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EXTRACTION PROCESS

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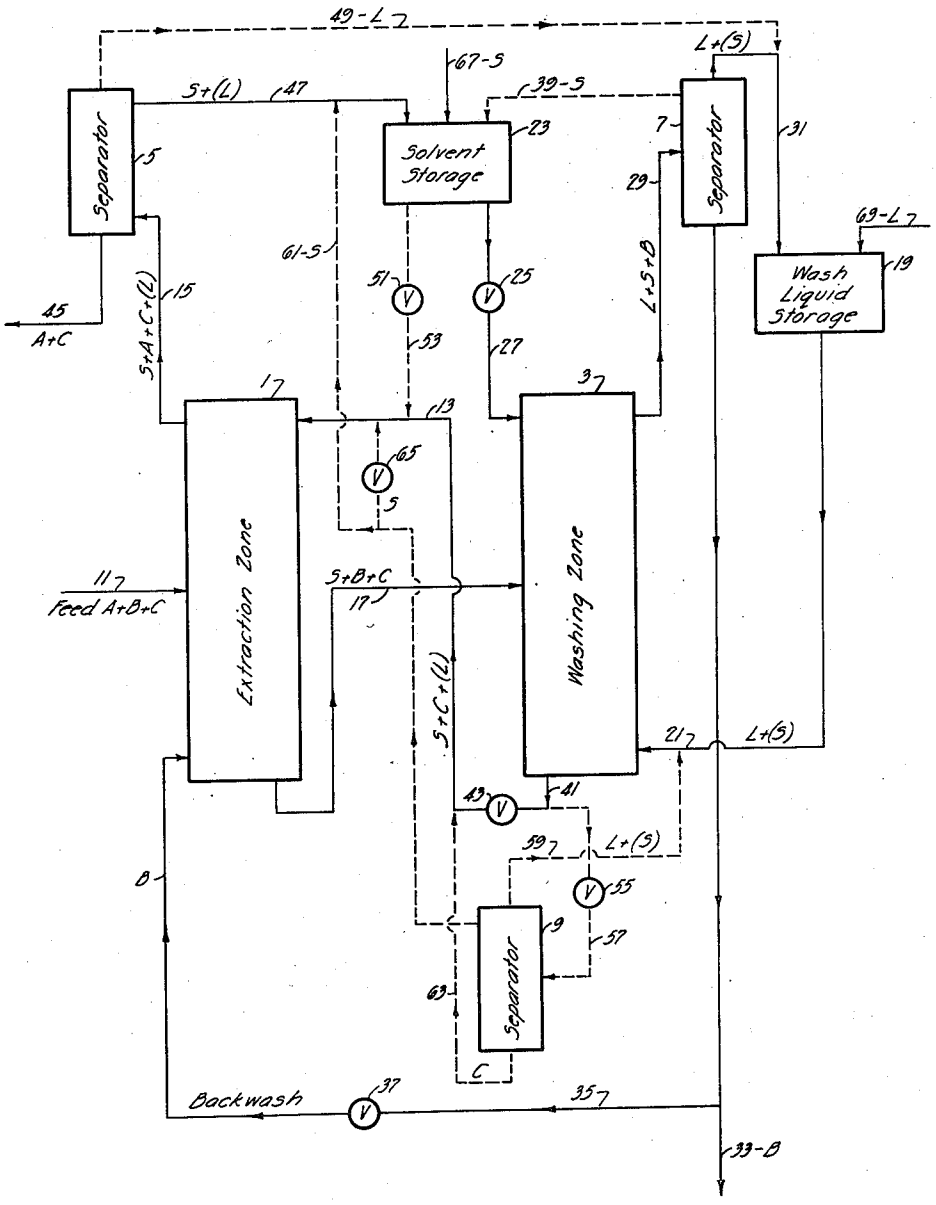

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EXTRACTION PROCESS

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This invention relates to an improved process for solvent extracting liquid mixtures, and more particularly comprises an improvement whereby a component of intermediate solubility is removed from a three component mixture.

It is frequently desired to remove the intermediate component from a liquid mixture of three components which are progressively more soluble in selective solvents, and to retain the least soluble and most soluble components. In applying the known methods of solvent extraction to such a purpose, it has heretofore been necessary to remove the two soluble components, further extract the mixture of these two components to separate them, and then recombine the desired components. This necessitates the use of two separation steps and is uneconomical.

It is an object of my invention to provide a process for withdrawing the intermediate component from a three component liquid mixture in an economical manner. It is a further object of my invention to provide an improved process of the type described in which only the component of intermediate solubility is removed from the feed mixture in the main extraction column in substantial amounts and in which such lesser amounts of the extremely soluble component as are withdrawn from the extraction zone are returned to it, thereby obviating the final mixing of the least soluble component after its withdrawal from the extraction zone with the most soluble component. It is a still further object of my invention to provide a process of the type described in which the desired components are combined prior to the step of separating them from the solvent, thereby making possible the elimination of one separation step.

With the above and other objects in view my invention consists of the combination of steps more fully described and illustrated in the following specification and drawing, and specifically pointed out in the appended claims, it being understood that numerous changes within the scope of the claims will be apparent and may be resorted to without departing from the spirit of my invention or sacrificing any of its advantages.

Referring to the drawing, which shows a flow sheet of one embodiment of my invention, 1 is an extraction apparatus, and 3 a washing apparatus. These may be single towers, provided with contact means or multistage treaters comprising mixers and centrifugal separating means for effecting the countercurrent contacting of two liquids or any other devices for the countercurrent treatment of the liquids. 5, 7 and 9 are three separating apparatus, such as distillation or fractionating columns, interconnected with pipes, as shown on the drawing and hereinafter described, and equipped with necessary valves, pumps, heaters, heat exchangers and adjuncts not shown.

In accordance with one mode of operation, a liquid mixture containing three components, A, B and C, respectively, of which A is the least soluble and C the most soluble in a selective solvent S, is introduced into apparatus 1 through pipe 11 and extracted with a solvent medium containing the solvent S introduced at 13. Under extraction conditions prevailing within the apparatus two phases are formed, one of which, the raffinate phase, is withdrawn through a pipe 15, and the other, the extract phase, through a pipe 17. The latter is hereinafter referred to as the first extract phase, and contains S, B and C in substantial amounts, and is conducted to apparatus 3, where it is washed with a solvent L supplied from a storage tank 19 through a conduit 21. Solvent L is selected to have physical and/or chemical properties which render it at least partially immiscible with the solvent S, in the presence of components B and C, permit it to dissolve the component B, and permit its ready separation from S, A, B and C by suitable means, such as fractionation, absorption, precipitation, etc.

The solvent L flows countercurrently to the first extract phase and under extraction conditions prevailing within the apparatus a second raffinate phase, consisting primarily of S and C (and a small amount of L), and a second extract phase are formed. In this operation solvent L washes out substantial amounts of component B, because B has a lower affinity for S than the component C. Solvent L will, however, at the same time dissolve a certain smaller amount of component C. According to the preferred mode of operation I prevent the retention of appreciable quantities of C by the solvent L by employing a double countercurrent operation in apparatus 3. In accordance with this operation fresh solvent S is introduced from storage tank 23 through valve 25 and inlet 27, so as to flow countercurrently to the rising extract phase. The scrubbed second extract phase which now consists substantially only of $L+S+B$ is withdrawn at 29 and conducted to the separator 7, which may be a simple distillation device which discharges L and S through conduit 31, and component B through conduit 33. A portion of the extract B

may be returned to extraction zone 1 through conduit 35 and used as a backwash, by adjusting valve 37. It will often be more efficient to exclude S from storage 19 and conduit 21. This may be accomplished by operating separator 7 as a fractionator, withdrawing substantially only L through conduit 31, and withdrawing S through conduit 39 to storage 23.

The second raffinate phase is withdrawn at 41, and flows through valve 43 to inlet 13 of apparatus 1, where it is used as the solvent medium, near the raffinate discharge point 15. This solvent medium is rich in the component C and contacts the phase rich in A produced in apparatus 1, and removes from it substantial amounts of the component B. Being already saturated with respect to C, this solvent medium will permit the component C to remain in the raffinate phase which is withdrawn at 15, and is for convenience hereinafter referred to as the first raffinate phase. This phase will also contain small amounts of S and of L (unless L is excluded from the solvent medium, as described below) which may be separated from the components $A+C$ in the separator 5. This separator can be a distillation apparatus, which discharges $A+C$ through outlet 45, and $S+L$ through conduit 47 to solvent storage tank 23.

Since the first raffinate contains but a small amount of L, and since it would be returned to the apparatus 3 with the solvent through conduit 27, this operation is quite feasible. It may, however, often be desirable to maintain the solvent in tank 23 in a substantially pure state. In this event the separator 5 may be a fractionator, and operated to discharge S through conduit 47 and L through conduit 49.

It is not always necessary that apparatus 3 be a double countercurrent apparatus. Thus, if the component C is not very valuable, or if its nature is such that it will not be substantially soluble in L, the valve 25 may be closed and/or a single countercurrent apparatus substituted for double countercurrent apparatus 3. In this case valve 51 will be opened and the fresh solvent introduced through conduit 53 to inlet 13, together with the second raffinate phase from conduit 41.

As a further modification, I may treat all or a portion of the second raffinate phase which flows through conduit 41 in separator 9 to remove L and/or S. According to the mode of operation, valve 55 is opened and second raffinate phase admitted to separator 9 through conduit 57. The valve 43 may be closed or adjusted to permit a portion of the raffinate phase to by-pass the separator 9. If the separator is operated as a simple distillation device, L and S are conducted through conduit 59 to conduit 21. If 9 is operated as a fractionator, L will be discharged through conduit 59 and S through conduit 61, which returns the latter to the storage 23. In either event component C is reintroduced to apparatus 1 through conduit 63 and inlet 13.

It should be noted that it is necessary to supply sufficient solvent S to the apparatus 1 to remove sufficient amounts of component B. If the separator 9 is operated to remove excessive amounts of S from the second raffinate it is necessary to satisfy this deficiency by partially opening valve 51 and/or valve 65, and/or operating the separator 9 to permit substantial amounts of S to be discharged through outlet 63.

Fresh solvents S and L may be introduced through conduits 67 and 69 to replenish the system.

Numerous modifications may be made in my process without departing from the spirit and scope of my invention. Although I have shown a situation in which the first raffinate phase is lighter than the first extract phase, and in which the second extract phase is lighter than the second raffinate phase, it is obvious that my process is applicable to situations in which these conditions do not obtain. The apparatus 1 and/or 3 may be provided with temperature control means for providing a temperature gradient, as is well known in the art, and may be provided with such other auxiliary aids as may be desirable. The apparatus 1 and/or 3 could further be operated according to a batch process. Although the extraction would not be as complete, the removal of the middle component would, nevertheless, be realized.

My invention may be employed to treat any three component mixture. An example is the treatment of a hydrocarbon mixture which contains paraffinic hydrocarbons, secondary olefines, and tertiary olefines, the secondary olefines having an intermediate solubility. To remove these secondary olefines a polar solvent may be used as the main solvent S. Examples of solvents of this type are liquid sulfur dioxide, furfural, nitrobenzene, chlorex, phenols, isoquinoline, etc. Mixtures of a solvent or with other solvents or with diluents may also be employed. A nonpolar solvent or a solvent of such polarity as to be capable of forming two liquid phases when mixed with the extract phase may be used as the secondary solvent L. Saturated paraffinic hydrocarbons, such as propane, or butane, or pentane, are examples of suitable secondary solvents. The solvent L should be selected so as to be easily separated from the olefines by distillation.

By the term "component" is designated any of the three portions of any liquid mixture which portions can be separated from each other by extraction with a solvent or solvent mixture. It will be evident that by the word "component" is meant not only one chemically pure substance, but that this word covers also a mixture of substances. A component is said to be pure if it does not contain any portion of a substance which should be present only in another component. In the present specification and claims, there is no difference in meaning between the words "solution", "liquid mixture" and "liquid solution".

I claim as my invention:

1. In a process for removing an intermediate component from a liquid mixture comprising three components of different solubilities in a first selective solvent, the steps of flowing said mixture in an extraction zone countercurrently to concurrent streams of said selective solvent and of a component which is more soluble than the said intermediate component under extracting conditions to form a first raffinate phase and a first extract phase containing said intermediate and more soluble components, separating said phases, subjecting said first extract phase in a washing zone to double countercurrent extraction with said first selective solvent and a second solvent under conditions to form a second extract phase which is rich in said second solvent and in said intermediate component and a second raffinate phase which is rich in the more soluble components and in said first selective solvent, and introducing at least a portion of said second

raffinate into the extraction zone to form the streams of selective solvent and of the more soluble component, whereby the first raffinate phase is enriched by the components of high solubility.

2. The process according to claim 1 in which a portion of the second raffinate phase is treated to reduce its solvent content before it is introduced into the extraction zone.

3. The process for treating a liquid mixture comprising three components of progressively different solubilities in a selective solvent to remove a component of intermediate solubility and produce a raffinate phase concentrated in the other components, which comprises the steps of extracting said mixture in an extraction zone with said solvent by flowing said mixture and solvent countercurrently to each other to form a raffinate phase and an extract phase, the latter containing said intermediate component and a more soluble component, separating the extract phase into portions, one of which is rich in the component of intermediate solubility and the other one of which is rich in the component of greater solubility, and enriching said raffinate by the component of high solubility by re-introducing the latter of said portions into the countercurrent extraction zone at a point so as to avoid countercurrent flow with respect to said solvent, whereby the washing out of said component of high solubility from the raffinate is suppressed.

4. The process according to claim 3 in which the treatment of the extract phase comprises the step of removing the intermediate component from the extract phase by washing it with a second solvent.

5. The process for removing an intermediate component from a liquid mixture comprising three components of progressively different solubilities in a selective solvent, comprising the steps of flowing said mixture in an extraction zone countercurrently to concurrent streams of said selective solvent and of at least one component of greater solubility than said intermediate component under extracting conditions to form first extract and raffinate phases, separating said phases, washing said first extract phase with a different solvent to form a second extract phase which is rich in the intermediate component, and a second raffinate phase which is poor in the intermediate component, and enriching said first raffinate by said component of high solubility by introducing at least a portion of said second raffinate phase into said extraction zone together with said solvent to form the stream of the more soluble component, whereby the washing out of the component of high solubility from the raffinate is suppressed.

6. The process for removing an intermediate component from a liquid mixture comprising three components of progressively different solubilities in a selective solvent, comprising the steps of flowing said mixture in an extraction zone countercurrently to concurrent streams of said selective solvent and of at least one component of greater solubility than said intermediate component under extracting conditions to form first extract and raffinate phases, separating said phases, washing said first extract phase with a different solvent to form a second extract phase which is rich in the intermediate component, and a second raffinate phase which is poor in the intermediate component, treating said second extract phase to produce substantially pure extract of intermediate solubility, introducing a portion of said substantially pure extract into the extraction zone as a backwash, and enriching said first raffinate by said component of high solubility by introducing at least a portion of said second raffinate phase into said extraction zone together with said solvent to form the stream of the more soluble component, whereby the washing out of the component of high solubility from the raffinate is suppressed.

7. A process for producing a mixture of paraffinic hydrocarbons and tertiary olefines comprising the steps of extracting a mixture of paraffinic hydrocarbons, secondary olefines, and tertiary olefines by flowing said mixture countercurrently to a polar selective solvent in an extraction zone to produce a raffinate phase and an extract phase, the latter containing both secondary and tertiary olefines, but substantially no paraffinic hydrocarbons of said mixture, separating said phases, contacting said extract phase with a solvent which, when mixed therewith, forms two secondary liquid phases in which the ratios of secondary to tertiary olefines are different, separating the resulting secondary phases, and enriching said raffinate phase by tertiary olefines by introducing the secondary phase in which the ratio of tertiary olefines to secondary olefines is greater into the extraction zone near the point of withdrawal of said raffinate phase from the extraction zone, located to avoid countercurrent flow of said introduced phase with respect to the polar selective solvent, whereby the washing out of tertiary olefines from the raffinate is suppressed.

8. The process according to claim 3 in which the liquid mixture comprising three components is a hydrocarbon mixture.

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