

Drop Coalescence Analysis In The Dispersion Band Of A Mixer Settler

Gerardo D. Riquelme, Francisco Femenías and Orlando Bezama
Chemical Engineering Department,
Universidad Técnica Federico Santa María
POBox 110-V, Valparaíso, Chile
Corresponding author email address: gerardo.riquelme@usm.cl

The behavior of the dispersion band in the settler of a pilot scale mixer-settler unit is analyzed. Drop to drop and drop to interface coalescence are studied by image analysis of digital video taken under different specific velocity. Dispersion band thickness, hold up and drop size distribution were determined along its length, and drop to drop and drop to interface coalescence kinetic were estimated. The analysis is focused in the differences of behavior of the dispersion band, when the settler is operating at a specific flow such that the dispersion band disappears before the end of the settler, dispersion band with a wedge profile, compared with the case when the dispersion band is in contact with the end of the settler, and drops are subject to compression with the wall (flooded settler). It was determined that drop size distribution in the dispersion band follows a log normal distribution, and the average drop sizes increase as a function of length and height in the dispersion band.

1. Introduction

Copper and iodine are recovered by solvent extraction of pregnant solutions obtained by leaching of copper minerals and Chilean nitrate ore, respectively. In both cases due to high volumetric flow of the aqueous pregnant solution, the preferred technology is the use of mixer-settler units. In this type of processes the entrainment of the organic and/or aqueous phases produces heavy economic losses and/or operational problems in downstream operations which usually compromise the economic results of the process or the quality of the product, or both. The main restriction in the design of the process is to minimize entrainment, rather than to maximize mass transfer, since the development of new organic extractants had considerably increased mass transfer in such a way that one or two mixer settler unit are enough to achieve the design recovery

Little has been published on the analysis of the phenomena that control the behavior of the settler, the coalescence of the dispersed phase in the dispersion band. Drop to drop and drop to interface coalescences are the phenomena which mainly determine the entrainment of the dispersed phase in the settler. The height of the dispersion band is the result of the dynamic equilibrium between the flow of the dispersion coming from the mixer, and the exit from the drops from the dispersion band by coalescence at the interface and of the continuous phase by gravitational settling. These two dynamic processes are the result of drop to drop and drop to interface coalescences. A well design mixer-settler will operates with a dispersion band characterized by a wedge shape, with its tip not reaching the end of the settler. Usually as result of operating with specific flow rates above the design value and/or as result of contamination of the phases or degradation of the organic phase, the dispersion band losses the wedge profile and reaches the end of the settler. The increase in height of the dispersion band produces an increase in the coalescence rate between the drop of dispersed phase and the interface, and the dynamic equilibrium is reestablished. However, entrainment of the dispersed phase is increased.

Vijayan (1975) made a good review of the work done so far, on drop to drop and drop to interface coalescence phenomena, no definitive conclusions was obtained on the influence of the drop diameter on the coalescence time. Blass et al. (1992) reviewed the hydrodynamic models of gravity settlers without coalescing aids concluding that model of settlers which are based exclusively on hydrodynamics are unsatisfactory. Pike et al. (1971) analyzed the properties of a dispersion band in a mixer-settler contactor concluding that further work need to be done in order to analyze drop size distributions at different points of the dispersion band. Hosozawa et al. (1973) modeled the dispersion band by applying queue theory, and validate the model with experiments done in a spray tower. Barnea et al. (1975) published a series of four papers in which analyze the structure of the dispersion band, the flow patterns of the dispersed and continuous phases within the dispersion band, the drop to drop coalescence time and the separation capacity of continuous settler, but no consideration was made on the entrainment of the dispersed phase. Jeffreys et al. (1970) studied how the average drop size increases along the dispersion band of the settler, for wedge shaped dispersion band. Based on these results they formulated a differential model based on drop to drop and drop to interface coalescence times. Hossain et al. (1983) performed experiments in order to analyze the hydrodynamics of mixer-settlers measuring drop size distribution in the dispersion band, under different operational conditions. They developed a correlation to estimate wedge length of the dispersion band, but no reference is made to analyze entrainment. Padilla et al. (1996) and Ruiz et al. (1996) studied the separation of liquid-liquid dispersions in deep layer gravity settler. They concluded that the thickness of the dispersion band is a function of the specific flow rate of the dispersed phase, that the average size of drops generated in the mixer is independent of the dispersed phase flow rate and that the drop volume distribution can be accurately represented by a log-normal distribution. No reference is made to entrainment of the dispersed phase.

Lately, Stevens (2006) reviewed recent advances in tools to investigate kinetics and coalescence in solvent extraction processes. Singh et al. (2008) analyzed the characteristics of the dispersion obtained in a continuous flow stirred tank by measuring drop size distributions under different operational conditions. As other authors had demonstrated the

conclusion is that the drop size distribution of the dispersion, leaving the mixer, can be accurately fitted by a log-normal distribution.

It had been well established that entrainment of the dispersed phase by the continuous phase increases exponentially with the increase in the specific flow rate entering the settler, Teh Cheng (1983). If a model to estimate entrainment is developed based on settling of drops while in the settler, the entrainment can be estimated as the amount of drops which are unable to settle toward the dispersion band within the length of the settler. This approach will generate a linear relation between entrainment and specific flow rate, which is unable to explain the exponential behavior experimentally obtained. Therefore, an analysis of the all phenomena occurring in the settler is required, especially in the dispersion band. This work presents the results of the analysis of the coalescence in the dispersion band, based on our experimental work done in a pilot scale mixer-settler.

2. Experimental

The experimental work was done in a single stage mixer-settler unit with organic and aqueous phases passing just once throughout the equipment. Escaid 103 was used as organic phase and water at pH 7 as aqueous phase. All experiments were done using an organic to aqueous phase ratio of 1:1. The experimental unit was made of transparent acrylic, 0.8 m long, 0.3 m width and 0.2 m height. The mixer, with a square cross section of 0.13 m by 0.13 m, was equipped with a pump-mixer impeller operated at 350 rpm. The experiments were run in such a way that when the mixer-settler unit achieves steady state operation, the dispersion band was filmed using a Watec CCD Camera Model WAT-902B. The camera was installed on a mobile car and allowed to film in a stationary position and to move with the same velocity as the dispersion band. The images were digitalized and velocity of the dispersion band estimated, and drop size distribution along three or four point along the length of the dispersion band, at different dispersion band height, was obtained.

3. Results and Discussion

Drop size distributions in the dispersion band, were obtained at different band height and different length in the settler. Figure 1 shows how the average drop size increases, as the dispersion band travel along the settler, until its disappearance by complete coalescence of the dispersed phase. The specific flow of $0.5 \text{ [m}^3/\text{hm}^2]$ is the larger value for which the dispersion band have a wedge shape. The analysis of these results shows that in general the average drop size increases along the dispersion band, either at its top or bottom. It must be considered that drops in this operational condition are free to flow along the settler with no other interference than the interface. Figure 2 shows the same results as Figure 1, but for specific flows of 3.0, 3.8 and $4.3 \text{ [m}^3/\text{hm}^2]$, which makes the settler to operate at flooded conditions. In these conditions the dispersion band had considerable thickness and drops in it are no free to flow in the direction of the general flow, because there is a wall that stops them at the end of the settler. The analysis of top and bottom average drop sizes along the length of the dispersion band show, that there is a steep decrease of the average drop size at

the bottom and stabilization at the top. This steep change can not be explained by assuming that the settler is collecting in the dispersion band the small drops that are settling to the bottom of the dispersion band from the continuous phase.

Figures 3 and 4 show the drop size volume distribution at different distance from the inlet of the settler. These distributions were analyzed and the experimental data correlated with a log-normal distribution drop volumes. As it can be realized from these figures the log-normal distribution accurately represents the drop size volume distribution near the inlet, but also how its behavior along the length of the settler. Figure 3 shows that when the dispersion band had a wedge shape the evolution of the drop size distribution along the dispersion can be predicted by a uniform increase in the average of the distribution and little changes in the standard deviation. Figure 4 shows the evolution of the drop size volume distribution when the settler is operating at the flooded conditions, in this case a specific flow of $4.3[m^3/hm^2]$. It can be realized that the drop size volume distribution does not increase its average when the dispersion band moves toward the outlet of the settler, and even a decrease of the drop size average is observed.

4. Conclusions

Measurement of drop size distribution have been performed for a kerosene-water system, in a pilot scale mixer-settler, changing the specific flow of dispersion in order to obtain wedge shape dispersion band in the settler and also a flooded operation. It has been shown that drop size volume distribution along the settler can be accurately represented by a log-normal distribution, not only in one point of the settler but along its length. It has been also shown that a large number of small drops are detected toward the end of the dispersion band, when the settler is operating at flooded condition. Experimentally has been found evidence of large drops explosions, apparently by compression, at the end of the dispersion band, when high specific flows are used. This can be the reason of the exponential increase in entrainment when the specific flow is increased, behavior that has been detected in industrial operations. Works is underway to further analyze these phenomena.

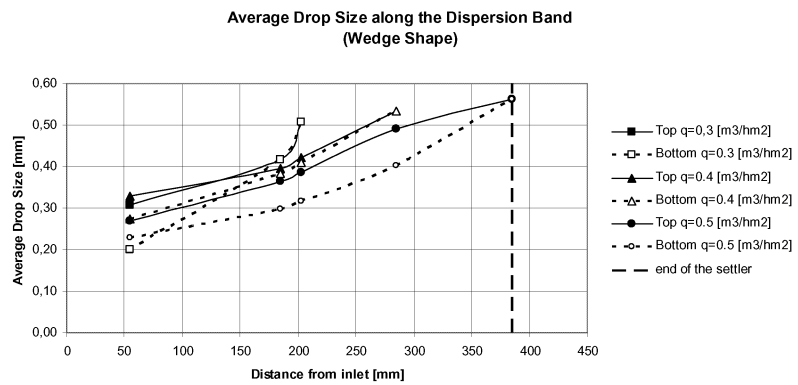


Figure 1. Evolution of the Average Drop Size along the Dispersion Band, when its shape is a wedge. Specific Flows between 0.3 and 0.5 $[m^3/hm^2]$

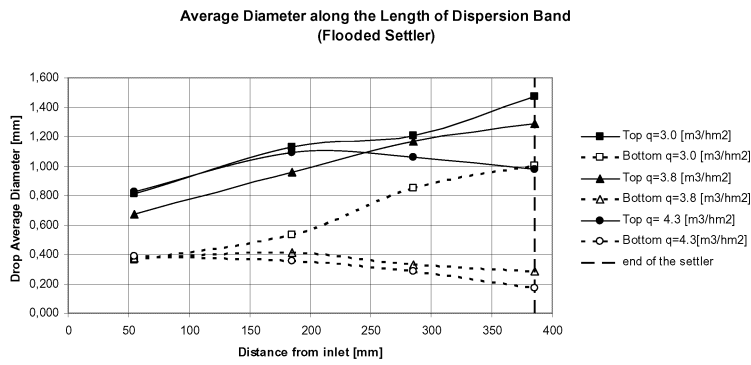


Figure 2. Evolution of the Average Drop Size along the Dispersion Band, when the settler is flooded. Specific Flows between 3.0 and 4.3 [m^3/hm^2].

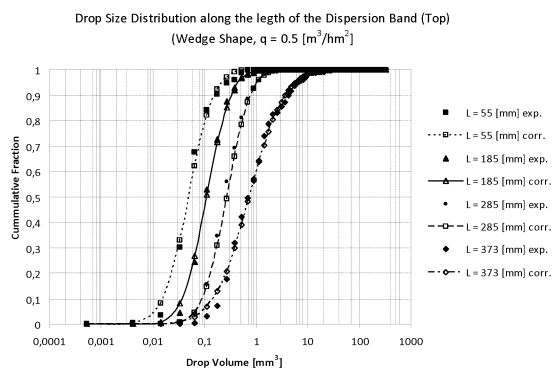


Figure 3. Drop Size Distribution along the length of the top of the Dispersion Band at Specific Flow $0.5 [\text{m}^3/\text{hm}^2]$.

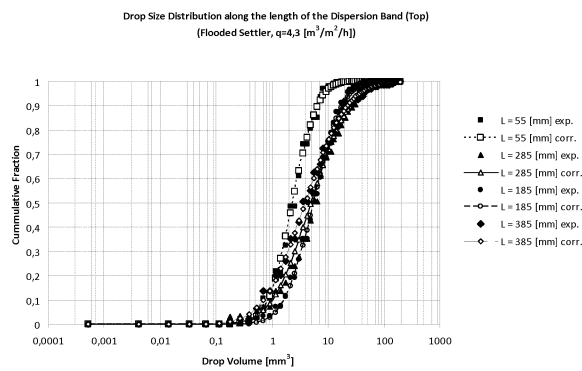


Figure 4. Drop Size Distribution at Specific Flow $4.3 [\text{m}^3/\text{hm}^2]$ at the Top of the Dispersion Band the interface of the Dispersion Band.

References

- Barnea E. and Mizrahi J., 1975a, Separation Mechanism of Liquid-Liquid Dispersions in a Deep Layer Gravity Settler: Part I. The Structure of the Dispersion Band. *Trans. Instn. Chem. Engrs.*, 53, 61-69.
- Barnea E. and Mizrahi J., 1975b, Separation Mechanism of Liquid-Liquid Dispersions in a Deep Layer Gravity Settler: Part II. Flow Patterns of the Dispersed and Continuous Phases within the Dispersion Band. *Trans. Instn. Chem. Engrs.*, 53, 70-74.
- Barnea E. and Mizrahi J., 1975c, Separation Mechanism of Liquid-Liquid Dispersions in a Deep Layer Gravity Settler: Part III. Hindered Settling and Drop to Drop Coalescence in the Dispersion Band. *Trans. Instn. Chem. Engrs.*, 53, 75-82.
- Barnea E. and Mizrahi J., 1975d, Separation Mechanism of Liquid-Liquid Dispersions in a Deep Layer Gravity Settler: Part IV Continuous Settler Characteristics. *Trans. Instn. Chem. Engrs.*, 53, 83-92.
- Blass E., Meon W., Rommel W. and A. Löbmann A., 1992, Is Hydrodynamic Modeling a Sound Approach for the Design of Gravity Settlers without Coalescing Aids?, *International Chemical Engineering*, 32, 601-618.
- Handbook of Solvent Extraction, 1983, Eds. Teh Cheng L., Baird H. I. and Hanson C., John Wiley & Sons, New York.
- Hosozawa M., Suzuki M., Tadaki T. and Maeda S., 1973, The Mechanism of Formation of a Droplet Bed at a Liquid-Liquid Interface, *Kagaku Kogaku*, 37, 402-408.
- Hossain K. T., Sarkar, S., Mumford C. J. and Phillips C.R., 1983, Hydrodynamics of Mixer Settlers, *Ind. Eng. Chem. Process Des. Dev.*, 22, 553-563.
- Jeffreys G. V., Davies G. A. and Pitt K. 1970a, Rate of Coalescence of the Dispersed Phase in a Laboratory Mixer Settler, Part I, *AIChE Journal*, 16, 823-827.
- Jeffreys G. V., Davies G. A. and Pitt K. 1970b, Rate of Coalescence of the Dispersed Phase in a Laboratory Mixer Settler, Part II. The Analysis of Coalescence in a Continuous Mixer Settler System by a Differential Model, *AIChE Journal*, 16, 827-831.
- Padilla R., Ruiz M.C. and Trujillo W., 1996, Separation of Liquid-liquid Dispersion in a Deep-layer Gravity Settler: Part I. Experimental Study of the Separation Process, *Hydrometallurgy*, 42, 267-279.
- Pike F. P. and Wadhawan S. C., 1971, Properties of an Emulsion Band in a Mixer Settler Contactor, *Proc. Int. Solvent Extraction Conference, ISEC, The Hague*, 1, 112-125.
- Ruiz M. C. and Padilla R., 1996, Separation of Liquid-liquid Dispersion in a Deep-layer Gravity Settler: Part II. Mathematical Modeling of the Settler, *Hydrometallurgy*, 42, 281-291.
- Singh K. K., Mahajani S. M., Shenoy K.T. and Ghosh S. K., 2008, Representative Drop Sizes and Drop Size Distribution in A/O Dispersion in Continuous Flow Stirred Tank, *Hydrometallurgy*, 90, 121-136.
- Stevens W. S. 2006, Interfacial Phenomena in Solvent Extraction and Its Influence on Process Performance, *Tsinghua Science and Technology*, 11, 165-170.
- Virayan S., Coalescence in a Laboratory Mixer/Settler Unit, Ph D. Thèse, Ecole Polytechnique Fédérale Lausanne, 1975.