Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Steiner, L. and Hartland, S.(1980) 'A New Type of Agitated Liquid/Liquid Extraction Column with Enhanced Coalescence Plates', Separation Science and Technology, 15: 4, 907 — 923
To link to this Article: DOI: 10.1080/01496398008076277
URL: http://dx.doi.org/10.1080/01496398008076277

SEPARATION SCIENCE AND TECHNOLOGY, 15(4), pp. 907-923, 1980

A NEW TYPE OF AGITATED LIQUID/LIQUID EXTRACTION COLUMN WITH ENHANCED COALESCENCE PLATES

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ABSTRACT

A new type of stator plates for the separation of mixing cells in agitated extractors is presented. These plates consist of thin vertical elements made of materials wetted by the dispersed phase. These elements form a lattice with an extremely high free area (over 90%) which hinders all flows other than the axial one. In this way, mixing cells are effectively separated while at the same time, the phases can flow through the column without flooding. The dispersed phase coalesces on the plate surfaces and occupies part of its free area, so that only the part necessary for the flow of the continuous phase remains free. Very high throughput and good separation efficiency were obtained during the preliminary experiments in a broad range of phase throughputs and ratios. The results were better than those achieved by comparable conventional columns.

INTRODUCTION

Counter-current extraction columns can be divided into two types: with, or without, mechanical power input. Today, columns of the first type are usually preferred because they are considerably smaller and more efficient. Mechanically agitated columns are provided either with rotating agitators or pulsators to accelerate extraction, thus homogenizing the contents and allowing a continual formation of new interfaces. It is impossible to say at present which of the two types of column is better, since excellent examples of both are available. It does however appear that the agitated column is more widely applicable and is therefore extensively used in industry.

No universal extractor currently exists which could be used for general application. The available types are designed for certain specific conditions, and it is hardly possible to maintain the same arrangement if another form of extraction is desired. The requirements for a successful extractor are clear however; it should possess high separation efficiency over as wide an extraction range as possible. Since the construction of extractors with numerous separation stages and small phase throughputs presents no great difficulty, the demand remains for good separation with the highest possible throughput. A suitable index for this is the extractor volume which is necessary for the separation efficiency of a theoretical stage at unit total throughput (the sum of both phases). This index is defined as the separation volume S,

$$S = (HETS)(A), \quad (1)$$

where HETS is the height equivalent of a theoretical stage, and A is the cross sectional area needed for unit total throughput.

Under favorable conditions, agitated columns can achieve small separation volume values, but further development is desirable. The improvement should serve mainly to broaden the effective region of operation. In this connection, it would be desirable to increase separation intensity (reduction in height equivalent of the theoretical stage) especially in the region of greater loads. In principal, agitated columns work either like a cascade of mixed cells, or as a continuous column which may be regarded as an improved type of spray column. The difference between these two cases is whether or not repeated coalescence and a new formation of drops occurs in each stage. It is obvious that mixer-settler operation is more intensive, and mass transfer rates are higher, because the interfaces are continually renewed. On the other hand, continuous columns of the second type can handle higher loads.

The free area of the stator plates have considerable influence on the load and separation efficiency of all agitated columns. If the area is small, one approaches the mixer-settler region, with drops remaining longer in each mixing cell and therefore being more likely to come into equilibrium and, under favorable conditions, to coalesce and re-form. If the free area is large, drops can circulate between the mixing cells. In this case, the probability of coalescence is less, backmixing increases, and the separation efficiency falls. On the other hand, a column with a larger free area can operate at higher loads, while the flooding limit is increased. Because of the great importance of the free area of the stator to the hold-up of the dispersed phase, columns have been designed with varying free areas along their heights to suit the conditions of the changing physical properties of the phases during extraction.

THE ENHANCED COALESCENCE (EC) COLUMN

It is apparent that the efficiency of an agitated column can be improved if stator rings are provided which effectively separate the cells lying one above the other but do not hinder the axial flow of the two phases. Moreover, they should promote coalescence, because only by repeated coalescence and redispersion can the mass transfer coefficients be high. Such a design has now been developed. In this design, the mixing cells are separated by means of a lattice made from tall, thin, vertical plates or from plates slightly inclined to the vertical. Furthermore, it is advantageous that these plates be made from a material (e.g., teflon or teflonized metal when oil is to be dispersed) which is wetted by the dispersed phase. The arrangement of these units can be varied. For instance flat, thin elements can be constructed to form a quadratic lattice, or a weblike construction, or a system of concentric cylinders can be used. Figure 1 shows

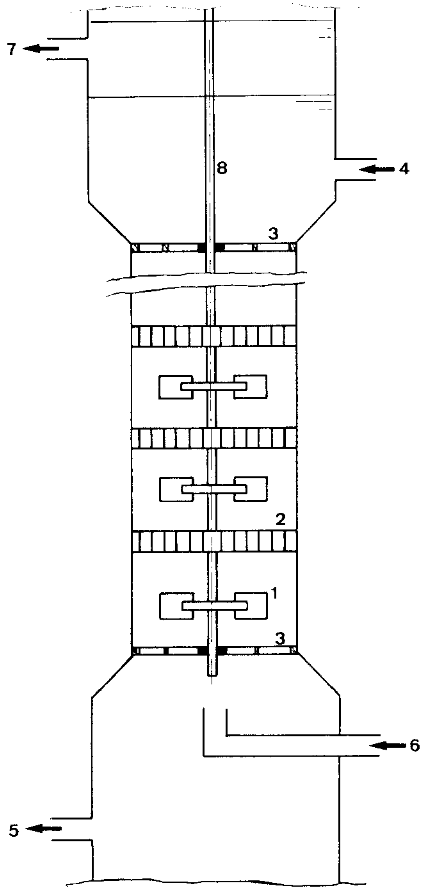

FIGURE 1. Arrangement of plates in column: 1 - agitator, 2 - EC plate, 3 - position of agitator shaft, 4 - input of heavy phase, 5 - discharge of heavy phase, 6 - input of light phase, 7 - discharge of light phase, 8 - agitator shaft.

the arrangement of plates in the column, whereas examples of possible plate constructions are depicted in Fig. 2. All these designs have the important feature that the lattice resists all flows except the axial one. The radial and tangential components are especially retarded, as shown in Fig. 3.

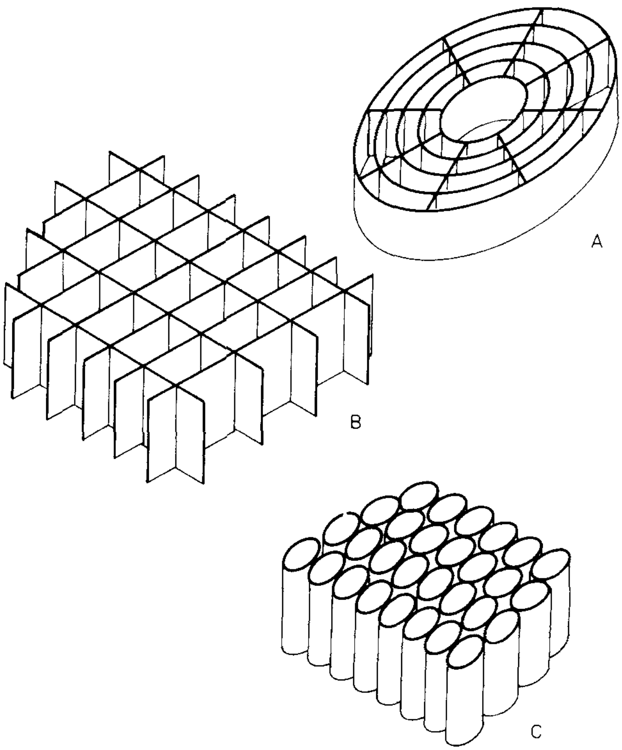

FIGURE 2. Possible constructions of the EC (Enhanced Coalescence) plate; a - concentric rings with ribs, b - square lattice, c - cylinder.

Because the lattice units are wetted by the dispersed phase, it coalesces in the apertures and blocks part of the free area, releasing itself from the top surface as large drops which are then redispersed by the next agitator. When the throughput is low, the greater part of the stator is blocked by the coalesced phase, and the free area remaining is just sufficient to allow the continuous phase to pass through. The stator thus operates in a similar way to a distillation plate without overflow, whose free area also depends on the load. In an EC column, the dispersed phase rises from the feed pipe to the first stator ring and

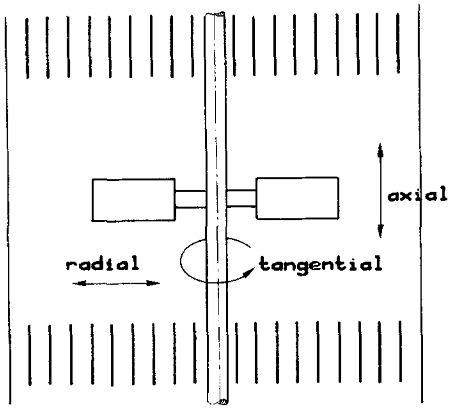

FIGURE 3. Types of flow produced by the agitator.

coalesces in its apertures. When the entire stator volume is full, large drops are released from the surface and broken down to smaller drops by the next agitator. These small drops circulate in the mixer cell until they are caught by the top or bottom stator ring and coalesced. By adjusting the width of the lattice apertures and inclining their surfaces to the vertical, it is possible to ensure that only a small fraction of the drops pass through the stator ring without coalescence occurring. Increased coalescence retards backmixing of the dispersed phase, which, even at higher throughput, continues to flow in the mixer-settler regime.

It is not yet known to what extent backmixing of the dispersed phase occurs, but it should not be more than in similar commercial types. In all probability the agitators play an important part, and those that promote radial flow should be used. Accuracy in the construction of a stator lattice is of great importance, particularly in regard to the symmetry of each element along the horizontal plane. This is made clear in Fig. 4.

The width of the apertures determines the load capacity of the entire column. With this type of stator plate, it is not the free area but the total surface area of the stator which determines the amount of coalescence and controls the behavior of the column.

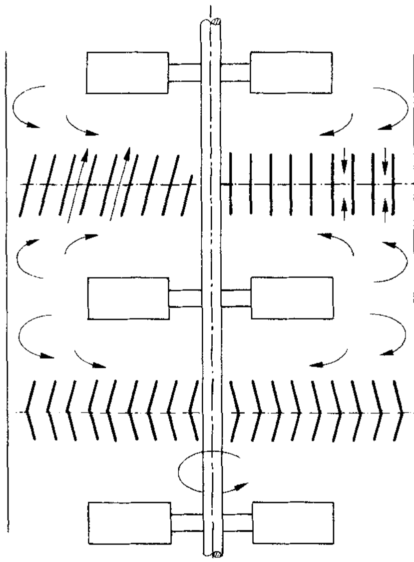

FIGURE 4. Operational position of the EC plate (without coalescence of dispersed phase).

If the apertures are too narrow, they remain closed, thus adversely affecting the dispersed phase throughput. If the apertures are too wide, coalescence and cell separation are inadequate for operation in the mixer-settler regime. In general, the load capacity of the EC column should span a broader throughput region than that of the usual commercial types.

EXPERIMENTAL

Model Column

In order to verify that the introduction of wetted surfaces does in fact improve performance, a model column, 50 mm in diameter, was constructed to obtain a preliminary estimate of the necessary channel width. Mass transfer experiments were carried out in the toluene-acetone-water system. Since a column of such small diameter cannot be compared directly with larger versions, tests were also conducted with conventional plates in the same apparatus. The column, which consisted of a glass tube of 50 mm diameter, was fed from tanks. The water and toluene flows were measured with Rotameters. Pure water and a toluene-acetone mixture were always used, so that mass transfer was from the dispersed toluene into the continuous water phase. The water was not circulated but, after passing through the column, flowed directly into the drain. The toluene was mixed with about 5% acetone in a 40-ℓ storage tank. After flowing through the column and through a water separator, it was collected in a second tank.

The column contained four stages. The blade agitators in each stage were 30 mm diameter and were mounted on a shaft at 60 mm intervals. Throughout the test phases, the shaft and all other column items remained unchanged; only the plates between the agitators were varied. The first arrangement consisted of conventional plates such as those used in the Oldshue-Rushton column, i.e., annular metal rings of 31 mm diameter, so the free area was about 40%. (With a column only 50 mm in diameter, it was not possible to consider very sophisticated designs.) After completing the comparison tests, three different EC plate designs had been examined. These are identified in Fig. 5.

Analysis of the test data was made assuming plug flow and linear equilibrium. Since the conditions other than plate design were constant, the test data could be compared directly.

It was found that the Type B EC plate (Fig. 5) yielded the best results with respect to theoretical stage height and throughput. The results of the Type C EC plate were also better than those of the Oldshue-Rushton comparison plate (Type A), but the difference was not great. The data presented in Figs. 6 and 7 show the theoretical stages attained per unit length of column (TS/m) and the dependency of the separation volume S on the load, B, which is defined as rate of total volume throughput (the sum of both phases) per unit area.

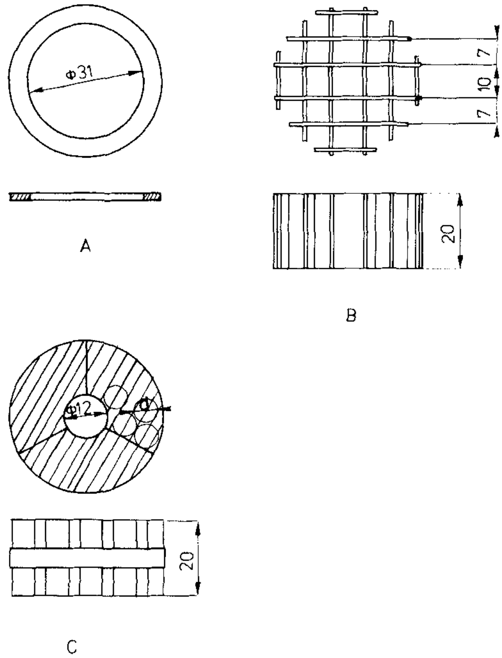

FIGURE 5. Plate designs examined on the small column model.

Intensive coalescence was observed visually on the EC plate, even at high stirring speeds. At lower and intermediate speeds, the rivulets of the coalesced dispersed phase, running from the top surface of the plate and being dispersed by the agitators, were clearly visible. The tube plates (Type C), perhaps because of the small diameter of the column, were not suitable, as they hindered the flow of the continuous phase. This is probably due to the filling of the spaces between the small cylindrical tubes with the dispersed phase, thus causing the water to be mainly transported down the mixer shaft or the column wall. Much better results were obtained when the plate with vertical plane surfaces was used. Loads above 60 m^3/m^2h were reached, and flooding tended to occur in the ends of the column rather than in the column itself. At high

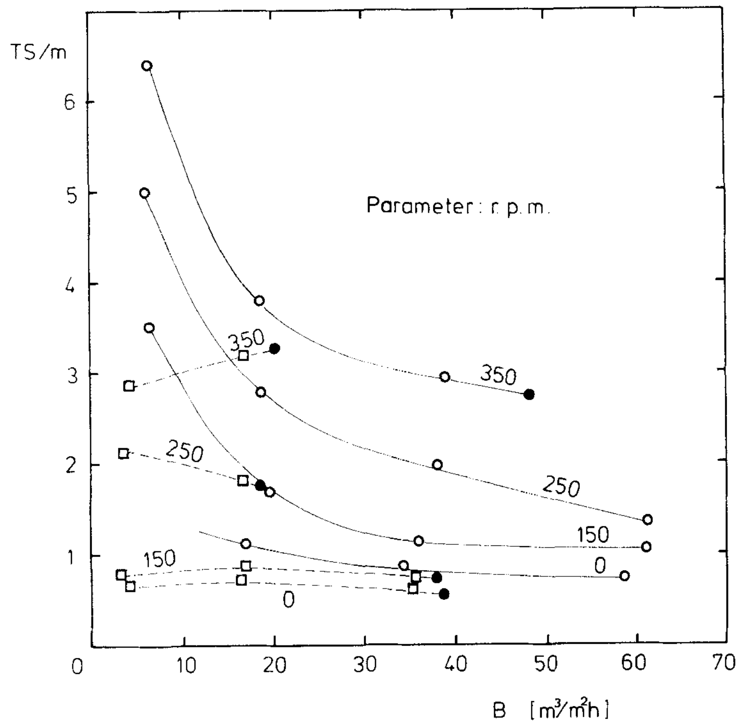

FIGURE 6. Results of mass transfer experiments with the 50-mm model column. Dashed lines - Plate A (Oldshue/Rushton); solids lines - Type B EC plate. The numerical values on the curves denote stirring speeds (rpm), and the solid points denote flooding.

loads a close-packed droplet dispersion formed under the lowest plate which eventually inverted, thus causing flooding to occur. Had the volume under the first plate been large, even higher throughputs could have been attained.

Although the number of stages per meter of column height was not very large with the Type B plate, one should not make a direct comparison between this rather primitive column and an industrial one. In any case, efficiency was up to twice as large as that obtained with conventional plates.

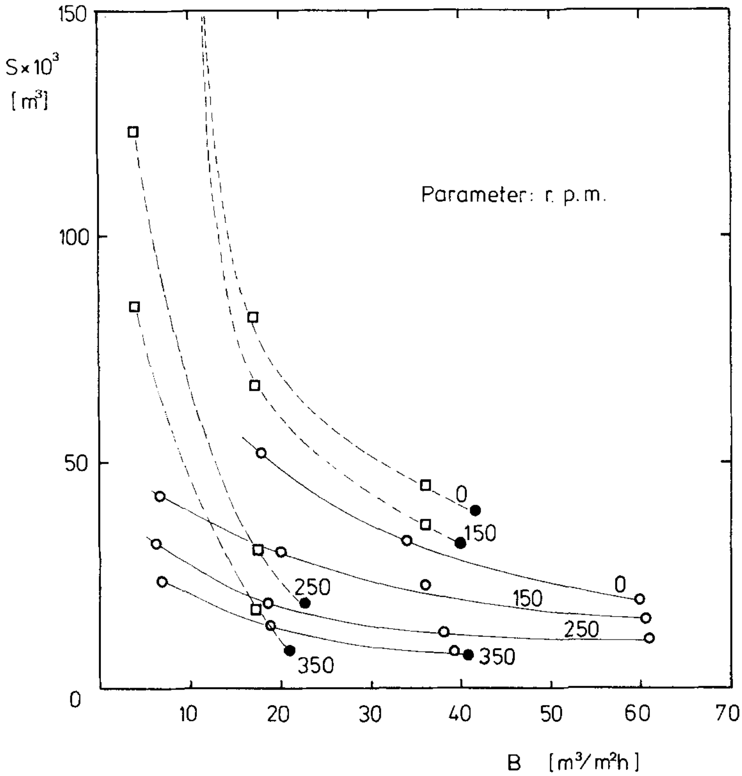

FIGURE 7. Comparison of the dependence of separation volume on stirring speed and load. Dashed lines - Plate A; solid lines - Type B EC plate. The numerical values on the curves denote stirring speeds (rpm), and the solid points denote flooding.

The column was also operated with stationary agitators; in this case repeated coalescence and rivulet formation could be readily observed. Had the plates been properly designed and spaced farther apart, it would have been possible to develop an effective column which would work without mechanical power input, operating in a mode somewhere between a non-pulsed perforated plate and a packed column. The value of the separation volume S reached with this column is already very encouraging, especially since the column had been designed expressly for use with agitators. As is seen from the data presented in Fig. 7, the best value was 0.024 m^3 at 58 m^3/m^2h. According to Reissinger (1), a value of about 0.015 m^3 was achieved with a special column packing, "Interpack," but with a load of 20 m^3/m^2h. It was also confirmed that the plates must be symmetrical in the horizontal plane; circulating flows caused by inaccuracies could be readily observed. Plates having two sections slightly inclined to each other, as shown in Fig. 4, should produce even more favorable results.

Pilot Plant Column

In view of the encouraging results shown above, an actual column 80 mm in diameter was constructed and exhibited at ACHEMA 1979 in Frankfurt (2). The column was fitted with 55-mm diameter blade agitators and Type B EC plates.

The toluene-acetone-water system was again used, and an automatic density measuring device was installed. In this operation, the toluene was directed from the column back to the feed container, then returned through a pump and Rotameter into the column again. A metering pump continually injected acetone into the feed to compensate for that which was washed out with water inside the column. To attain this stabilization with respect to acetone concentration required about 30 min, depending upon the flow; after this, the column operated at steady-state conditions. The attainment of steady-state conditions was indicated by monitoring the densities of the exit streams. The water that was discharged from the column was directed into the drain.

Throughputs, end concentrations, hold-up, and backmixing in the continuous phase were determined for this column. Mass transfer measurements indicated that there is an optimal agitator speed for every load; too low or excessive speeds resulted in the mass transfer being less, as displayed by the data presented in Fig. 8. The optimum could not be reached at loads above 30 m^3/m^2h because flooding occurred under the lower plate of the column.

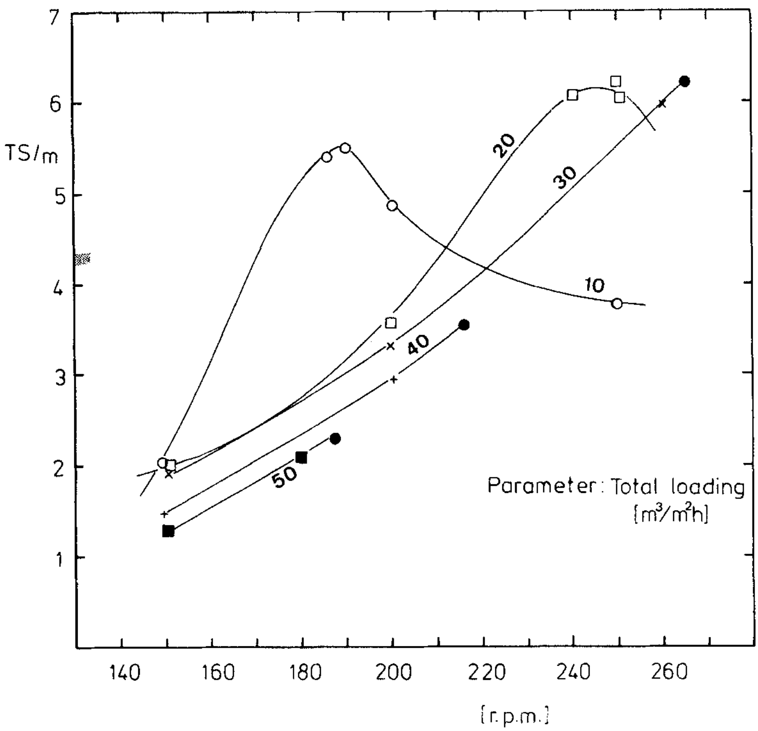

FIGURE 8. Mass transfer results measured on the 80-mm column; the numerical values associated with the individual curves denote column load, whereas the solid data points denote flooding in the lower dome of the column.

This flooding is caused by the first agitator producing streams that are not compensated by an adjacent agitator. The dispersed phase is consequently driven into the lower section of the column, where it coalesces, and possibly causes an inversion of the phases and hence flooding. (In subsequent research, an attempt will be made to design the ends of the column so that they counteract the effects of the first and last agitators.)

At lower throughputs, the streams of coalesced dispersed phase leaving the plates and being redispersed by the agitators could be observed. At higher speeds, the column was completely filled with small drops, rendering further observation impossible. From the dependence of the separation efficiency on the agitator speed, it may be inferred that when speeds are too high, less coalescence and more mixing occurs. The results of the mass transfer experiments are presented in Fig. 9. The lower curves were obtained at constant agitator speed. For the upper curve, the speed was varied until the maximum efficiency was obtained. The last two values were limited by coalescence in the lower part of the column, but the remaining values represent the actual

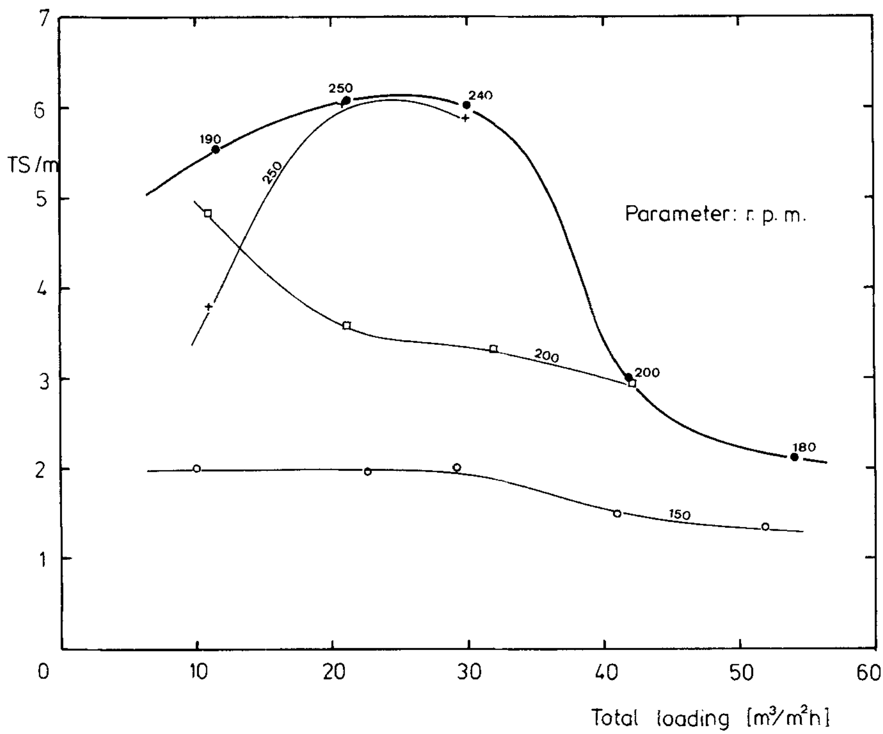

FIGURE 9. Summary of mass transfer measurements on the 80-mm column. The numerical values attending the curves and the individual solid data points represent agitator speeds (rpm).

maximum given by the characteristics of the plates themselves. If the column were correctly designed, the sharp decrease in efficiency between 30 and 40 m^3/m^2h would be eliminated. Backmixing in the continuous phase was about the same as that found in other agitated columns and, as expected, was dependent upon agitator speed and throughput. More results are needed, however, before a reliable correlation can be obtained. In the meantime, a relationship suggested by Bauer (3) for the Kühni column may be used. This relation is given by

$$\frac{E}{u \cdot h} = 0.59 + 0.0249\ (d/h)^{0.33} \cdot (d^2n/Du), \qquad (2)$$

where E is the dispersion coefficient in the continuous phase, u is the actual rate of flow of the continuous phase in the column, h represents the stage height, d is the diameter of the agitator, n represents the stirring-speed of the agitator, and D denotes the diameter of the column. The free area of the plate is not included in this correlation because in this case it has no specific meaning. A comparison with the original equation for a Kühni column with a free area of 40% is given in Fig. 10. It is evident that backmixing is similar in the two cases.

In order to examine the characteristics of the column with other systems, a few experiments (without mass transfer) were conducted with the system methyl isobutyl ketone and water. In contrast to the toluene-water system, this system had a much lower interfacial tension; as a consequence, column performance was improved. Throughputs up to 100 m^3/m^2h were attained and fewer difficulties arose at the lower end of the column. Dispersion was easily achieved and small, regular-sized drops were produced even at lower agitator speeds. It was also observed that, with a load of 60 m^3/m^2h and phase ratios between 1:20 and 20:1, the column operated without difficulty. From this one can conclude that a system with a higher interfacial tension yields conservative values which are exceeded by those of other systems.

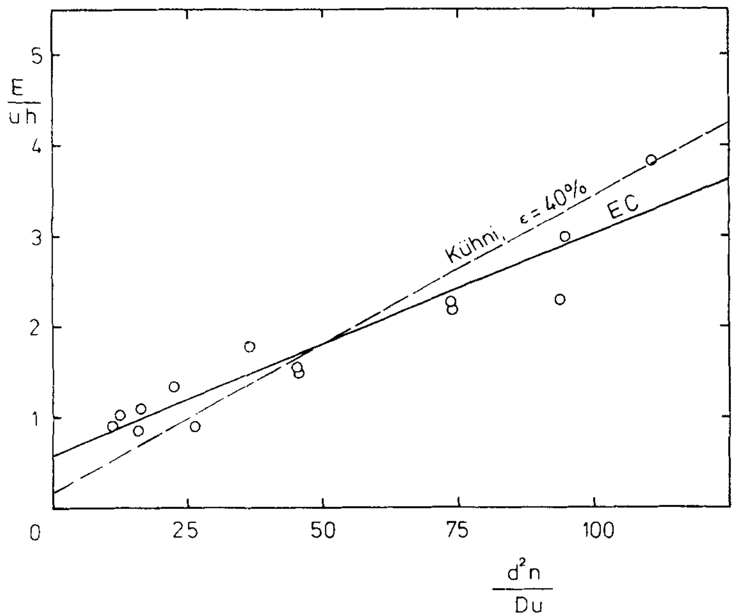

FIGURE 10. Backmixing in the continuous phase compared with that of a Kühni column having plates with 40% free area.

An additional, rather significant observation that was made was that all of the EC plates operated virtually identically, so that the use of additional plates to obtain a uniform dispersion was unnecessary. This result was to be expected, since each agitator is fed by continuous streams of the phase to be dispersed and breaks them down into drops. As a consequence, the operation of all of the agitators is equal, and the column operates more uniformly than other types.

CONCLUSIONS

Preliminary experiments have confirmed that the enhanced coalescence plates provide better separation efficiency, and much higher throughputs, then conventional plates. They effectively separate the mixing regions even though their free area is about

90% and increase the efficiency of the stages through repeated coalescence and redispersion of the dispersed phase. The experiments carried out confirm that the column is more efficient than existing agitated columns. In fact, the efficiency of practically every type of column could be increased by the insertion of wetted surfaces of the type described. In this way the improvement claimed could be clearly demonstrated. It should also be possible to develop a large diameter EC column without mechanical power input. The results obtained so far suggest that such a column should be able to compete with existing non-pulsating perforated plate columns.

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