IMPROVEMENT ON SEPARATION EFFICIENCY OF A NEW DESIGN OF MIXER-SETTLER BASED ON PHASE INVERSION

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ABSTRACT

Over the past twenty years mixer-settlers were widely used in hydrometallurgy and many designs have been proposed with general aim to decrease the area required. The size of the settler seems to be a critical factor in mixer-settler design. The aim of this paper is to present an improvement on separation efficiency of a new design of mixer-settler based on phase inversion. The new equipment has been applied to treat a waste water contaminated with fine petroleum-in-water dispersions or emulsions.

Phase inversion can easily be realized when the initial continuous phase is transformed into drops far larger than the droplets in the primary dispersion.

The efficiency of separation of the equipment depends on variables like oil in water concentration, agitation into mixer chamber, volumetric ratio between organic and aqueous phases, total co-current flow and height of organic bed.

In this work a statistical design is employed to determine how independent variables could influence the separation efficiency of the equipment when treating referred waste waters.

INTRODUCTION

Over the past twenty years mixer-settlers were widely used in hydrometallurgy and many designs have been proposed with general aim to decrease the area required.

The main advantages of these equipments are : strong operational loads, easy operation and maintenance, and simple start-up. As it is known, the rate of mass transfer across a phase boundary is a function, among other variables, of the drop size distribution or interfacial area between phases. Up to a point, the smaller the drop size the greater the rate of mass transfer. Coupled with increasing dispersion however, increased coalescence time required, and therefore the settler size becomes the critical factor in mixer-settler design. The size is governed by throughput limitations imposed by the rate of coalescence of the dispersed phase. As settling problems related to solvent loss and/or entertainment were shown to be the major cost factor in the design, attempts have been made to increase the settling rate and therefore throughput, resulting in an increase in the overall economy (Ritcey and Ashbrook, 1979). Finally in order to achieve good overall settling velocity together with good flexibility many designs such as the Lurgi Multi-Tray Settler* and the Segmental Circular Settler have been proposed with the general aim to decrease the area required while maintaining high throughput and efficiency. Thus shortening the distance between the droplets and the interface increases the rate of coalescence.

It is known that the use of the so-called phase inversion method has given excellent results in the separation of primary and secondary emulsions, allowing a decrease in the settling area while maintaining high throughput and efficiency (Hadjiev and Kyuchoukov, 1989; Aurelle et al., 1991; Paulo et al., 1994; Paulo, 1996).

Some previous studies have shown that in separators based on this principle it is possible to increase separation efficiency when increasing throughput. The new equipment has been recently applied to treat industrial waste waters contaminated with fine petroleum-in-water dispersions or emulsions (Chiavenato, 1999).

In this work a statistical design is carried out in view of determine how dependent variables could influence the separation efficiency of the equipment when treating referred waste waters.

EXPERIMENTAL

Phase inversion principle

The operating principle of the unit is given in Fig. 1. Primary dispersion (1), produced in the mixer by mechanical agitation, is forced through a distributor with a perforated plate (2) separating both mixer and settling zone. The original continuous phase becomes the dispersed phase inside the settler. The settling zone is filled with an organic phase (3) identical to the originally dispersed one. During dispersion at the perforated plate big "carrier" drops (6) which diameter D are formed containing fine "carried" droplets of the preliminary dispersed phase. The motion of these big drops towards the interface (4) is governed by a residence time $t_R = H/V_S$, where H is the height of the organic bed and V_S is the sedimentation velocity of the drops. The carried droplets (less dense) carried by the carrier drops (more dense) tend to ascend inside the carriers, thus concentrating in the upper part and coalescing in the organic bed. The non-coalesced droplets can still be recovered near the interface (4).

It is important to notice that each carrier works as a micro-decanter, thus decreasing the distance to the interface.

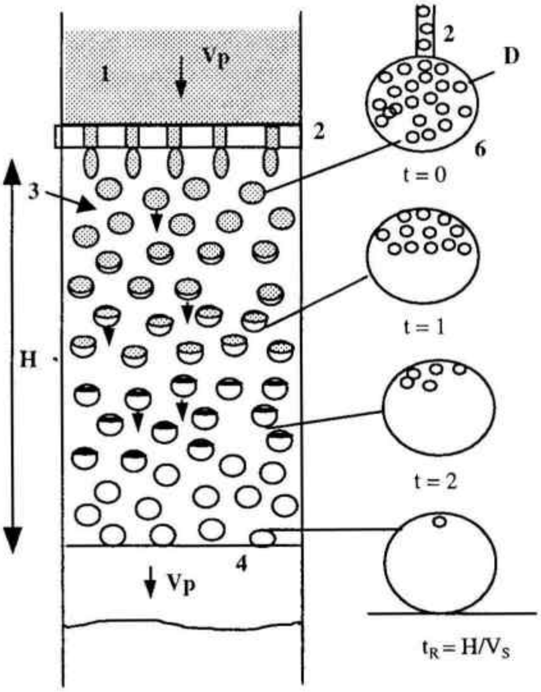

Figure 1 - Phase inversion principle.

A drop-drop coalescence can take place inside the carrier drop resulting in an increase in the ascending velocity and therefore reducing the coalescence time (Paulo, 1996).

Systems and analysis

The aqueous continuous phase studied is common in the petroleum industry. It consists of an oil-in-water emulsion with drops up to 10 µm in diameter. The oil concentration of these emulsions can reach 2000 ppm which is prohibitive for direct discharge on environment. Environmental legislation recommends an oil concentration not exceeding 20 ppm in Brazil. Thus, it is usual to perform a treatment for these waste waters. The organic phase contacted with the aqueous to transfer oil to dispersed droplets, is a solvent. Table I presents some physical properties of the system.

Table I - Physical properties of the system

Product	Density $[10^3\ Kg/m^3]$	Viscosity $[10^{-3}\ Kg/ms]$	Surf. Tension $[10^{-3}\ N/m]$	Int. Tension $[10^{-3}\ N/m]$
O/W emulsion	1,055	0,65	55,20	27,29
Solvent	0,760	1,00	22,27	
Oil	0,874	1000,0	---	---

(Data by Chiavenato, 1999)

Analysis of the feed and treated water were carried out by a gravimetric technique after solvent extraction with n-hexane.

Experimental design

A factorial design was used to determine the influence of oil concentration into feed and total flow upon efficiency of phases separation. The experimental design was made according to (Box et al. 1978).

RESULTS AND DISCUSSIONS

Figure 2 shows the efficiency of separation as function of total flow for several oil concentrations in aqueous feed. Results show that an increase in total flow leads to an increase in efficiency of separation. This tendency is particularly notable to lower oil concentrations in feed, e.g. 88 to 200 ppm. This is an interesting and curious result that means a better performance of the equipment when charged. Previous works with the new equipment have reported similar results. This fact represents a great advantage over conventional mixer-settler units which reduce efficiency of separation with throughput.

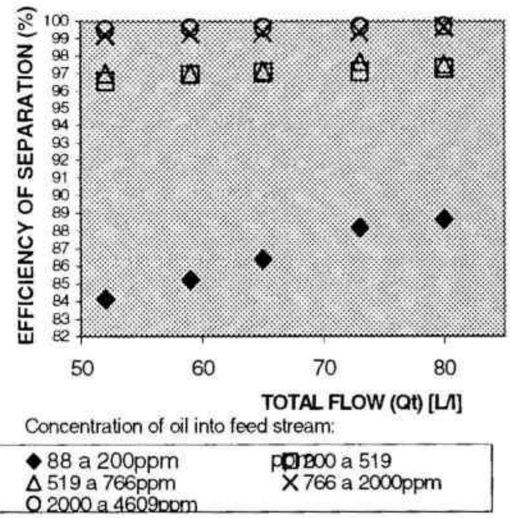

Figure 2 - Efficiency of separation as function of total flow to several oil concentration into feed.

Attempts to explain this observation are based on size of carriers drops generated at the perforated plate. An increase in the total flow leads to an increase in the velocity of carrier drops (Vp, Figure 1) resulting in small drops. By diminishing carrier drops diameter, sedimentation velocity, Vs, will be also diminished. Associated residence time t_R will increase leading to more time of coalescence of carried drops with their continuous phase into settler (3, in Figure 1).

To compare the influence of two independent variables studied until this date, a statistical design was carried out. Table II shows results of efficiency of separation to certain operational conditions displayed in the same table. It can be seen that new equipment presents an efficiency of separation of practically 100% for concentrations in oil up to 820 mg/L.

Table II - Data for statistical experimental design

Assay	Concentration in oil (mg/L)	Total Flow (L/H)	Efficiency of Separation (%)
1	360	59	96.53
2	820	59	99.25
3	360	73	97.10
4	820	73	99.38
5	640	66	97.65
6	640	66	96.97
7	640	66	97.13
8	140	66	86.13
9	1000	66	99.67
10	640	52	96.99
11	640	80	97.52

The design used was a 2^2 factorial plus Central Composite Design and two levels were evaluated (-2 and +2). The dependent variable was the efficiency of separation. The errors were associated with the coefficients at the central point. Values for the minimum and maximum levels were set on experimental data (Chiavenato, 1999). Statistical analysis of the data was performed using software Modreg (Barros Neto et al., 1995) and Statistical Design (Statsoft, EUA).

Statistical treatment allowed the fitting of the data to the following quadratic model:

$$Y=\beta_o+\sum_{i=1}^{k}\beta_i X_i+\sum_{i=1}^{k}\beta_{ii}X_i^2+\sum_{i=1}^{k}\sum_{j=i+1}^{k}\beta_{ij}X_iX_j \quad (I)$$

where Y is the dependent variable, β_i the model coefficients, and X_i the independent variables, oil concentration in feed (mg/L) and total flow.

Table III shows values of the coefficients for the quadratic model represented by Equation I.

The dependence of efficiency of separation in terms of the two independent variables studied could then be displayed by means of a three-dimensional graph as shown in Fig 3.

Table III - Calculated coefficients of the Quadratic Model represented by Eq. I.

Coefficient	Efficiency of Separation (%)
β_0	98.0384
β_1	2.63500
β_2	0.14670
β_3	-1.1080
β_4	0.04800
β_5	0.11000

According to the data from Table III the coefficient β_1 presents a more significant value than the others ones. At the same time, negative value of β_3 means a region of maximum for efficiency of separation.

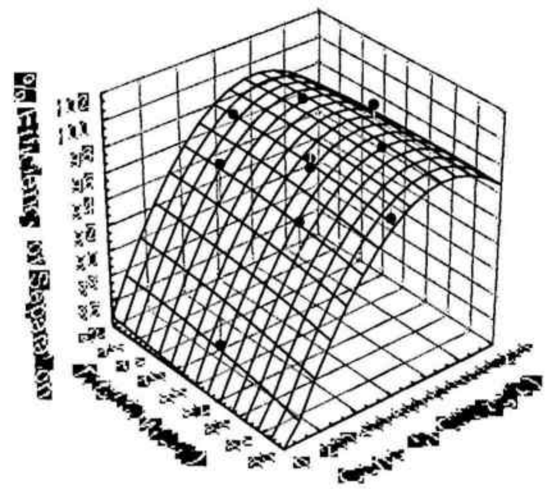

Figure 3. Effect of concentration in oil associated with total flow upon efficiency of phase separation.

Figure 3 clearly shows the magnitude of the influence of oil concentration into feed upon efficiency of phase separation. Besides it denotes a maximum efficiency of separation in the range of '800 to 1000 mg/L of oil into aqueous feed.

Statistical design shows a variance of 84,31% (Table IV). Data from F-test presented in this table are close to the literature. A high value for F-test in the absence of fitting can indicate a high statistical significance.

Table IV Analysis of Variance Using Modreg

Origin of variation	Addition of squares	Degrees of freedom	Means squares	F-Test
Regression	110,839	5	22,168	
Residual	20,632	5	4,127	5,37
Absence of fitting	20,380	3	4,793	
Net error	0,253	2	0,126	53,74
Total	131,472	10		
Variance (%) = 84,31				
Maximum reported Variance (%) = 99,81				

CONCLUSIONS

The new equipment proposed for treatment of oil-in-water emulsions has shown advantages with relation to conventional ones. To this new application the device worked better when loaded and this fact is relevant to an industrial application of the equipment. Besides high throughputs the device presents a vertical design which can be advantageous where plant lay-out is reduced e.g., oil-well on off-shores.

By treating contaminated waste waters with up to 4600 ppm in oil, cleaned waters with less than 20 ppm in oil were obtained, which accomplishes limitations imposed by environmental legislation.

Results from Statistical Design (Modreg and Statistic softwares) show the best separation in the range of 820 to 1000 mg/L of oil into aqueous feed. According to statistical studies carried out with experimental data, total flow has no influence upon efficiency of separation in relation with oil concentration into feed.

Authors should continue the work studying the influence of other independent variables as agitation, organic/aqueous ratio, and height of organic bed, upon efficiency of separation.

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