CASE STUDY, PILOT PLANT TESTING RESULTS IN REDUCED COST FOR LIQUID-LIQUID EXTRACTION COLUMN

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INTRODUCTION

It has been previously reported[1,2,3] that the most accurate method used to design liquid-liquid extraction (LLE) columns is via pilot plant testing and empirical scale-up. Furthermore, optimized column design via pilot plant testing can also result in significant capital and operating cost savings for the end user as well. This paper will cover a case study for the design of a liquid-liquid extraction column. An application is presented in which water is used as the extraction solvent for removing methanol from an organic feed stream. The original extraction column design, based upon estimating techniques only (no previous test experience), called for a rotating disc contactor (RDC column) with specified diameter and agitation zone height. Pilot plant testing was used to (1) demonstrate that a Karr Column is the more appropriate column type for this application, and (2) provide the data for the design of a significantly smaller production size column. The result was a cost savings of 35% for the end user due to the design of a Karr Column based upon the pilot plant testing.

APPLICATION

A "Request for Quotation" was received for a liquid-liquid extraction column to extract methanol from an organic acrylate stream using water as the solvent. The design criteria for this application are summarized as follows:

Organic Feed Rate	37,100 lb/hr (4675 GPH)
Water Rate	11,900 lb/hr (1427 GPH)
Methanol Concentration:	
Organic Feed	2.56 %
Water (recycle)	0.53% (via distillation)
Raffinate	< 0.1% (specification)

Suppliers were requested to provide a bid for a 78" diameter RDC column with 35 agitated stages. An initial quotation for the RDC column was prepared and presented to the end user.

In reviewing this application, the author was not comfortable with the RDC column. Previous experience with similar organic-water systems revealed that this process could be very susceptible to emulsification. The RDC column is not the best choice for these type systems due to the high sheer nature of rotating discs[4]. Rotating discs provide relatively poor mixing action and must be operated at high speed and sheer in order to generate sufficient surface area (small dispersed phase particle size) for effective operation. The Karr Column with a reciprocating plate stack imparts relatively low sheer and generates a more uniform dispersed phase particle size. This makes the Karr Column the optimal choice for systems that tend to emulsify[5]. With the potential for significant size and equipment cost savings, the client agreed to conduct a pilot test in a Karr Column before proceeding with the purchase of the production column.

Prior to pilot testing, liquid-liquid equilibrium data was generated using a standard "Shake Test" procedure[6]. The results from the shake tests revealed that the distribution coefficient for methanol in this organic-aqueous system was a relatively constant value of 6.7 for the range between 2.5% and 0.1% methanol in the aqueous phase. The concentration of methanol in the aqueous phase is 6.7 times the concentration in the organic phase. Thus, the Kremser equation can be used to calculate the number of theoretical stages required as follows:

$$n_S = \frac{Log\left[\left(\frac{x_f - \frac{y_s}{m}}{x_n - \frac{y_s}{m}}\right)\left(1 - \frac{1}{E}\right) + \frac{1}{E}\right]}{Log\, E}$$

			Solute Free Value
Where:	x_f	= solute concentration in feed	0.0256
	x_n	= solute concentration in raffinate	0.0009
	y_s	= solute concentration in solvent	0.0053
	m	= distribution coefficient	6.7
	E	= extraction factor (S/F x m)	0.33 x 6.7 = 2.2
	n_s	= number of theoretical stages	calculated

n_S = 6 theoretical stages

PILOT PLANT EQUIPMENT SET-UP AND PROCEDURE

The equipment set-up for the pilot plant test is shown on the attached Figure 1. Essentially, this set-up consists of the following:

- One-inch diameter x 8' plate stack height Karr Column with 2" diameter expanded ends top and bottom. Shell is glass and the plate stack assembly is 316SS. The column includes a variable speed drive (manual control) for operation between 0 and 400 strokes per minute (SPM). A manifold system for the water inlet location was used as shown in Figure 1 to provide the option for 6' or 8' agitated height. *NOTE: The height was selected based upon an assumption of ~12" height per theoretical stage.*

- The organic feed and water solvent were received in 55-gallon drums. These were charged to the pilot Karr Column directly from the drums using a dip tubes and variable speed metering pumps (FMI pumps). Flow rates were set and checked using

graduated dropping funnels and stopwatch.

- The bottoms flow rate (aqueous extract phase) and interface control was via an FMI pump with manual adjustment. Overhead (organic raffinate phase) was allowed to flow by gravity to receiver.

Test Procedure

The following procedure was used to operate the pilot Karr Column:

- Initially, DI water was "spiked" with 0.5% methanol and 0.5% acrylate to simulate the expected recycle "solvent" for plant operation.

- The organic acrylate feed and water were charged directly from 55-gallon drums into the extraction column. The operation was at room temperature.

- For startup each day, the column was first filled with clean acrylate (no methanol) and the agitator speed was set at a slow, initial setting.

- Once the column was full, the acrylate feed and water pumps were turned on and both flow rates adjusted to the desired rates. The agitator speed was then adjusted to the desired set point.

- The bottoms pump was turned on and adjusted to establish an interface in the bottom-disengaging chamber of the column. The bottom take-off rate was continuously monitored and adjusted to maintain a constant interface level.

- The test supervisor specified conditions for each run. Feed and solvent rates were confirmed every 30 minutes throughout each run. At the completion of a run, raffinate and extract flow rates were measured and samples of each were taken for analyses.

- During startup each day, a total of five (5) column turnovers were performed before the initial samples were taken. A column turnover is defined as the total column volume divided by the combined feed and solvent rates. After the first run, and following adjustment of the variables, a total of three (3) turnovers were performed before additional sampling.

TEST RESULTS AND DISCUSSION

A summary of the key operating parameters and the results (raffinate methanol concentration) are provided in Table 1. An overview of the testing follows:

- Runs 1 – 3 were made with an agitated height of 6' and standard 2" plate spacing (#1) throughout the entire plate stack. The results show that even with the maximum

agitation speed before flooding of 100 strokes per minute (SPM), the target raffinate concentration of less than 0.1% methanol was not achieved. This indicates that more height is required for this arrangement. However, visual observation of the performance within the extraction column revealed that the dispersed phase droplets (heavy water phase) were relatively large and flowed down the column to a point approximately 6-12" above the organic phase inlet. This was where flooding began, i.e. a second interface formed in this region of the agitated zone. Above the flood point, the column was observed to have relatively low dispersed phase holdup.

- Based upon the observation of flooding and dispersed phase holdup, the plate stack was removed from the column and the plate spacing was adjusted. Starting from the bottom of the column there was one foot with 6" plate spacing, followed by one foot of 4" plate spacing and then the remainder of the column was at 2" plate spacing. This plate stack arrangement (#2) was used for all additional runs.

- Runs 4-9 were made with the modified plate stack (#2) and 6' agitated height. A review of Runs 4-9 reveal that not only was the capacity increased (feed rate increased from 150 to 180 cc/min), but significantly higher agitation speed could be imparted to the system without causing a flooding condition. The results reveal that an agitation speed of 200 SPM was necessary in order to achieve the required number of theoretical stages and produce raffinate with < 0.1% methanol.

- One additional run, at a feed rate of 210 cc/min (Run 11), also generated raffinate with < 0.1% methanol.

PRODUCTION COLUMN DESIGN

The scale up procedure for the Karr Column has been reported previously[5]. Based upon discussions with the end-user it was decided that the results from Run 9 would be used as the scale up point to the production column even though a higher rate (Run 11) worked successfully. This would insure that the production column had plenty of future capacity potential. The conditions for Run 9 were as follows:

Organic Feed Rate	150 cc/min
Water Solvent Rate	45 cc/min
Specific Throughput	560 GPH/ft^2

In liquid-liquid extraction columns, specific throughput is used to designate capacity. Specific throughput is defined as the feed + solvent rates divided by the cross sectional area of the column. For the Karr Column the capacity scale up is 1:1, thus the diameter of the production column is calculated as follows:

$$\text{Area} = \frac{\text{Feed Rate} + \text{Solvent Rate}}{\text{Specific Throughput}}$$

$$\text{Area} = \frac{(4674 + 1427)\ \text{GPH}}{560\ \text{GPH} / \text{ft}^2}$$

$$\text{Area} = 11\ \text{ft}^2$$

$$\text{Diameter} = 45\text{"}$$

The scale up height of the Karr Column is calculated as follows:

$$H_{Production} = (D_{Production} / D_{Pilot})^{0.38} \times H_{Pilot}$$

$$H_{Production} = (45 / 1)^{0.38} \times 6\ \text{feet}$$

$$H_{Production} = 26\ \text{feet}$$

COLUMN COMPARISON AND SAVINGS

A side by side size comparison between the original RDC specified in the Request for Quotation and the Karr Column designed via the pilot plant test program is shown in Figure 2. Based upon this comparison, it is evident that the Karr Column is significantly smaller than the RDC column originally specified for this application.

The quoted price for the Karr Column was 35% less than the quoted price for the RDC column. This price difference does not include additional installed cost savings due to the smaller column, such as less support structure, less piping requirements and lower foundation loading.

Finally, it must be pointed out that there is no evidence that the requested RDC column would meet the performance criteria specified by the end users. However, the pilot plant testing demonstrated the performance of the Karr Column. As such, the Karr Column was quoted with a process performance guarantee, whereas the RDC column did not include such a guarantee. In the end, the end user purchased and installed the Karr Column designed from the pilot test data. At the time of this paper, that Karr Column had been meeting capacity and efficiency performance for more than 10 years.

CONCLUSION

Liquid-liquid extraction columns require pilot plant testing for accurate design of production size columns. Pilot plant testing is critical for performance, and it can result in significant capital cost savings for the plant installation as demonstrated in this case study. The S/F ratio was not a variable in this test program. However, pilot plant testing often is used to optimize the S/F ratio vs column height in order to minimize the operating cost for an extraction system (i.e. minimize the solvent recovery costs).

LITERATURE CITED

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3. Glatz, D., Parker, W., "Portable Extraction Column for Generation of Scale-up Data". AIChE National Meeting, November 8-12, 2004.

4. Holmes, T., Karr, A., Cusack, R., "Performance Characteristics of Packed and Agitated Extraction Columns", AIChE Summer National Meeting, August 17, 1987.

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6. Glatz, D., Parker, W., "Extraction Enhancement, One Step at a Time", Chemical Engineering, November 2004.

FIGURE 1

PILOT KARR COLUMN SET-UP

METHANOL EXTRACTION FROM ACRYLATE

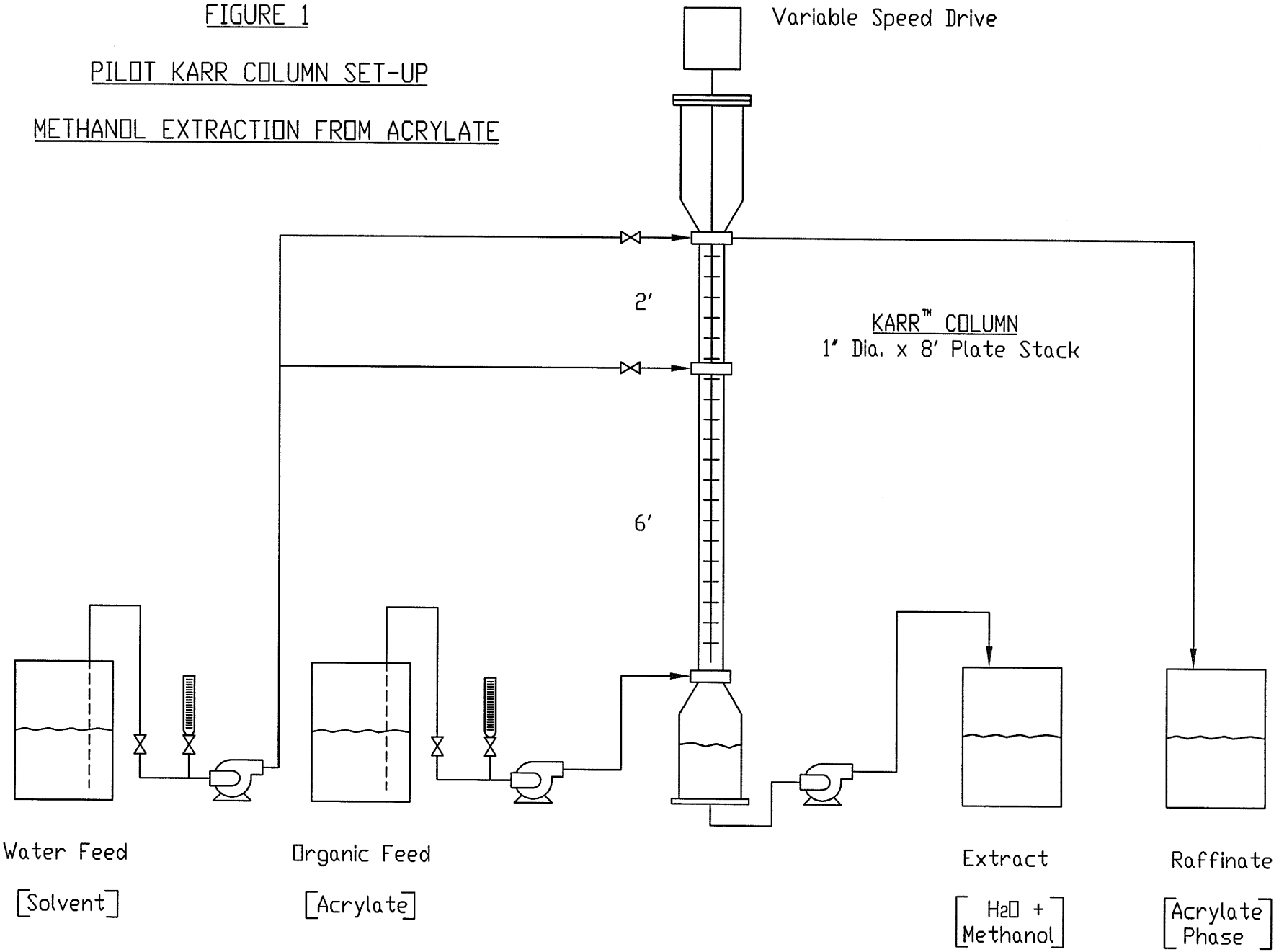


TABLE 1

Karr Column Pilot Plant Test Results

Methanol Extraction from Acrylate

RUN No.	Plate Stack	Organic Feed Rate (cc/min)	Water Feed Rate (cc/min)	Agitator Speed (SPM)	Interface	Raffinate Methanol Conc. (%)	Raffinate Water Conc. (%)
1	1	150	45	75	Bottom	0.165	2.83
2	1	150	45	100	Bottom	0.124	2.55
3	1	150	45	110	Bottom	Column Flooded	
4	2	150	45	110	Bottom	0.169	2.78
5	2	150	45	140	Bottom	0.112	2.72
6	2	180	54	100	Bottom	0.203	2.90
7	2	180	54	125	Bottom	0.146	3.08
8	2	180	54	150	Bottom	0.118	2.66
9	2	180	54	200	Bottom	0.078	2.73
10	2	180	54	220	Bottom	Column Flooded	
11	2	210	63	175	Bottom	0.084	2.65

Notes:

1" diameter x 6' plate stack height Karr Column used for all runs

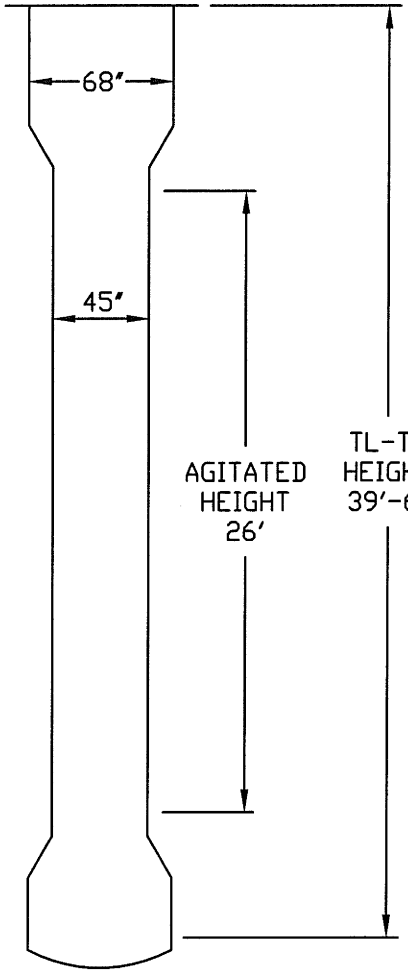
Plate Stack #1 = constant 2" plate spacing throughout

Plate Stack #2 = from bottom, 1' of 6" spacing, 1' of 4" spacing, remainder at 2" spacing

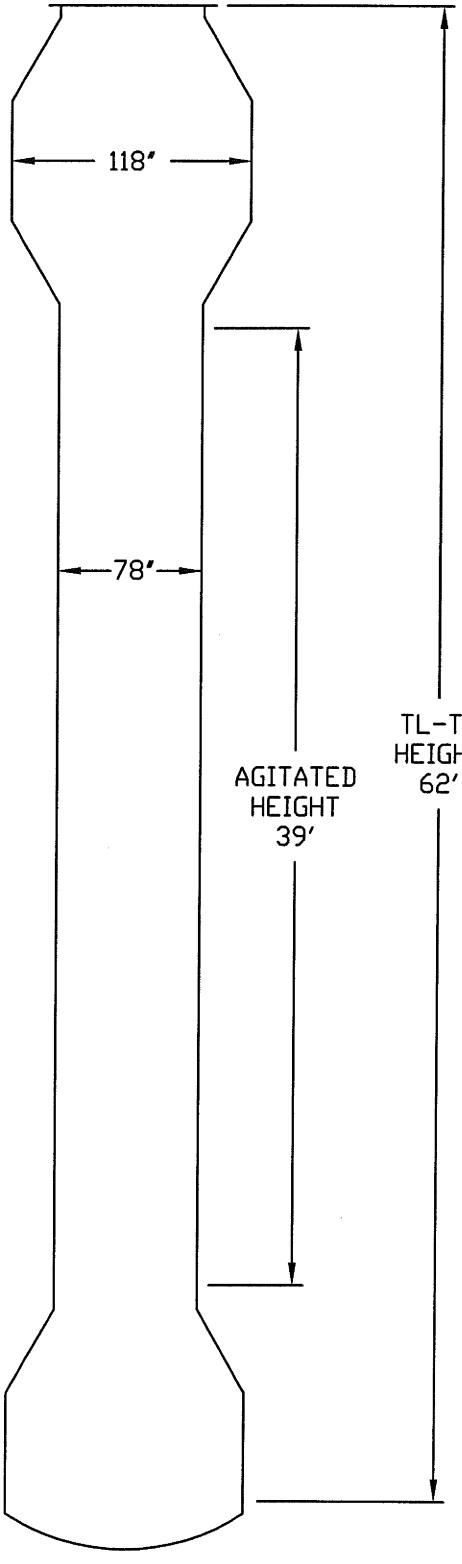
Feed is organic acrylate with 2.5% methanol

FIGURE 2

KARR™ vs RDC COLUMN SIZE COMPARISON



KARR COLUMN



RDC COLUMN