CHAPTER 6

RESULTS AND DISCUSSIONS

The experimental data are presented in Appendix A.1 to A.9. The results and discussions of iodine transferred from aqueous solution to dispersed phase are as follows :

6.1 Flooding Determinations

The determination of random packing effect results areshown graphically in Fig.17. Stainless steel rings were used to determine this effect at af between 0.3 to 1.5 cm/sec. and amplitude was given at 1 cm. The results indicate that flooding capacities decrease with increasing af as expected and that flooding capacities are not affected by random packing. These results should eliminate the factor of random packing effect for both flooding data and efficiency data for this experimental work and published work. Indeed the inclusion of a random packing effect would have been difficult to realize.

Jackson(3) has indicated that the variation of surface properties influence flooding capacities, such as the different effect of ceramic and polyethylene rings on flooding capacities inFig.1. Jackson made his experimental studies based on the ternary water-benzoic acid-toluene and polyethylene rings and ceramic rings as packing materials. These results were confirmed in this study on the ternary water-iodine-carbon tetrachloride using stainless steel packings and unknown teflon-like plastic material.

Figure 18 shows the variation of flooding capacities with af using stainless steel rings as packings. Each lines represent constant frequencies at 22, 30, 44 and 85.7 pulse per minute. Flooding capacities decrease with increasing amplitude. Flooding capacities affected by af using stainless steel rings as packings with the amplitude as parameter is shown graphically in Fig.19. For straight line representing a constant at 2, 3 and 4 cm., the flooding capacities increase linearly as the frequency increases.

There are several remarks that should be made concerning the flooding behavior of the columns. Of importantsto point out is that unlike the work of Jackson the agitation parameter for the pulsed packed column can not be represented by the combined parameter af for flooding data. In this investigation it was found that the amplitude a and frequency f play influences on flooding behavior which are different. Had this influence been of the same order the four straight lines of Fig.18 would have been a single curve.

In an attempt to explain this different influence it is worthwhile to look at two sets of data points of Fig.18 namely at af=2.86 cm/sec. the set of data which gives a higher flooding rate represents a low amplitude(a=2.0cm) and a high frequency (f=85.7 pulse/min.) whereas the sets of data points giving lower flooding rates represent a high amplitude (a=4.0 cm) and a lower frequency (f=44 pulse per minute). These two sets of points give different flooding rates although the pulsation velocity is the same. Visual observations of the low a high f set of data indicate the higher mean drop diameter. A higher mean drop diameter would imply that the average rate of fall of drops is higher which would tend to increase the flooding limit. A small drop diameter implies a low passage velocity and more chance of not passing through the equipment. Another point left as yet untried is the hold-up of dispersed phase at these two sets of values. Higher or lower passage velocities are related to hold-up, it is believed that for low passage velocities of dispersed phase, the hold-up is greater than the other case and this increases continuous phase flow rate which in turns untrains dispersed phase in its direction until dispersed phase is not longer capable of passing through the column and flooding occurs.

Figure 19, a peculiar observation may be made, which represents constant amplitude increases with frequency of pulsation indicating higher flooding limits at higher frequency. Putting this observation another way, flooding limits increase with increased agitation (an increased resistance) and creates a paradox. The only answer to this phenomena is that the separate influencesof a and f on flooding are very much different and the optimal set up for maximizing capacities of columns is low amplitude and high frequencies. This generalization being made notably for the system and operating ranges on hand.

For flooding characteristics determination of plastic rings as packings, Fig.20 and 21 show the graphical results as regression lines. The same trend of results are as Fig.18 and 19 for stainless steel rings as packings, however, quantitative differences were observed.

The flooding characteristics comparisons between stainless steel and plastic rings are also shown in Fig.22 and 23. It can be concluded that at various constant values of a and f, plastic rings show better performance than stainless steel rings on flooding capacities. A likely explanation is that plastic rings are preferentially wetted by the organic phase (carbon tetrachloride) and adhesive property changed the droplets into extended films on the plastic surface and this arrangement allowed the continuous phase to meet with less flow resistance from the dispersed phase as it would otherwise have met with a stainless steel packing. Consequently, the tendency to drag the carbon tetrachloride dispersed phase droplets in the direction of the continuous aqueous phase is thus greater using stainless steel packings.

At various constants of a, plastic rings also showed better performance on flooding capacities than stainless steel rings throughout the range of af used.

Chantry and coworkers(4) have indicated that the use of higher frequency and lower amplitude would result in lower HTU and less consumption of power. They also indicated that higher frequency tended to create finer dispersions and lower amplitude tended to create lower axial mixing. However, they have not indicated the resluts in flooding capacities, but from this work it may be indicated that besides the Chantry indication, the higher flooding capacities were obtained as well.

The result of Jackson's work on flooding capacity influenced by different packings is shown in Fig.1. Jackson has indicated that the regression lines were obtained, flooding capacities decreased with increasing af. It may be noted that a single line was obtained at each of packings. It may be possibly described that Jackson may use the single value of frequency and vary the amplitudes made af increase, so the results for the other value of f were not obtained. However, the authors did not give the detailed experimental data in their works.

5.2 Efficiency Determinations

The column efficiency was determined from the concentration difference between phase in and out by using the following equation, $(Y_{in} - Y_{out})/Y_{in}$.

Figure 24 shows the variation of efficiency affected by af and extraction factors over the range of af 0.3 to 6.0 cm/sec. and extraction factors 14.22, 7.11

and 3.56 for plastic rings as packings. The continuous phase flow rate was given at 12.6 l/h. or superficial velocity 0.12 cm/sec. At each extraction factors, the efficiency increases with af. It may be noted that efficiency increases rapidly for low af and reaches assymtotic values for higher af. It is observed that it is unnecessary to pulse beyond af 6.0 cm/sec. as the assymtotic values has nearly been reached. For pulsed columns maximum pulsation is about 8.0 cm/sec. The optimal results for plastic rings was at an af of 4.0 cm/sec. at any extraction factor, for higher af than this optimal value, the efficiency obtained was increased by not more than 1-2%. For the variation of three extraction factor values, higher extraction gave higher efficiency due to the higher dispersed phase flow rate. Low energy consumption was obtained with higher extraction factors for higher efficiencies, but the final selection of the extraction factors rests on economic considerations of solvent storage costs and solvent recovery costs.

One extremely important result obtained from efficiency determination is that the pulsing parameter af correlated well with extraction efficiency. This result is in sharp contrast with flooding result behavior. This does not necessary mean that the two results are contradictory as efficiency is measured at 50-60% of flooding.

conditions and the two measurements are radically different in nature.

Figure 25 shows the variation of efficiency with af and extraction factors over the range of af 0.3 to 6.0 cm/sec. and extraction factors 14.22, 7.11 and 3.56 for stainless steel packings. The continuous aqueous phase flow rate was set at 12.6 liter/hr. or superficial velocity 0.12 cm/sec. For extraction factors 14.22 and 7.11, the curves obtained tend to increase in efficiency with increasing af, for another extraction factor (3.56) a maximum efficiency was obtained at af 4.0 cm/sec. and the maximum efficiency was 43%. The maximum points were not obtained by the extraction factors 14.22 and 7.11, but the curves increase rapidly for low af until an asymtotic value was reached. The optimal efficiency for extraction factor 14.22 and 7.11 were 78%and 66.5% respectively. For stainless steel, higher efficiencies were obtained by increasing the extraction factor as expected.

The graph for extraction factor 3.56 in Fig.25 is especially interesting as it indicates a loss of efficiency for af greater than 4.0 cm/sec. This same phenomenon has been observed for pulsed perforated plate columns and is explained by the following reasoning. Mass transfer efficiency is governed by the equation $N = K.a.\Delta C$. where N is mass transfer rate, K is mass transfer coefficient , a is area of mass transfer and ΔC is defined as concentration difference of solute between continuous phase and dispersed phase. If K is assumed to remain constant with pulsation, it is noted that a the interfacial surface area increases with pulsation which should result in increased extraction efficiency. However it is also known that increased pulsation increases backmixing and the overall effect of backmixing is to significantly decrease the concentration potential Δ C. For af greater than 4.0 cm/sec. the decrease of mass transfer rate dueto backmixing play an greater influence than the increase of mass transfer rate due to increased interfacial area. It should be noted that for pulsed perforated plate columns the optimal pulsation would occur at the higher pulsation than pulsed packed columns.

Figure 26, 27 and 28 show the efficiency comparisons for both stainless steel and plastic rings as packings. The efficiency comparisons at extraction factors 14.22, are shown in Fig.26, plastic rings gave higher efficiency than stainless steel by about 15% at any af. It may be possible to offer the following explanations. In the case of plastic rings, higher hold-up of dispersed phase were obtained because plastic rings were preferentially wet by solvent phase which made carbon tetrachloride dispersed phase adhesive on the plastic surfaces as extended film which resulted in increased interfacial area. Another explanation is perhaps a higher residence time of drops in the equipment which translates into a higher dispersed phase hold-up. Hold-up studies on polyethylene pulsed perforated plate columns(33) indicate that wetting characteristics of material inside the column change hold-up values. For the case of plastic material and an organic dispersed phase, the hold-up is reported higher than for the case of stainless steel packings.

The efficiency comparisons at extraction factors 7.11 and 3.56 are shown in Fig.27 and 28, plastic rings also gave higher efficiency than stainless steel by about 10-15%.

Sobotik and Himmelblau(28) studied the effect of plate wetting characteristics and direction of mass transfer in a pulsed sieve-plate column. The use of stainless steel or polyethylene plates gave about the same rates when the acetic acid (as solute) was transferred from the water dispersed phase to the continuous ketone phase. However, substantially higher rates were observed for acid transferred from the continuous ketone phase to water for polyethylene plates. An alternative plates arrangement gave results intermediate between the performance of all polyethylene or all stainless steel plate columns. In the same way, the results for iodine transferred from continuous water phase to carbon tetrachloride dispersed phase were obtained by giving higher performance for plastic rings. Thus it may be possibly concluded that a similar phenomena should be obtainable when using plastic or stainless steel rings when iodine is transferred from the carbon tetrachloride dispersed phase to the continuous aqueous phase.

Jackson et al.(3) has indicated that for the direction of solute transfer from the continuous phase to the dispersed phase the polyethylene packings gave heights of transfer unit which were about 25% lower than those for the sieve-plate column, with ceramic packingsthe least effective. It is significant to note that pulsing energy input with polyethylene packing was one-fourth that of the sieve-plate column according to Jackson. The transfer rates were also somewhat better for plastic rings. Then it may be concluded that for this work the polyethylene packing should obtain the better performance than that of stainless steel plates. So the polyethylene rings gave better performance than both stainless steel packings and stainless plates. As a final remark it may be said that wetting characteristics of columns play an important role in influencing both the flooding characteristics and efficiency of column. It has been shown in this investigation that for solute transfer from a continuous aqueous phase to a dispersed organic phase an organic wetting material would be preferable. For the case of back extraction wetting characteristics do not influence the extraction efficiency according to Sobotik and Himmelblau.(28)

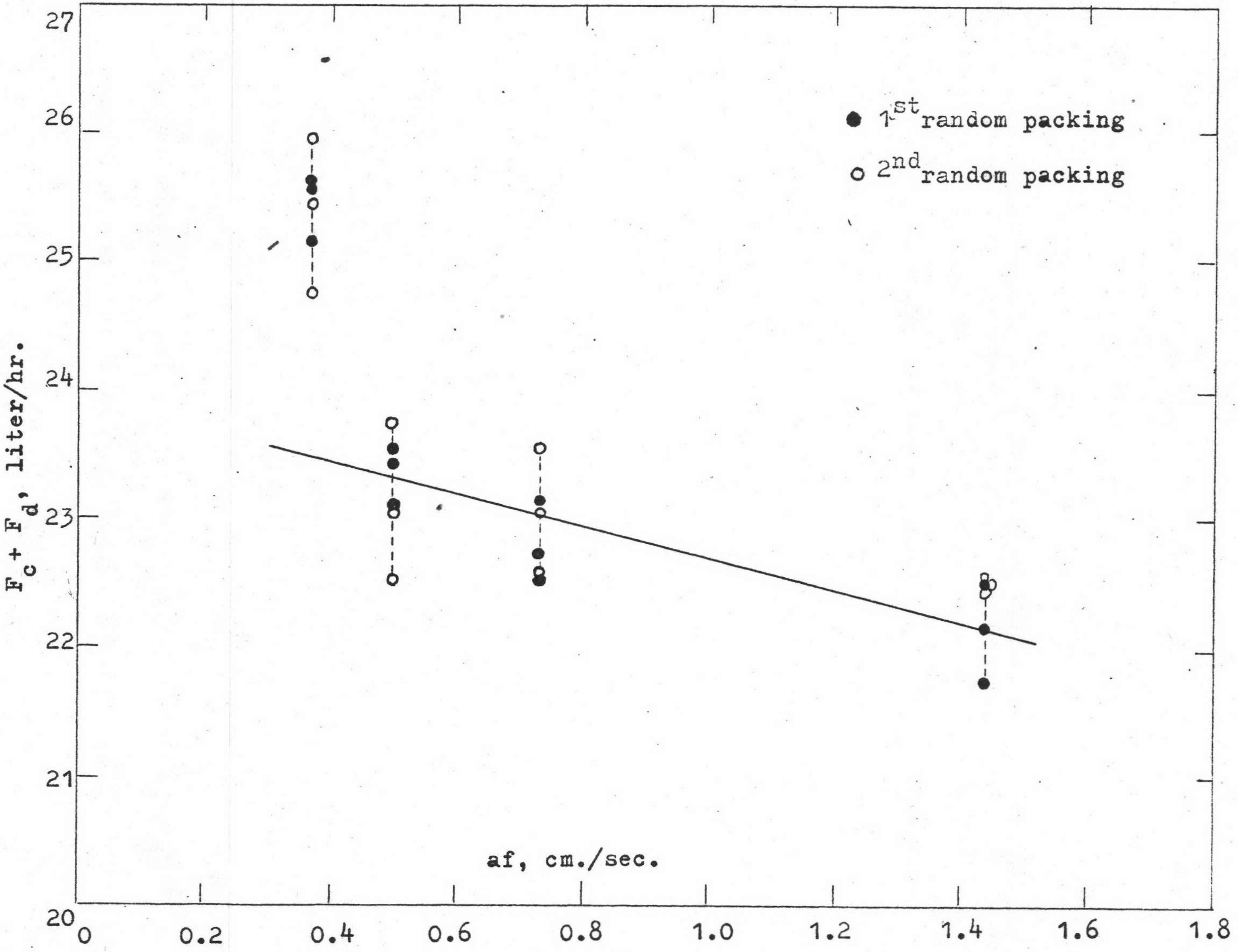

Figure 17 Flooding characteristics of pulsed packed column with 1/2-in. stainless steel rings at a=1 cm.

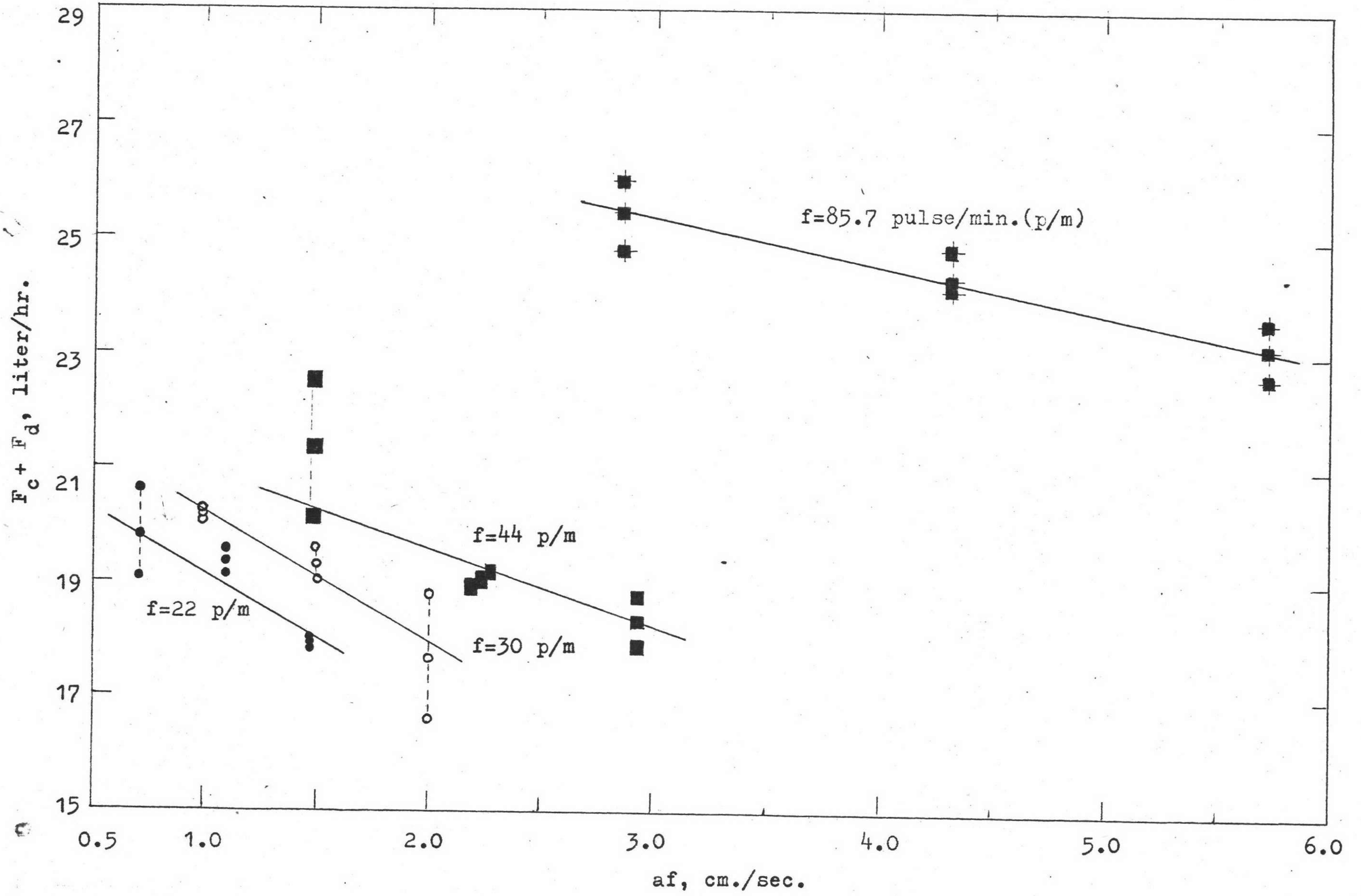

Figure 18 Flooding characteristics of pulsed packed column with 1/2-in.stainless steel rings at various frequencies.

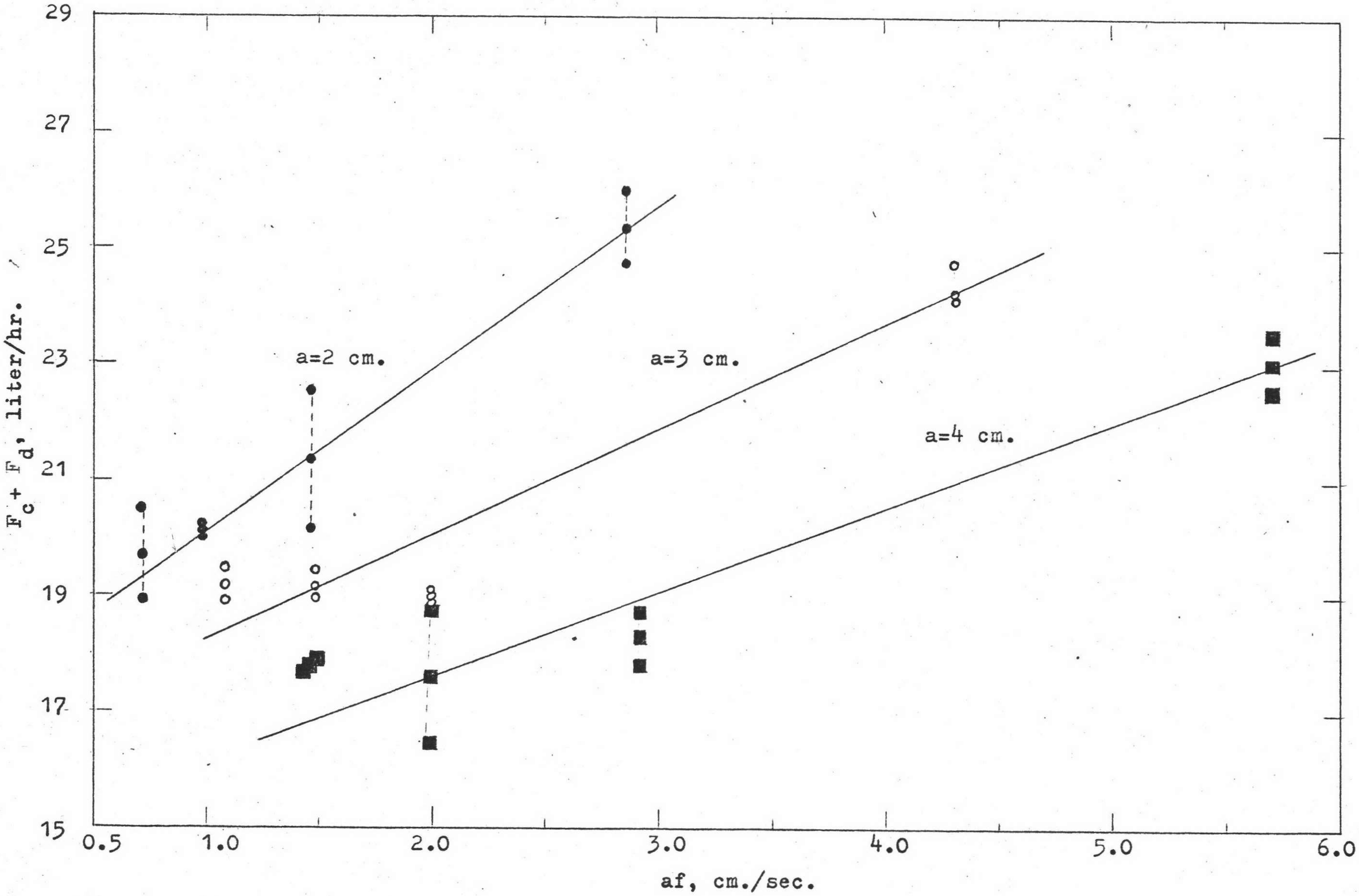

Figure 19 Flooding characteristics of pulsed packed column with 1/2-in. stainless steel rings at various amplitudes.

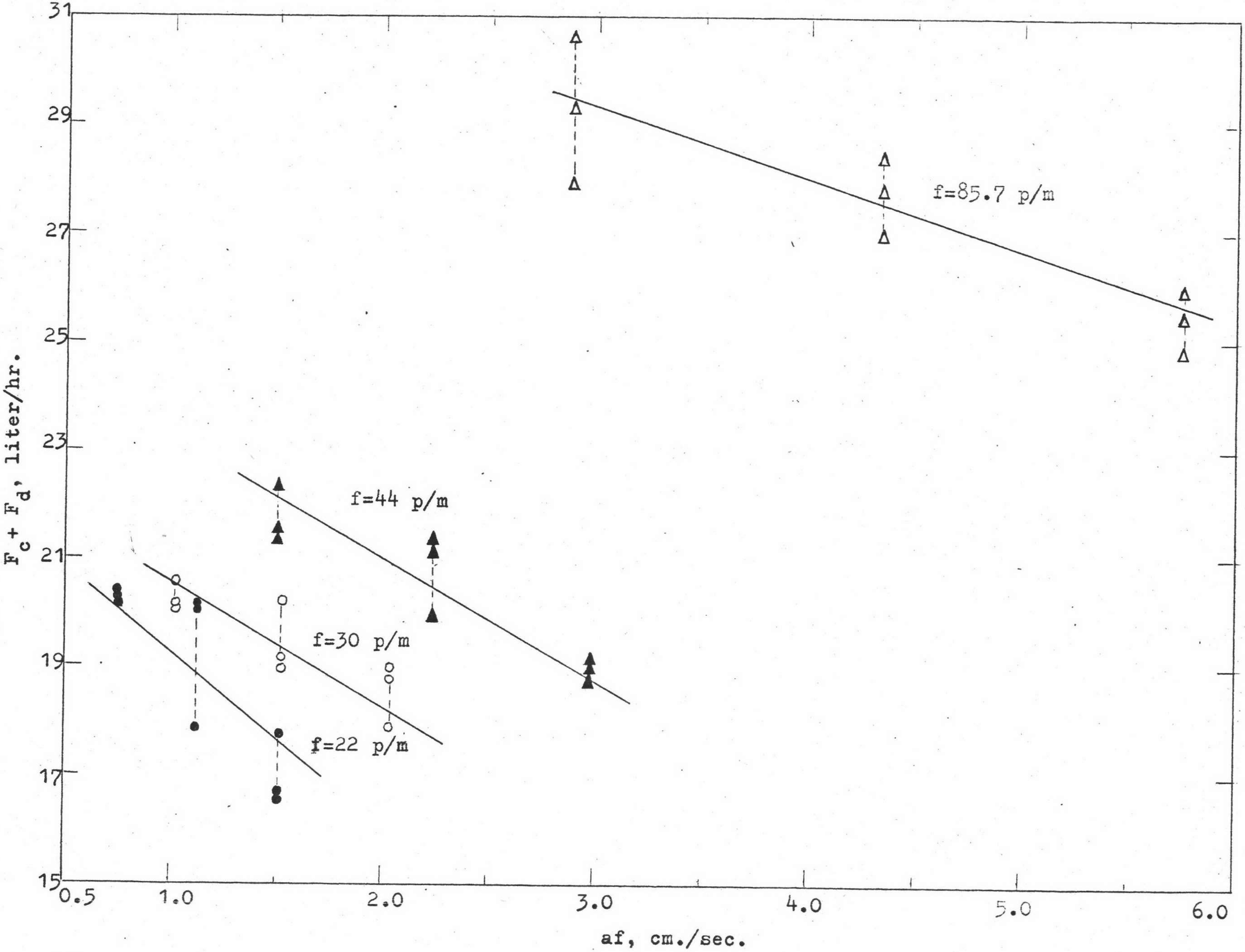

Figure 20 Flooding characteristics of pulsed packed column with 1/2-in.plastic rings at various a

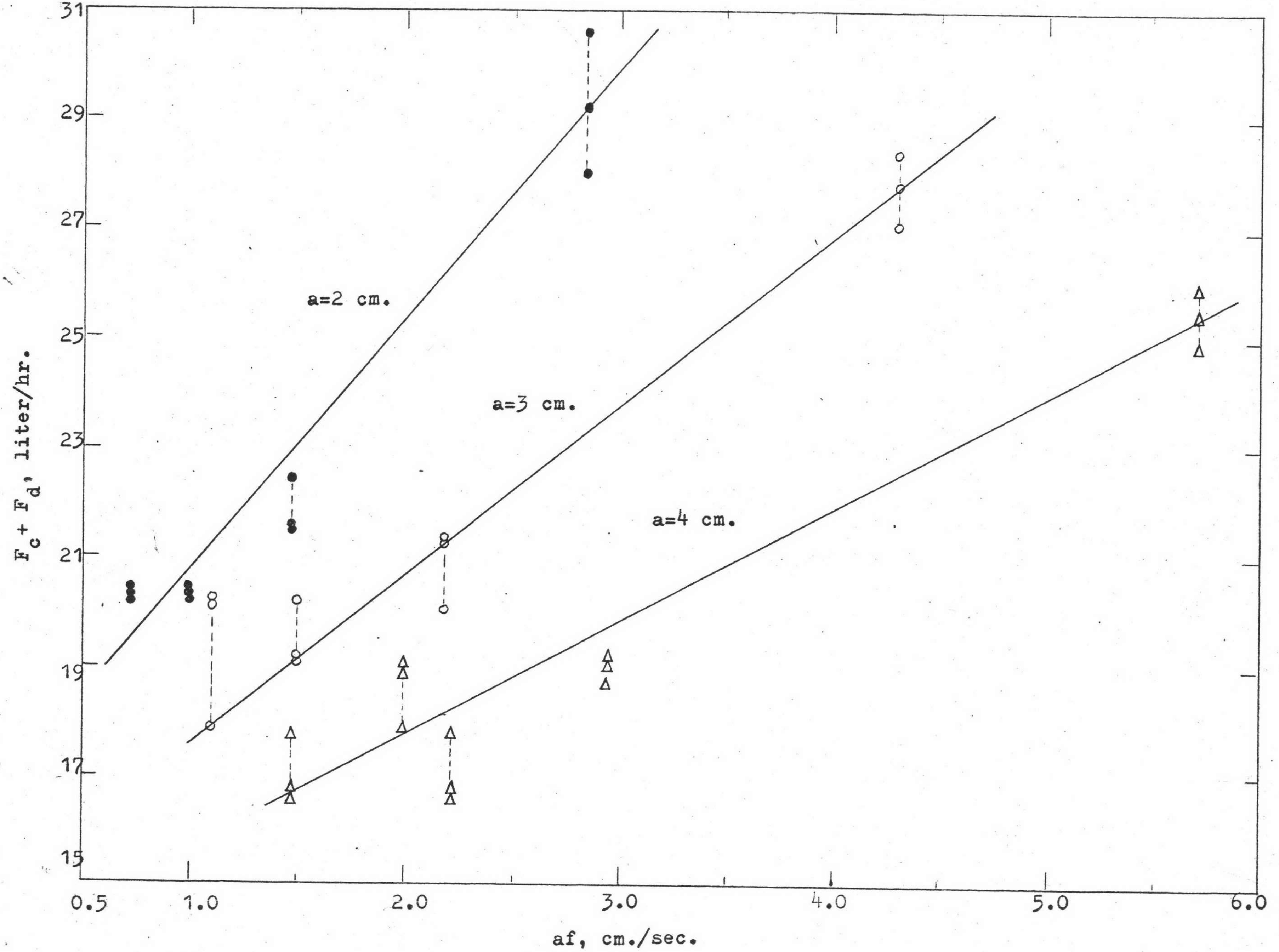

Figure 21 Flooding characteristics of pulsed packed column with 1/2-in.plastic rings at various f

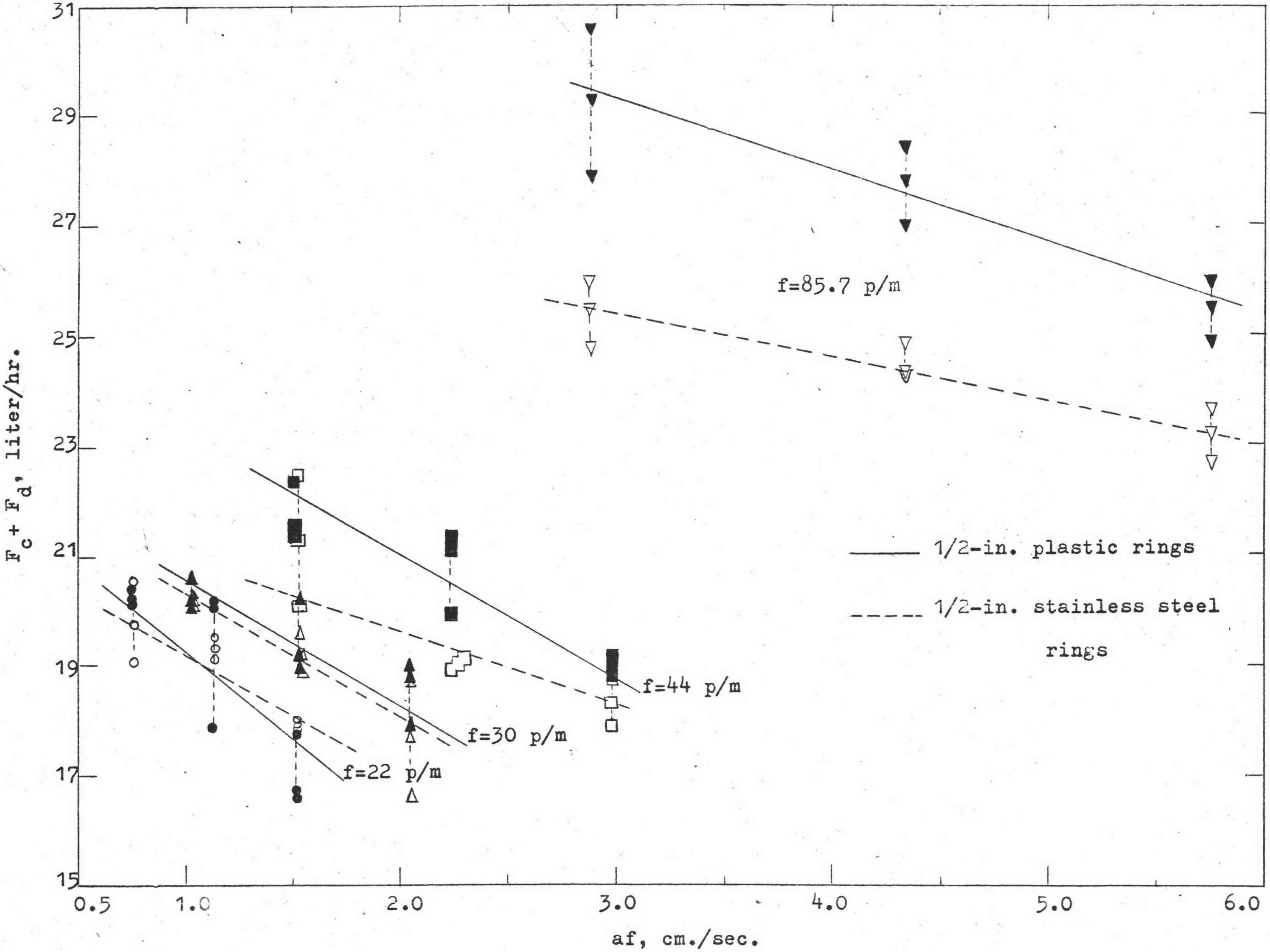

Figure 22 Flooding characteristics comparison between plastic and stainless steel rings at various f

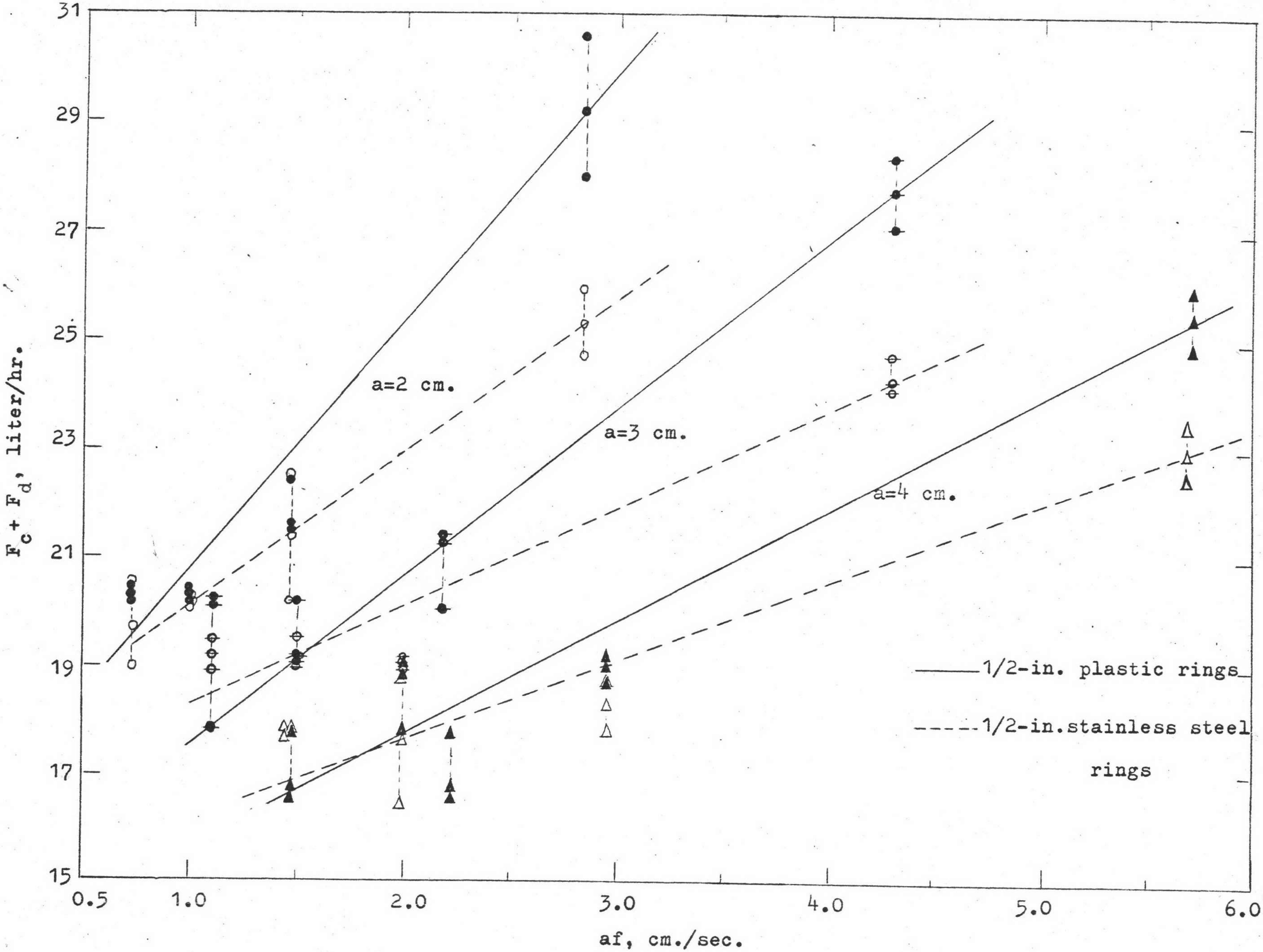

Figure 23 Flooding characteristics comparison between plastic and stainless steel rings at various a

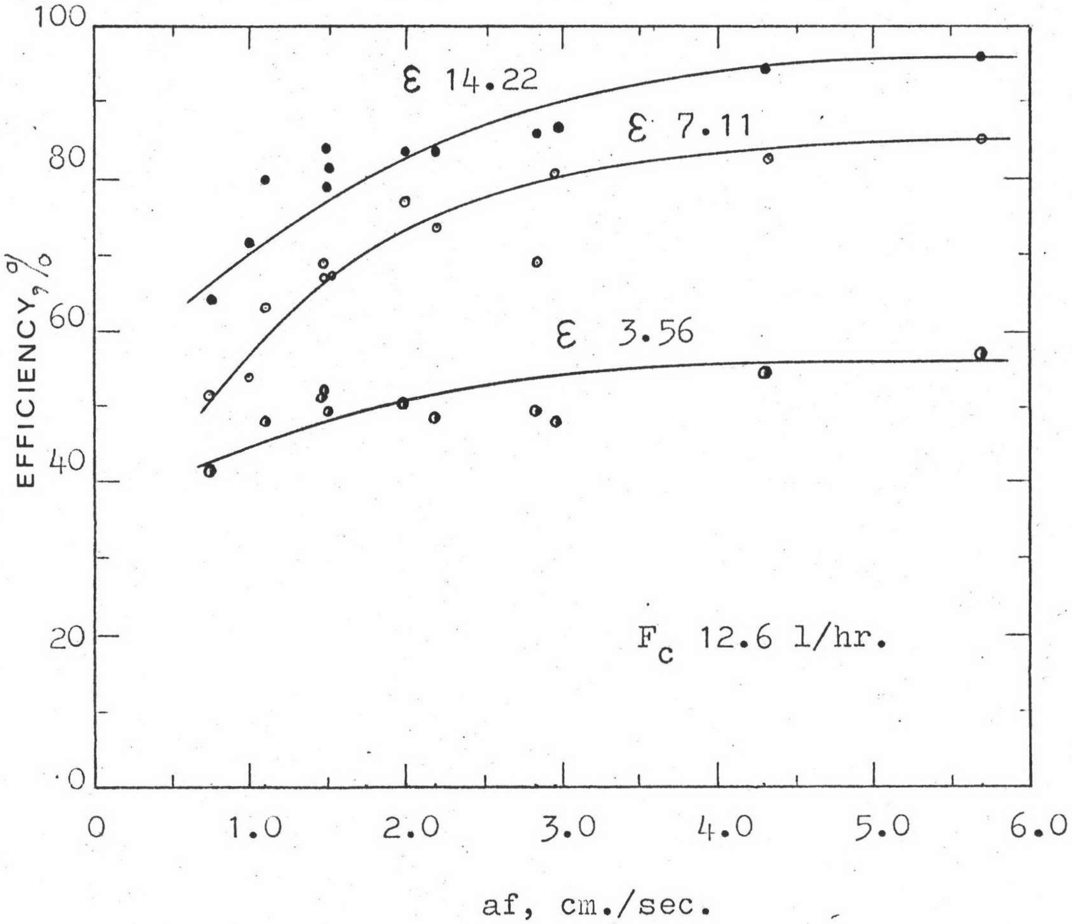

Figure 24 Efficiency determination of pulsed packed column with 1/2-in. Plastic rings.

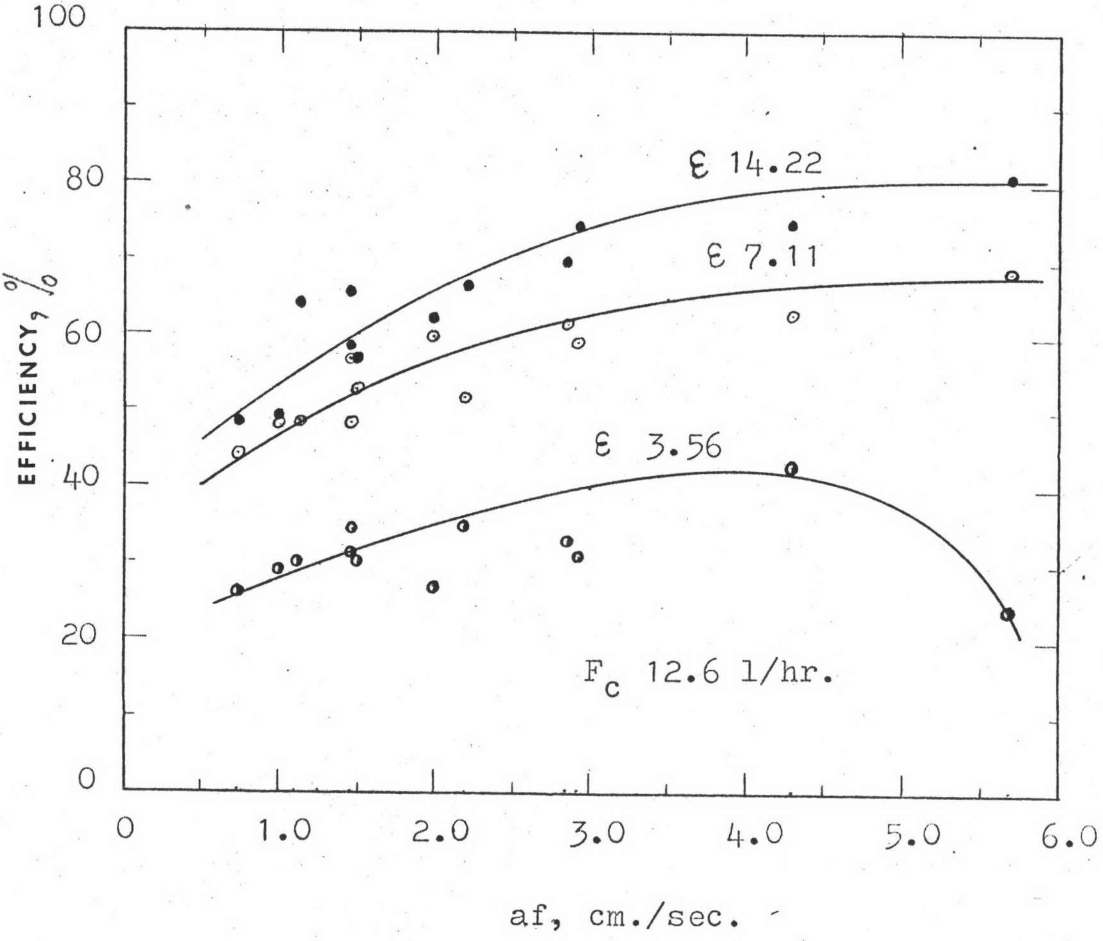

Figure 25 Efficiency determination of pulsed packed column with 1/2-in. Stainless steel rings.

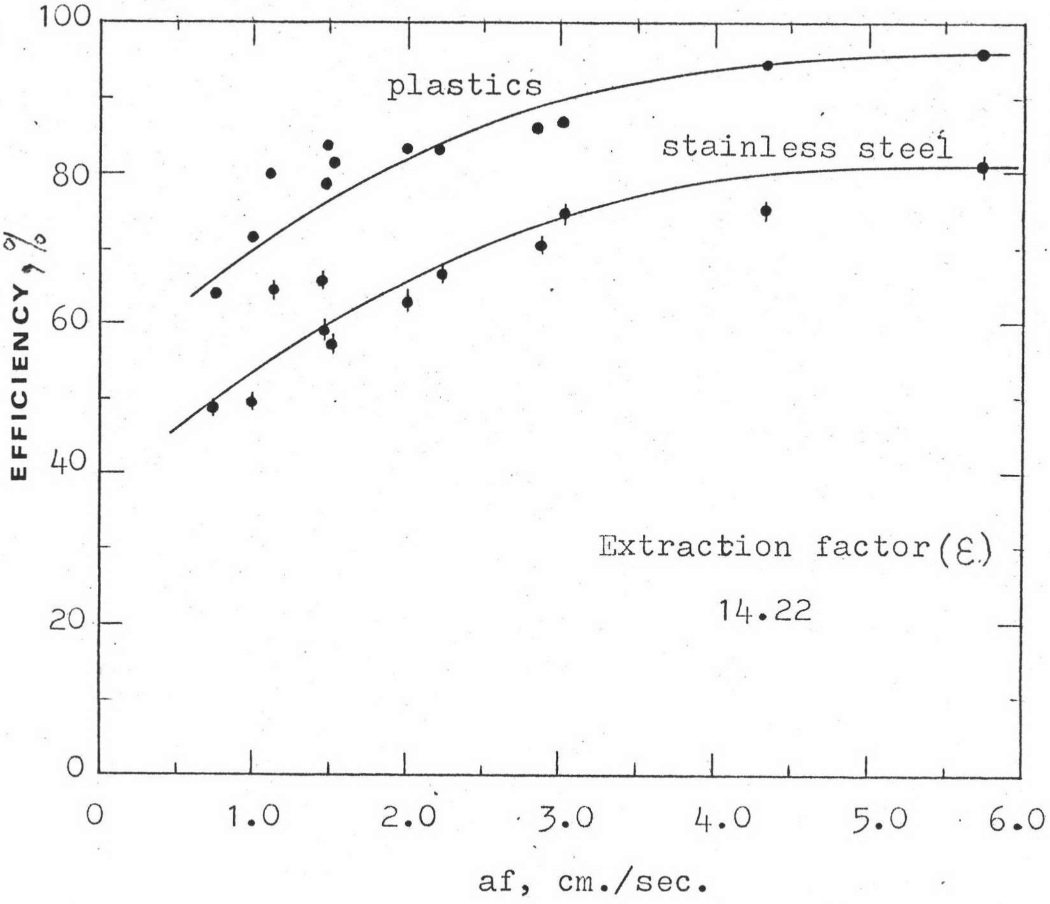

Figure 26 Efficiency comparison of pulsed packed column with 1/2-in. plastic and 1/2-in. stainless steel rings.

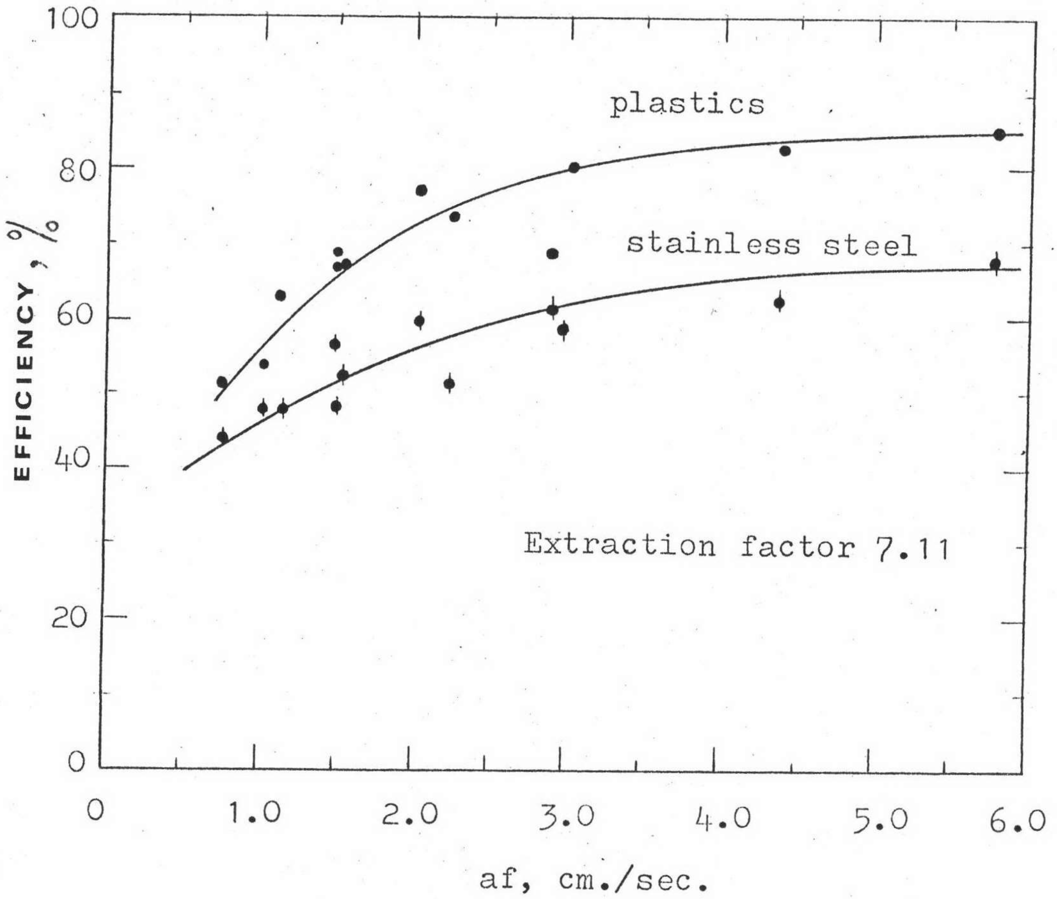

Figure 27 Efficiency comparison of pulsed packed column with 1/2-in. stainless steel and 1/2-in. plastic rings.

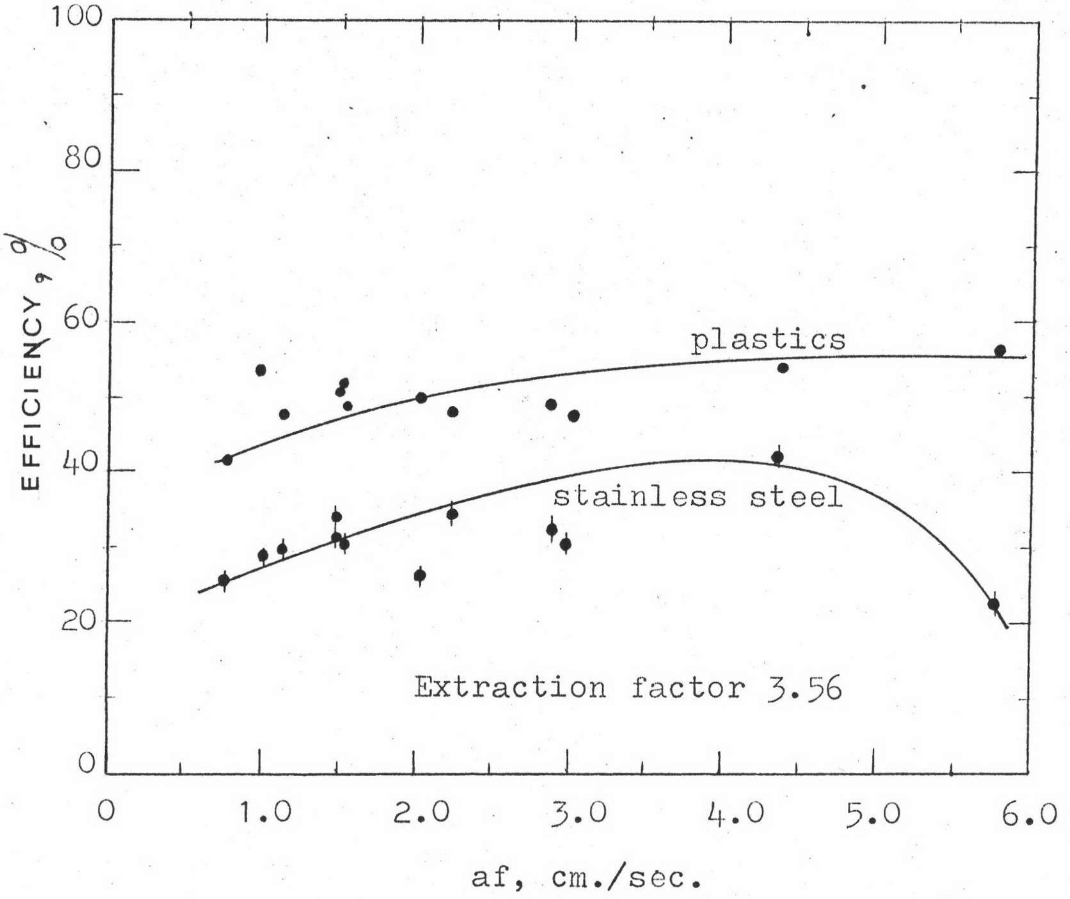

Figure 28 Efficiency comparison of pulsed packed column with 1/2-in. stainless steel and 1/2-in. plastic rimgs.