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Electrical Field Contactor for Solvent Extraction

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ABSTRACT

Electrostatic coalescers have been extensively used in solvent extraction units. However, the multiple properties of electrical fields may be applied to the realization of liquid-liquid contactors in which mixing and phase separation are caused by electrostatic effects.

Under the action of an electric field an emulsion is submitted to various effects : drop disintegration and promoted coalescence respectively for high and low field intensities, modified displacements resulting from electrostatic forces and change of the mass transfer coefficients.

The last effect (mass transfer) has been studied with a suitable device : the LEWIS cell in which an electric field is applied perpendicular to the interface. Enhanced copper extraction was observed, using the LIX 65N extractant.

The study of these basic mechanisms resulted in the design of an efficient electrostatic extraction column.

The hydrodynamic study confirms the feasibility of applying high electrical voltages to two-phase systems containing a significant amount of conductive dispersed phase provided that the electrodes are coated with a suitable insulating layer.

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The drop size spectrum and the retention may be controlled by the field strength in a two phase flow column. The field strength adjustment between the electrodes is obtained by simple voltage setting and it is easy to alternate the dispersion and coalescence zones.

Additional development is necessary before designing an industrial contactor : examination of other chemical systems, other operating conditions (higher flow rate, uneven field on each stage ...) and determining mass transfer rates.

INTRODUCTION

Electrostatic industrial applications are more and more numerous, for instance : electrostatic painting or printing, separation processes such as dust precipitation. Recently, a new application has been considered in the field of solvent extraction.

The performance of agitated liquid-liquid contactors depends on their hydrodynamics which is controlled by three mechanisms : break-up, motion and coalescence of drops.

An electric field influences these three mechanisms, for it may :

- disintegrate drops by electrostatic dispersion at sufficient intensities ;
- increase or decrease drop velocity due to the additionnal electrical forces ;
- promote coalescence, under certain conditions by changing drop interactions.

It also modifies mass transfer coefficients by induced turbulence near drop interface.

These different effects have been systematically studied (1,2,3) ; they will be investigated in the first part of this report. We shall also describe a typical device (LEWIS cell) we have designed for this purpose and to measure extraction mass transfer kinetics in electric fields.

An electric field can be applied to a mass transfer operation in several ways.

First, the electrostatic extraction processes require low conductivity continuous phases because the field strength must be high enough to promote significant phenomena (at least a few tens of volts per cm).

In this case an electric field can be used for emulsion disengagement ; electrostatic settlers are already extensively used, particularly, in the oil industry. However, an electric field can be applied in the contactor itself :

- to improve any function of the existing contactors. For example, KNEULE and NEMEC (4) have added electrical coalescence stages regularly distributed in a rotating disc contactor.
- to realize both mixing and phase separation. A few original apparatus have been suggested (5, 6, 7, 8). Their chief advantages are better reliability due to the abscence of moving mechanical parts and rated mass transfer efficiencies. A better knowledge of the basic mechanisms has lead to the design of a new model of electrostatic extraction column. Some of its characteristic hydrodynamic features will be described below.

BREAK-UP, MOTION AND COALESCENCE OF DROPS IN AN ELECTRICAL FIELD

Drops dispersed in a continuous immiscible phase are prone to take two kinds of charges :

- polarization charges induced by an external electric field;
- true charges, generated for example by electrode contact charging. These charges are dissipated by conduction with a time constant equal to the relaxation time of the continuous phase. Their influence on mass transfer has been extensively studied by THORNTON and by BAILES who showed their favorable effect (5,9). But the relaxation time of many continuous phases used in solvent extraction, particularly in hydrometallurgy, is much less than a fraction of a second so that true charges have no noticeable effect. Therefore we will not consider them here.

Drop polarization has three main consequences :

- a nonuniform electrostatic pressure opposed to the interfacial pressure all over the surface causes drop deformation. When it overcomes the cohesion forces, the drop disintegrates :

- a new external force acts on the drops : the dielectrophoretic force, which is given by :

$\vec{F} = (\vec{m}.\vec{grad})\vec{E}$ ($\vec{m}$ is the drop dipole moment)

- a dipole-dipole interaction between drops tends to line them up and bring them closer.

Using literature data, we shall quantify these electromechanical effects. We assume that each particle is a perfect conductor (this being the case for every liquid-liquid system that includes an aqueous dispersed phase with ionic soluble species) immersed in a linear isotropic dielectric medium.

Electrostatic Dispersion

Various studies have been devoted to the analysis of the equilibrium and stability of an isolated neutral conducting drop subjected to an electric field (11,12). They have shown that the drop elongates in a nearly prolate ellipsoidal shape. Its eccentricity is an increasing function of the WEBER electrostatic number :

$$|1| \quad W_{e_E} = \frac{\varepsilon \varepsilon_o E_o^2 d}{\gamma}$$

d	drop diameter
E_o	field intensity
γ	interfacial tension
ε_o	vacuum permittivity
ε	continuous phase permittivity

The variation of this deformation is represented in figure 1, according to ROSENKILDE (10). The drop disintegrates when it reaches a value which represents the instability point. The critical value above which instability occurs and the maximum stable deformation are :

$$|2| \quad W_{e_E} = 0.409 \qquad |3| \quad \frac{a}{b} = 1.838$$

The corresponding critical field strength for the experimented phases are given in Table 1.

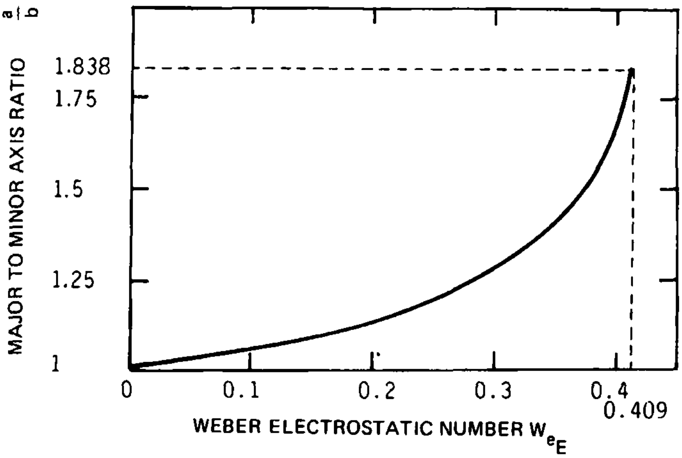

FIGURE 1. Major to minor axis ratio versus the WEBER electrostatic number.

TABLE 1

Numerical Examples

Physical electrical properties of phases - see Table 3	
critical field strength according to equation |2| drop radius $r=1.5 \times 10^{-3}$ m	$E = 4.3 \times 10^{2}$ kV/m
dielectrophoretic force |5| to weight ratio $\Delta V = 10$ kV $R_{ext} = 3 \times 10^{-2}$ m $R_{int} = 10^{-2}$ m $R = 1.5 \times 10^{-2}$ m	0.70
dipole force |6| to weight ratio $r_1 = r_2 = 1.5 \times 10^{-3}$ m $s/r_2 = 0.1$ $E_o = 2 \times 10^{2}$ kV/m	0.08

Dielectrophoresis

We assume that the field strength (E) is lower than its critical value and that drops have a spheroidal shape. Their dipole moment is :

$$\vec{m} = kv\vec{E}$$

v = drop volume

k = polarizability

$k = 3\varepsilon\varepsilon_o$ for a conducting drop.

In a nonuniform field they are subjected to the dielectrophoretic force :

$$\vec{F} = 1/2 \; kv \; \vec{\text{grad}} \; E^2 \qquad |4|$$

The main examples of nonuniform fields have been published by POHL (12) : in the field created between an inner wire and an external coaxial cylinder electrode the force is radial, directed toward the center and has the following value :

$$F = \frac{1}{2} \Pi\varepsilon\varepsilon_o \; d^3 \; \frac{\Delta V^2}{R^3 \ln^2 \dfrac{R_{ext}}{R_{int}}} \qquad |5|$$

ΔV potential drop

R_{int}, R_{ext} outside and inside radii

R distance from the considered point to the axis.

A numerical example of this force intensity compared to the drop weight is given in Table 1 .

Dipole Coalescence

DAVIS calculated the forces acting on two nearby neutral conductive spheres placed in a uniform field (13). The force components are the following :

$$F_z = \varepsilon\varepsilon_o \; r_2^2 \; E_o^2 \; (F_1 \cos^2 \psi + F_2 \sin^2\psi) \qquad |6|$$

$$F_x = \varepsilon\varepsilon_o \; r_2^2 \; E_0^2 \; F_3 \sin 2\psi$$

r_1, r_2 radii of the spheres

s distance between the spheres

ψ angle between $\vec{E}$ and z axis (see figure 2).

The coefficients F_i, i = 1,3, are series limits which depend only on the relative geometry of the system (r_1/r_2, s/r_2). Values corresponding to the case where $r_1 = r_2$ are shown in Table 2 (13).

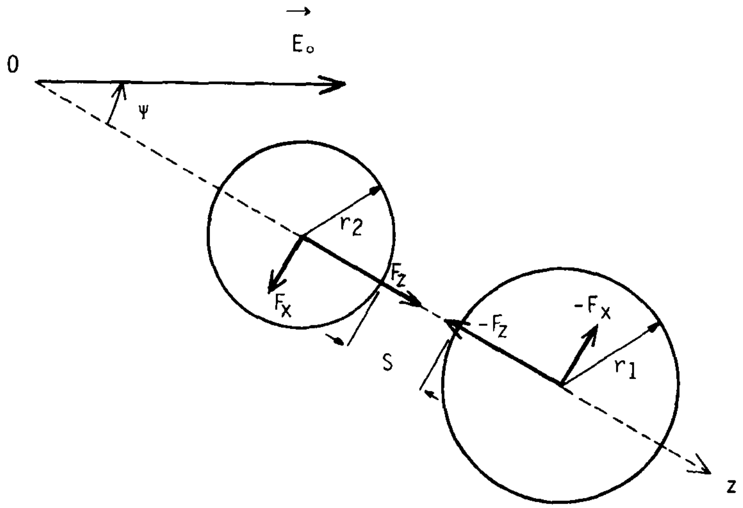

FIGURE 2. Dipole forces acting on two spheroidal drops in a uniform electric field.

TABLE 2

s/r_2	F_1	F_2	F_3
10.0	0.00030	- 0.00014	- 0.00014
1.0	0.0927	- 0.0330	- 0.0391
0.1	1.400	- 0.0945	- 0.2248
0.01	9.554	- 0.1032	- 0.3756
0.001	59.49	- 0.1041	- 0.4652

The attraction is optimum as the z axis and the field line up. Its value is very low when s is greater than r_2 and considerably increases when the drops get closer (see Table 2).

All the figures given in this table are consistent with our experimental results (1) (2).

MASS TRANSFER IN AN ELECTRICAL FIELD

Literature Survey

We saw that the normal electric stress component deforms the drop interface. KOMORI et al. have proposed the use of an intermittent field to yield periodic elongation of drops and thus increase the overall mass transfer coefficient. As to the tangential component it generates a circulating fluid motion (15). BERG et al. have shown that the resulting renewal of the boundary layer has the same favorable effect (16).

As for the extraction of a metallic ion, BAILES suggested that the field induced orientation of extractant molecules at the interface could enhance the rate of reaction (17). Electrophoresis would also have an influence by increasing diffusion of ionic solutes.

AUSTIN (18), in his experiments using a plane interface cell, pointed out the rise by a 1 to 6-10 factor of the mass transfer rate for partially miscible binary systems (18).

Using the same equipment, we expected to obtain some data on the relative importance of the different electric field effects by comparing mass transfer kinetics with or without applied electric field.

Apparatus

We selected the copper LIX 65N extraction system (19). It includes an aqueous solution of copper chloride and an organic mixture of LIX 65N extractant in ESCAID 100 diluent. The phase agitation on each side of the interface is produced by two metallic paddles. A central teflon disc prevents the vortex occurrence. The electric field is created by application of a potential difference between the agitators, Samples of the organic phase are taken at regular time intervals with a hypodermic needle. Copper samples were analysed by atomic absorption spectroscopy.

Results

Figure 3 represents the variations of the copper concentration in the organic phase for different electric voltages :

O kV, + 10 kV, - 10 kV

The electric field promotes mass transfer. The effect is more important when the field is directed towards the aqueous

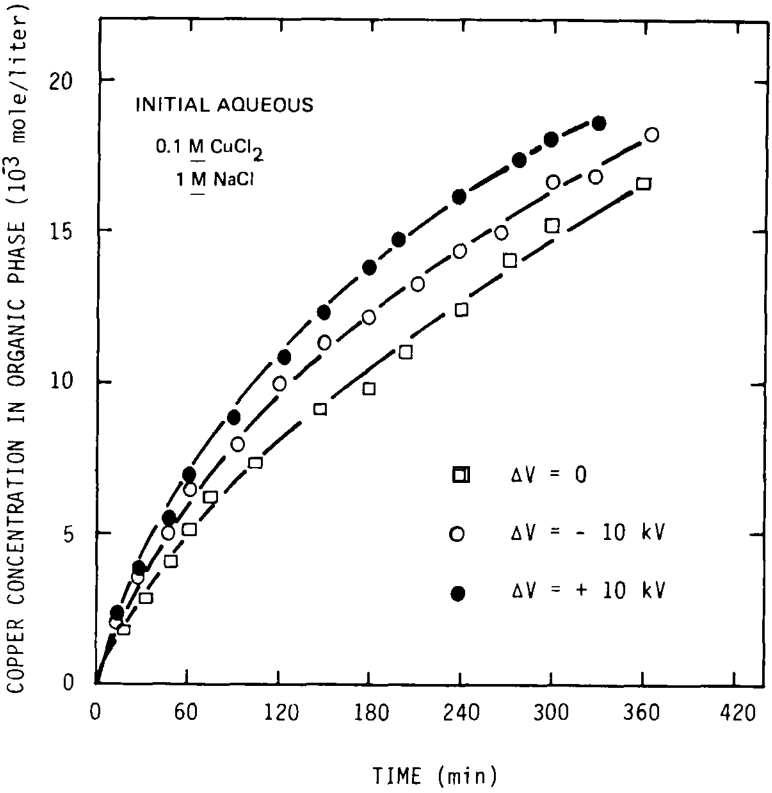

FIGURE 3. Organic phase copper concentration plotted versus time for the voltages O, + 10, - 10 kV

phase (positive voltage). These experimental data are not sufficient to define a predominant electrostatic mechanism. Another phenomenon, electromigration of chemical species, modifying the concentrations of positive or negative ions in the boundary layer and thus the reaction equilibrium, intervenes in the extraction process.

Further experiments are in progress.

ELECTRICAL FIELD CONTACTOR

Literature Survey

THORNTON and BAILES were the first to study a single-stage electrical field contactor (5,6). The dispersed phase is disintegrated when forced through a horizontal perforated plate under high direct voltage. The small droplets produced in this way travel through the continuous phase and coalesce at the interface at the bottom of the column equipped with a grounded grid. The main problem of electrostatic extraction is to avoid the short-circuit between the electrodes due to conduction paths formed by drop bridges the dispersed phase usually being conductive. BAILES partially solved this problem by lifting the upper nozzle plate out of the continuous phase, so as to maintain a vapor space between the electrode and the liquid surface. The MURPHREE efficiency with a 3 kV/cm field intensity was twice that without electric field for the n-butyl acetate-acetone-water system.

KOWALSKI et al. , AGAIEV et al. selected a column design with vertical rod electrodes (7,8) coated with a thin polyethylene layer to prevent short-circuits. The electric field is transverse, alternative and of higher than critical intensity. For washing out acetic acid from xylene or carbon tetrachloride with water, the extraction rate in a 3 kV/cm field was five times higher than without any field (7). For removing aromatic hydrocarbons in a fuel oil by furfural extraction, the HETS in a 5 kV/cm field was half that in a RASHIG ring packed column operated in the same conditions (8).

Although these equipment with no moving parts are attractive, their behavior is not well understood and further investigation is necessary. The overall mass transfer efficiency has been measured but little attention was devoted to the hydrodynamics. It is our purpose to develop this point.

A laboratory column has been designed and dispersed phase hold-up and droplet sizes were measured.

Apparatus

In a countercurrent column, the drop diameter has to be greater than the stationary one. The dielectrophoretic force acting on the drop under a nonuniform field may add to the gravity force and decrease this diameter. The droplet size in a column may be lowered by using this force, allowing an increase of the interfacial area and hence of the mass transfer. Nevertheless a high gradient in large parts of a column lead to technological sophistication and a gravity-induced countercurrent flow was prefered.

Electrodes are made of stainless steel rods, 10 cm long. These rods are coated with a thin layer of teflon to eliminate short circuit risks. Eight stages of electrodes are assembled in a Perspex column, 3.9 cm in diameter and 120 cm long (see figure 4).

An autotransformer (220 V-0/230V) feeding the primary of a H.T. transformer (230-15000V) is used as the power supply. The phases are fed by two variable flow rate pumps. The dispersed phase is introduced at the top through a cone shaped diffuser and a perforated plate (nine 1 mm diameter holes)

The hold-up (ratio of dispersed phase volume to total phase volume) is measured by the interface displacement method : the flow is stopped by rapidly closing valves and the entrapped dispersed phase level is measured after decantation.

Emulsion pictures are taken at different heights.

The dispersed and continuous phases are respectively an aqueous acid solution and a commercial diluent. These chemical phases belong to a high interfacial tension system (see table 3). The dispersed aqueous flow rate varies between 0 and 18 l/hr. The aqueous to organic flow rate ratio is maintained at 40.

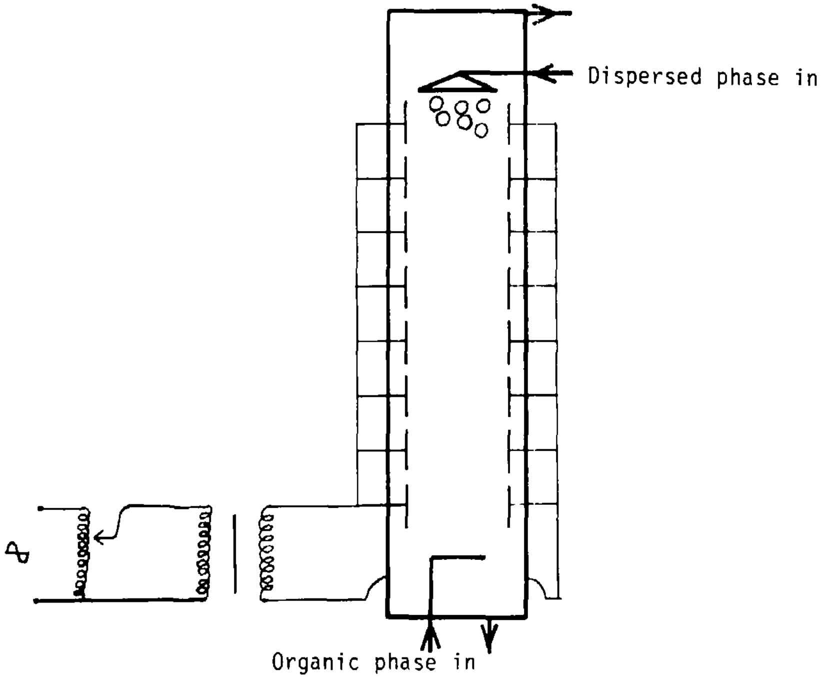

FIGURE 4. Schematic diagram of the column.

TABLE 3

Phase properties at 22°C

density (10^3 kg m^{-3})	ρ_c = 0.87 ρ_d = 1.10
viscosity (mPa s)	ν_c = 1.11 ν_d = 1.35
interfacial tension (N m^{-1})	$\gamma = 33.9 \times 10^{-3}$
conductivity (Ω^{-1} m^{-1})	$\lambda_c = 8.3 \times 10^{-10}$
relative permittivity	ε_c = 2.4

The subscripts c and d refer respectively to the continuous and the dispersed phase.

Results

The same voltage is applied to the eight stages. The field does not vary all along the column. The man dispersed phase hold-up is plotted versus the dispersed phase flow rate for several applied voltages (see figure 5). Three pictures of the emulsion in the column, taken at 0 - 3.75 - 11,25 kV voltage and 6 l/hr aqueous flow rate are presented (see figure 6).

When the voltage is less than approximately 5 kV, the field all over the interelectrode space is below the critical value (value linked to the droplet size without any field by equation |2|), hence the coalescence is favored. Droplet dilatation may be observed from a 100 volts voltage. Between 2 and 5 kV, the water drops have completely coalesced after a 40 cm fall (1/3 of the column) and the dispersed phase flows as thick discs (see figure 6-b-). The larger the flow rate, the more important the decrease of the mean hold-up compared to the fieldless operation of the column (see curves -a- and -b- figure 5).

For voltages between 5 and 11 kV, the field strength is higher than the critical value in part of the interelectrode space (close to the electrodes) and the droplets are partly disintegrated : the hold-up increases slightly.

Above 11 kV, the field is everywhere greater than the critical value, drops burst violently as soon as they are fed in. As KOWALSKI mentioned (7), droplet recombinations and ruptures occur all along the column and a stationnary droplet spectrum sets up (see figure 6 -c-). Droplet disintegrations as well as continuous phase electroconvective motions generate bidimensional turbulence very favorable to mass transfer. Pictures show that :

- for a constant aqueous flow rate, the higher the electrical field, the smaller the mean droplet diameter and the higher the hold-up (see curve -c- and -d- figure 5).

- for a constant voltage, the higher the flow rate, the larger the drop diameter. This explains why the relative increase

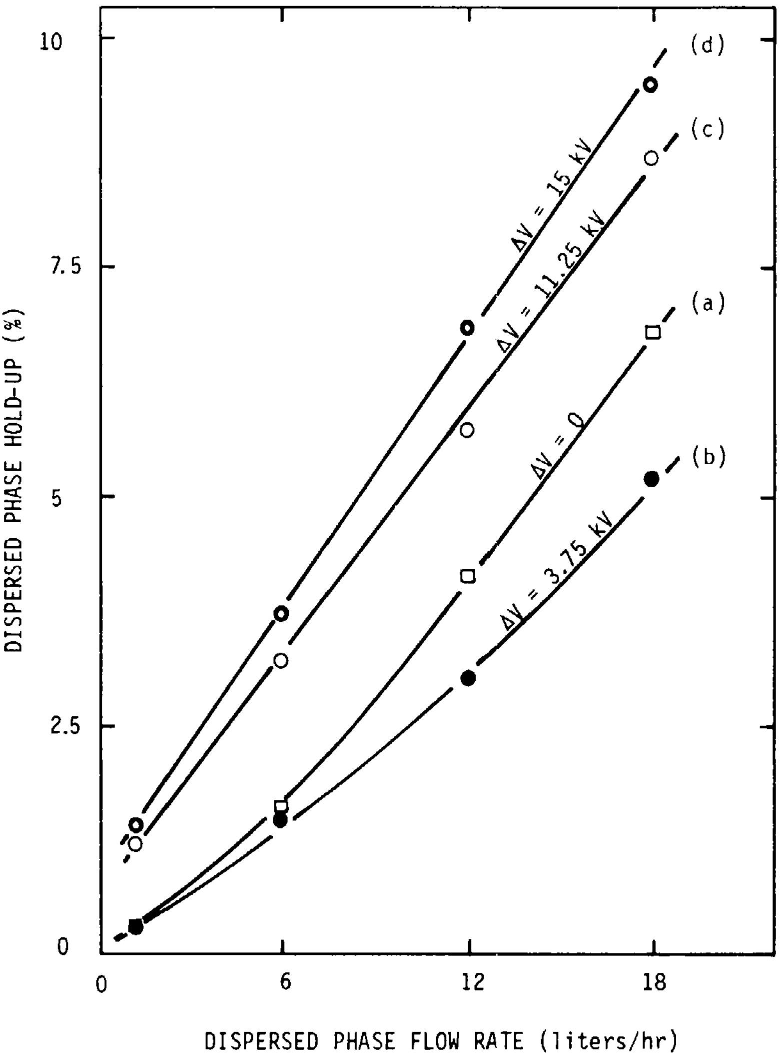

FIGURE 5. Mean dispersed phase hold-up in the column versus the aqueous flow rate for several interelectrode voltages.

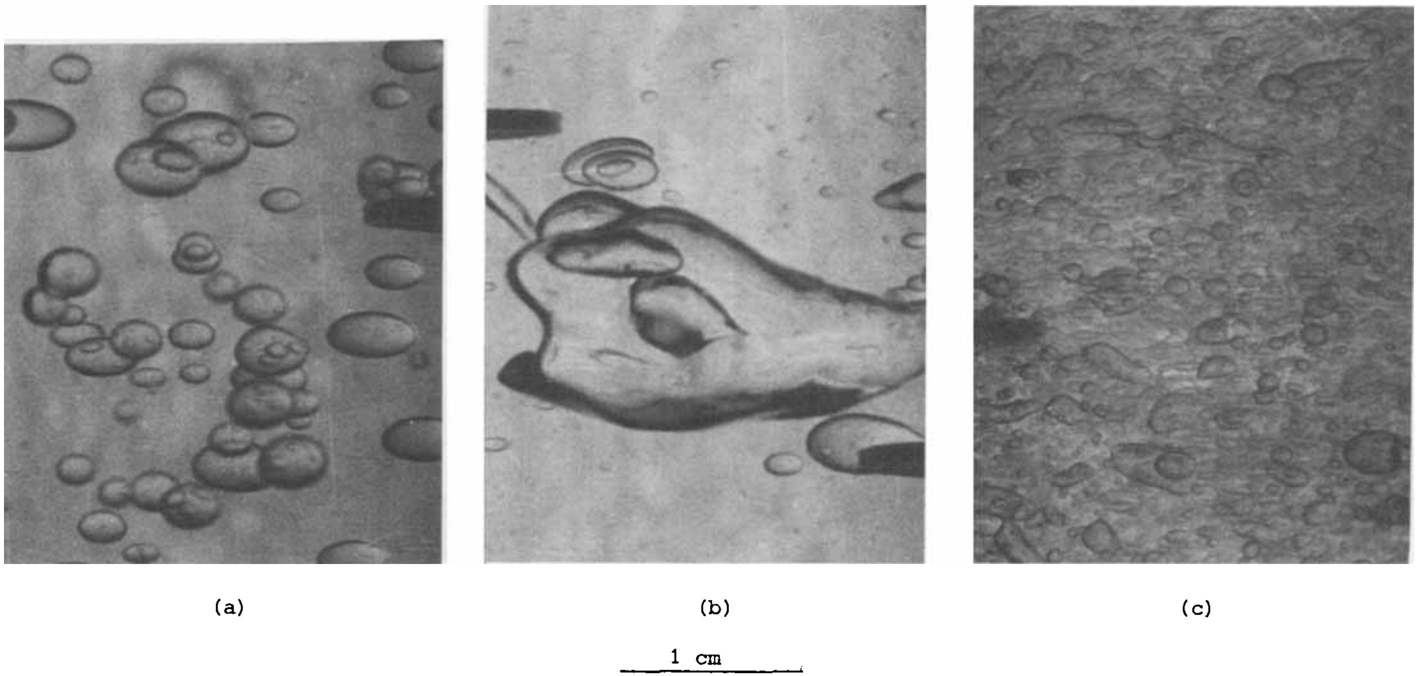

FIGURE 6. Emulsion drop size in the seventh stage of the column (aqueous flow rate : 6 liters/hr)

(a) without field

(b) with coalescing field ΔV = 3.75 kV ($W_{c_E} < W_{c_E}$ crit.)

(c) with dispersing field ΔV = 11.25 kV ($W_{c_E} > W_{c_E}$ crit.)

of the hold-up compared to fieldless operation values decreases (see figure 5). The reason is that the hold-up increases with the flow rate : droplets are closer, dipolar forces are more intense and electrical coalescence is more favored than rupture.

CONCLUSION

The application of a suitable intensity electrical field to two phase liquid-liquid systems leads to various phenomena of great interest for the solvent extraction operation. These phenomena have been summarized. Among them, we have considered the mechanical effects of drop rupture, displacement and coalescence, and the local influence of the field on mass transfer. The value of the forces has been made more accurate and confirmed by the experiment.

An electrostatic extraction column has been constructed and tested. Thanks to insulated coated electrodes, the use of this contactor can be extended to many others multiphase systems. Its hydrodynamic behavior showed that the emulsion characteristics could be easily controlled by means of the applied voltage.

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