Knitmesh And Duplex-Nylon Type Coalescence Aids Use In Phase Disengagement

Hamit Topuz, W.L.Wilkinson

Abstract: This study shows how dispersions consisted of droplet sizes ranging from 100 microns and above of immiscible liquids in agitated vessels coalesced and settled back to their phases by employing commercially known as knit-mesh made from stainless steel and nylon. These components known as higher surface energy and lower surface energy contained coalesce aids respectively. In addition, to compare, coalesce aid made purely from commercially known as duplex-nylon also used. The experimental set up was 1:3 scale of a single stage mixer-settler unit of the already existing unit which was in use at BNFL Springfield Works. The liquid –liquid system made from 20 % tri-butyl-phosphate (TBP) technical grade of odorless kerosene forming the light organic phase or solvent phase and, 5% M nitric acid forming the heavy aqueous phase. The solvent phase contained 70 gram of uranium per liter. Uranium contained phase was supplied by above mentioned company.

Index Terms: knitmesh; duplex- nylon; single stage mixer-settler; immiscible liquid dispersion; tri-butyl-phosphate.

1 Introduction

IN general, in recovery and purification of uranium and other precious elements from their ores or complexes in hydrometallurgical operations were performed in large size mixer-settlers. The design and constructions of such equipment were initiated from the scale up of small sized laboratory version of such devices by certain parameters. This has been a common approach for such a long time and even by now. As studied by Jeffrey et al. [10]. A recent development in the design put forward by Jackson and co-worker [11]. Although, this has been going on for so long, yet not established well so that one could easily adopt such devices for such purposes off the shelf. The reason for this is because of there are many criteria that must be considered before landing on a safe and most versatile optimum design in such applications and this is a live fact. Therefore, the factors that has direct bearing on optimum capacity calculations must be carefully and scientifically selected and determined for a successful overall experimental unit for ultimate purposes. In this work ,commercially known as “knit mesh” coalescence aid manufactured from stainless steel and nylon was exercised in a single stage mixer-settler 1/3 of the already existing unit which is in use in BNFL in Springfield Works in order to increase the capacity requirements were investigated. In addition, a second type of coalescing aid made from pure duplex nylon was also studied for further evaluation of the performances of two different types of such aids over one another used for the same purposess.

2 Concept and Literature Survey

The coalescence of dispersion of immiscible liquids in agitated vessel has been studied for a range of liquids and geometries under both batch and continuous conditions.

- *Hamit Topuz, is currently in Department of Industrial Engineering, Maltepe University, Istanbul, Turkey. Tel: +90 216 626 10 50 E-mail: hamittopuz@maltepe.edu.tr*
- *W.L.Wilkinson Department of Chemical Egineering, University of Bradford, England, The UK.*

Preliminary experiments were carried out first in a small-scale single mixer – settler unit operating with the 20% TBP/OK, 0.05M nitric acid, and uranyl-nitrate system. In practice dispersions comprised of droplets of varying sizes. Separation of the phases of two immiscible liquids is a very important operation in many fields of engineering. There are many methods [1] available and these studies first deals with gravity settlers and the efficiency of knit-mesh packing’s which can be used with such settlers, Davies et al. [3, 4]. In this work phase disengagement and behavior of unstable primary dispersions which consisted of droplets larger than 100 micron in diameter were studied in knit-mesh pads by Holywell [2]. Eckert and Gormely [12] studied the factors affecting the phase separation in a similar set up for copper extraction. Bozzano and Dante [13] studied droplet coalescence in immiscible liquid systems. Mylnek and Resnick [14] reported a work when the rate of coalescence is very low a limited number of collusion takes place. Recently, Carulucci and Kraume [15] worked on dispersion phase influence on droplet formation in stirred liquid-liquid systems. Further, King and et al. [16], K.Kumar Singh and et al. [17], John A Boxall and et al. [18], Stephanie Nachtigall and et al. [19], Topuz and Wilkinson [9], Michal and Soos [20], Mohd Izzudin et al. [21, 22], Young-Sei Lee [23], Gholam Samani and et al. [24], and Paul E. Rueger and et al. [25] investigated different aspects of dispersion created in stirred vessels of various geometries met in the literature of concern.

3 Experimental

3.1 Equipment and Chemicals Used

In this work, single-stage mixer-settler unit was picked up. It is constructed from all transparent material commercially known as “Perspex” Its length is 105 cm, wideness is 15 cm, and its height is 30 cm. The experimental set is shown in in Figure 2. The chemicals used % 20 tri-butyl-phosphates, 80% odorless kerosene (OK) technical grade as the solvent or organic phase and 5% M nitric acid making the aqueous phase .Physical properties of chemicals is shown in Table.1. Besides, in actual working conditions in the existing plant 70 grams of uranium per liter contained in the solvent phase. The mixer and tank properties are shown in Figure 1. In order to stop mass transfer from one to the other phase both liquids phases were treated together in the unit for a long time.

4 EXPERIMENTAL STUDY PROGRAM

To investigate the thickness of dispersion band in the unit, the following parameters were taken under observation. i-Total flow rate, ii-Organic-to-Aqueous rate, iii-Types of Dispersion, iv-Temperature, v-Mixer Speed During experimental study, uranium concentration was changing, but no reverse effect of the concentration change of uranium was detected.

Table 1. *Physical Properties of liquids used at 20^0C*

Liquid	ρ (g/cc)	$\mu(cp)$	σ(dyne/cm)
20% TBP/OK	0.82	3.00	14
0.05M HNO_3	1.03	1.02	-
Cyclohexanol	0.96	68[1]	4.500
Distilled water	1.00	1.00	-
MIBK[2]	0.800	0.57	10.50

(1)- Hand book of Chemistry,1976
(2)- Taken from I.C.T
*- Interfacial tension against aqueous phase –(0.05 M HNO_3)

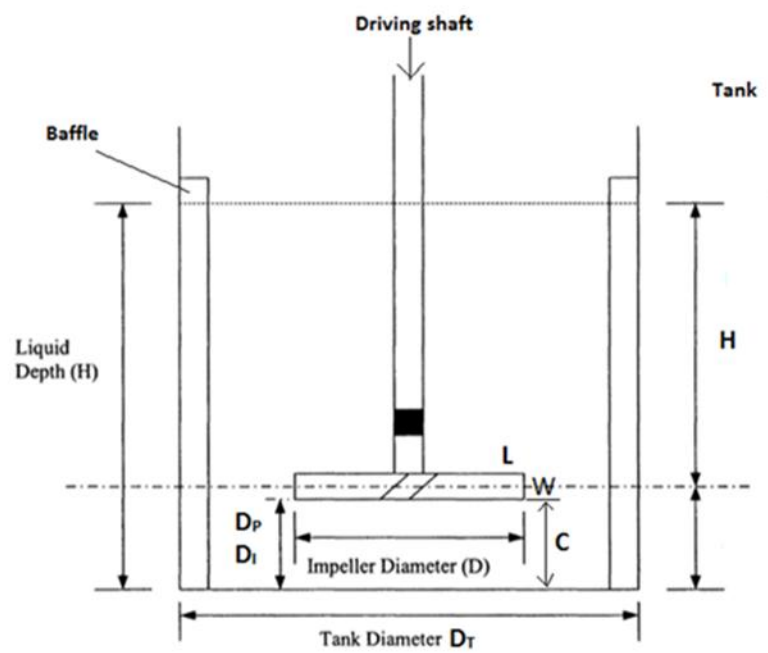

Figure 1. *Tank properties*

D_T:D_I=3.0	J:D_I=0.10
C:D_I=1.0	H:D_T=1.0
L:D_I=0.25	W:D_I=0.20
D_P:D_I=3:4	

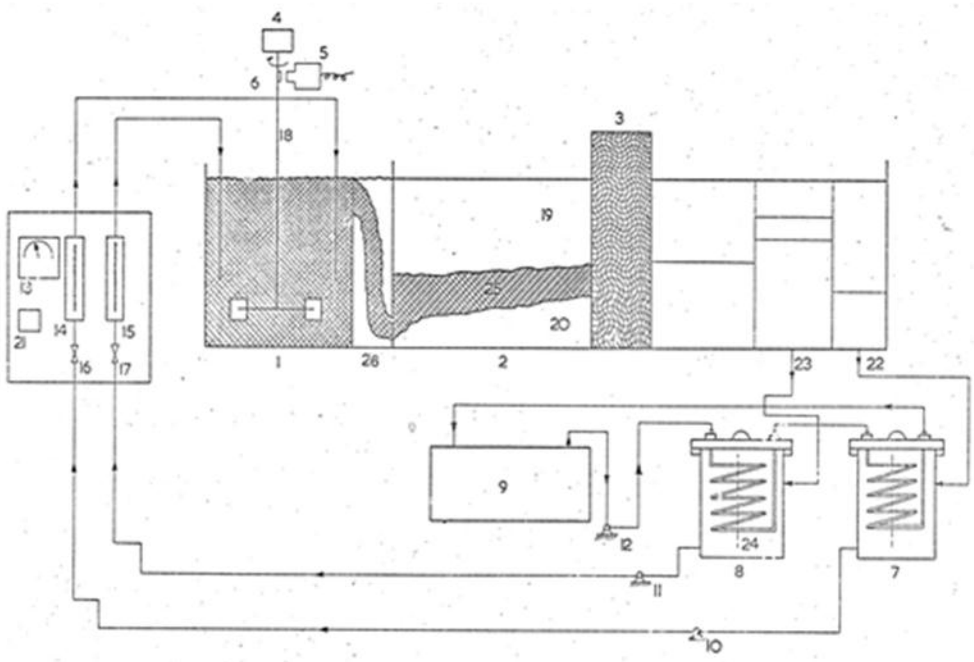

Figure 2. *General flow diagram of experimental and a pad position in the settler.*

TABLE 2: *PARTS LIST FOR FIG.1*

Item number	description	Item number	description	Item number	description
1	*Mixing vessel*	10	*Solvent pump*	19	*Solvent phase layer*
2	*Settling department*	11	*Aqueous pump*	20	*Aqueous phase layer*
3	*Knit-mesh pad*	12	*Hot water pump*	21	*Switch board*
4	*motor*	13	*Electronic tachometer*	22	*Solvent outlet*
5	*Magnetic probe*	14	*Solvent flow meter*	23	*Aqueous outlet*
6	*A piece of ferrous metal*	15	*Aqueous flow meter*	24	*Heating elements*
7	*Solvent storage tank*	16	*Solvent feed valve*	25	*Dispersion band*
8	*Aqueous storage tank*	17	*Aqueous feed valve*	26	*Mixed phase port*
9	*Temperature controlled bath*	18	*Impeller*		

5 RESULTS OF PHASE DISENGAGEMENT EXPERIMENTS

5.1 Qualitative Observations
When lower feed flow used the formation of dispersion band is very weak and little such that it may only extend to the middle of the settler. The appearance of the band looks as if a knife edge larger at the closer distance to the mixer as the band far from the mixer it became less thicker than the head side and it just looks like a knife. On the other hand, as the feed flow increased, the emulsion band got substantially increased and it was so apparent and visible such that the emulsion band taken up the entire length of the settler. Further, it looks like a good shape of a rectangular.

5.2 Performance observation with no coalesce aid present in the unit
To understand the performance of the system without any aids present in the unit, a large number of experimentations were conducted. Only the ones notable were discussed below.

5.2.1 The Effect of mixer size
The effect of impeller size on the dispersion band thickness was investigated using three different sizes of six-flat-bladed turbine impellers as shown in Figure 1. Both types of emulsions were formed at various speeds and phase ratios in no case a phase inversion occurred. At lower speeds, using the smaller size of impeller, the intensity of mixing was low but with the larger impeller much more rigorous mixing was observed. The results are given in Figure 3 and 4. The trends are expected. In all cases the emulsion band increases, approximately linearly with throughput. The result also show that more rigorous mixing results in poorer settling.

5.2.2 The effect of emulsion type
The type of dispersion formed was easily detected by visual inspection. A substantial improvement in phase settling was seen when aqueous –in-oil dispersion was used. A detailed work was done by Topuz and Wilkinson [9].

5.2.3 Effect of solvent to aqueous ratio

Emulsion band thickness in the settler was considerably increased with a solvent/aqueous ratio of 1:2 compared with solvent /aqueous ratio of unity using aqueous in oil dispersions. A reverse situation occurred when the dispersed phase was changed to solvent at the same solvent/ aqueous ratio. The results were shown in Figures 5 and 6. The results show that the phase ratio solvent to aqueous is of importance on the operating performance and in the design of a mixer-settler. The result shows that better settling obtained when the minor phase was dispersed as shown by Topuz and Wilkinson [9].

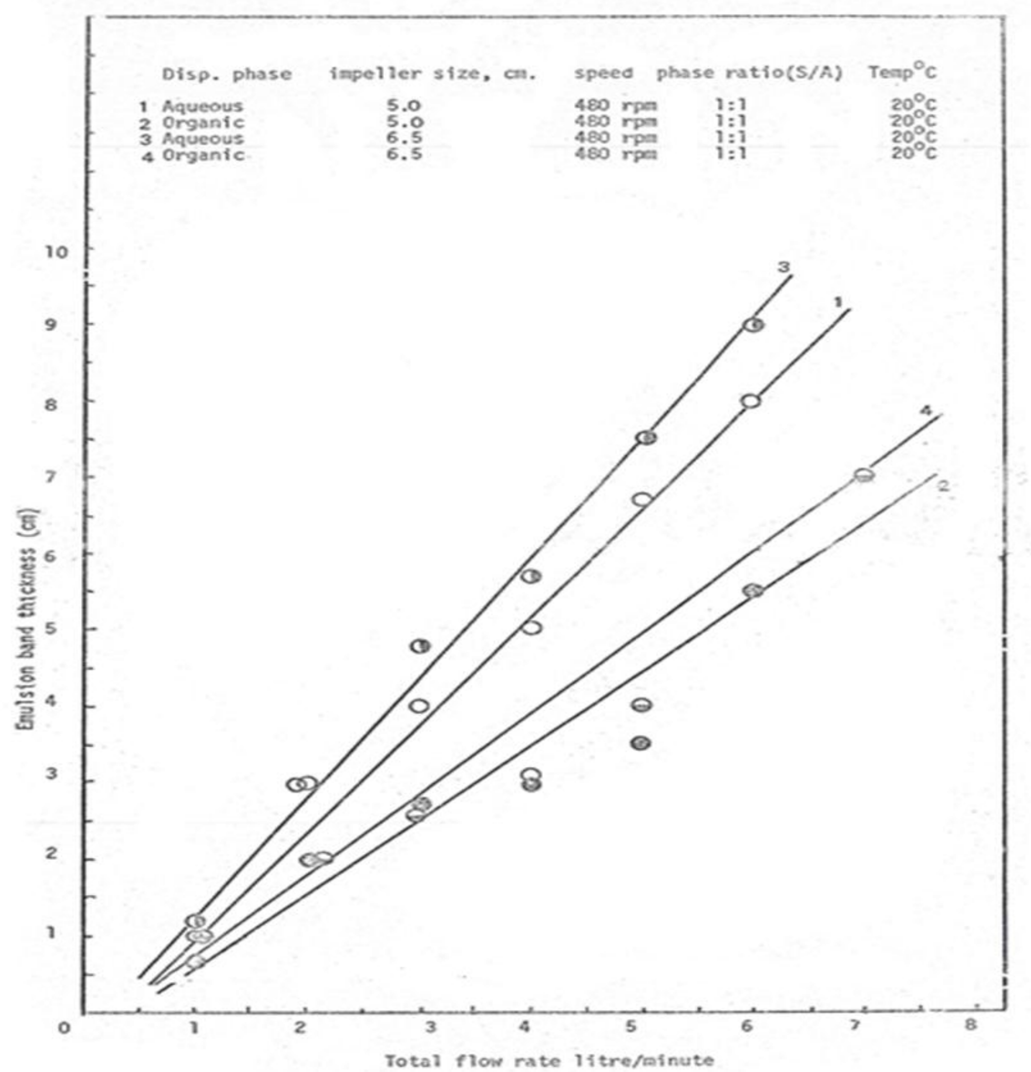

Fig. 3. *Effect of impeller size and emulsion type*

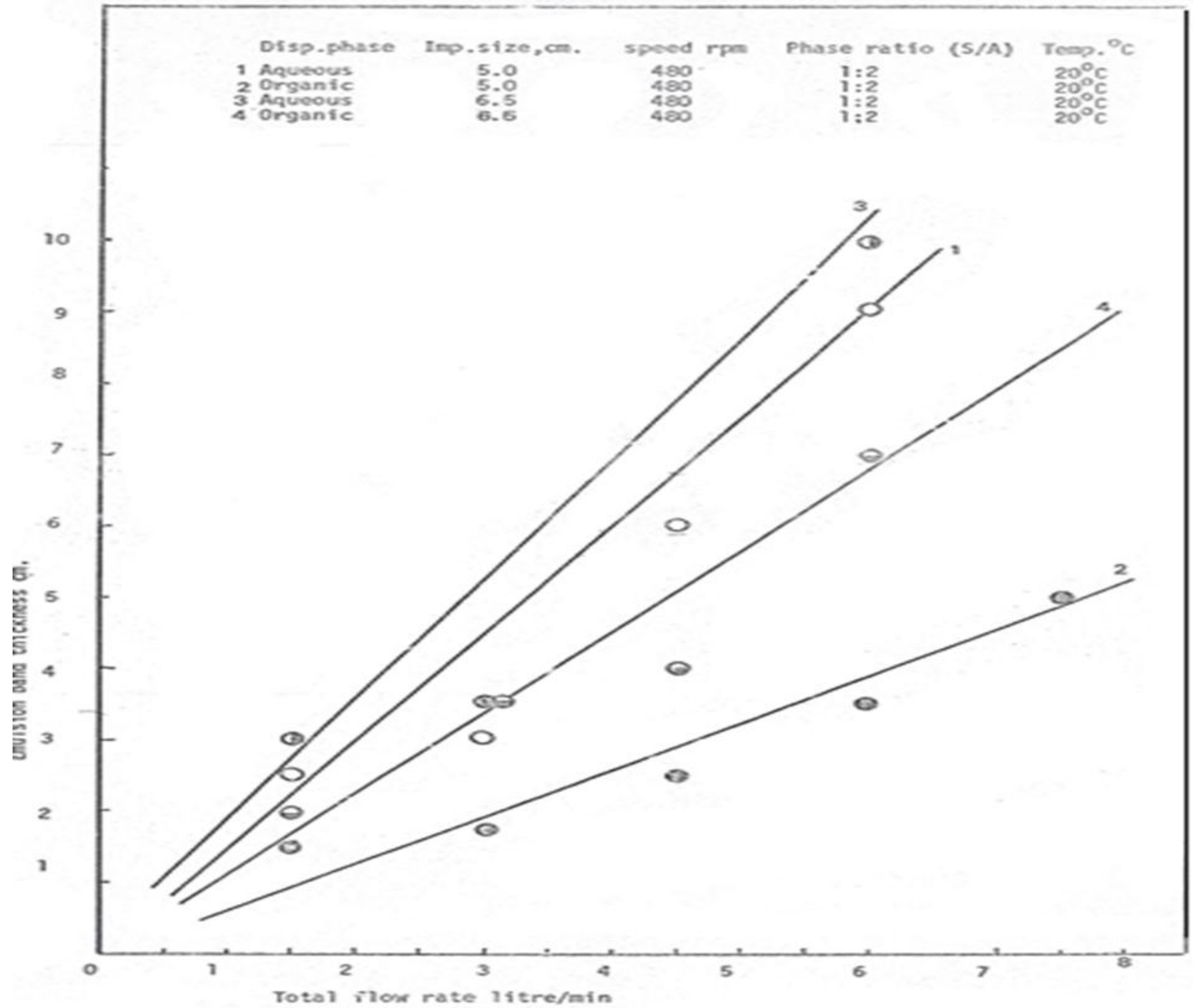

Figure 4. *Effect of impeller size and emulsion type*

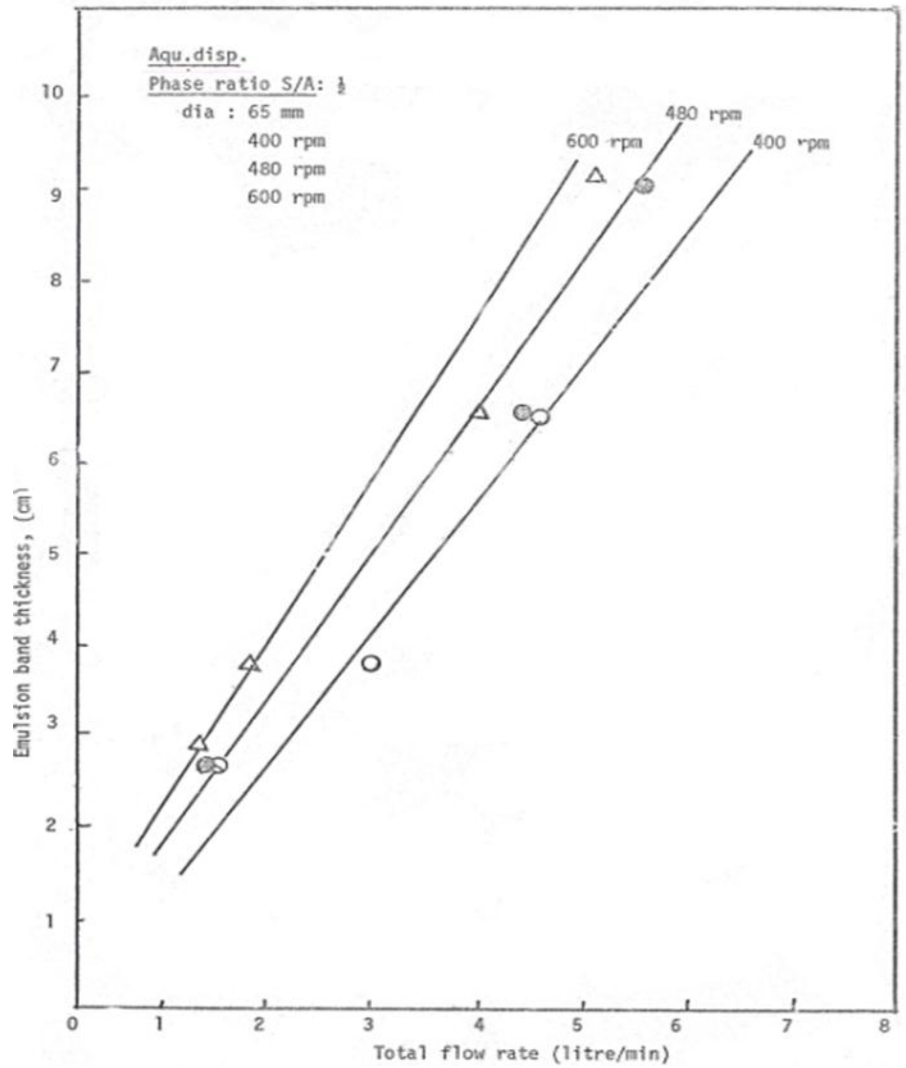

Figure 5. Effect of mixer speed and phase ratio when aqueous dispersed

5.2.4 Effect of mixer speed

The effects of mixer speed are illustrated in Figures 5 to 8. The results generally indicate that the emulsion band thickness increased with increasing impeller speed for both types of dispersions. Evidently, the effect of emulsion band thickness and so maximum throughput is quite significant for the aqueous dispersion but not for the solvent dispersion. This means more intense mixing gives emulsions with poorer settling characteristics. Entrainment noticeably is worse at the higher speeds. The results are in good agreement with Barnea and Mizrahi [5 - 7], and Ryon et al. [8].

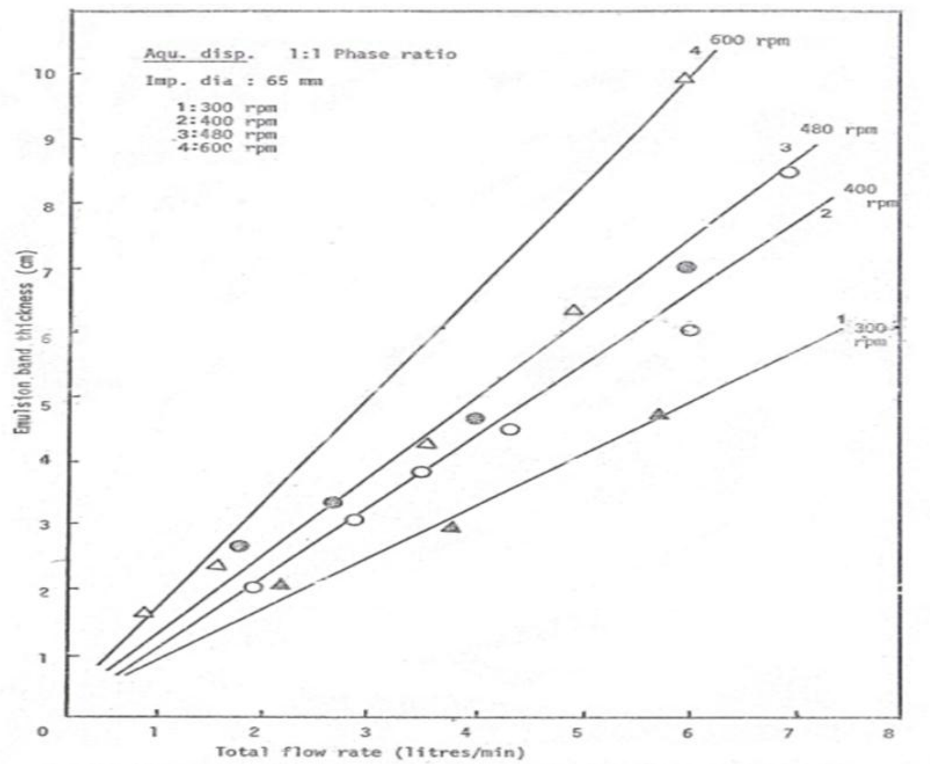

Figure 6. *Effect of mixer speed and phase ratio when aqueous dispersed*

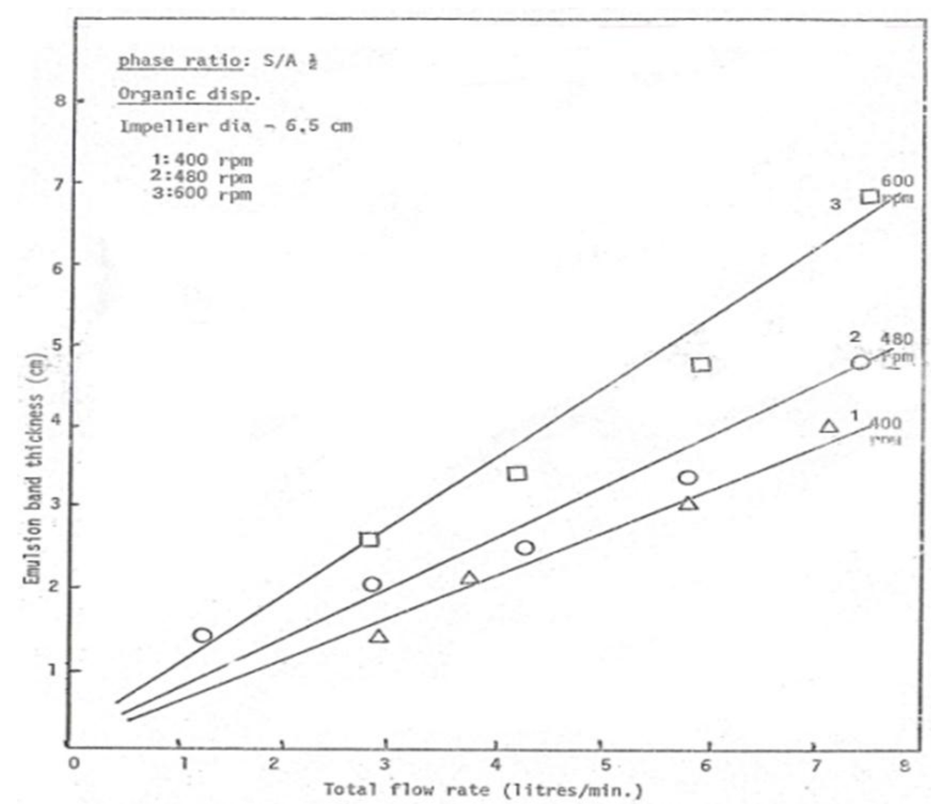

Figure 7. *Effect of mixer speed and phase ratio when organic dispersed*

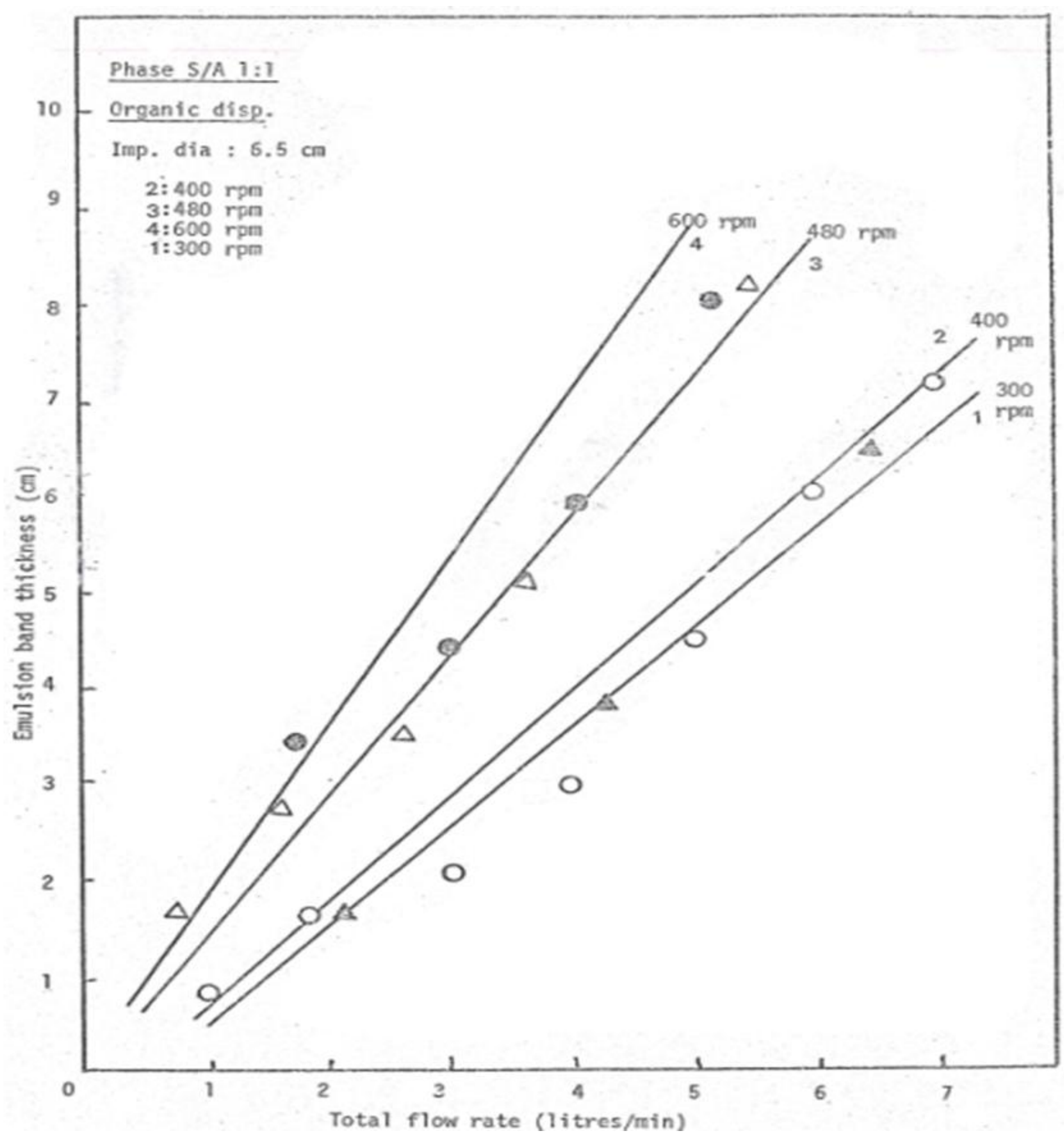

Figure 8. *Effect of mixer speed and phase ratio when organic dispersed*

5.2.5 Effect of Temperature

The effect of temperature on phase disengagement over a range of temperatures from 200 to 400 C at various flows was studied. The heating arrangement was shown in Fig. 2. The results are given in Figures 9 and 10. The effects of mixer speed and phase ratios when different phases dispersed are given in Fig. 7 and 8.

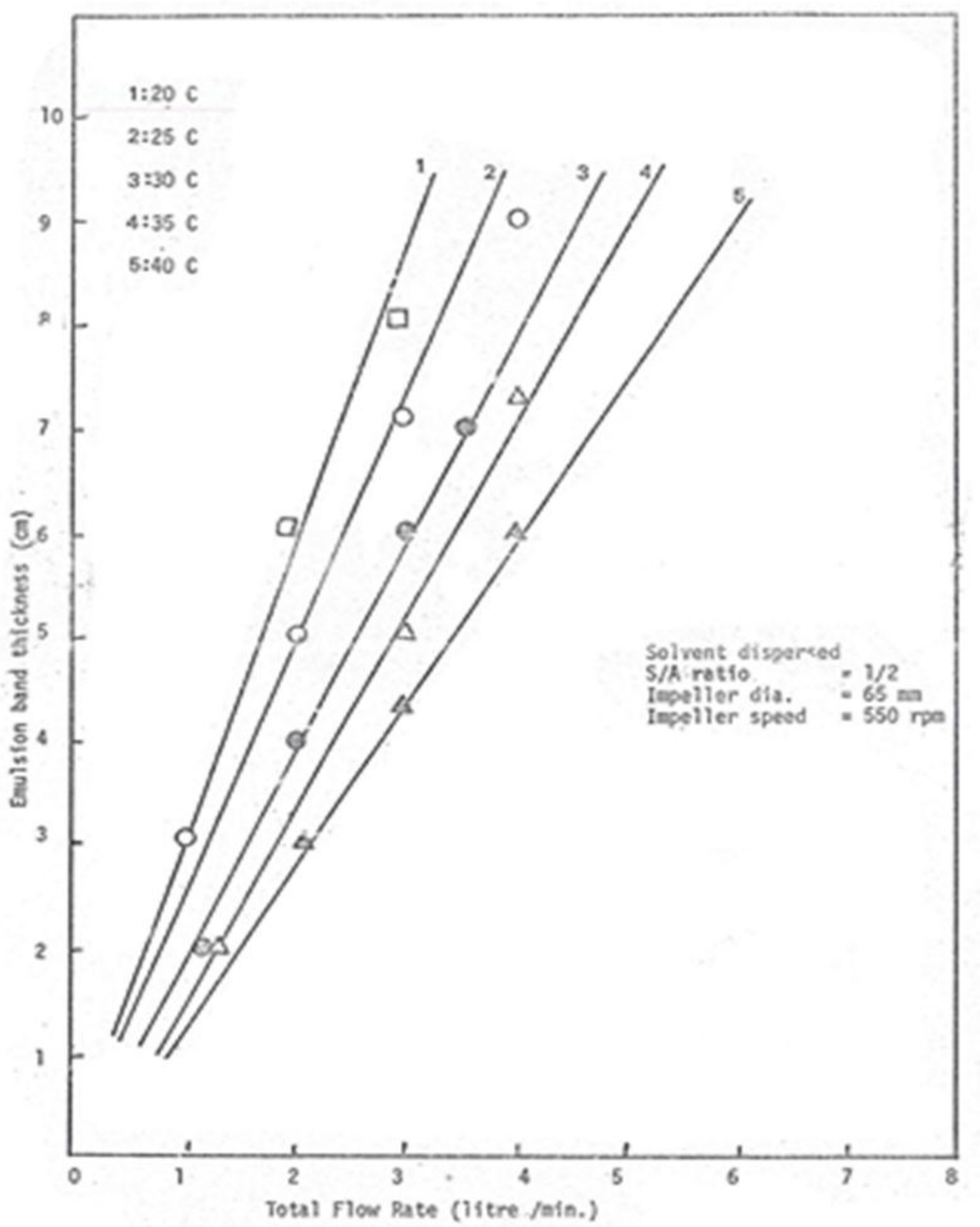

Figure 9. *Effect of temperature (organic dispersed)*

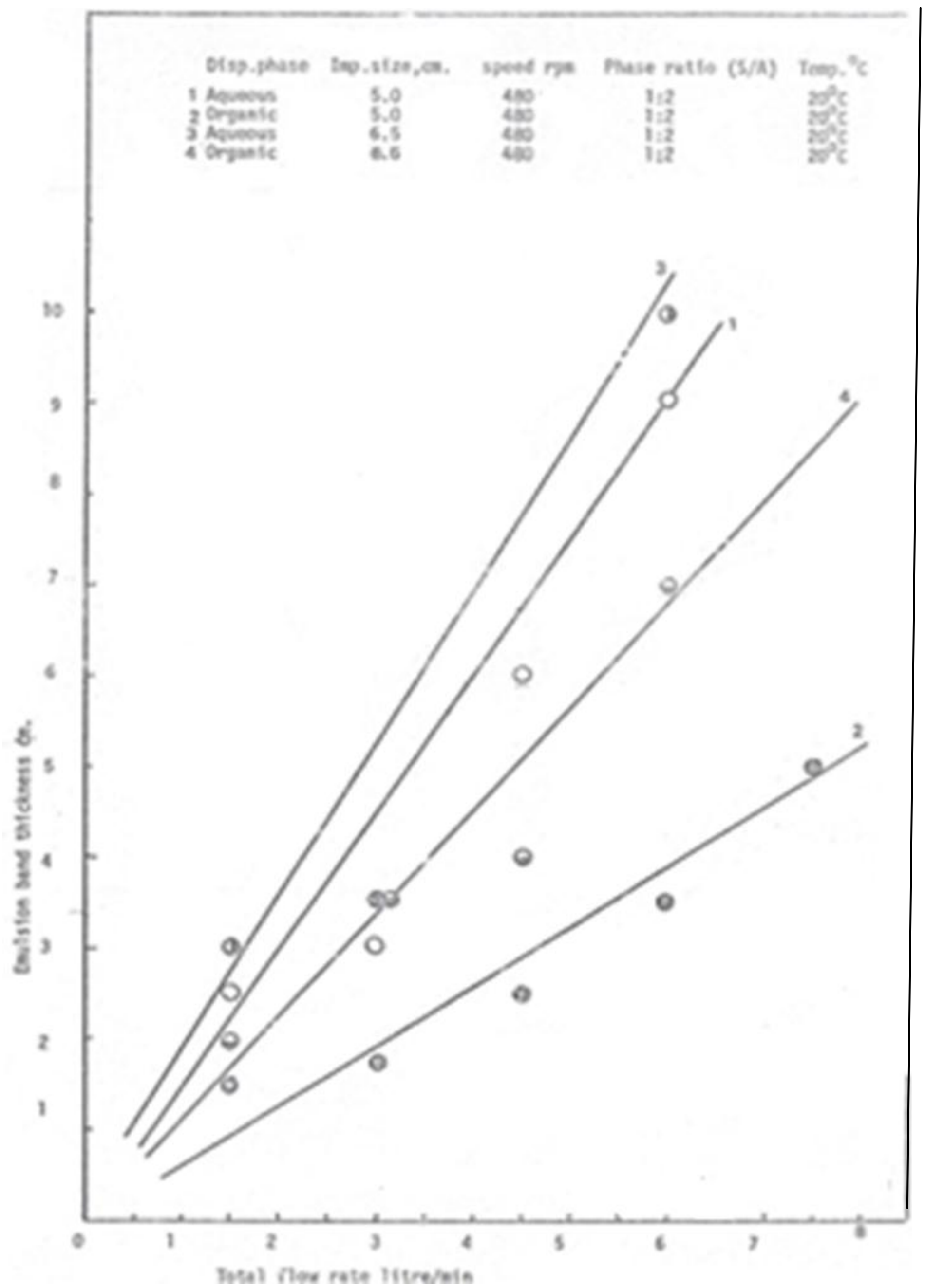

Figure 10. *Effect of temperature impeller size and emulsion type*

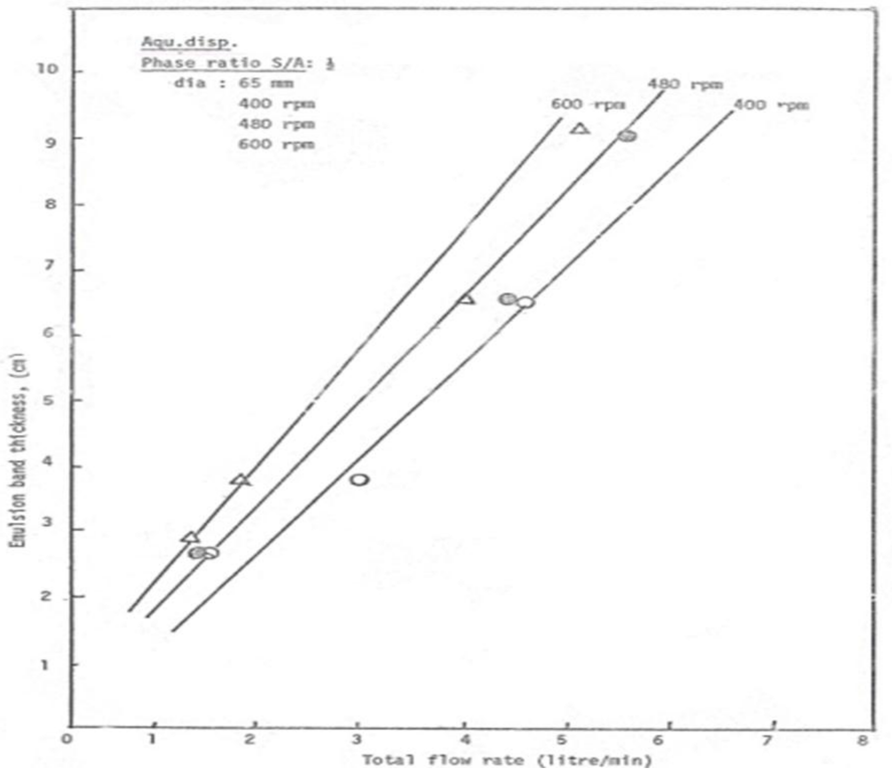

Figure 11. *Effect of mixer speed and phase ratio when aqueous dispersed*

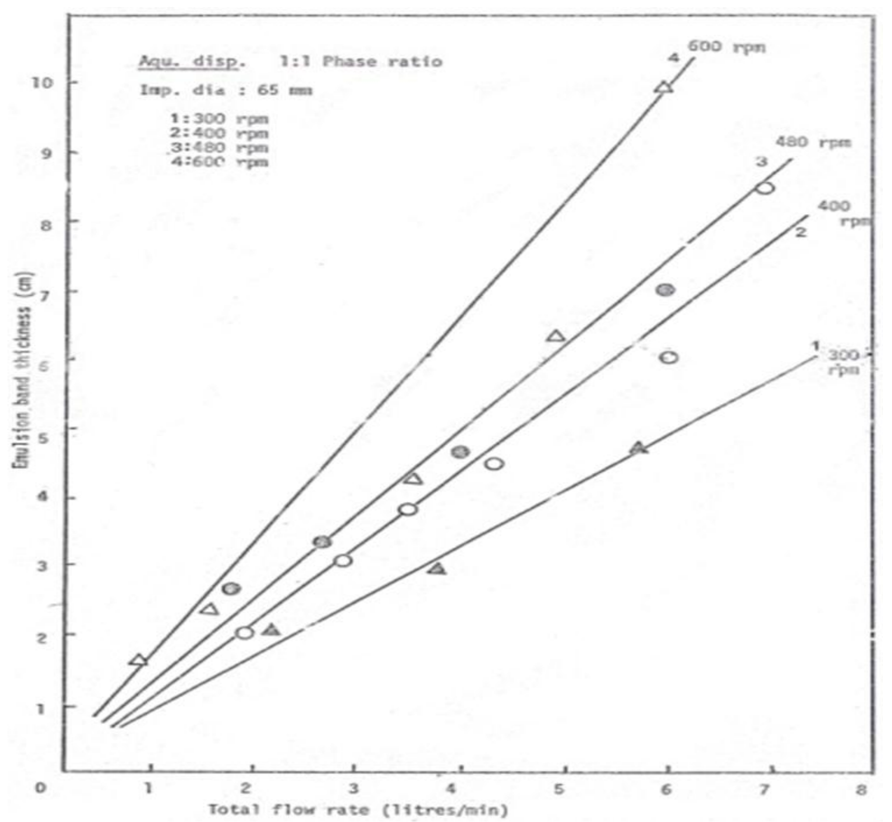

Figure 12. *Effect of mixer speed and phase ratio/ when aqueous dispersed*

These show that the emulsion band thickness decreased almost linearly with increasing temperature. This is in line with literature records. The higher the temperature the better the settling profiles. The results indicate that increasing temperature by 200 C is doubling of the settling rate. The results derived from temperature effect experiments showed good agreement with Barnea and Mizrahi [5 – 7], and Ryon et al. [8].

5.2.6 Effect of Entrainment

When working with solvent –in-aqueous emulsions both phases showed signs of entrainment and this again became worse as the pad moved close to the mixed phase port. When working aqueous to solvent phase emulsions, the aqueous phase behind the pad was always clear. The entrainment was very low, but the solvent phase was cloudy, especially the pad was placed close to the mixed – phase port. The results are shown in Figure 13. Topuz and Wilkinson [9].

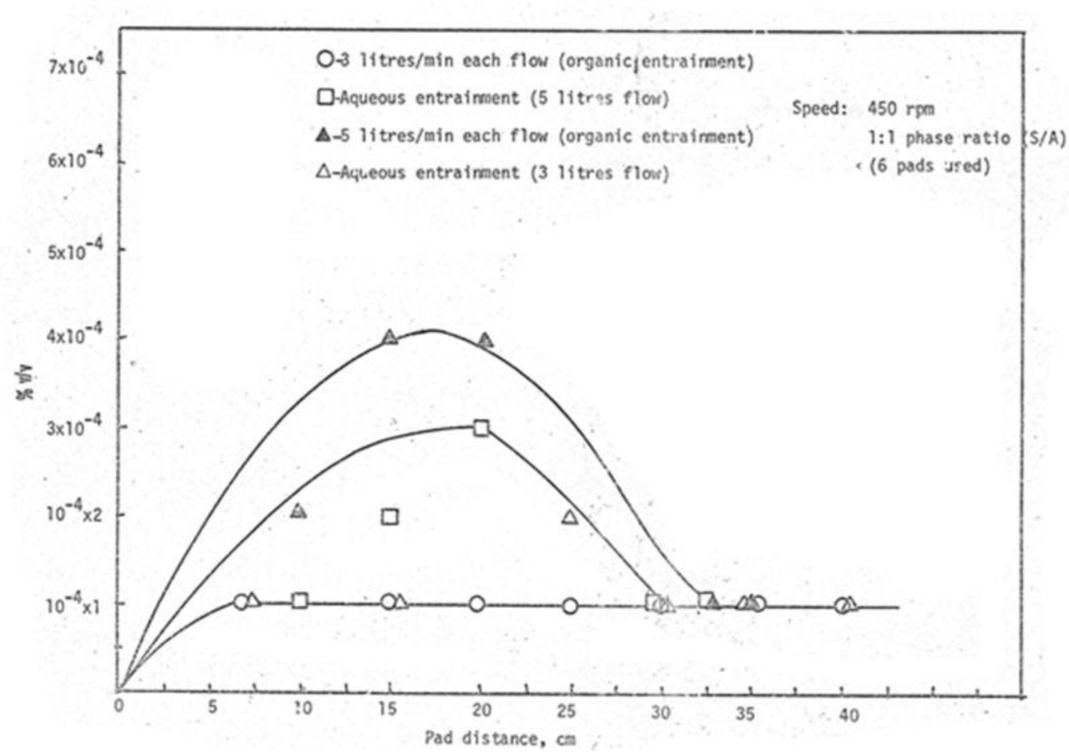

Figure 13. *Entrainment of Phases*

6 PERFORMANCE WITH NYLON PADS IN THE SETTLER

A pad equal in size to the duplex pad was made up from domestic nylon pan scrubbers and the investigations were repeated under the same conditions as before. The effect of coalescence was equal to that of the duplex pad in every case. Surface wetting properties of the material do not appear to be very significant. However, due to the more dense structure of the nylon pads the pressure drop was increased to a maximum of 2.5 cm compared with 1.5 cm, as resulted from the knit mesh pad. Topuz and Wilkinson [9], and the chemical Engineer [1].

7 DISCUSSION OF RESULTS

Studies derived from both experimental works indicated that with no pad present in the unit maximum working flow capacity was found to be 6 liters. This flow produced an emulsion band of 7 cm band thickness. The rate of coalescence in the unit was calculated to be 4 m3/h per m2 of the settler unit. When knit mesh used the rate of coalescence 4 m3/hm2 was increased to 10 m3/hm2. This means the rate of coalescence has been increased by a factor of 2.5. The corresponding emulsion band thickness was observed to be 20 cm. This high band thickness can only be exercised in a deep settler unit. It became clear that both pads should be placed at a certain position within the settler unit. Because this directly influence the capacity of the unit. Pressure drops across on both pads not a critical issue and therefore do not create an engineering problem. i.e. for a larger capacity unit design. Further, it was revealed that the surface properties of both pads seem to be not an important parameter. Finally, both aids may be successfully and safely applied to such separation and purification operation in hydrometallurgical processing technologies.

8 ACKNOWLEDGEMENT

I would like sincerely to acknowledge late Professor W.L. Wilkinson's guiding whom has shown me during this project under his supervision in Chemical Engineering Department as The Chairman School of Post-Graduate of Chemical Engineering Department at University of Bradford, England, The UK. I would also like to acknowledge Turkish Ministry of Education for the financial support for this study to make possible.

9 ADDRESSES

Correspondence concerning this paper should be addressed to Dr. H. Topuz, Maltepe University, Engineering And Natural Sciences, Başıbüyük, Maltepe, Istanbul ,Turkey

10 Nomenclature

D_I	*Impeller size (cm)*
D_T	*Tank size (cm)*
μ	*viscosity, cps*
σ	*interfacial tension dynes/cm*
ρ	*density, gr/cm^3*
I	*Impeller size*
T	*Tank size*
H	*liquid level in tank, cm*
C	*impeller height off tank bottom*
J	*baffle width*

References

[1] The Chemical Engineer,No.266,Oct.1972

[2] A New Packing for Gas- Liquid and Liquid-Liquid Separation Processes ,by Knitmesh Ltd.,Greenfield,Holywell,Flintshire,U.K.

[3] G.A.Davies,Jeffreys,G.V., New Packings for Liquid-Liquid Separtors and Liquid Extractors, Chemical Processing ,Nov.1972.

[4] G.A.Davies,Jeffreys,G.V.,,Coalescence of Drops in Packing-Factors affecting the Separation of droplet Dispersions,Filration and Separation,July / august,1969.

[5] Barnea,E,and Mizrahi, Trans. Instn. Chem. Engrs., Vol.53,Part II ,IV,1975.

[6] Barnea,E, and Mizrahi, Trans .Instn. Chem. Engrs., Vol.53,Part III ,1975.

[7] Barnea,E,and Mizrahi,Trans.Instn.Chem.Engrs.,Vol.53,Part I ,IV,1975.

[8] Ryon,A.D.,Daily,F.L. and Lowrie,R.S. ORNL Oak Ridge ,Tenn. US Atomic Energy Comm.Oct.,1960.

[9] H.Topuz,Ph.D.Thesis ,Bradford University , England,The UK.,1978

[10] Davies,G.A.,Jeffreys,G.V.,Ali,F.,Design and Scale-up of Gravity Settlers,The Chemical Engineer,Vol.25,Nov.1970.

[11] Jackson, F.D., Newrick, G. M., Warwick, G.C.I.,A Recent Development in the Design of Hydrometallurgical Mixer-Settlers, Inst. Chem. Engrs. Symposium Series No.42.

[12] N.L.Eckert and L.S. Gormely,Phase Separation in an experimental Mixer-settler, Ins.Chem.Eng. Res .des, Vol.67, March (1989).

[13] G.Bozzano, M.Dente M.,Mechanism of drop Coalescence at the Interface of two Immiscible liquids,20th European Symp.on Computer Aided process Engineering,(2001)

[14] Y. Mylnek, and W. Resnick , AIChE J,18,1,122 (1972)

[15] G. Carlucci, and M. Kraume, Drop Size Distribution in Liquid/Liquid Systems Influenced on the Dispersed phase. Master final project ,Matr.No.331007Techische .Universitat Berlin- Institut fur Prozess-Und Verfahrenstechnik.

[16] B.A. King, and T.W. Winward, and D. L. Bjorneberg, Am. Soc. Agri. and Bio.engrs.,Vol.30(3), (2014).

[17] K.K.Sing,S.Madhusudan Mahajani,and S.Ghosh. Hydrometallurgy, Feb.2008

[18] John A. Boxall ,Carolyn A. Koh,E. Dendy Sloan, Amedeu K. Sum, and David , T. Wu., Ind. Eng.Chem.Res.,2010,49(3), pp1412-1418

[19] Stephanie Nachtigall, Daniel Zedel, Mattias Kraume, Analysis of drop deformation in turbulent flow, Chinese Journal of Chemical Engineering,2015CrossRef.

[20] Michal Vonka, Miroslav soos, Characterization of liquid-liquid dispersion with variable viscosity by coupled computational fluid dynamics and population balances, AIChE J, 2015,61,8,2403 Wiley Online Library.

[21] Mohd Izzudin Izzat Zainal Abidin, Adul Aziz Abdul Raman, Mohamad Iskandr Mohamad Nor, Dep. of Chemical Engineering, Faculty of Engineering, University of Malaya, 50603, Kuala Lumpur, Malaysia, Ind. Eng. Chem.res.2013,52(46),pp 16085-16094.

[22] Mohd Izzudin Izzat Zainal Abidin, Adul Aziz abdul Raman, Mohamad Iskandr Mohamad Nor, Experimental Investigation in Liquid-Liquid dispersion System:Effects of Dispersed Phase ,Viscosity and Impeller Speed, Industrial & Engineering Chemistry Research,2014,53,15,6554CrossRef.

[23] Young-Sei Lee, Characteristic of Mass Transfer Volumetric Coefficient and Sauter Mean Diameter in a Liquid-Liquid Agitated Vessel,Korean Chemical engineering research, 2012,50,5,913CrossRef.

[24] M.Gholam Samani,A.Haghi Asl,J.safdari,M.Torab - Mostaedi, drop size distribution and mean drop size in a pulsed packed extraction column, Chemical Engineering Research and Design2012,90,12,2148CrossRef

[25] Paul E. Rueger, Richard V. Calabrese, Distribution of water into oil dilute systems. Chemical Engineering Research and Design, 2013, 91, 11,2122CrossRef.