## ALIEN PROPERTY CUSTODIAN

## EXTRACTING ALIPHATIC ACIDS

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No Drawing. Application filed January 27, 1941

The present invention relates to improvements in the extraction of aliphatic acids, particularly to the extraction of acetic acid from dilute aqueous solutions thereof.

The present application is a continuation-inpart of my application Serial No. 275,513, filed May 24, 1939.

It has already been known that aqueous solutions of aliphatic acids may be extracted countercurrently with organic liquids, such as ethyl 10 acetate, in columns provided with filling material such as Raschig rings.

In my application Serial No. 173,949, now Patent No. 2,211,087, I have already described an improved process for increasing the extraction yield 15 in such a process wherein the relative linear velocities of the dilute aqueous solution of the acid and of the extraction agent were generally maintained greater than .2 cm. per second with reference to the total cross-section of the extraction 20 column. Therefore, in order to obtain the maximum extraction effect it was necessary to select an extraction column which was suitable for the desired throughput. If, however, only one extraction column was available and it was desired 25 to lower the throughput, the amount of acids in the extracted aqueous wastes increased greatly and the efficiency of the apparatus considerably decreased. On the other hand, if the desired throughput was so great that the relative velocities of the liquids in the extraction column exceeded the upper limit the extraction effect was also lowered.

This is a great disadvantage as it is highly impractical for a plant to have different extraction 35 columns available to take care of the variations in the amount of the dilute acids which are to be extracted at different times.

If the same extraction column were employed and the desired throughput below that described above, it is necessary to increase the amount of extraction agent employed to an uneconomical degree. In other words, to counteract the lessened extraction effect it is necessary to increase the proportion of the extraction agent employed considerably. In accordance with my invention it is possible, however, to obtain excellent yields of extraction for different throughputs even when employing the same apparatus.

I have discovered that the extraction effect 50 which is obtained does not merely depend upon the diameter of the extraction column employed, but also upon the height at which the level between the two liquid phases, that is the level between the aqueous layer and the layer of extrac- 55 tain throughput is desired the level between the

tion liquid, is maintained within the column and that if a maximum extraction effect is to be obtained in an extraction column the height of this level must be adjusted for the throughput desired. Therefore, when the throughput of the liquids through the extraction column is altered the level between the liquid phases in the column must also be altered until maximum extraction effects are obtained for the desired throughput. The level between the liquid phases is easily altered by temporarily altering the amount of extraction agent or aqueous acid introduced into the extraction column. For example, if the column of the extraction agent is to be increased, it is merely necessary either to increase the flow of the extraction agent into the extraction column temporarily or to decrease the flow of the aqueous acid into the column temporarily. On the other hand, if the level between the phases is to be altered in the other direction, it is merely necessary to increase the flow of the aqueous acid or decrease the flow of the extraction agent temporarily. After the most favorable height for the level between the liquid phases is ascertained, this is then maintained for the entire extraction. The level between the liquid phases will remain constant as long as the amount of liquids introduced and withdrawn from the extraction column are constantly maintained at the proper ratio for the extraction.

Previously the level between the phases within the extraction column has been maintained at a certain height without regard to the desired throughput of the liquids through the apparatus. For example, it has been usual to extract dilute acetic acid with ethyl acetate in such a manner that the aqueous layer was restricted only to the lower portion of the extraction column, namely, the portion which was not provided with filling material. In accordance with the present invention, however, it has been found that the lower the desired throughput the higher should be the level between the aqueous acid phase and the ester phase and such level may be within the 45 portion of the column containing the filling material and even near the top of the extraction

It is not necessary to ascertain the proper level between the liquid phases in the extraction column each time the desired throughput is altered. It is possible, for example, to ascertain the proper level for a series of different throughputs through the apparatus employed and prepare a chart therefrom and then whenever a cerliquid phases may be adjusted to the proper level with reference to such chart.

The extraction column employed in accordance with the present invention is preferably provided with means indicating the level between the two liquids with the column, so that the adjustment of the level between the liquid phases to the proper height is facilitated.

The following example serves to illustrate the present invention:

## Example

An extraction column was employed which was 10 meters high and possessed a diameter of 1040 mm. The column was provided with cylindrical filling bodies having a maximum diameter of 35 mm. Into this column 4000 liters of 17% aqueous acetic acid and 1200 liters of ethyl acetate were fed countercurrently in such a manner that the ratio between the heights of the layer of aqueous acid and the layer of ester was 1:9. The aqueous layer was beneath the zone of the extraction column which contained the filling bodies. The aqueous wastes withdrawn from the column contained only 0.06%-0.08% of acetic acid, which means that the extraction yield amounted to 99.3%. By changing the throughput to 2000 liters of dilute acetic acid and 6000

liters of ethyl acetate in the same apparatus while maintaining the conditions of the extraction the same otherwise, the amount of acetic acid in the aqueous wastes withdrawn from the extraction column increased to over 1%. The extraction yield was therefore decreased to about 94%. By increasing the height of the line of division between the liquids to such a degree that the ratio between the height of the layer of 10 aqueous acid and the height of the layer of ethyl acetate was 3:1 so that the line of dlvlsion was within the zone of the extracting column containing filling bodies, the amount of acetic acid contained in the wastes withdrawn 15 from the column was lowered to below 0.1% and the extraction yield was increased to 99.0%-99.5%. The most advantageous ratio between the two layers of liquids may, in every case, be easily ascertained for a given throughput in an 20 extraction column by determining the amount of acid in the aqueous wastes. The desired ratio is substantially maintained after once adjusted If the throughput of the respective liquids is not substantially altered.

The above-described process is also applicable in the extraction of other aliphatic acids with known organic solvents.

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