INVESTIGATION OF THE PERFORMANCE CHARACTERISTICS OF A MIXER-SETTLER EXTRACTOR

by

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A THESIS

submitted to

OREGON STATE COLLEGE

in partial fulfillment of
the requirements for the
degree of

MASTER OF SCIENCE

June 1961

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Date thesis is presented January 27, 1961

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ACKNOWLEDGMENT

The author wishes to express his appreciation to Dr. C. E. Wicks and Professor J. S. Walton for their help and suggestions during this investigation. The assistance of Mr. R. C. Mang was also appreciated for his work in building the apparatus.

Grateful acknowledgment goes also to Oregon State College whose assistantship and facilities made this work possible.

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INTRODUCTION

The proposed study for this thesis was to construct and put into operation a pump-mix, mixer-settler liquid-liquid extractor. Data were taken on the change in extraction efficiency with impeller speed at a given set of feed conditions and the change in efficiency with total feed rate at a given impeller speed.

Liquid-liquid extraction is one of the important unit operations; it is used in various processes in the field of atomic research and development. Since liquid-liquid extraction or solvent extraction, as it is sometimes called, deals with mass transfer a contactor must be developed which disperses one phase into another or provides some method of contact in which the mass transfer can take place. The ideal contactor should provide as many of the following characteristics as possible: (a) minimum investment for both associated equipment and building, (b) maintain a high efficiency over a wide operating range, (c) have high efficiency with low hold-up, (d) simple controls, (e) minimum maintenance, (f) be flexible for process variation, (g) provide reliability of operation, and (h) be adaptable to easy replacement.

At present there are four types of contactors used to some degree in research and production. These are: (a) vertical flow columns which include spray, packed, and perforated, (b) oscillary flow vertical columns such as a pulse column, (c) mixer-settler type, and (d) centrifugal contactor. All of these mentioned contactors have certain advantages over the other types hence there is no contactor that will be the most efficient for every job. A detailed evaluation of the relative merits of the four types of contactors used in the atomic research field has been published (6, p. 1-12).

The mixer-settler type of liquid-liquid extractor has been used for over fifty years. During this time there have been many alterations in design of the apparatus. All of the various types provide one thing in common, namely, contact in individual stages. The variations employ common shaft for all stirrers versus individual shaft for each stage; vertical versus horizontal arrangement of the stages; cocurrent versus countercurrent settling within each stage; and the control of the ratio of phase in the mixing chamber independent of the flow rates. In most cases overall flow is countercurrent. Co-authors from the University of California have given a detailed description of twelve types of mixer-settler designs (3, p. 188-197).

SOME OPERATING CHARACTERISTICS OF THE PUMP-MIX MIXER-SETTLER EXTRACTION UNIT

During the last decade a considerable amount of work has been done under government contract on mixer-settler equipment. The horizontal box type equipment, which was studied most, has several desirable characteristics for use in the field of atomic research. At present most of the extractors are of a pilot plant size. Depending upon the size of the unit, total feed rates have been as low as 0.05 to 4 milliliters per minute (1, p. 1358) up to 873 milliliters per minute (14, p. 469).

The major difference in the various types of horizontal contactors is found in the mixing section and method of transporting the two phases to successive stages. A unique and successful type of extractor is the pump-mix mixer-settler. This extractor was developed at the Knolls Atomic Power Laboratory at Schenectady, New York in August 1948. Each mixing section has an impeller which is designed like a centrifugal pump. The section is divided into two compartments with one on top of the other. The tip of the impeller projects through a hole in the plate separating the two sections and draws up the heavy phase from the previous stage and disperses it into the continuous light phase. The hole separating the two sections has a diameter larger than that of the impeller thus forming an annulus. The capacity of the impeller is designed to be greater than the flow rate of the heavy phase. The

net result is that part of the mixed phase is recirculated through the annulus and there is a make and break effect between the interface and the impeller tip. This recirculation is necessary to provide adequate mixing (11, p. 41). An extensive study was made of the pump-mix impeller; the total flow and head were given as a function of the impeller speed and the size of the recirculation hole (13, p. 1-24). The above study was made with an impeller five inches in diameter with a tip of 1-1/4 inches I.D. and 1-1/2 inches O.D. The recirculation hole varied from 1-3/4 inches to three inches in diameter and the impeller speed reached 500 rpm. With this impeller action mixing and pumping is provided at each stage while the light phase flows through the bank by gravity. With this type of apparatus, by nature of the design, the light phase is always the continuous. The inventors of the unit have considered altering the design to disperse the light phase into the heavy phase but this change has not been necessary to satisfy the process requirements.

With the impeller providing the mixing action, an increase in the speed of the impellers will increase the degree of mixing. Impeller speeds have been used as high as 10,000 rpm. (16, p. 18) with lowest values in the low hundreds. The limiting upper speed would be that which produces an emulsion difficult to separate, thus increasing the hold-up time required. It was pointed out (16, p. 9) that a variation in speed of about $\pm$ 10 percent whether for the individual stages or over-all operation results in only a small change in stage efficiency. In many systems which can tolerate vigorous

mixing, blades have been attached to the impeller to increase turbulence. It was found that placing mixing blades on the impeller yields higher efficiency at all speeds (5, p. 203).

Some method of baffling is required between the mixing and settling sections to quiet the turbulent action and it is advisable to have a baffle on the light phase port in the mixing section. Coplan, Davidson, and Zebroski (2, p. 406) pointed out that with various baffle and blade arrangements the efficiency of a unit can vary 77 percent to 99 percent. In the above case the direction of rotation was also important.

One of the most difficult problems in establishing stability in a mixer-settler is providing a proper interface level in the settler. The pump-mix impeller provides good operating characteristics and interface control except when there is a small difference in density between the two phases. Since the pumping action of the impeller may undergo unpredictable variation an interface of low density differences is difficult to control. For example, a system with a density difference of 0.03 grams per milliliter must have good impeller control because deviation of 1/32 inch in the impeller height would produce a one inch change in the level of the interface (7, p. 639).

Many types of problems have arisen from the use of the horizontal mixer-settler but most of them have been solved. One of the initial problems was that of evaporation of volatile liquids. In one such case (1, p. 1359) a blotter perforated for the impeller and placed over the top of the bank

reduced the evaporation to a tolerable minimum. Air vents to the product containers were also filled with glass wool.

There appears to be no limit as to the possible number of stages in a series of mixer-settler units. A fifty-six stage mixer-settler unit was put into operation at the Ames Laboratory (7, p. 640). The pump-mix mixer-settler unit well satisfies the condition of flexibility for process operation. With feed ratio varying from 1:10 to 10:1, high efficiencies were reported in the same unit (2, p. 406).

A common feature of all operating units was the excellent efficiencies reported. Over a wide range of operating conditions many of the unit reported an average operating efficiency of around 90 percent. The efficiency was affected by three operating conditions: insufficient mixing, backmixing, and by-passing. The first of these conditions was easily overcome by increasing the speed of the impeller or adding mixing blades of various configurations. Backmixing was reduced by inserting a baffle on the light phase port in the mixing section. Backmixing was found to occur in all designs tested except those with an antechamber for the entering streams to all the mixing sections (3, p. 196). By-passing was also reduced by application of baffles and was eliminated in units with antechambers.

W. G. Mathers and E. E. Winter (8, p. 99-104) have proposed another type of mixer-settler which operates with an air stream. The mixing and pumping energy was provided by a stream of air admitted to the bottom

of each mixer. The efficiency of this unit was somewhat parallel to that of the pump-mix unit. It appears to be a versatile unit with low power requirements and no moving parts. F. Roberts and B. T. Ball (10, p. 6-20) have given a good review of horizontal mixer-settler equipment. Their review discussed eleven different types of apparatus. One section concerned the characteristics of six impeller designs in operation in the various units. They also proposed a mixer-settler with individual cylindrical stages. Their proposed unit consisted of a series of concentric tubes with the mixing being done in the inner tubes and the settling section being formed by the outside annulus.

It has been pointed out here that the pump-mix mixer-settler satisfies many of the chracteristics of the ideal contactor. The equipment can be compact and if shielding was required its height would be low when compared to that of a column. It has demonstrated a high efficiency over a wide operating range and process variation. Another important feature was the ease of obtaining phase samples from the stages. In most cases the samples are extracted directly from the top of each unit. With the simple design and placement any number of stages can be added on or taken from a unit quickly. The mixer-settler is then, a simple but efficient device for liquid-liquid extraction.

DESCRIPTION OF THE APPARATUS

The apparatus consisted of a three stage horizontal pump-mix mixer-settler liquid-liquid extractor. The design of the impeller and individual stages was adapted from a model used by the Bureau of Mines located in Albany, Oregon. The rest of the construction and design was that of the author. The unit was constructed of 304 stainless steel, glass and teflon. This provided maximum flexibility for possible systems which can be studied in the apparatus with a minimum amount of corrosion.

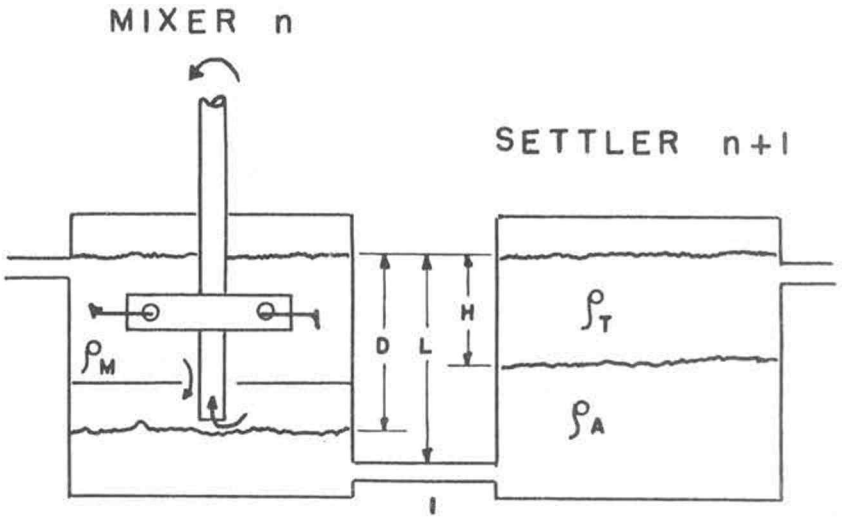
For future reference the stage which the feed entered was designated as number one and the solvent entered number three or the last stage. Also in this work the feed was always the light phase and the solvent was always the heavy phase.

Each stage consisting of a mixing and settling section was placed in a unit so the mixing section of one stage was adjacent to the settling section of the next stage. Flow through the unit was countercurrent. By nature of the design of the apparatus the heavy phase was always the dispersed phase and the light phase was always the continuous phase. The heavy phase was pumped from stage to stage by the impellers and the light phase flowed through the bank by gravity. Very little hydraulic head was required for the light phase to flow through the bank. It was hard to estimate the actual difference in the height of the light phase in the three settlers. The liquid level in the last

stage was at the bottom of the exit port because it drained out at that port without restriction. In the other two stages the settler port was completely covered with the liquid and one could not usually distinguish a height differential even though there was one.

The feed and the solvent were pumped with two Eastern Industries Model E-1 pumps which were controlled by powerstats. The flow was regulated manually by either a needle valve or the powerstat using two rotameters from the Brooks Rotameter Company as indicators. The light phase was indicated by a 6-15-2 rotameter with a glass ball and the heavy phase by a 4-15-2 tube with a stainless steel float.

The impellers were driven by individual belts from a common shaft. The speed of the shaft was regulated by a Variable Speed Zero-Max Torque Converter Model 142 X Revco Incorporated. The speed of the impellers could be increased up to 3000 rpm. The direction of rotation was the same in all of the stages with respect to the light phase port in the mixer. The impellers were held in place by two pillow blocks. These permitted raising and lowering the impellers to any desired height. The impeller height adjustment was necessary to regulate the height of the interface in the succeeding stage. This relationship between the impeller height in the n th stage and the interface level in the n + 1 stage can easily be shown and is illustrated in Figure 1.



P = PRESSURE

ΔP = PRESSURE DROP OF RECIRCULATING MIXED PHASE

ρ = DENSITY

AT POINT 1:

$$P = D(\rho_M) + (L-D)\rho_A - \Delta P = H(\rho_T) + (L-H)\rho_A$$

SOLVE FOR H:

$$H(\rho_A - \rho_T) = D(\rho_A - \rho_M) + \Delta P$$

$$H = \frac{D(\rho_A - \rho_M) + \Delta P}{(\rho_A - \rho_T)}$$

EQUATION 1

FIGURE 1 RELATIONSHIP BETWEEN IMPELLER HEIGHT AND INTERFACE HEIGHT

At steady state the only variables in equation 1 would be H and D. The above equation neglects the effects of pressure drop through the ports and the connecting piping and assumes the corresponding densities to be constant. These seem to be valid assumptions under the operating conditions. Thus the height of the interface in the second and third stages is controlled by the height of the impeller in stage one and two respectively. The interface in the first stage is controlled by the adjustable weir on the heavy phase product from the first stage. This adjustable weir was a simple leg made out of glass.

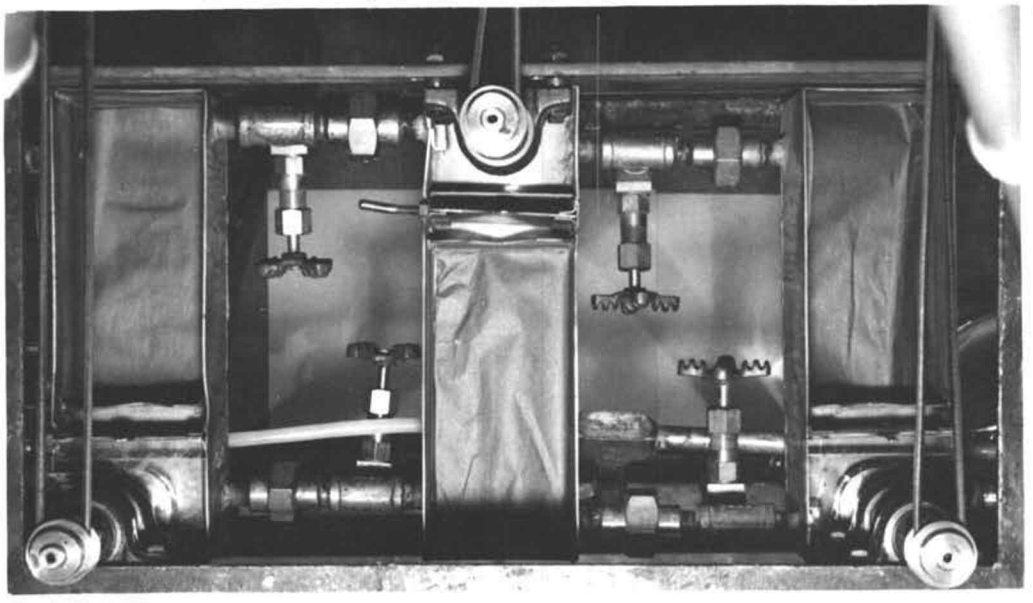
Glass plates were used to cover the mixing sections in each stage to prevent mass loss by splashing because of vigorous mixing in that section.

Thin walled teflon tubing was placed in the feed lines near the rotameters to provide a flexible line to permit removal of the rotameters.

The only baffles used were placed on the light phase port in the mixing sections of stages two and three. It was of simple box like construction.

The feed and products were stored and collected in glass carboys.

Figure 2 is a picture of the completed apparatus and figure 3 gives detailed description of the impeller and individual stage design.



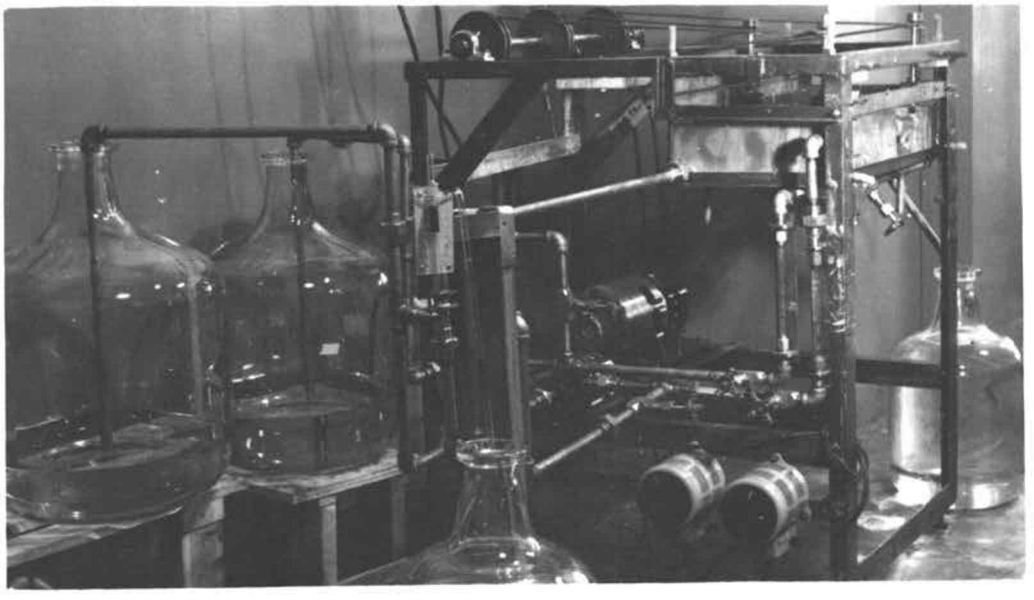


FIGURE 2 TOP AND SIDE VIEWS OF APPARATUS

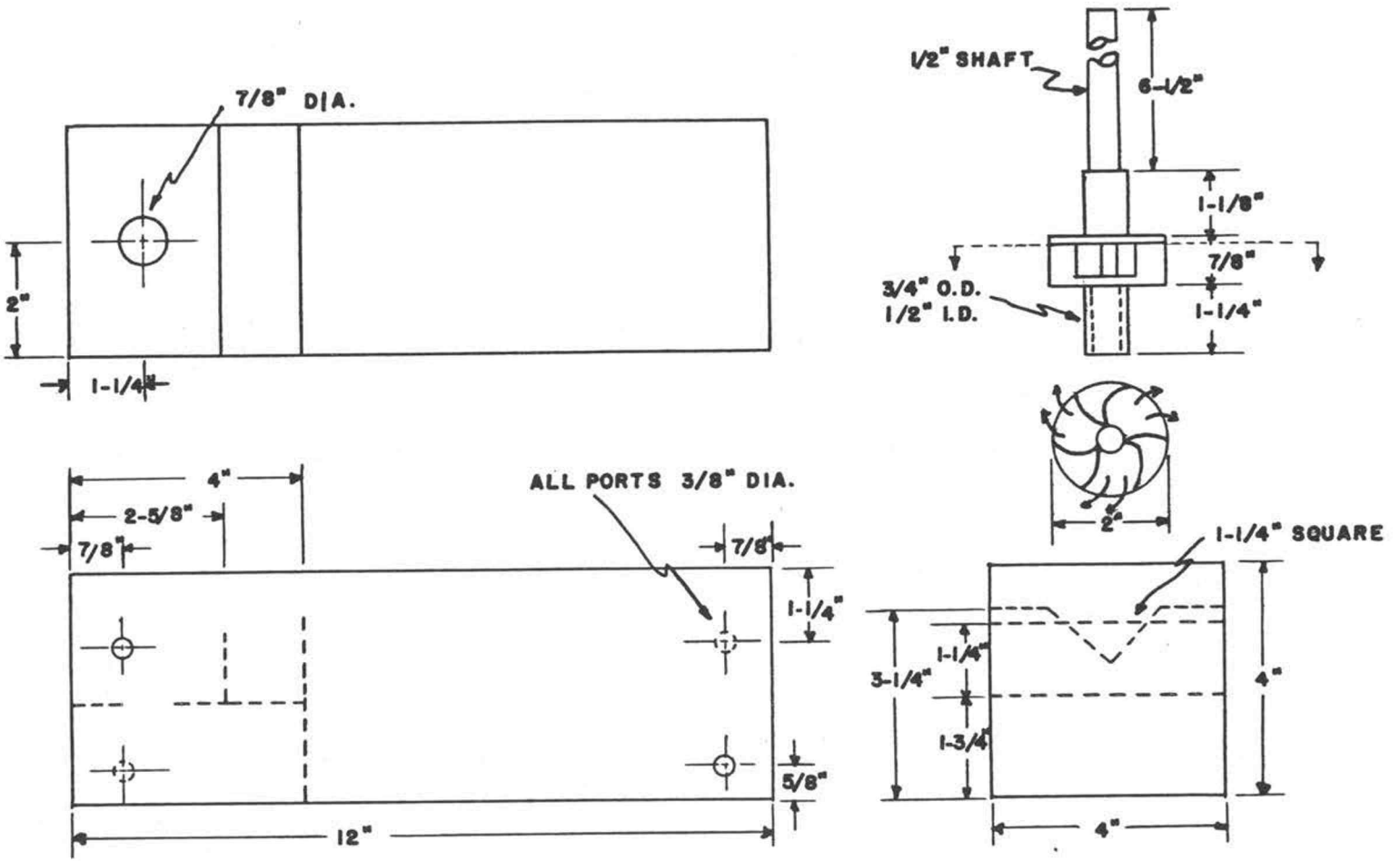


FIGURE 3 INDIVIDUAL STAGE AND IMPELLER DESIGN

DESCRIPTION OF SYSTEM STUDIED

The system studied in this work was toluene-water-acetic acid. This system was chosen because of the availability of equilibrium data and chemicals to be used. The chemicals used were glacial acetic acid, toluene U. S. P., and distilled water. The analysis was done using pelleted sodium hydroxide and standardized with potassium acid phthalate.

Woodman's (15, p. 1283-1286) equilibrium data were used. The data, which were taken at 25° centigrade, are given in table 2 of the appendix. The error in the above mentioned equilibrium data was stated as less than 0.5 percent. Table 3 in the appendix gives the tie line data. The tie lines were made by analyzing for acetic acid only. The tie line data were tested for consistency by methods developed by Hand (4, p. 1983) and Othmer and Tobias (9, 693-696) and was found acceptable. Figures 3 and 4 represent the equilibrium diagram and the x-y diagram (tie line data) from the data by Woodman. The experimental work and analysis of this thesis were done at room temperature which varied from 22-25° centigrade over the period of time the runs were made.

With the given equilibrium data and experimental apparatus the operating conditions were necessarily limited. With the aqueous phase as the solvent the location of the operating line on the x-y diagram would be limited

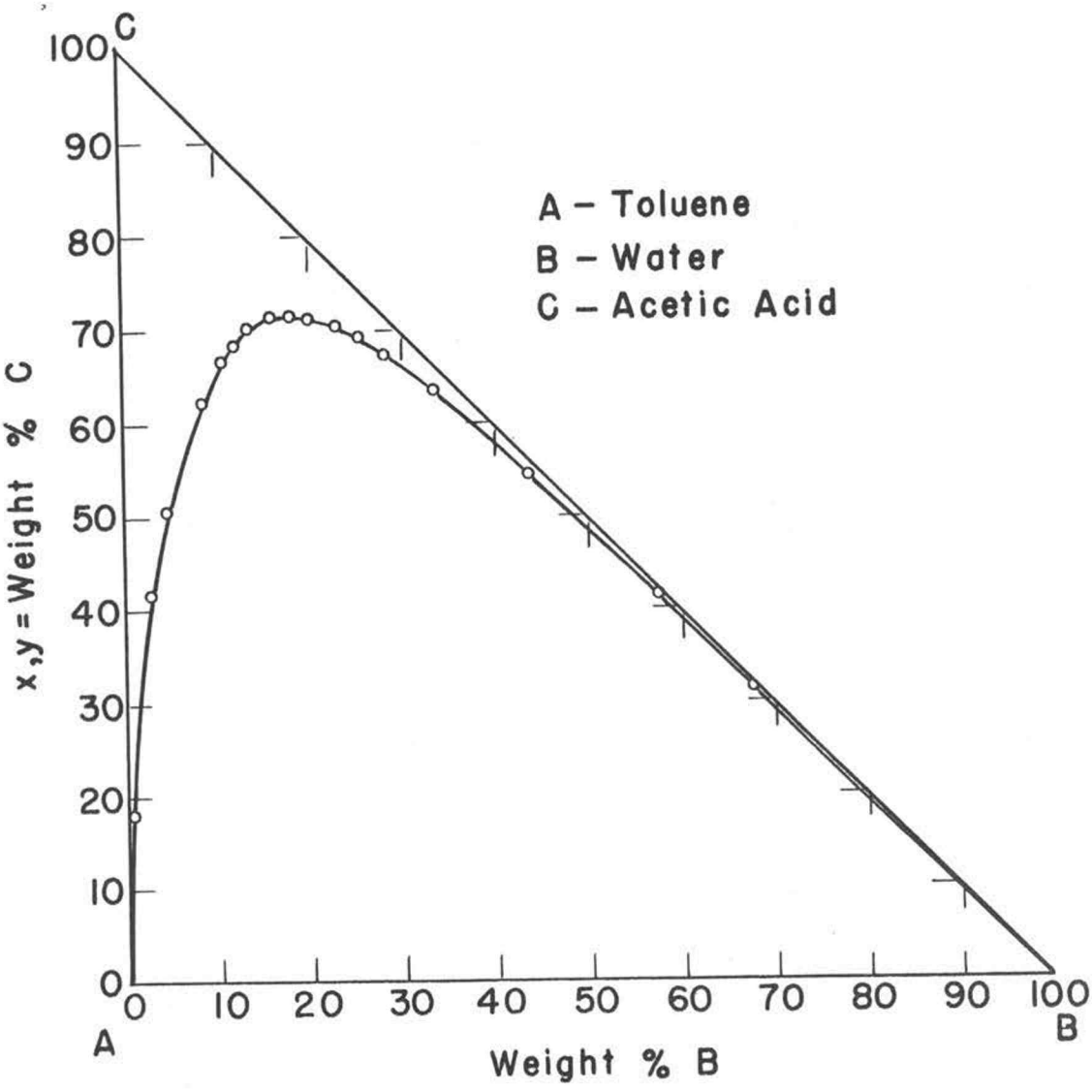


FIGURE 4 EQUILIBRIUM DIAGRAM FOR TOLUENE–WATER–ACETIC ACID AT 25° C.

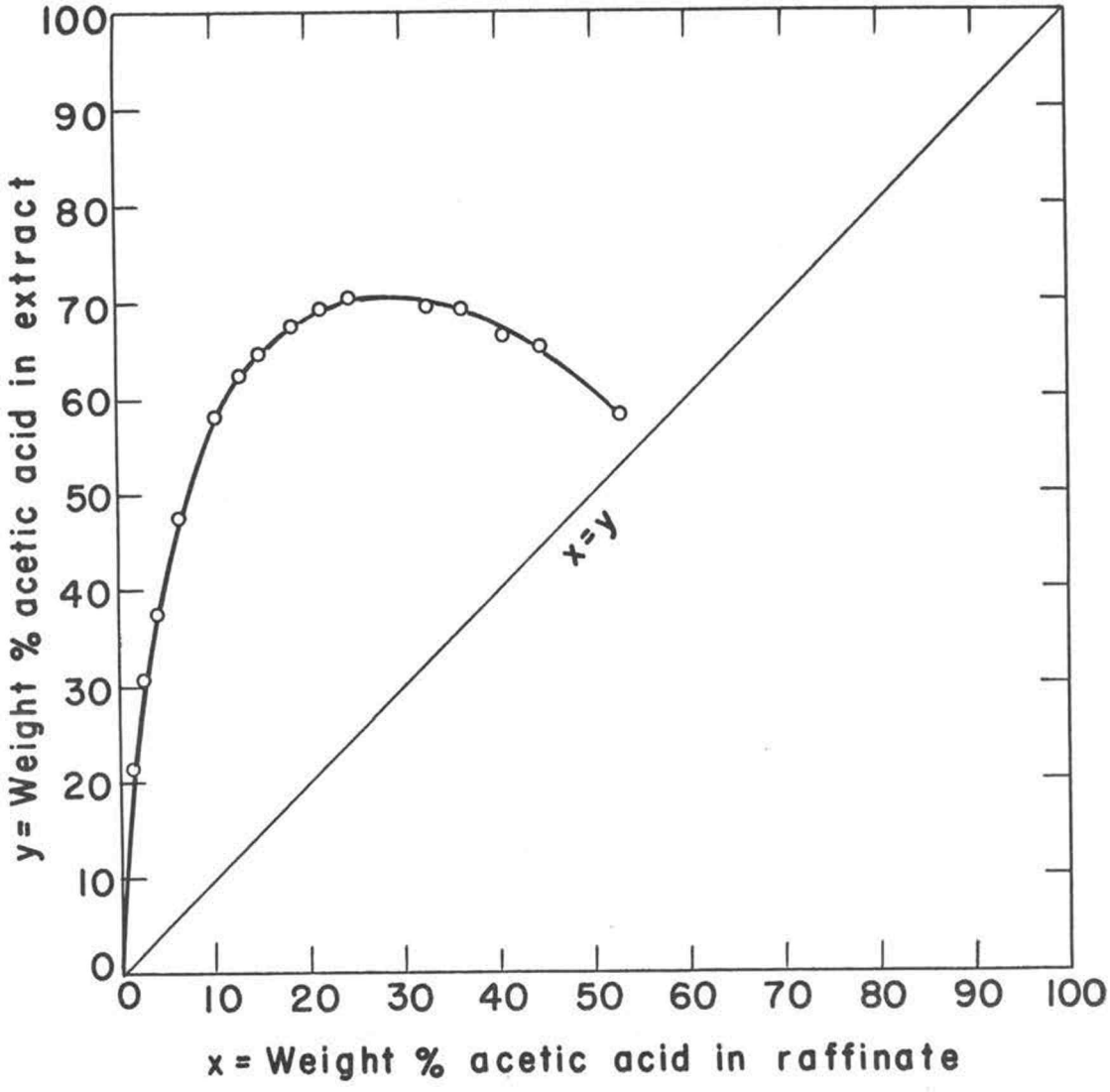


FIGURE 5 TIE LINE DATA FOR TOLUENE-WATER-ACETIC ACID AT 25° C.

when the number of stages is three. Under these circumstances the feed composition was arbitrarily taken as 23.00 percent acetic acid and the solvent as 58.75 percent acetic acid with both of these at equilibrium conditions.

The proposed study for this thesis was to determine the change in extraction efficiency with changing impeller speed using the above feed compositions. The ratio of the feed rates was again arbitrarily chosen within the limiting range to be 4.1:1 mass ratio feed to solvent. The second series of runs were conducted to determine the change in efficiency while varying the total feed rate with the feed compositions, mass ratio, and impeller speed variables held constant. This speed was chosen from the results obtained from the first series of runs.

The number of theoretical stages was determined graphically on the x-y diagram. The operating line was obtained from the triangular equilibrium diagram by the method described by Treybal (12, p. 404-407). The efficiency or extraction efficiency was defined as the number of theoretical stages divided by the actual stages times one hundred. Figures 6 and 7 show determination of the efficiency for run number seventeen. All other efficiencies were determined by this method.

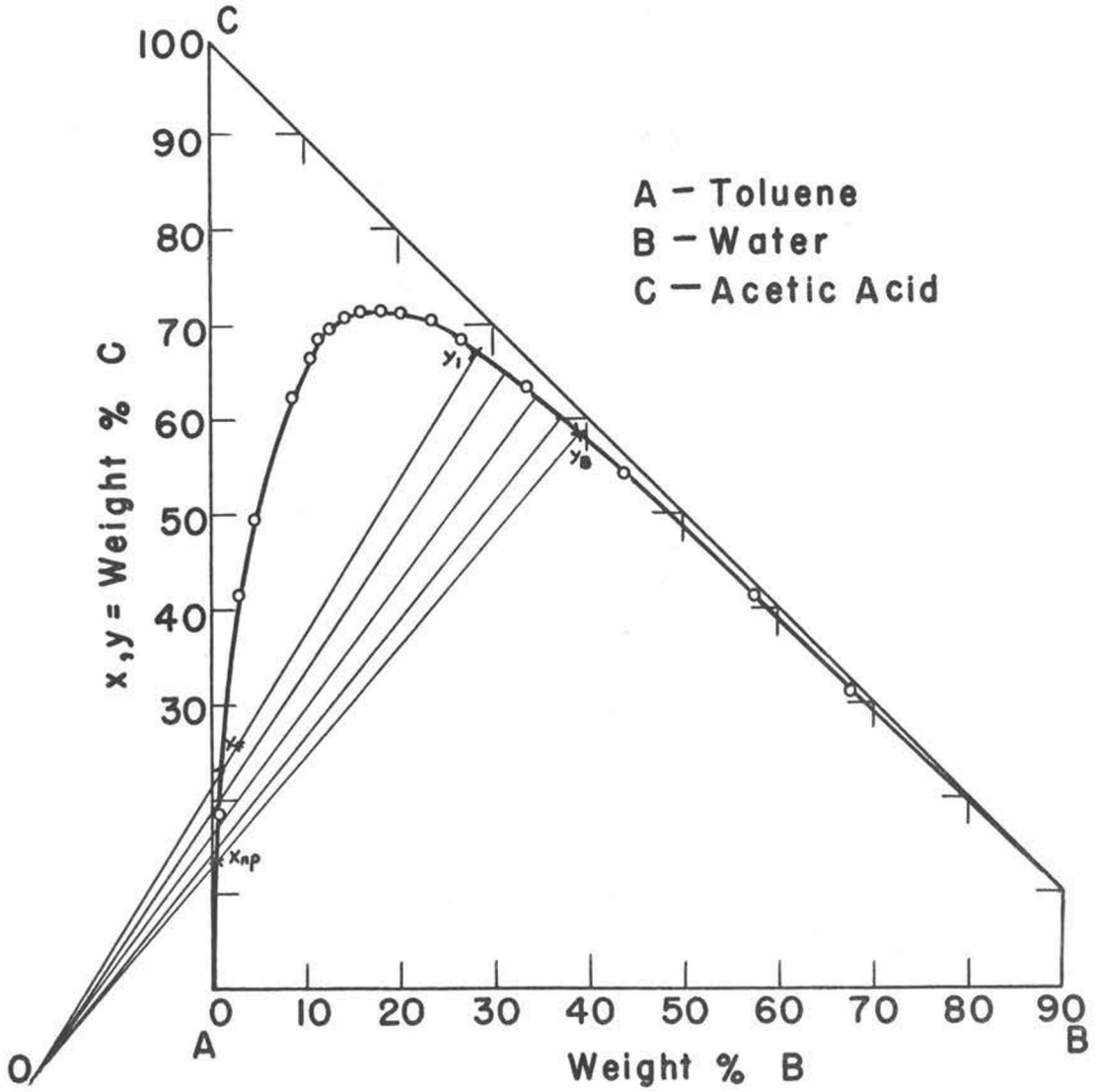


FIGURE 6 DETERMINATION OF OPERATING LINE FOR RUN NUMBER 17

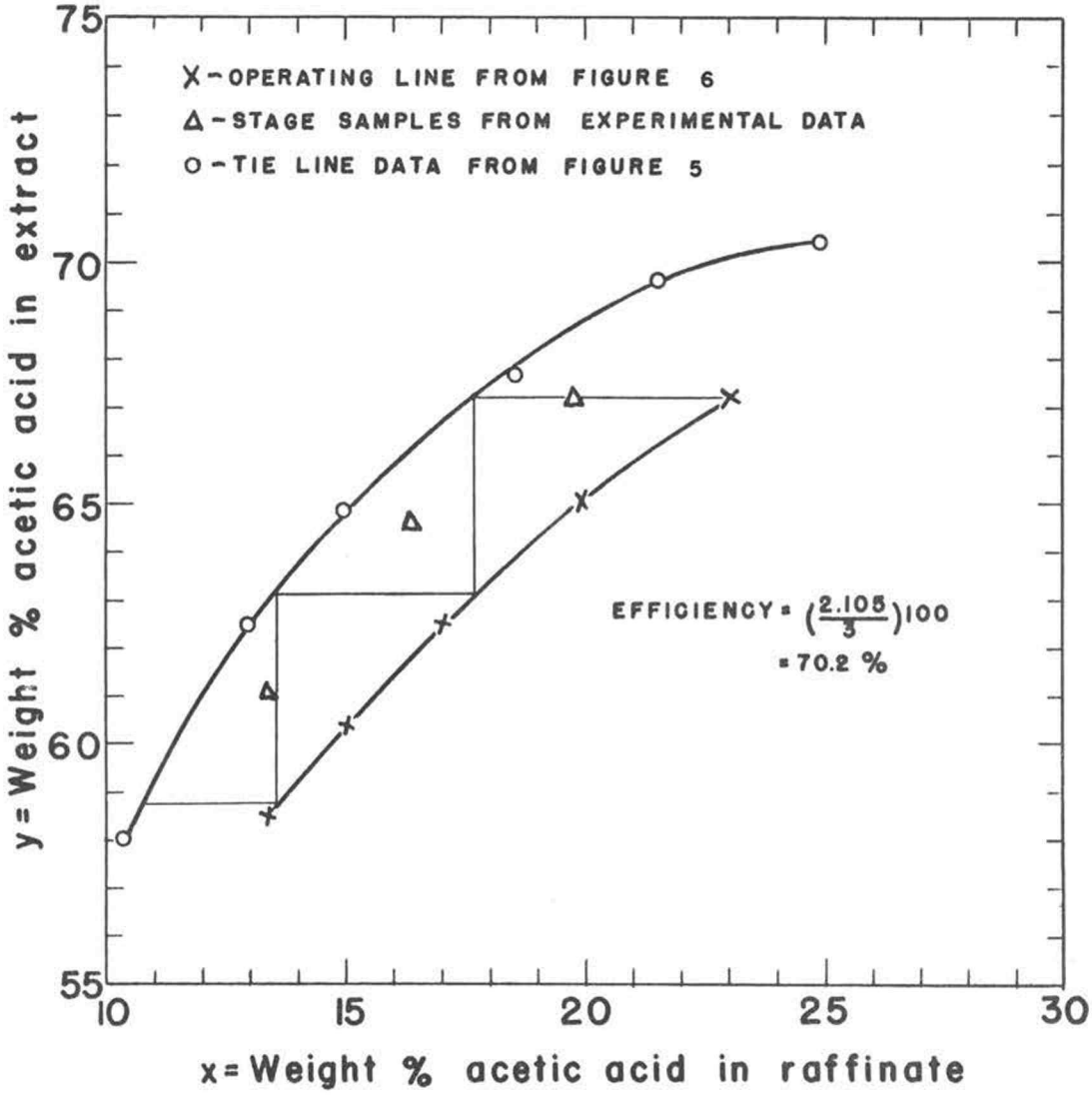


FIGURE 7 DETERMINATION OF OVER-ALL EFFICIENCY FOR RUN NUMBER 17

OPERATIONAL PROCEDURE

Before beginning each run the feed rate and the rpm of the impellers were predetermined. In all cases the composition of the feed and the solvent was the same and the mass ratio of the two was constant. Before each run the feed compositions were brought to equilibrium conditions to ensure proper location of the feed compositions when determining the number of theoretical stages.

Before the feed pumps were started the impellers were adjusted to the proper speed. The desired feed rates were set using the rotameters for indication. The rotameters were previously calibrated with the given feeds. The height of the adjustable weir on the first stage and the impeller heights for all stages were then adjusted to provide approximately equal heights for the interfaces. Once this was done the only adjustment to be made during a run was to maintain proper feed rates.

During the duration of the run which lasted between three and one-half and four hours the speed of the impellers was taken three or four times with a tachometer to provide an average value for the revolutions during the duration of the run. Individually the impellers did not change more than 1.80 percent and the largest deviation from the average was 2.05 percent.

During the last twenty to forty minutes of the run the products were collected in a flask. The composition of this sample was checked against

a sample at the end of the run to determine if steady state was reached. The collected samples indicated a steady state condition was reached within the duration of the run. The mass rates of each stream were determined to give a check in the total material balance.

Component material balances were not made because of the nature of the analysis. The total material balance between the feed rates and the products collected at steady state showed a maximum deviation of 2.77 percent. The feed rates were believed to have fluctuated by at least this amount.

At the end of a run samples from intermediate points were taken by introducing a fifty milliliter pipet directly into the settlers in each stage. The two product samples were taken from the outlet points.

ANALYTICAL PROCEDURE AND DISCUSSION

Samples were analyzed to determine the concentration of acetic acid. The organic or raffinate phase was titrated with 0.9916 normal sodium hydroxide with sample sizes from five to ten milliliters. The amount of base required varied from approximately fifteen to twenty-five milliliters. The aqueous or extract phase was titrated with 5.4652 normal sodium hydroxide and the sample sizes were eight to ten milliliters. The volume of base required was again in the range fifteen to twenty milliliters. In all cases the samples were titrated twice or until the difference between two titrations was 0.05 milliliters or less. This gave a maximum error of 0.3 percent. The titrations were done to a phenolphthalein end point.

After the titrations the density of each sample was determined with a pycnometer. With the normality and the density of each sample known the weight fraction of acetic acid was determined.

Samples were taken with three different pipets, a five milliliter graduated, a ten milliliter volumetric, and a ten milliliter graduated pipet. Densities were taken with the same pycnometer and weighed on the same scales. Titrations were made with two ten milliliter micro burets.

Difficulties encountered in getting correct analysis was the most troublesome problem during this work. After fifteen runs were completed, it was found that an error was present in the results obtained to that time.

For the first sixteen runs a base of approximately one normal was used in titration of both aqueous and organic phases. Under these circumstances a small sample of the aqueous phase was required if approximately twenty milliliters of base was to be used in the titration. A 1.5 milliliter sample was chosen. Four, two milliliter graduated pipets were used for the sampling. It was discovered that the four pipets did not deliver exactly the same volume of sample. By using the different pipets the concentration of one sample could vary as much as 0.4 percent weight of acetic acid. This error although small made an error in the overall efficiency of the unit up to 20 percent. This error coupled with the fact that adequate measures were not taken during the initial fifteen runs to maintain the base free from carbon dioxide gave inaccurate and inconsistent results.

The first ten runs gave consistent results in the range of 86 to 98 percent efficiency with increasing impeller speed at a given feed rate and mass ratio. These were later proven to be in error due to the errors in the pipets and the changing normality of the base. These results also led the author to believe that the operations were conducted in the proper range of impeller speed and feed rates. This explains why the final runs were made at the selected feed rates and impeller speeds. Runs eleven to sixteen were at a given impeller speed (selected from the results obtained from the first erroneous series of runs) and operated at different total feed rates at a given mass ratio. Inconsistency in this series of runs led to the discovery of the two above

mentioned sources of error which were eliminated for the final eight runs made under the same conditions as the first two series of runs.

RESULTS

Table 1 gives the results obtained for the runs that were made after all apparent errors were corrected. Two graphs were made with the data of table 1 as shown in figure 8 and 9. Figure 8 is a plot of efficiency versus impeller speed at a set feed ratio and rate. Figure 9 shows the change in efficiency with total feed rate at a set impeller speed and feed composition and mass ratio.

Both of the graphs were best represented by straight lines at an overall efficiency of 66 percent. The individual stages have approximately the same efficiencies.

The results shown in these two curves were not expected. Other investigators (1, p. 1361) (4, p. 204) (2, p. 407) employing other systems have found that efficiencies increase with increasing impeller speed and decrease with total feed rate. However, with the system investigated and within the range of feed ratio and impeller speeds in this investigation the efficiencies did not change. From the data obtained several conclusions can be drawn: (a) the impeller speed might be increased for this system, and (b) the total feed rate can be greatly increased thus reducing the residence time. However, one can not eliminate the possibility that the impeller speed is at the optimum value even at the lower range. The only way to provide an answer to this question would be to increase greatly the impeller speed and if no

TABLE 1 Experimental Data

Run Number	Aqueous Phase 1	Organic Phase 1	Aqueous Phase 2	Organic Phase 2	Aqueous Phase 3	Organic Phase 3	Aqueous Feed	Organic Feeds
17	67.24%	19.71%	64.59%	16.23%	61.06%	13.36%	22.98%	58.56%
18	67.43%	20.12%	65.19%	16.66%	62.04%	13.65%	23.02%	58.60%
19	67.04%	19.41%	64.73%	16.19%	61.82%	13.48%	23.03%	58.75%
20	66.90%	19.36%	64.70%	16.14%	61.82%	13.46%	22.96%	58.71%
21	67.49%	20.18%	65.49%	17.13%	62.72%	14.15%	23.02%	58.64%
22	67.08%	19.54%	64.89%	16.31%	62.05%	13.57%	22.99%	58.75%
23	67.01%	19.41%	64.68%	16.20%	61.88%	13.52%	22.81%	58.75%
24	66.99%	19.31%	64.52%	16.07%	61.81%	13.49%	22.86%	58.75%

Figures represent weight percent acetic acid

Run Number	Aqueous Feed	Organic Feed	Products Total	Weir Height	Room Temp.	Impeller Height 1	2	3	R.P.M.	Eff.
17	43.7	178.2	222	6.8	23°	19.5	19.0	21.0	439	70.2%
18	52.25	213.9	266	6.7	23°	19.0	19.0	21.0	442	66.3%
19	34.8	142.6	177	6.8	24°	19.0	19.0	21.5	442	65.5%
20	30.6	124.7	158	6.8	25°	20.5	19.0	21.5	445	66.0%
21	23.5	96.5	123	6.65	24°	22.5	21.0	21.5	449	63.9%
22	39.2	160.8	203	6.85	24°	21.0	21.0	21.0	452	63.9%
23	30.6	124.7	158	6.88	24°	21.0	20.0	20.5	562	65.5%
24	30.6	124.7	160	6.9	24°	20.5	21.5	21.5	292	65.4%

Feed rates given in grams per minute
Weir height was read from scale independent of apparatus
Impeller height was measured from top of mixer section to top of impeller and figures represent thirty-seconds of an inch

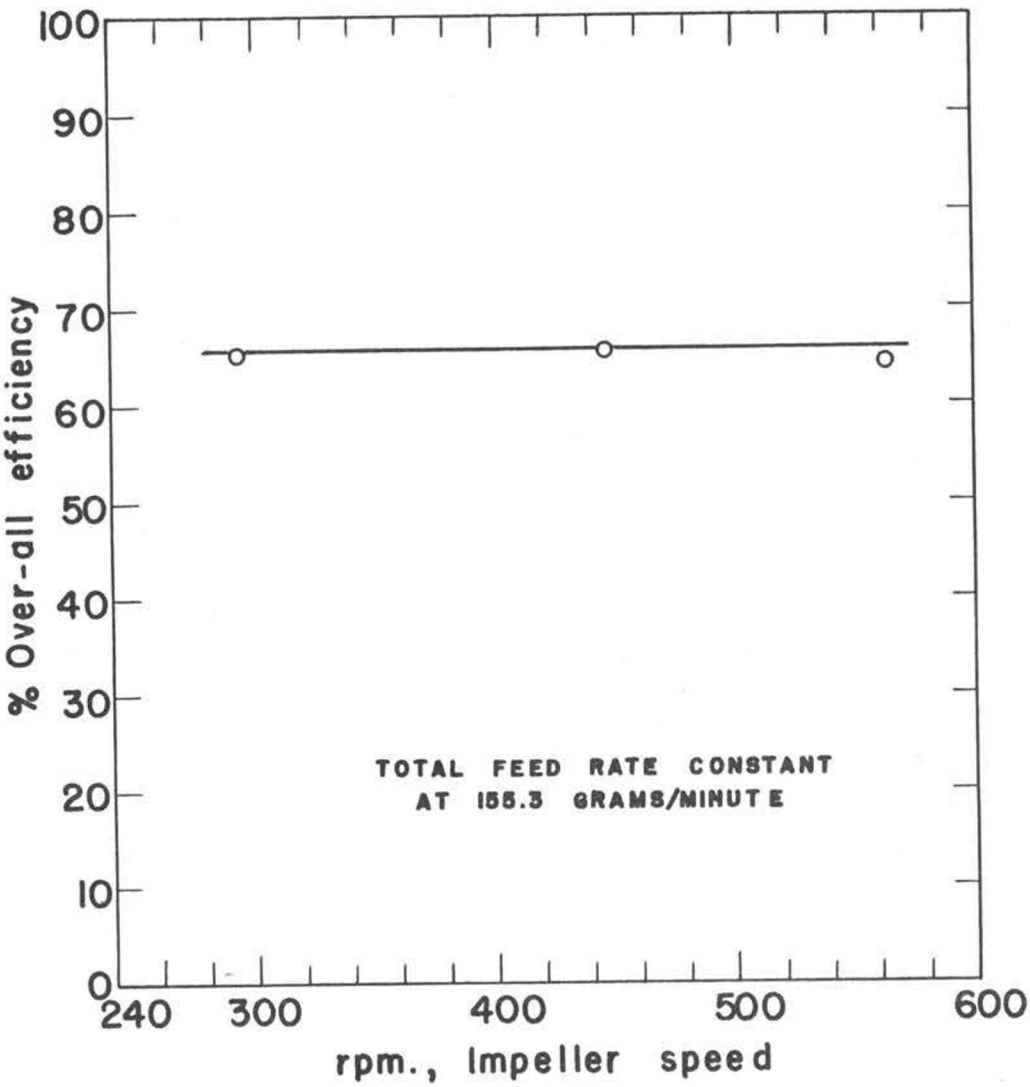


FIGURE 8 CHANGE IN EFFICIENCY WITH IMPELLER SPEED

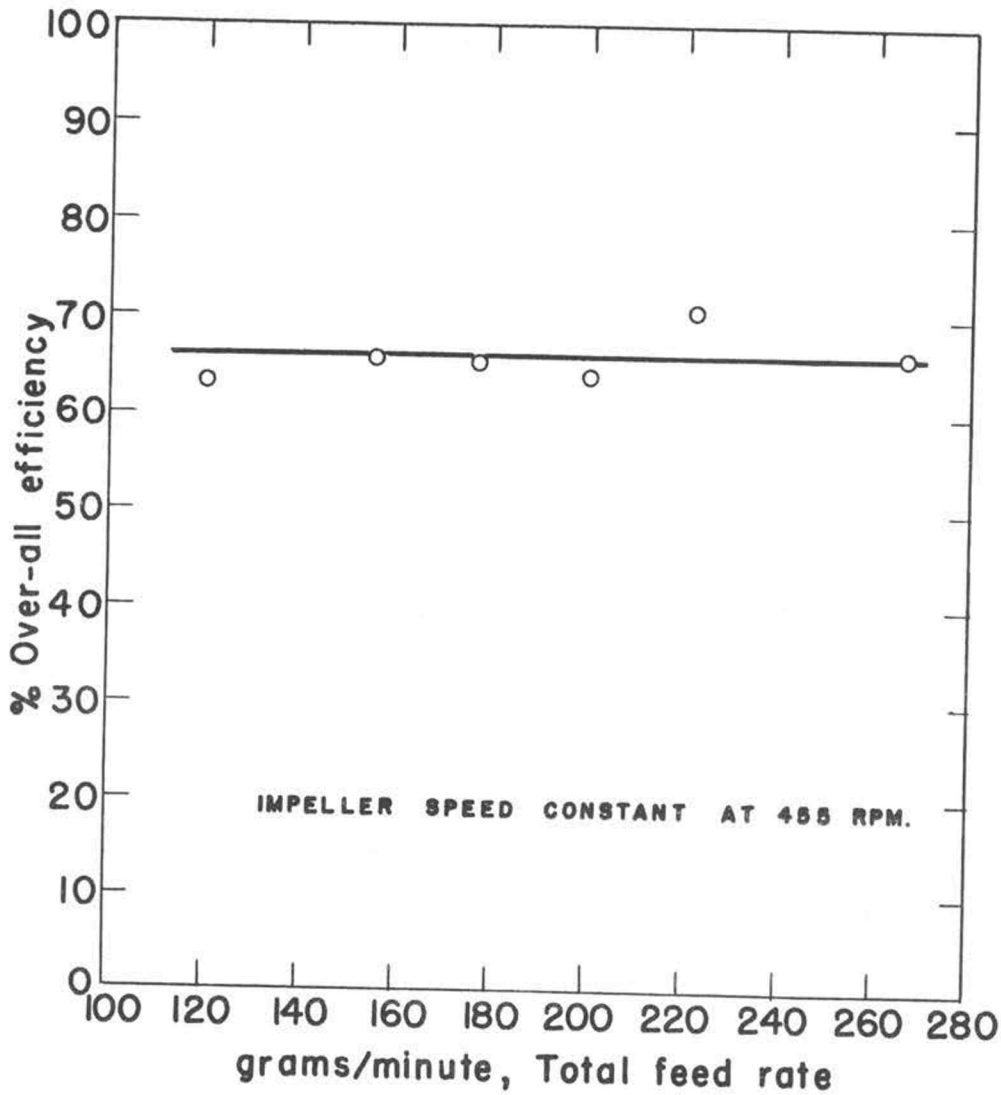


FIGURE 9 CHANGE IN EFFICIENCY WITH TOTAL FEED RATE

change in efficiency is noted it would indicate that very little agitation is necessary to provide for adequate mass transfer. This may not be the correct answer but it can not be eliminated as a possibility.

It is also noted that without additional baffle and fin arrangements the limiting efficiency may not be much higher.

RECOMMENDATIONS FOR FUTURE WORK

The following recommendations are made for possible improvements in the apparatus. If faster impeller speeds are to be used a seal will have to be made around the glass cover plate and the mixing section. While operating at approximately six hundred rpm there was difficulty in preventing splashing and hence a material loss from the mixing section. At speeds below this figure this problem was not encountered.

If at all possible it would be desirable to have the feed pumped using positive displacement pumps. Under the present set up the feed rate fluctuated and frequent adjustment was necessary.

It might also be advantageous to have a method of measuring the height of the liquid in the settlers as well as the height of the interface.

Future improvement may also include the installation of various baffle arrangements for the mixing section as it was noted in the introductory section that the efficiency was increased in every case where mixing blades and baffle configurations were used.

Larger feed and product containers would permit easier operation over a wider range of feed rates.

With these improvements it would be expected that some effect of feed rates and impeller speeds on efficiencies might be found, and it is suggested that future investigators of this system may wish to study these variables.

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APPENDIX

Table 2

Equilibrium Data by Woodman

a-% water by weight

b-% toluene by weight

a	b
0.4721	81.3979
1.474	66.866
2.966	55.124
4.594	45.84
4.689	44.72
6.871	35.38
8.507	29.27
9.809	25.28
10.82	22.34
11.53	19.89
11.93	19.30
12.02	19.48
12.38	18.37
12.83	17.43
12.98	17.53
13.56	16.07
14.50	14.62
16.00	12.54
17.10	11.32
18.00	10.29
20.08	8.662
21.76	7.415
23.23	6.342
24.34	5.97
25.30	5.454
25.80	5.308
26.52	5.02
28.13	4.446
33.42	2.975
43.89	1.419
57.5967	0.6533
67.8344	0.2856

Table 3
Tie Line Data by Woodman

Aqueous Layer	Toluene Layer
Wt. % HAc	Wt. % HAc
0	0
21.38	1.374
30.87	2.797
37.69	4.072
47.50	6.363
58.05	10.35
62.52	12.98
64.86	14.98
67.70	18.55
69.71	21.50
70.55	24.88
69.85	32.96
69.40	36.13
66.68	40.64
65.31	44.41
58.46	53.07