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Operating Characteristics of Pulsed Plate Columns

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ABSTRACT

Droplet coalescence and wetting phenomena have a considerable effect on the dispersed-phase holdup of pulsed solvent extraction columns. Changes in these phenomena with time may cause loss of the stable mode of operation even though operating variables remain constant. The influence of retarded coalescence and changes in the wetting behavior of the column plate cartridge on pulsed column operating characteristics are discussed. A method for eliminating the wetting parameter is proposed.

INTRODUCTION

The pulsed solvent extraction column consists of a vertical tube in which two immiscible liquids pass in counter-current flow. The presence of perforated plates spaced equidistantly and the application of pulsation cause one of the liquids to disperse into drops while the other liquid remains as the continuous phase. This dispersion provides the surface area which is necessary for material transfer between the two phases. The net transfer of material accomplished at the operating solution flow rates is termed the separation capacity of the column.

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In practical application it is frequently found that the separation capacity decreases with time and/or the set volumetric flow rates are no longer accommodated by the column. In this report difficulties of this type are described which occured during experimental operation. The ultimate goals of the experiments are to demonstrate the applicability of pulsed plate columns in cases with large differences between dispersed and continuous volumetric flow rates and to obtain design data for potential application in an industrial nuclear fuel reprocessing plant.

OPERATING BEHAVIOR

The functional performance of pulsed columns requires the plate cartridge and the column wall to be wetted by the continuous phase and repelling of the dispersed drop phase /1/.

Figure 1 shows the operating characteristics of a pulsed sieve plate column in a specific PUREX system: aqueous nitric acid vs. 30 vol. % tributyl phosphate in n-alkane. The overall volumetric flow rate has been plotted versus the pulse frequency. Beyond the upper curve the column can no longer operate. The volumetric flow rates are so high that the two phases can no longer pass through the column in an ordered counter-current flow. The dispersed phase together with the continuous phase leave the column and the operating failure defined as flooding is attained. In the three zones below the enveloping curve, column operation is possible.

For illustration, photographic pictures of the three regions of operation are shown in Figure 2. The mixer-settler region is characterized by relatively large drops which rise quickly, accumulate below the column plates, and frequently coalesce. This region occurs at low pulsation frequencies. By raising the pulse frequency or throughput the dispersion region is attained which is characterized by an uniform drop distribution in terms of time and location. If the pulsation or volumetric flow rates are further increased, an unstable region is reached where very dense populations

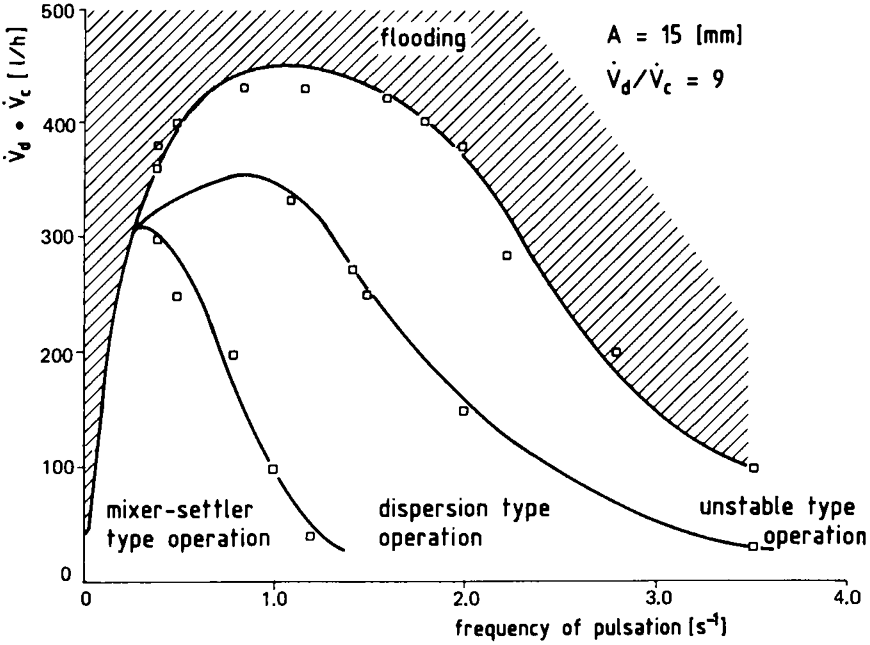
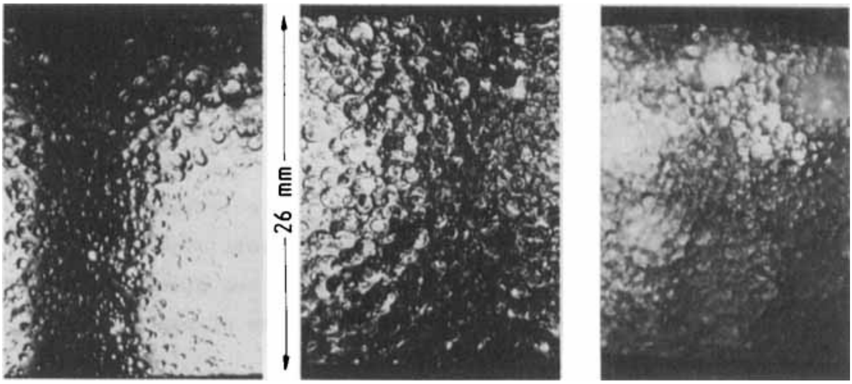


FIGURE 1 : Pulse column operating characteristics, aqueous continuous



mixer - settler type dispersion type unstable type

FIGURE 2: Typical droplet patterns

of drops locally coalesce into large clusters of dispersed phase. By passing the following perforated plate, redispersion is possible and this results in an unstable fluid dynamic behavior (right picture in Fig. 2).

In practice, the dispersion region is considered to be the preferred mode of operation. The point of operation is usually placed approximately in the center of this region in order to have a sufficient safety margin with respect to flooding and to be able to safely accommodate fluctuations in throughput and pulsation. For the aqueous-phase-continuous (organic dispersed) mode of operation the point of column operation was defined for an overall volumetric velocity of 2.5 liter per hour and square centimeter at a pulse frequency of 1 s^{-1} and a pulse amplitude of 15 mm.

At this point of operation a mean dispersed-phase holdup of $\bar{\varepsilon}$ = 22.5 % is established, as shown by the four curves in the center of Figure 3. The plots have been obtained using columns of different height and diameter although for identical flowsheet conditions, cartridge designs, and pulsing conditions. It can be seen that the holdup varies slightly with the height of the column, but all columns show similar trends and mean holdup values. This condition is to be regarded as the initial state established at the beginning of an experimental campaign with new column cartridges and clean phases. At some later time during the operation the column operation departs from its design range although external operating conditions remain unchanged. The holdup decreases (and the drop size increases), moving to the left side of Figure 3 away from the shaded dispersion zone. Large-volume drops are observed for the holdup curve marked by inverted triangular symbols. These drops quickly move upward through the space between two plates during an upward pulse and some of them coalesce below the plates during the downward pulse.

At still later times, designated by the outermost left curve, organic drops several millimeters in size and irregular in shape are formed, and these contain inclusions of small drops of aqueous phase. After a few days of continued operation a condition is

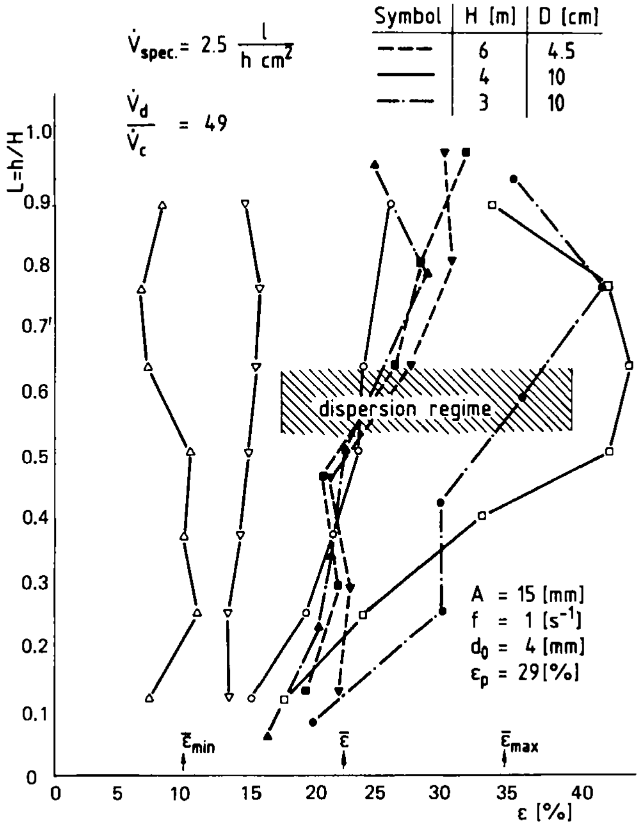
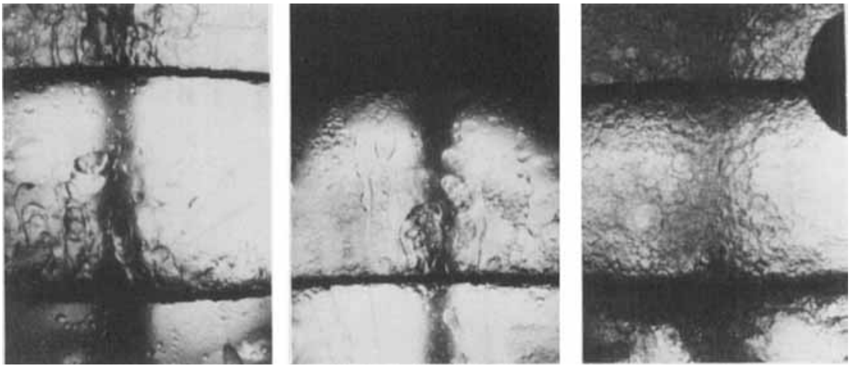


FIGURE 3: Holdup behavior as a function of column hight

reached which is shown in Figure 4. Large finger-shaped forms of organic phase are found in the lower half of the column. In the middle of the column a mixed phase region is established by the inclusion of aqueous drops. The difference in densities between the dispersed and the continuous phases becomes gradually smaller due to the inclusion of aqueous drops in the organic phase. Counterflow is increasingly hampered. This, finally, leads to an ill-defined foam in the upper half of the column. The column becomes blocked and floods because the blockage propagates downwards in the column as a result of foam formation.



bottom of column middle of column top of column

FIGURE 4: Deranged fluid dynamic behavior, uranium loaded phases

The two curves on the right-hand side of Fig. 3 are characterized by a completely different type of fluid-dynamic behavior. Normally, drop coalescence is observed for holdup values > 40 % because of dense drop packing and the unstable operating condition shown in Figure 1 would be expected. However, this condition is not attained in this example. Here a multitude of drops are observed with the majority having drop diameters < 1 mm. Although the impression is conveyed that the drops contact each other, no coalescence into large drops can be recognized. With this large quantity of drop phase, countercurrent flow decreases more and more while buoyancy becomes less; the drop residence times in the column are very long. The continuous feed of dispersed phase at the bottom of the column finally causes such a dense drop packing in the bottom part of the column that the continuous phase now occurs only as lamina between the organic drops. Countercurrent flow vanishes, the column floods and blockage formation in this case proceeds from bottom to top.

Consequently, two cases have been identified which, despite constant external parameters, may influence the fluid-dynamic behavior of the column in such a way that the throughput must be reduced or even stopped. In the first case, a steady reduction in holdup and an increase in the drop volume are observed in the course of operation. In the second case, the holdup gets larger and by the absence of drop coalescence no unstable operating range can be established.

TEST RESULTS

Coalescence Behavior

The absence of drop coalescence despite the high holdup (i. e., despite dense column packing) suggests that coalescence is retarded. Although the organic drops are separated from each other only by thin lamina of aqueous phase, they do not combine. This state is observed both in the precence and absence of uranium.

Measurements of fluid density, viscosity and interfacial tension reveal slight differences between the disturbed and the undisturbed systems. However, if measurements of the overall coalescence time are performed, differences become significant.

The coalescence time for the mutually saturated system consisting of low-concentration acid and organic phase are 53 seconds for the organic drop phase and 60 seconds for the aqueous drop phase (Table I). In the system saturated with 17 gU/l in the aqueous phase and 80 gU/l in the organic phase, the respective times of coalescence are 74 and 86 seconds, respectively; i. e., they are clearly longer (Table II). Thus, the presence of the third component, uranium, decelerates the velocity of coalescence. In both sets of data, the drops are noted to coalesce more quickly if the surrounding phase is aqueous than when the surrounding phase is the organic phase.

The coalescence of a drop with another drop or at a plane phase boundary is determined by the outflow and breakaway of the film of the continuous phase /2/. Due to the viscosity of the continuous phase, the resistence to the outflow of the film is a factor de-

TABLE I: Coalescence Time, Test System: (0.25 - 2.50) m HNO_3 - 30 vol. % TBP

condition	coalescence time [s]		holdup $\bar{\varepsilon}$ [%]
	org. phase= drop phase	aq. phase= drop phase	$\frac{\dot{V}_d}{\dot{V}_c} = \frac{196}{4}$; d_0 = 5 mm ε_p = 0.46
new phases:			
2.50 m HNO_3	53	63	
0.50 m HNO_3	47	62	
0.25 m HNO_3	41	51	
pulse column phases time of testing			
1 day	50	63	15.8
5 days	53	58	16.0
210 days	53	61	16.0
disturbed system	64	67.5	21.75
org. phase cleaned with activated carbon	52	63	
used storage vessel	59	67	
3 days	59	67	17.5
6 days	54	57	
13 days	54	70	16.7
60 days	53	59.5	16

TABLE II: Coalescence Time, Uranium Loaded System: (2.5 - 3) m HNO_3 - 30 vol. % TBP

pulse column phases, time of testing	coalescence time [s]		holdup $\bar{\varepsilon}$ [%]	flowrate $\dot{V}_d/\dot{V}_c$
	org. phase= drop phase	aq. phase= drop phase	(d_0 = 4 mm, ε_p = 0.29)	
disturbed system	134	94	operation not possible	
7 days	78	87	34	180/20
23 days	83	93	flooding	192/8
28 days	77.5	94	27.2	192/8
67 days	76	94	23.6	180/20
clean phases	73.5	86	22	192/8

termining coalescence. Thus it seems to be logical that the coalescence of the organic drop in surrounding aqueous phases is brought about more easily than in the other case ($\eta_{org.} = 2\eta_{aq.}$).

According to /3/ the physical material properties are not the only variables influencing coalescence; mobility and vibration of the phase boundary may rather be influencing factors. The "rigid, immobile" phase boundary may be the reason why after the collision of two drops the thin liquid film cannot flow out to a critical thickness within the time available which would permit bursting and coalescence. This might be an explanation not only for the longer times of coalescence in the presence of uranium but also for the disturbed systems. It can likewise be imagined that the effect of electrical double layers might be of some importance in this context; the longer time of coalescence associated with higher acid molarity points in this direction. As a matter of fact, retarded coalescence in both cases leads to an increase in holdup. The formation of large drops is hindered, or does not take place at all, and the spectrum is shifted towards smaller drops. With the volumetric flow rate of dispersed phase remaining unchanged, this phenomenon leads to an increase in drop density in the column. Since the integral velocity of small drops is smaller than that of large drops, the drop residence time gets longer and the holdup increases.

The denser packing of drops has the same effect on the surrounding continuum as an increase in viscosity. A relationship given in /4/ for solid spheres is extended in /5/ considering that the drops are not actually rigid.

$$\eta = \eta_0 \; (1 + 2{,}5 \; \emptyset \; |(p + \tfrac{2}{5})/(p + 1)|) \qquad (1)$$

Where η_0 is the viscosity of pure continuous phase, $\emptyset$ is the volumetric ratio of drop phase and continuous phase and p is the ratio of viscosities of the two phases.

The influence of holdup on the viscosity is evident from Eq. (2).

$$\varepsilon = \frac{\phi}{\phi + 1} \qquad (2)$$

For a ratio of viscosities between the organic and aqueous phases of p = 2 and for holdup values of 20, 40 and 70 %, one obtains for the aqueous-continuous mode of operation η-values of 1.5 $\eta_{aq.}$, 2.3 $\eta_{aq.}$ and 5.7 η_{aq}. The continuous phase becomes more and more viscous in its behavior and the counterflow of the drops is more and more hampered. The intact system reacts by coalescence and succeeds in passing through the instable zone (Fig. 1) before it ceases to perform. By contrast, the system hindered in coalescence does not enter unstable range of operation. Consequently, the column departs from its working range earlier.

The influence of drop coalescence on column behavior is also confirmed by the tendency observed in experiments carried out in the organic-phase-continuous mode of operation (Fig. 5). For a clean HNO_3-TBP system, coalescence times of about 60 seconds are measured

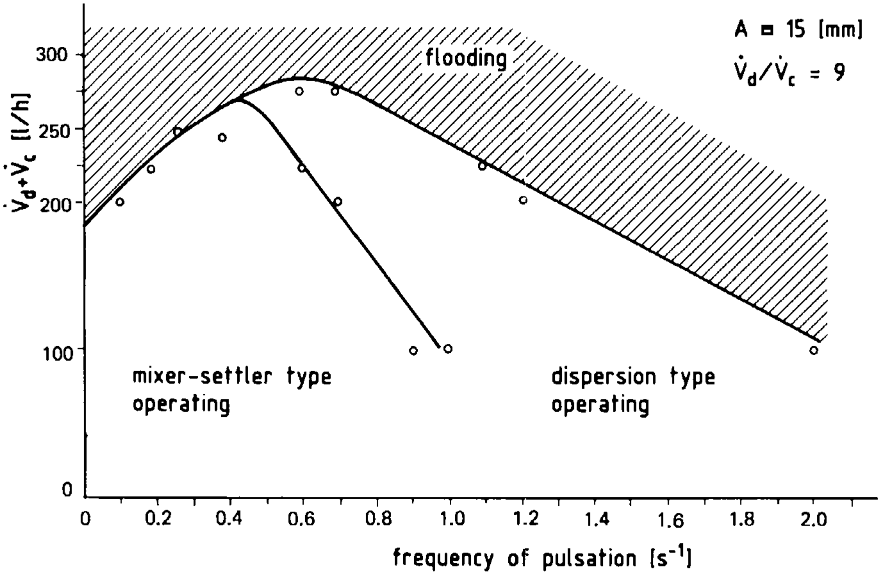


FIGURE 5: Pulse column operating characteristics, organic continuous

(Table I). As shown by the figure, the unstable range of operation characterized by drop coalescence is not observed. The consequence is that, (compared to Figure 1) the maximum throughputs measured for aqueous-continuous operation are far from being reached here. So, the essential result to be retained is that an increase in the time of coalescence leads to a narrowing of the operating band.

Wetting Behavior

A phenomenon related to coalescence, although very different in its effect on column behavior, is the experimental finding that the holdup steadily decreases with time of operation and finally leads to column flooding, although the external parameters are not subjected to variations. The reason lies in the different wetting behavior of the cartridge as a function of the time of operation /6/.

Molecular orientation at the liquid-solid boundary plays a role similar to that at the liquid-liquid phase boundary. For instance, unsymmetrical structures in the molecular unit may lead to the development of polarities in a competing environment /7/. Under this influence the molecules at the phase boundary become oriented to satisfy the criterion of minimum energy. The equilibrium condition between the surface of the solid and the respective liquid may be disturbed. Various molecules face the solid phase boundary; they may be adsorbed in a selective manner and form an adsorbed film covering the surface. Thus, the interface energy of the solid must be reduced by an energy amount due to the adsorbed film.

Obviously such adsorbed layers influence the equilibrium of tensional stresses at the point of contact described by Young's equation. In case of two immiscible liquids A and B and a clean solid phase boundary S one obtains for A as the drop phase /7/

$$\sigma_{SB(A)} - \sigma_{SA(B)} = \sigma_{AB} \cos \Theta_{SAB} \tag{3}$$

The subscripts in parentheses indicate that the liquids are saturated with respect to each other.

The angle Θ is called the contact angle. In practice, angles $> 90^o$ are attributed to poor wetting. The liquid cannot wet the surface of the solid if contact angles take values of about 150^o. On the other hand, wetting is described as good if small contact angles are measured.

The wetting behavior is determined by the "advancing - receding" method /8/. It requires determination of the contact angles for two cases: (1) for the drop of aqueous phase in surrounding organic phase (water advancing), and (2) for the organic drop in surrounding aqueous phase (water receding).

The wetting behavior is characterized by the difference of the cosine values

$$\cos \Theta_{rec.} - \cos \Theta_{adv.} = \Delta \qquad (4)$$

Negative Δ-values are assigned hydrophilic behavior, while positive Δ-values are assigned hydrophobic behavior.

The material of the plates used in the aqueous phase continuous mode of operation is stainless steel DIN 1.4301. To attain good wettability, these plates are subjected to standardized cleaning procedures prior to their being installed. For the HNO_3-TBP system, $\Delta = -1.9$. Accordingly, the sheet metals are clearly hydrophilic in their behavior at the time of installation into the column.

After a period of operation the column plates show a completely different behavior according to Figure 4, pictures 2 + 3. For these cartridges, $\Delta = 1.75$. The wetting behavior with respect to the aqueous phase has moved from the hydrophilic to the hydrophobic state with the result that the dispersed organic phase wets the column plates instead of the continuous aqueous phase.

To clarify the processes, a number of specific wetting tests were made. The non-charged and the uranium charged liquid systems were studied for their time effect on contact angle. A distinction was made between an open and a closed system. In the open system, room air had free access to the surface of the organic phase during the whole period of testing. In the closed system the access of air was considerably hindered by a well fitting cover.

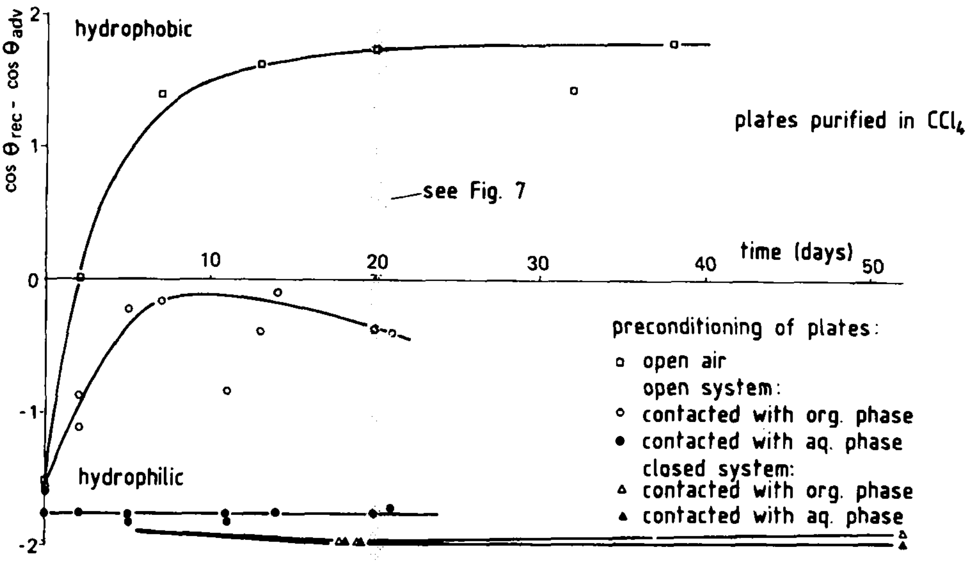


FIGURE 6: Wetting behavior, stainless steel - 0,25 m HNO_3 - TBP

Figure 6 shows the trend of the wetting behavior with time for an initially clean saturated two-component system: 0.25 m HNO_3 against 30 % TBP/n alkane. The sheet metal plates were treated in carbon tetrachloride in an ultrasonic bath and air dried before insertion into the system. The values on the ordinate describe the condition of wetting before introduction into the contacting vessel at the beginning of the test.

The upper plot shows the wetting behavior of stainless steel 1.4301 exposed to air. Within a few days the hydrophilic wetting behavior changes into the hydrophobic condition. The center plot in Figure 6 shows the behavior of the metal surface when inserted into the organic phase. Within about five days wetting changes from a clearly hydrophilic into a rather neutral, indifferent state. The sheet metal exposed to a long contact with only the aqueous phase (lower plot) behaves differently; it conserves the original good wetting behavior with respect to the aqueous medium. These effects are observed in the "open systen".

For the case where access of air is prohibited at the upper organic phase boundary ("closed system"), both the sheet metal contacted with the aqueous phase and that contacted with the organic phase stay in a clearly hydrophilic wetting condition constant with time.

Figure 7 shows characteristic individual drops after a contact time of 20 days, developing from a joint initial condition. In the upper part of the figure the influence of air is shown on the wetting behavior. The heavy drops of the aqueous phase appear on the upper side of the sheet metal. Under the influence of the surrounding organic phase the drops form contact angles of approximately 150°. A slight inclination of the sheet metal plate away from the vertical axis causes the drops to roll off, demonstrating that no wetting takes place. On the same plate the organic drop behaves in a completely different way if surrounded by the water phase. Contact angles of 25° are measured. The organic phase wets the surface so adequately that even on the upper side of the sheet metal an elongated drop develops.

The central part of Figure 7 shows the condition prevailing in the "open system". In the first diagram drops of aqueous phase can be seen surrounded by the organic phase. The left-hand sheet metal side was in contact with the aqueous phase; elongated drops are observed with small contact angles. The right-hand half of the sheet metal had been contacted with the organic phase for 20 days and here the contact angles of the drops are about 90°. The second diagram shows organic drops on the same plate surrounded by the aqueous phase. On the left-hand side where the sheet metal was contacted with the aqueous phase during the experiment large contact angles are seen for the organic drops, indicating that wetting is poor. By contrast, the angle of contact for the part contacted with the organic phase is 100°. For the sheet metal exposed to the organic phase the difference of cosine values (Δ) is -0.4, which corresponds to an indifferent wetting situation; for the sheet metal contacted with the aqueous phase, Δ is -1.7, a value equivalent to a hydrophilic wetting behavior.

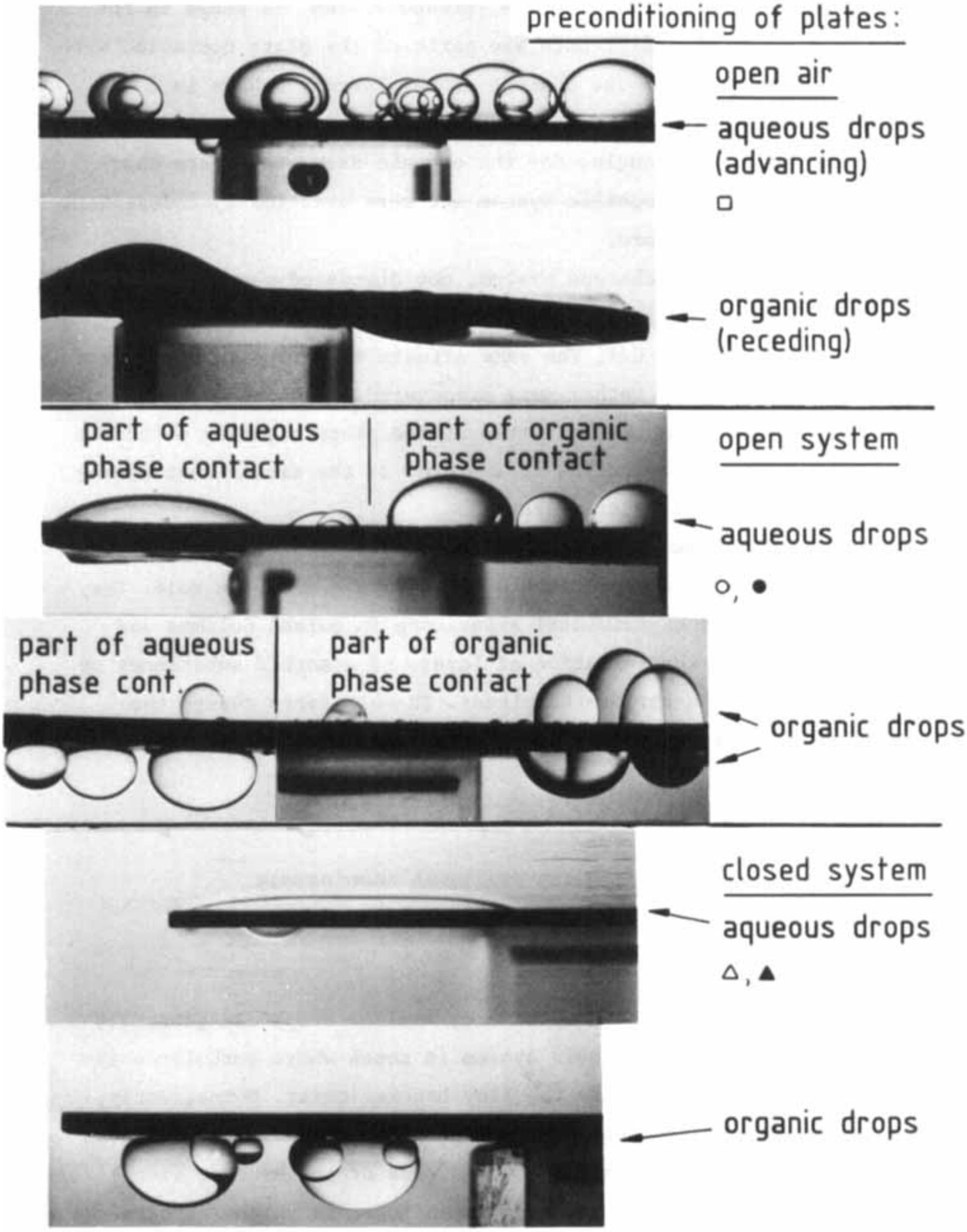


FIGURE 7: Shape of drops (contact angle), stainless steel 1.4301 - 0,25 m HNO_3 - 30 vol.% TBP, exposure time: 20 days

The wetting behavior of the "closed system" is shown in the bottom part of Figure 7. Here the parts of the plate contacted with either the organic or the aqueous phases largely behave in a similar way. Very small contact angles are seen for the aqueous drop and very large angles for the organic drop. These are characteristic of a hydrophilic system and were practically independent of the time of exposure.

In the uranium charged system, not discussed above, the aqueous 3 m HNO_3 phase contained 18 g U/l and the organic 30 vol. % TBP phase contained 90 g U/l. The same effects as discussed above were found, but they were rather more pronounced.

If one performs the last step of the plate cleaning procedure by using H_2SO_4 the situation is improved to the extent that hydrophobicity occurs at later times.

The tests have shown that the wetting behavior is not constant with time and that the presence of air plays a decisive role. They fully confirm the experimental experience in pulsed columns and indicate clearly the formation of layers of adsorbed substances on the surface of the perforated plates. These effects change the wetting behavior and thus give rise to a modified fluid dynamic behavior /9/.

DISCUSSION AND PRACTICAL CONCLUSIONS

Coalescence

Regarding drop-drop coalescence, smaller drops are generally observed in the liquid-liquid system in zones where turbulence is high and larger drops where the flow becomes quiet. Consequently, in the aqueous-phase-continuous pulsed plate column, organic drops are generated above the plate and the zone of coalescence lies below the plate. In this latter zone an increase in the dispersed phase fraction is observed which leads to an additional damping of the turbulence of the continuous phase and promotes coalescence /10/.

Where the tendency to coalesce decreases, an increase in holdup occurs.It is very frequently observed that if previously used and drained equipment parts are again integrated into the process, residues of organic phase in combination with air give rise to the observed inhibitions of coalescence. Analyses, done so far, do not indicate the influencing effects, neither do solvent degradation products explain the observed coalescence behavior. The substances that cause resistence to coalescence are assumed to reside in the organic phase because retardation of coalescence can be eliminated if the organic phase is passed through an activated carbon bed. However, because of the high expenditure required, this procedure cannot be proposed for commercial application. A remedy may be provided by designing receiver, collection, and buffer tanks in such a way that they can be emptied completely, leaving no residues.

If coalescence retardation is not serious, it can be compensated for by a reduction of the pulse frequency. However, retardation of coalescence frequently causes such an increase in holdup that the volumetric flow rates must also be substantially lowered. According to experience gathered by the author, this degradation in performance becomes weaker with time, but since it can occur over several weeks, it ultimately means a considerable reduction in the capacity of the solvent extraction separation system.

Wetting

The fluid-dynamic stability in the aqueous-phase-continuous mode of operation calls for hydrophilic wetting behavior of the column cartridge. During operation layers of adsorbed substances are formed on the perforated plates which gradually transform the wetting behavior into the hydrophobic fluid-dynamic unstable condition. The wettability of the perforated plates thus undergoes variations with the history of the metal surface. In /11/ a similar behavior is found for the air-dried stainless steel sheet metal and the stainless steel sheet metal wetted with the aqueous and organic phase, respectively. It can also be found that no clearly defined limits exist. The author also has detected - in agreement with these observations - that there is obviously an influence exerted by the initial phase contact on the further development of the wetting behavior on the metal surface. In both cases not more than this simple statement can be made; this report presents no in-depth study of the phenomenon because it has not been possible to systematize findings and measurements of contact angles to the necessary extent.

The author /11/ has attempted to purify the column by hot etching and carbonate washing under pulsation. He also has disassembled the entire cartridge and purified each plate with a commercially available degreaser and wishes to underscore that the latter method has been the more effective one, and he draws the reader's attention to the fact that an adaptation of this method to practical use in industrial-scale plants is desireable.

The findings described here show that plate purification in an ultrasonic bath can restore the initially hydrophilic condition. Direct use of the ultrasonic method in the column is suggested. Purification is particularly effective when the cartridge is directly exposed to ultra sound. This is possible if a central rod is rigidly welded to the equidistant perforated plates (Fig. 8), and the rod is extended through the top of the column to a sonic transducer.

The sound velocity c is a material constant set by the Young's modulus and density. The sound frequency f and the wavelength λ are coupled via this velocity:

$$c = \sqrt{\frac{E}{\rho}}\ ; \quad c = \lambda \cdot f \qquad (5)$$

The wavelength is defined by the requirement of having the greatest possible uniformity of sound intensity on the equidistant plates. This is achieved if the wavelength λ and the plate distance H correspond in the following manner:

$$H = \lambda;\ \frac{\lambda}{2};\ \frac{\lambda}{4};\ \ldots \qquad (6)$$

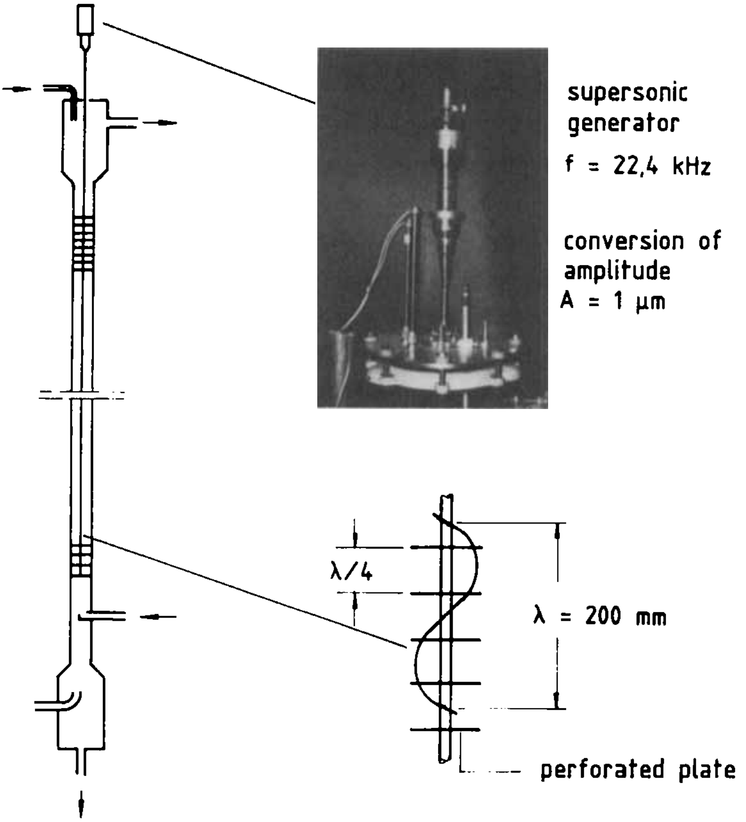


FIGURE 8: Supersonic cleaning of cartridge

For the experimentally used plate distance H = 50 mm the required wavelength is $\lambda = 200$ mm by setting $H = \frac{\lambda}{4}$. This wavelength corresponds to a frequency f = 22.4 kHz for the stainless steel cartridge. An appropriate amplitude transformation to A = 1 µm offers a configuration corresponding to the usual range of applications of industrial-scale purification systems.

This method /12/ has been successfully tested over more than two years of operation on columns of 100 mm inner diameter and 4 m cylindrical length by using three supersonic pulses per day, each with a duration of 3 seconds. Steadily reproducible holdup values show that the influence of wetting has been eliminated. By preservation of the hydrophilic wetting condition of the column cartridge the prerequisites have been provided for stable operation in terms of fluid dynamics and thus for a constant effective separation power. The extremely small expenditure in equipment is an asset for the practicability of the method proposed.

The coalescence behavior and the wetting behavior have not yet been studied in detail until now and, in the presence of mass transfer events, obviously become even more complicated. However, to gather knowledge of more general validity that goes beyond plant-specific concepts, an in-depth study of this complex subject will be necessary. - From the practical point of view the change in wetting behavior during operation time is a significant disadvantage. Stopping the process and cleaning the column are the procedures in practice. By the proposed ultrasonic column cartridge cleaning this disadvantage is eliminated.

NOMENCLATURE

A = amplitude, entire pulse stroke
D = column diameter
E = Young's modulus
F = column cross section
H = column height
= plate distance
L = normalized column height
V = volume
$\dot{V}$ = volume flow rate
$\dot{V}_{spec} = \frac{\dot{V}_d + \dot{V}_c}{F}$ = specific capacity (volumetric velocity)

c = sound velocity
d_o = diameter of holes
f = frequency
h = hight
p = ratio of viscosities

Greek Letters

Δ = difference

Θ = contact angle

$\phi = \frac{V_d}{V_c}$ = volumetric ratio

$\varepsilon = \frac{V_d}{V_d + V_c}$ = dispersed phase holdup

ε_p = fractional free area of plates

η = viscosity

λ = wavelength

ρ = density

σ = interfacial tension

Subscripts

c = continuous phase

d = dispersed phase

aq. = aqueous phase

org. = organic phase

adv. = advancing

rec. = receding

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