mixer was gradually increased because both the phases ran over through the overflow to the settler in the form of dispersion with the same flow rate that which the pure phase flowed into the mixer. On increasing the holdup of dispersed phase, the character of the dispersion flow in mixer changed. At constant revolutions, the intensity of turbulence considerably decreased. For the holdups above 90%, the surface of dispersion was quite smooth with visible streamlines. This process went on until the phase inversion took place.

After the change of the type of dispersion, the highly turbulent flow was immediately renewed, the eddies were well visible in the dispersion and the droplets of dispersion flew away from the surface. The change of flow character was so conspicuous and rapid enough to enable us to detect visually the phase inversion. The described[4] detection in terms of measurement of changes in electric conductivity of dispersion gave identical results with the visual one. Immediately after the phase inversion, the pumping from the settler was stopped, and the holdup in the mixer was measured.

RESULTS

The values of critical holdups are summarized in Table I. They were constant in the given range of impeller revolutions. The reproducible dependence on the impeller revolutions and size was measured in the systems given in Table II.

TABLE I
Critical holdups

System no.	Aqueous phase	Organic phase	Critical holdup		Viscosity ratio μ_o / μ_w	Impeller revolutions, s^{-1}	
			ϕ_o	ϕ_w		o-disp.	w-disp.
1	water	BRDZ	94	95	1.2	20 ~ 30	20 ~ 30
2	0.05 M KCl	BRDZ	89	95	1.2	20 ~ 30	20 ~ 30
3	0.05 M KCl	BRDZ	86	95	1.2	20 ~ 30	20 ~ 30
4	water	mineral oil	96	39	46	20 ~ 30	20
5	0.05 M KCl	mineral oil	96	39	46	20 ~ 30	20
6	0.05 M KCl	mineral oil	96	37	46	20 ~ 30	20
7	water + glycerol	mineral oil	97	90	3.1	20 ~ 30	20 ~ 30
8	water + glycerol	BRDZ	76	97	0.081	16.7	20 ~ 30
9	water + glycerol	mixture #1	95	94	0.87	20 ~ 30	20 ~ 30
10	water + glycerol	mixture #2	96	96	1.08	20 ~ 30	20 ~ 30
11	0.05 M KCl	mixture #1	98	87	12.8	20 ~ 30	20 ~ 30
12	0.05 M KCl	mixture #2	98	86	15.9	20 ~ 30	20 ~ 30
13	water	butanol	81	65	3.1	16.7	16.7
14	water	heptane	68	62	0.43	20	20
15	0.03 M H$_2$SO$_4$	0.2 M HDEHP	92	88	1.3	20 ~ 30	20 ~ 30
16	0.05 M H$_2$SO$_4$	0.2 M HDEHP	92	88	1.3	20 ~ 30	20 ~ 30
17	0.15 M HNO$_3$	0.27 M HDEHP	78	89	1.4	16.7 ~ 25	20 ~ 30

Note: mixture #1 – mineral oil : BRDZ = 0.769 : 0.231 (v/v); mixture #2 – mineral oil : BRDZ = 0.862 : 0.138 (v/v).

In systems no. 1, 2, 3, 13 the measurements were carried out in two types of mixers differing in the surface wettability. No significant differences were found between the values of critical holdups.

TABLE II
Dependence of critical holdup on conditions of agitating

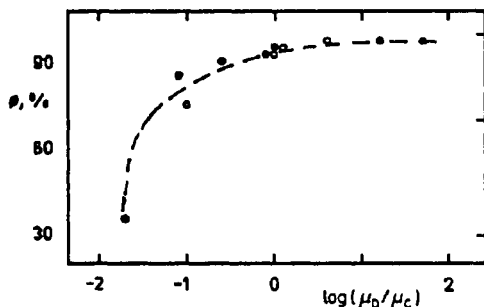| Disp. phase	Cont. phase	Impeller diameter, m	Revolutions s^{-1}	Critical hooldup of dispersed phase, %
Butanol	water	$4 . 10^{-2}$	8.33	87.2
		$4 . 10^{-2}$	10.0	84.7
		$4 . 10^{-2}$	13.3	83.5
		$4 . 10^{-2}$	16.7	81.0
Water	butanol	$3 . 10^{-2}$	30.0	62.1
		$4 . 10^{-2}$	8.3	72.8
		$4 . 10^{-2}$	10.0	71.1
		$4 . 10^{-2}$	13.3	70.7
		$4 . 10^{-2}$	16.7	65.2
		$7 . 10^{-2}$	8.3	71.5
0.27 M HDEHP	0.15 M HNO$_3$	$3 . 10^{-2}$	25.0	70.3
		$3 . 10^{-2}$	33.3	74.9
		$4 . 10^{-2}$	13.3	77.5
		$4 . 10^{-2}$	16.7	78.2
		$4 . 10^{-2}$	20.0	78.6
		$4 . 10^{-2}$	25.0	78.8
		$7 . 10^{-2}$	6.7	71.7
		$7 . 10^{-2}$	10.0	71.4

FIG. 1
Dependence of critical holdup on log (μ_D/μ_C); ● aqueous phase dispersed, ○ organic phase dispersed

The changes of critical holdup of dispersed phase in dependence on the ratio of viscosities were investigated in the system aqueous phase – aqueous glycerol solution; organic phase – mixture of BRDZ + mineral oil. The dependence of critical holdup on the ratio of phase viscosities is illustrated in Fig. 1.

The measured critical holdups of dispersed phase are, unlike the hitherto published values, comparatively high for most systems. Their dependence on the impeller revolutions is not conspicuous and manifests itself only in systems with lower values of critical holdups. The impeller diameter may influence the values of critical holdups even in case that the dependence on revolutions in not observable. The explanation of some contradictions in the previously published data might be found in the effect of geometry of the equipment used. The dependence on the ratio of viscosities is significant just only in the case when the viscosity of continuous phase considerably exceeds the viscosity of dispersed phase.

SYMBOLS

C	continuous phase
D	dispersed phase
o	organic phase
w	aqueous phase
ϕ	critical holdup of dispersed phase at which phase inversion takes place
μ	viscosity

Abbreviations

BRDZ	paraffinic redistillation residue with 3% aromates; flash point above 200 °C
HDEHP	solution of bis-(2-ethylhexyl)phosphoric acid in BRDZ

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