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Gullwing Liquid Extraction / Separation Modus-Operandi

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Abstract: *Traditional Mixer-settlers are still widely used in the Chemical Process Industry for the operation which requires high capacity and the few stages like fermentations, mixing & separating the two phases. The obvious disadvantages of mixing & settlings are the capital cost, space requirement and the inventory of material held up in the equipment. Consequently an economic design of contactor would be producing the most efficient contacting combined with rapid complete separation of the two phases in the smallest possible volume and time. While there are number of alternatives to the mixer-settler out of which the GULLwing Extractor appears particularly promising due to its simplicity, compact nature and energy efficiency. The GULLwing Contactor is novel design of equipment which works by turbulent jet contacting, combined with instantaneous hydro-cyclone separation for extraction of solute from one solvent to another where the two solvents are immiscible and have a density difference. The fundamental reason, why in the GULLwing Contactor mass transfer coefficient is increased, is due to high relative velocity between two phases which reduces interfacial film resistance. Modified design of batch as well as two stages continuous Contactor has been successfully developed and optimised for a test system. Compared with traditional mixer-settler the GULLwing Contactor reduces processing time and energy consumption it was found for batch Contactor it requires only 11% of the power W/g/m^3 and 12% of the energy J/g/m^3 and for continuous Contactor it requires only 14% of the power W/g/m^3 and 10% of the energy J/g/m^3. Similarly total extraction on batch Contactor is 7.68 g/sec/m^3 and on continuous Contactor is 75.9 g/sec/m^3 as compared to 4.21 g/sec/m^3 of the mixer-settler. The batch Contactor gives 82% more extraction and continuous Contactor gives 18 times more extraction for the same system with the same chemical composition as compared to traditional mixer-settler. The GULLwing Contactor would be well suited to hazardous biochemical, nuclear fuel extractions, irradiated fuel processing, and food processing.*

Keywords: *Gullwing Contactor, Liquid-Liquid Extraction, Separation Technique, EA-TCE & WATER system.*

1. INTRODUCTION

Extractions are used when we want to separate substances. One way this can be done is by using a solvent in which a desired substance dissolves in and the undesired substance does not dissolve in. Apparently, the solubility is a function of how well a substance dissolves in a solvent which was around 8%. Liquid extraction or solvent extraction refers to an operation in which the components of a liquid mixture are separated by contacting it with a suitable insoluble liquid solvent, which preferentially dissolves one or more components, as described by [1]. In liquid-liquid extraction, the separation of the components of a solution depends upon the unequal distribution of the components between the two immiscible liquids [2]. A typical liquid-liquid extraction operation utilising differences in solubilities of the components of a liquid mixture may be described as consisting; contacting the feed with a solvent, separation of the resulting phases and removal and recovery of solvents from each phase. In the liquid-liquid extraction operation, the liquid mixture to be treated and a suitable insoluble solvent are contacted intimately. The constituents of the liquid mixture are distributed between the phases resulting into some degree of separation (which refer to be improved by a multistage contact), and then the phases are separated from one another based on the density difference. The basic separations & extraction are presented in the Figure (1) which shows that the three phases Predicted means calculated & Experimental findings of liquid-liquid extraction system.

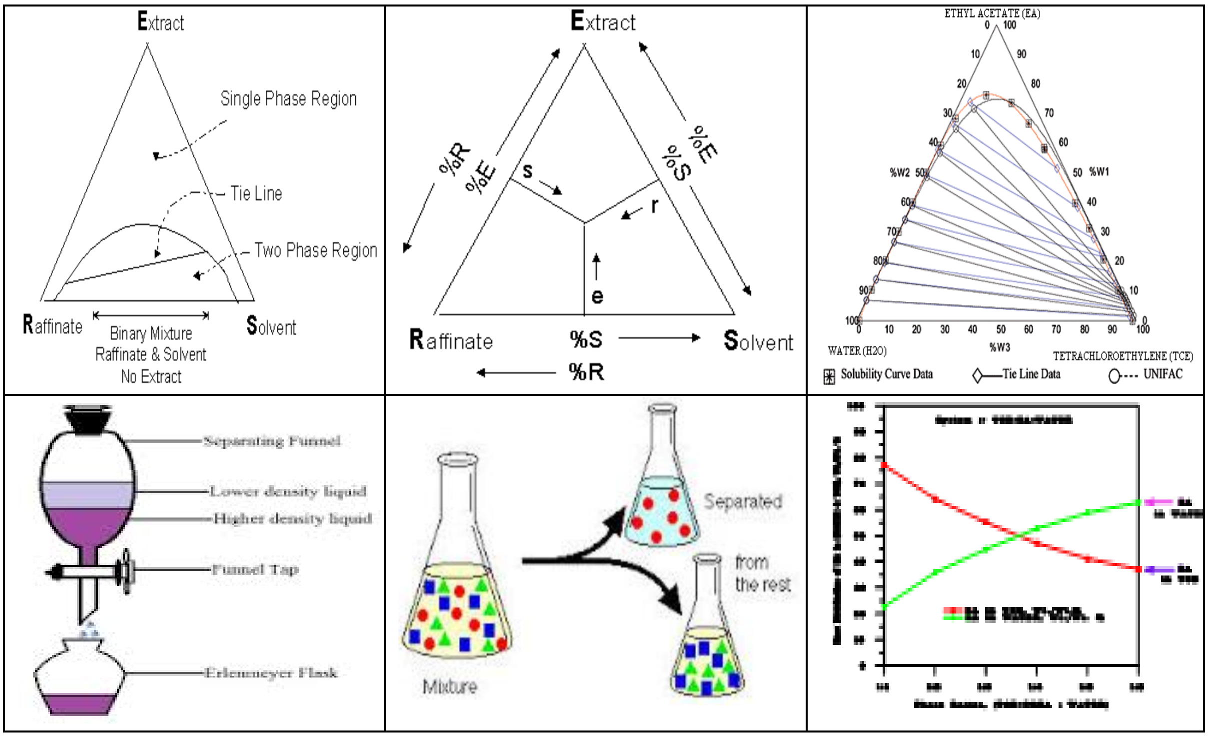


Fig1. *Ternary Diagram for Predicted and Experimental System of Ethyl Acetate (EA)- Water (H_2O) – TetraChloroEthylene (TCE), Binodal curves and TIE lines.*

According to a qualitative of the economic operating range chart of various classes of Contactor as shown by [3], it concluded that the GULLwing Contactor somewhere between the Centrifugal Contactor Gravity-flow and mixer-settler. Figure (2) suggested a selection guide for use in choosing industrial Contactor for any available process. From the figure it should be clear that there are several specific extraction processes for which a change in the method of extraction or in operating conditions may be justified on energy saving grounds, for example, the replacement of traditional mixer-settler with GULLwing-Contactor.

Liquid-liquid extraction, also known as solvent extraction and partitioning, is a method to separate compounds based on their relative solubilities in two different immiscible liquids, usually water and an organic solvent. It is an extraction of a substance from one liquid phase into another liquid phase. Liquid-liquid extraction is a basic technique in chemical laboratories, where it is performed using a separating funnel as shown in Figure (1). In other words, this is the separation of a substance from a mixture by preferentially dissolving that substance in a suitable solvent. By this process, a soluble compound is usually separated from an insoluble compound. Solvent extraction is used in nuclear reprocessing, ore processing, the production of fine organic compounds, the processing of perfumes, and other industries.

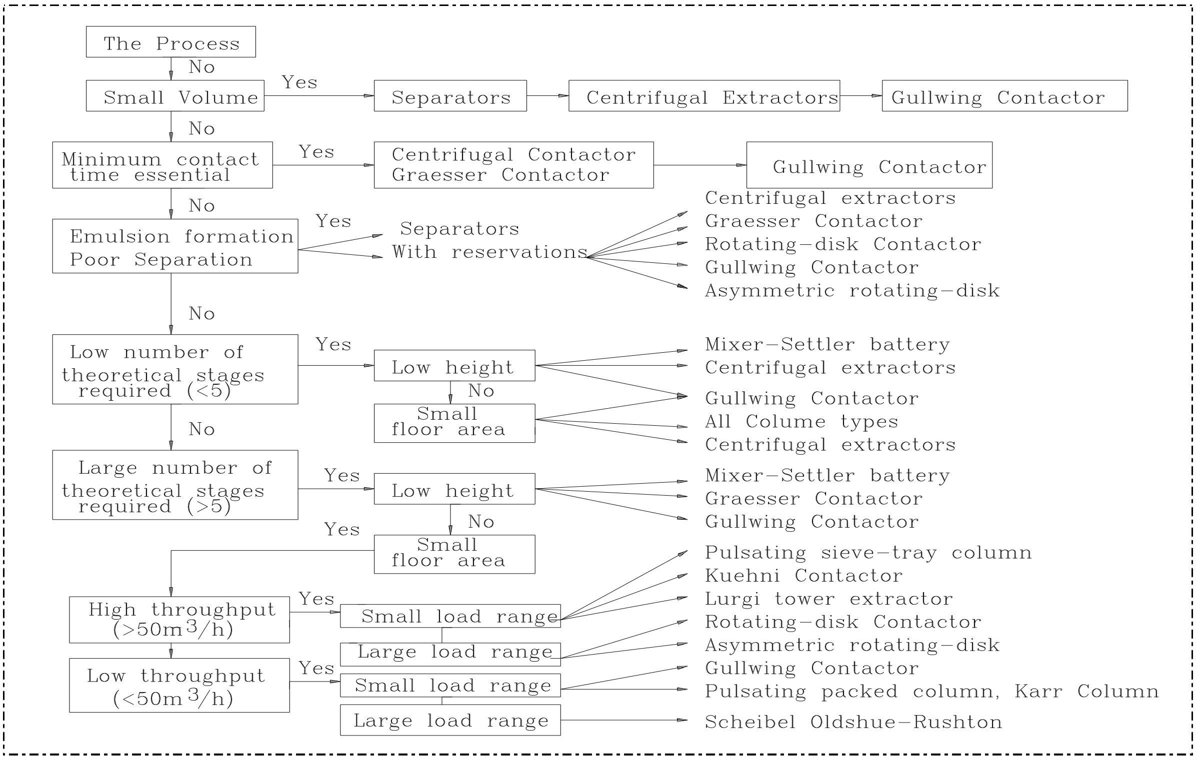


Fig2. *Decision Network for Selection of Liquid-Liquid Contactors for any available process, from Ricci and Staff [3], now including GULLwing Contactor.*

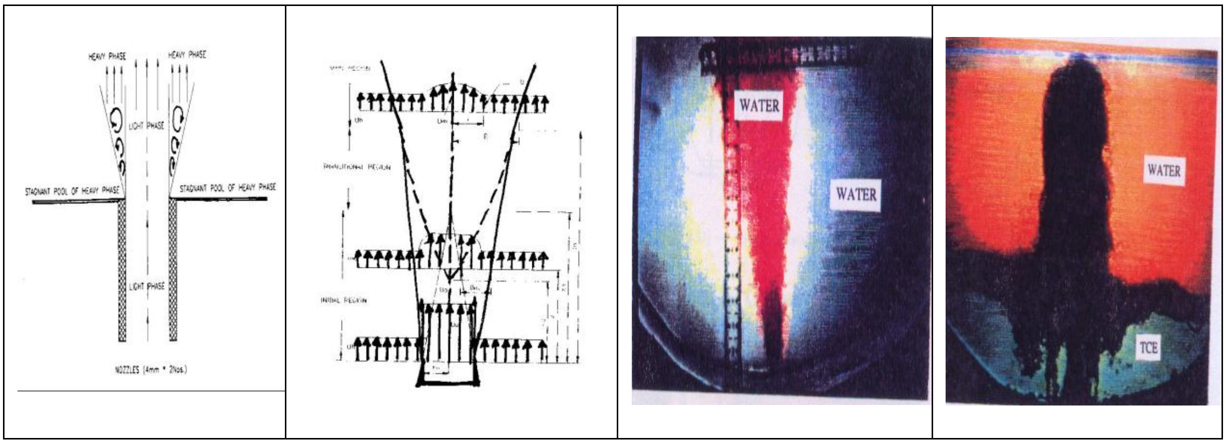


Fig3. *Turbulent Jet Behaviour in Water & TCE*

The turbulent contacting (extraction) zone is to produce high relative velocities between the two liquid phases so as to accelerate diffusion controlled extraction processes as described by Treybal [4]. This is the heart of the Contactor, the other zone are ancillary to it, droplets of the heavy phase that are not separated under the Gullwing may be carried over by the light phase in its radial as well as axial motion towards the separation zone of the Contactor. At the same time adjustable Gullwing promotes radial escaping of light phase from the sides of the Gullwing and allowed major portion of heavy phase droplets to coalesce and descend under the influence of gravity into the heavy phase pool for further contact and re-entrainment by the jets of light phase.

The present work is part of a continuing development of an economic design of Contactor which attempt to achieve interphase mass transfer and phase separation essentially simultaneously. The design has now been refined and it is shown in figure (3). The major change from the previous design is that the GULLwing is no longer fixed to the vessel but can be moved up and down with the help of adjustable GULLwing Hanger. This also allowed fluid to escape radially from the edge of the GULLwing as well as axially. In the present study the performance of jet Contactor is studied by extracting ethyl acetate EA ($CH_3CO_2C_2H_5$) from TetraChloroEthylene TCE ($Cl_2C = CCl_2$), into water establishing a driving force for the mass transfer from TetraChloroEthylene to water jets. EA, & TCE, both chemicals are considered to be safe and therefore we choose this system with solubility high 8%, from [4].

2. SCALE UP & DOWN

Scale-up means increasing the scale of a liquid-liquid extraction for the laboratory scale to the pilot scale to the production scale. It is the tune of the extraction technologist to increase the scale of a extraction without a decrease in yield or if a yield reduction occurs, to identify the factor which gives rise to the decrease and to rectify it as explained in [5]. The accuracy of scale-up techniques is only as good as power and correlations, so it is expanding some considerable time to test the validity of potential correlation in the extraction in questions. Particularly for Chemical jet GULLwing and air lift vessels tend to be scaled up on basis of geometric similarity, constant liquid velocity. The major difference will be the length of vessel resulting in increased pressure at back of larger vessels, which complete set up of the GULLwing contactor is shown in figure (4).

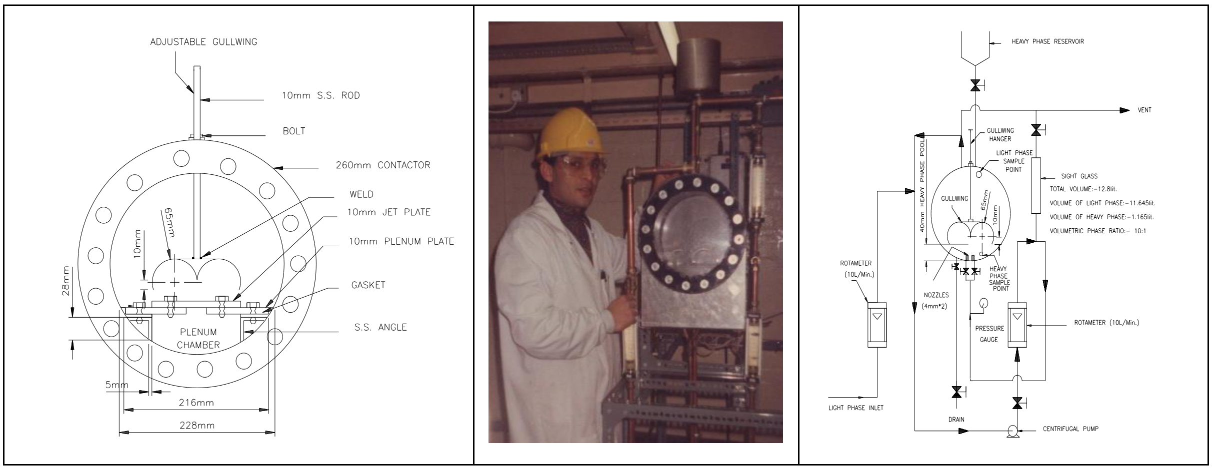


Fig4. *Schematic Flow Diagram of the Batch Gullwing Contactor*

3. EXPERIMENT

The Contactor is essentially a horizontal cylindrical vessel along the axis of which are two semi-circular pipe sections joined together to form a Gullwing. Immediately below the Gullwing is the number of nozzles through which the light phase is recirculated. The jets of the light phase contact break up and entrain the heavy phase liquid, which lies as a heavy phase pool in the lower part of the vessel. Contacting is mainly dependent on what happens at the jets, whereas separation depends on what happened in the hydro cyclone under the Gullwing [5]. The entrained droplets of the heavy phase are propelled upwards by jetted light phase on to the underside of the Gullwing. Here the vertical motion is converted into circular motion [6] and under each half of Gullwing a semi-hydro cyclone effect must be established as shown in figure [5].

Observations were made with increasingly larger nozzles, from 5mm to 8mm diameter, indicated that even with quite low linear velocity of the light phase at the nozzle exit (i.e., lower than 2m/s). The heavy phase were dispersed by the increasing mass flow rate of light phase to such an extent that the two phases emulsified and became impossible to separate. The optimum combination of extraction and separation was obtained using 4mm diameter nozzles with a light phase velocity of 2m/s at the nozzle exit with 40mm pool of heavy phase without the entrainment. Series of experiments were carried out to achieve optimum height of the Gullwing from top of the heavy phase pool depth which would generate the most effective hydro cyclone in terms of the degree of separation of light phase and heavy phase.

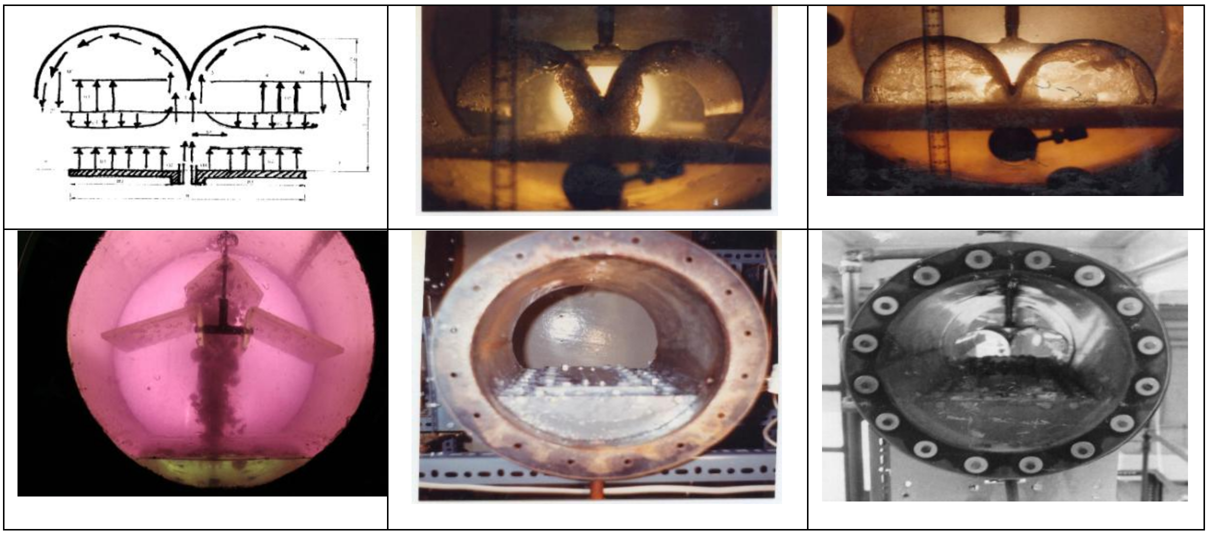


Fig5. *Jets Behaviour under the GULLwing*

The vertical position (height) of the Gullwing from the jet is found that 60mm height of the Gullwing above the jet exit gave the shortest possible time for 99% extraction without any carryover of TCE in the light phase at 2m/s L.P. Samples were taken at measured time interval from heavy phase pool for ethyl acetate content and from light phase for tetrachroethylene content until an equilibrium concentration was achieved, and analysis of these samples were carried out by using gas chromatography [7].

4. RESULTS & CALCULATIONS

In order to assess the progress of extraction towards equilibrium of EA in the Gullwing Contactor, equilibrium curves for different phase ratios were established by utilising conventional GC apparatus. The extraction performances of the Contactor were calculated from the extraction efficiency index based equation (1).

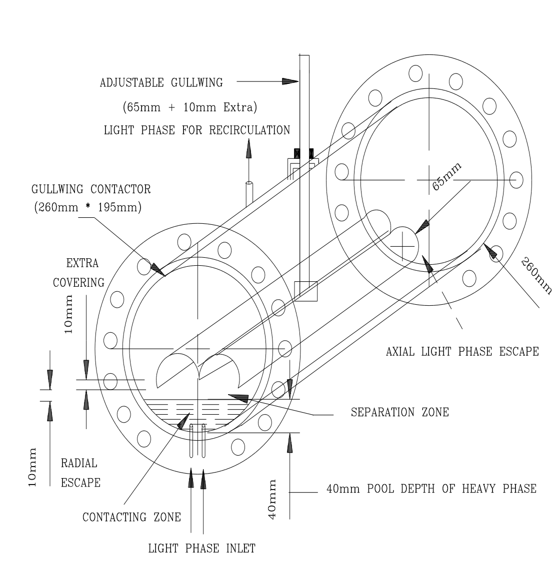
Degree of Extraction (D.O.E.) :- $$Et = \frac{Ci - Ct}{Ci - C*} \times 100 \qquad (1)$$

The data was obtained for batch Gullwing Contactor with 80mm Gullwing which is closed to walls of the Contactor, by keeping L.P. velocities constant at 2m/s with 40mm heavy phase pool depth for analysing the samples for EA content and Degree of Extraction is calculated with the help of equation (1). A known volume of the light phase samples under different operating conditions were collected from the pilot-plant (rig). The light phase samples were further extracted with known volume of chloroform (5ml). Then by using a burette the extract was separated. Five micro litres 5μL of this extract then fed to the chromatograph for TCE content. After 8 to 10 minutes the output print from chromatopac is obtained. This result then compared with standard output print from chromatopac. Then total carry over in parts per million (ppm) of solvent (TCE) from chromatograph were computed by using equation (2). Several geometric parameters of Gullwing Contactor such as different types and various diameters, the position of the Gullwing, numbers and types of the nozzles had been investigated first on the batch Contactor. The optimum design parameters were obtained from the batch Gullwing Contactor was used to design a two stage multijet continuous counter-current Gullwing Contactor.

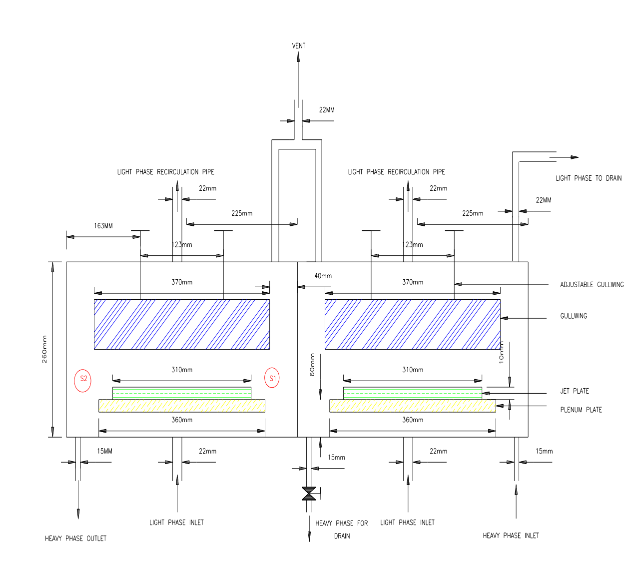
Total Carryover of TCE (ppm) = 0

$$\frac{Volume\ of\ Chloroform}{Total\ Volume\ of\ Light\ Phase} \times \frac{Peak\ area\ of\ TCE}{Peak\ area\ of\ \mathrm{Standard}\ Sample} \times 5 \times 10^{6}\ \ (\mathrm{ppm}) \qquad (2)$$

It has been shown by both Rizvi [8] that D.O.E. could be greatly influenced by the rate of circulation of light phase. Therefore addition of more jets in the present design by increasing the number of jets but due to the space limitation on the Contactor it was very difficult to increase the number of light phase jets , this problem of mechanical nature is solved by introducing a Plenum Chamber at the bottom of the Contactor. The design of the Plenum Chamber is shown in figure [6], which appears as Plenum plate and Jet plate both were assembled at the bottom of the Contactor [9]. Design eliminate individual injection of light phase altogether, and it shows that the Plenum Chamber is the part of the Contactor with Jet plate which is drilled with 20*4mm staggered jets per stage is shown in figure (6).



3-D sketch & Design.



Drawing showing Sectional Elevation

Fig6, *Design & DRAWING of GULLwing Contactor and Sectional Elevation of the Plenum Chamber which Observed as the part of the Gullwing Contactor*

A consequence of staggering the jets about the axis is that all the light phase flow from each jet was directed under one or other sides of the Gullwing and not split as in the previous work. The advantage of the Plenum Chamber is that by detaching Jet plate from the Plenum plate and it can be easily modified. Counter current extraction of EA was carried out by using solvent (WATER) which enters at the end of the Contactor farthest from the feed point (entry point of TCE + 5% EA), and two phases pass counter-current to each other. By using direct method of light phase injection. The light phase was recirculated through 20*4mm staggered jets per stage. This arrangement was expected to increase the system's overall performance due to the fact that the fresh light phase (EA free) would have direct impact on the heavy phase mixture leaving the stage 1 as per Schoen [9] , thus by enhancing the concentration differences between the phases (driving force for mass transfer), the total concentration difference i.e. stage extraction was evaluated by using Murphree's stage efficiency equations (3 & 4). The Murphree stage efficiency [10], could be defined as the ratio of the actual concentration change of that phase within the stage to the change that would have occurred if equilibrium had been reached.

Therefore the Murphree stage efficiencies for stage 1 and stage 2 in the light of above definition could be expressed by overall efficiency;

Stage1. $$(E_{MS})_1 = \frac{C^*_{EA(in)} - C^*_{EA(1)}}{C^*_{EA(in)} - C^*_{EA(1)eq.}} \times 100 = 60.1\% \quad (3)$$

Stage2. $$(E_{MS})_2 = \frac{C^*_{EA(1)} - C^*_{EA(out)}}{C^*_{EA(1)} - C^*_{EA(out)eq.}} \times 100 = 90.7\% \quad (4)$$

$$\text{Overall Efficiency} = \frac{C^*_{EA(in)} - C^*_{EA(out)}}{C^*_{EA(in)} - C^*_{EA(out)eq.}} = 96.2\% \quad (5)$$

5. DISCUSSION

The Gullwing near to the jets and in the lower or upper portion of the Contactor forms two essentially circular cross sectional areas running under the length of the Gullwing. Because the jets direct the mixture of the heavy and light phase upwards and underneath the peak of the Gullwing. The fluid in the hydro cyclone on the right hand side rotates clockwise and in the other anti-clockwise. The spaces formed by the Gullwing and heavy phase pool depth by raising or lowering the Gullwing from the heavy phase pool. This adjustment of the Gullwing aimed at control of the entrainment, because this allowed light phase to escape radially from the edges of the Gullwing as well as axially from the ends of the Gullwing. Series of experiments were carried out to achieve optimum height of the Gullwing from top of the heavy phase pool depth which would generate the most effective hydro cyclone in terms of the degree of separation of light phase and heavy phase. The vertical position (height) of the Gullwing from the jet is found that 60mm height of the Gullwing above the jet exit gave the shortest possible time for 99% extraction without any carryover of TCE in the light phase at 2m/s L.P. velocity.

Table [1] *:- Power and Energy Consumption at various Jet Velocities in the Gullwing Contactor with 80mm Closed Gullwing to reach 99% of Equilibrium.*

Exp. Nos.	**Volume flow Rate(Q)**	**Time**	**Pressure (P)**	**Power Req.**	**Energy Req.**	**Volume flow Rate(Q)**	**Time**
	L/min	m^3/s	sec	lb/in^2	N/m^2	kW/m^3	kJ/m^3
1	1.5	$25*10^{-6}$	2760	1.9	$13.1*10^3$	0.025	70.56
2	2.0	$33*10^{-6}$	1980	2.0	$13.8*10^3$	0.036	70.39
3	2.5	$42*10^{-6}$	1380	2.1	$14.5*10^3$	0.048	65.6
4	3.0	$50*10^{-6}$	1080	2.2	$15.2*10^3$	0.059	64.1
Carryover of TCE starts to appear in WATER							
5	3.5	$58*10^{-6}$	810	2.5	$17.2*10^3$	0.077	63.08
6	4.0	$67*10^{-6}$	480	2.7	$18.6*10^3$	0.097	46.6
7	4.5	$75*10^{-6}$	330	3.2	$22.1*10^3$	0.130	42.9
8	5.0	$83*10^{-6}$	240	3.7	$25.5*10^3$	0.170	40.8
9	5.5	$92*10^{-6}$	180	4.2	$28.9*10^3$	0.210	37.4
10	6.0	$99*10^{-6}$	135	4.7	$32.4*10^3$	0.250	33.8
11	6.5	$10.9*10^{-6}$	105	5.2	$35.9*10^3$	0.310	32.6
12	7.0	$11.7*10^{-6}$	90	5.7	$39.9*10^3$	0.360	32.4

A comparison was carried out between the batch as well as continuous Gullwing Contactor and a conventional Mixer-Settler by selecting their optimum operating conditions such as 40mm heavy phase pool depth, 2m/s jet velocity and 65mm + 10mm extra edges of the Gullwing at 60mm height from the jet exit without any carryover of TCE in WATER.

The agitation speed of the control Mixer-Settler is varied between 200 and 400rpm, corresponding to variation of 175-1047 W/m^3 power consumed and found that the 250rpm is the best speed of the agitator at which total time for contacting and separation was minimum as compared to other speeds. The relative performances of the Gullwing Contactor and Mixer-Settler are shown in figures [7]. The agitator Contactor consumes more power and energy compared with jet Contactor system because; in an agitator Contactor the whole of the contents of the mixing vessel are in motion, while in case of the jet mixing system only a small portion of the two phases near the jet is in motion, and in agitator drive system the power consumed to achieve certain degree of extraction is directly proportional to the third power of the rpm of the agitator, on the other hand the power consumed by the jet system is directly proportional to the product of the volumetric flow rate and the pressure reading at the distributor.

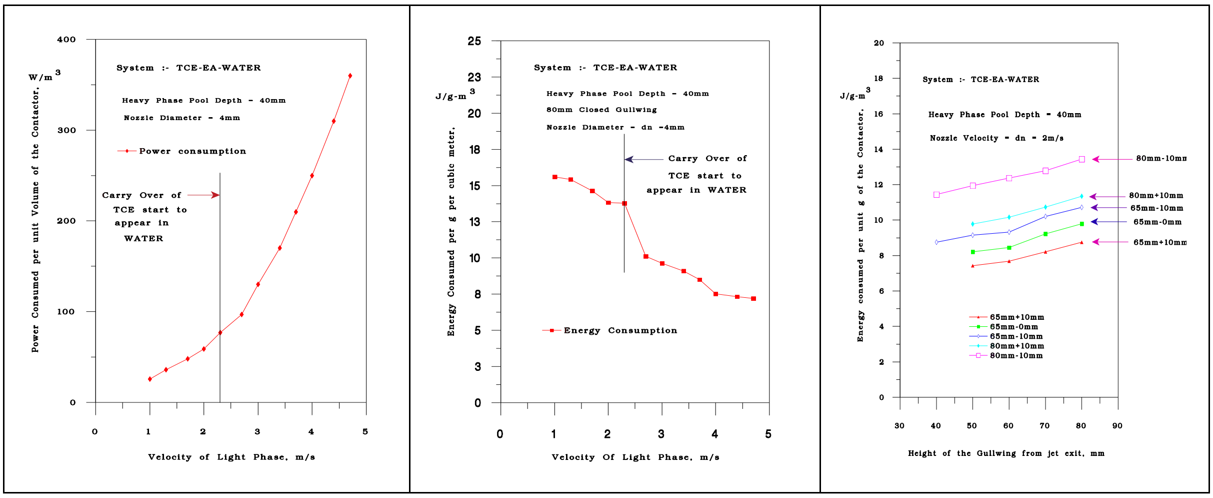


Fig7. *POWER & ENERGY Consumptions by GULLwing Contactor at different VELOCITIES and at different DIAMETER of the GULLWINGS.*

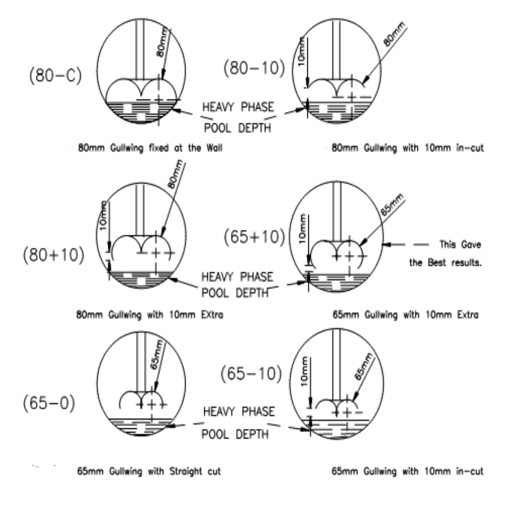
The Gullwing Contactor is competitive with the traditional mixer settler and can be used to replace the traditional-mixer settler because Gullwing-Contactor has following advantages over traditional mixer-settler:-

- Small operating volumes and therefore low solvent and low capital costs.
- Low energy and control expenditures.
- -extra stage easily added to existing system.
- -suspended solid matter easier to handle than in other extractors.
- -low height of extractor and required small floor area.

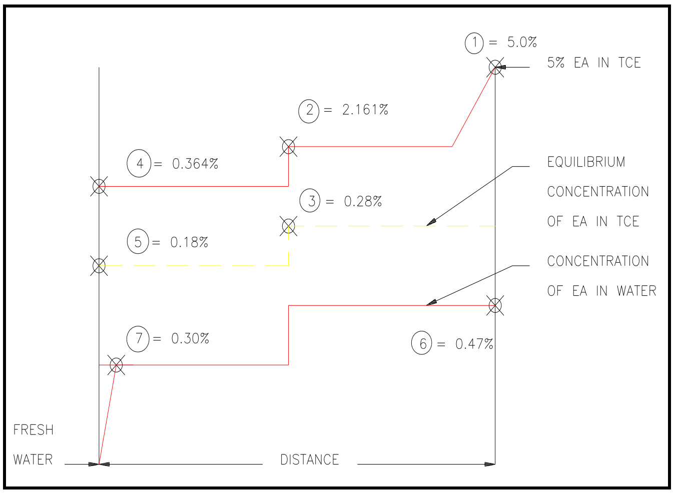
Comparison between Centrifugal extractors and Gullwing Contactor:- In the centrifugal extractors, residence time can be reduced and phase separations accelerated by application of centrifugal force instead of gravity. Because of their precision construction, the capital cost of centrifugal extractors is higher than that of Gullwing Contactor, and centrifugal extractors require greater maintenance than Gullwing Contactor. However centrifugal extractors are compact and have a relatively high throughput in small geometric space similarly Gullwing Contactor is compact and high relative throughput is possible in small geometric space. The centrifugal extractors are particularly useful when contact time must be short as for chemical unstable systems, when product inventory must be kept a minimum, or when liquids tend to emulsify or are generally difficult to separate. Similarly Gullwing Contactor could be applied for the unstable system having with short contact time and when liquids tend to emulsify.

6. CONCLUSIONS

Gullwing Contactor is also very simple in installation as it is based on standard pipe sizes. The only moving parts are associated with the circulation pump. Compared with agitator shafts seals, pumps are more leak proof and thus the Gullwing Contactor is well suited to extraction/reaction systems that require good sealing for Biochemical environment. Furthermore, for exothermic or endothermic reactions the pumps can also be used to pass the light phase through an external heat exchanger injecting cooled or heated light phase at the point of reaction. The distribution ratio (D) is equal to the concentration of a solute in the organic phase divided by its concentration in the aqueous phase. Depending on the system, the distribution ratio can be a function of temperature, the concentration of chemical species in the system, and a large number of other parameters. Note that D is related to the ΔG of the extraction process which is shown in Figure [8].



Position (Height) of the Gullwing



Concentration distribution of EA at 60mm height of 65mm+10mm Gullwing with 2m/s light phase inlet Velocity.

Fig8. *Two stage counter current Continuous Gullwing Contactor with different HEIGHTS & DIAMETERS.*

Compared with Mixer-Settler the Gullwing Contactor reduces processing time and energy consumption. It is found that for batch Contactor it requires only 11.4% power $W/g/m^3$ and energy $J/g/m^3$ and for continuous Contactor it requires only 10.6% power $W/g/m^3$ and energy $J/g/m^3$. Similarly total extraction on batch Contactor is 7.68 $g/sec/m^3$ and on continuous Contactor is 75.9 $g/sec/m^3$ as compared to 4.21 $g/sec/m^3$ of the Mixer-Settler. Thus the batch Contactor gives 82% more extraction and continuous Contactor gives 18 times more extraction for the same system with the same chemical composition as compared to traditional Mixer-Settler. The Gullwing Contactor would be well suited to hazardous Biochemical, & chemical environments such as Nuclear fuel extraction and irradiated fuel processing, food-processing and fine biochemical industries.

6.1. Abbreviations

Ci Initial EA concentration in TCE

Ct Concentration of EA in TCE after elapse of time (t)

C* Equilibrium concentration of EA in TCE

6.2. Acronyms

E_{MS} Murphree Stage Efficiency.

$C*_{EA(in)}$ Entering heavy phase concentration, weight %, wt./wt.

$C*_{EA(1)}$ Leaving heavy phase concentration, weight %, wt./wt.

$C*_{EA(out)eq.}$ Equilibrium concentration, weight%, wt./wt. ppm, Parts per million.

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REFERENCES

[1] Gavhane, K.A., "Mass Transfer-II", Nirali Prakashan, Pune, (2008).

[2] Thornton, J.D., "Science and Practice of Liquid-Liquid Extraction", Vol. 1 , pp.40-147, 244-337, 492-591 & Vol. 2, pp.353-367, Clarendon Press, Oxford, (1992).

[3] Ricci, L & the Staff of Chem. Engg., "Separation Techniques, liquid-liquid Systems", pp.215-57, McGraw-Hill, New-York, (1980).

[4] Treybal, R.E., "Liquid-Extraction", pp. 541, McGraw-Hill, New-York, (1963).

[5] Kothari, D.C., "Application of Gullwing Contactor for Liquid-Liquid Extraction", M.Sc. Thesis, The University of Leeds, Leeds, United Kingdom, (1994).

[6] Bailey, J.S., & Bhatia, S.C., "Biochemical Engineering", CBS, New Delhi, (2009).

[7] McGreavy, C., Pitt, M.J., Preece, P.E., Rizvi, S.Z.H. & Sachs, G.E., "Experimental results from Gullwing Contactor / Separator", IChemE., London, U.K., (1991).

[8] Rizvi, S.Z.H., "Hydrodynamics of Liquid-Liquid Extraction processes in the Gullwing Contactor", PhD Thesis, The University of Leeds, Leeds, (1989).

[9] Schoen, H.M., "Counter-Current Extraction", Pregamon Press, Oxford, (1962).

[10] Rousseau, R.W., "Multistage Contactors, 'Number of Trays, Graphical Approach (Murphree Stage Efficiency)" in 'Handbook of Separation Process Technology', pp. 60,354-356 & 405-466, John-Wiley & Sons., New-York, (1987).

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