June 27, 1939. D. S. McKITTRICK 2,164,193

COUNTERCURRENT CONTACT APPARATUS

Filed Oct. 19, 1937

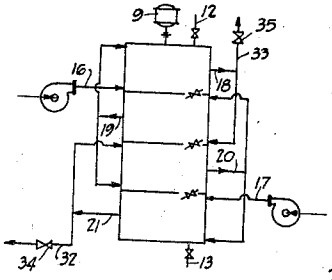


FIG. 3

FIG. 1

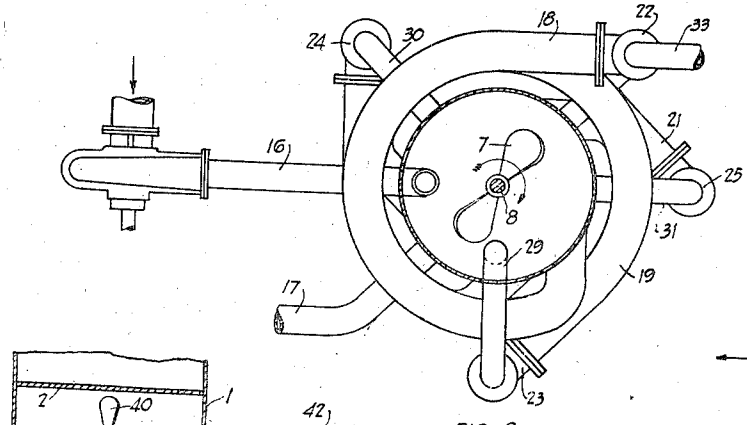


FIG. 2

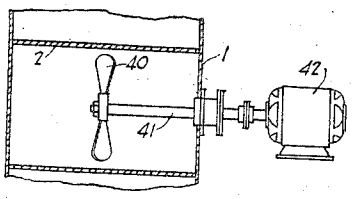


FIG. 5

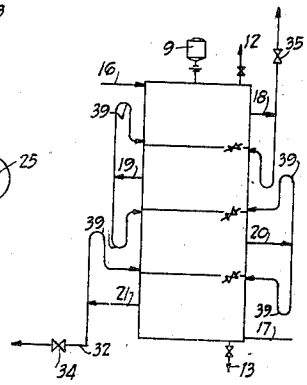


FIG. 4

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By his Attorney

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UNITED STATES PATENT OFFICE

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COUNTERCURRENT CONTACT APPARATUS

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9 Claims. (Cl. 261—108)

This invention relates to improvements in apparatus for countercurrent contact treatment or extraction of one liquid with another partially or wholly immiscible fluid of a different specific gravity which may be either a liquid or a gas. It is particularly applicable to the solvent extraction of hydrocarbon oils with a solvent which will selectively dissolve certain constituents thereof, but may be applied in other countercurrent treatments, such as the extraction of essential oils with solvents such as alcohols, or the treatment of fluids with chemicals, like the contacting of oil with aqueous caustic soda.

It is an object of the present invention to provide an apparatus adapted to contact two at least partially immiscible liquids of different specific gravities, or a liquid and a gas, passing in countercurrent to one another, through a series of mixing phase separating operations. An important object is to provide an apparatus of the type described which is simple in operation, and which will require a minimum of regulation and attention by the operator. A further object is to provide an apparatus in which the fluids flow through the successive stages by gravity, without the use of pumps between stages. A further object is to arrange the levels of the connecting conduits to provide a high differential in pressure between adjacent stages, whereby the treatment of fluids differing only slightly in specific gravities is facilitated and the throughput is increased. Other objects of the invention will appear from the detailed description of the invention.

In operating the apparatus of the present invention, the two fluids, e. g., oil and a selective solvent, are continuously brought together in countercurrent flow, and intimately mixed in a mixing chamber; the mixture is flowed through an elongated conduit wherein the mixture separates into fluid phases of different specific gravities, and the separated phases are continuously withdrawn from the apparatus.

The apparatus in its prefered embodiment comprises a vertically elongated housing divided by horizontal partitions into a series of superimposed mixing chambers, each provided with agitating means, such as stirrers. The lighter and heavier fluids are preferably fed into the mixing chamber at different levels. While the heavier liquid may be fed into the mixing stage at a level higher than the lighter fluid, and the resulting emulsion may be withdrawn at any level, according to the preferred embodiment the heavier liquid is introduced near the bottom of the mixer, and lighter liquid near the top of the mixer, and the emulsion is withdrawn at an intermediate level, since this arrangement increases the pressure differential between stages and thereby increases the rate of flow in the apparatus. Each mixing chamber is connected to an elongated settling tube of sufficient diameter to permit the mixture or emulsion withdrawn from the mixing stage to flow at a velocity sufficiently low to prevent turbulence and of sufficient length to effect the stratification of the mixture; the settling tubes may be horizontal or slightly inclined. The discharge end of each settling tube is connected to a vertical conduit, as by means of a T fitting, whereby the lighter phase formed in the settling tube may flow upwardly, and the heavier phase downwardly. Each upwardly extending branch of the vertical conduit, except in the case of the uppermost stage, is connected to the next higher mixing stage; and each downwardly extending branch, except that from the lowermost stage, is connected to the next lower mixing stage.

The invention will be further understood from the following detailed description, taken together with the accompanying drawing, which illustrates three preferred forms of the apparatus, it being understood that the devices illustrated are exemplary only and that numerous changes in the arrangement and locations of mixing chambers and conduits may be made without departing from the spirit and scope of the invention, as defined in the appended claims.

In the drawing, Figure 1 is an elevation view, partly in section, of the countercurrent contact apparatus; Figure 2 is a sectional view taken on section line 2—2 of Figure 1; Figure 3 is a schematic flow diagram showing the arrangement of conduits; Figure 4 is a flow diagram, similar to Figure 3, showing a modified arrangement of the conduits; and Figure 5 is a fragmental vertical sectional view of one mixing stage, showing a modified arrangement of the agitating device, connecting conduits being omitted.

Referring to Figures 1 to 3, the apparatus comprises a vertically elongated tower 1 provided with vertically spaced horizontal partitions 2, dividing the tower into mixing chambers 3, 4, 5 and 6. Mixing means, such as rotating paddles 7, are provided in each mixing chamber. These may conveniently be mounted on a vertical shaft 8, extending the height of the column through the partitions and rotated by means of a motor 9, the partitions being provided with suitable packed bearings housing the shaft 8 to prevent the flow of fluid between mixing stages. It is a

feature of this arrangement that small leaks between adjacent mixing chambers do not appreciably impair the usefulness of the device, since fluid seeping through the packing will be retained within the system.

Each adjacent pair of mixing chambers is interconnected by means of a by-pass conduit **10**, provided with a valve **11**, the openings being located adjacent the surfaces of the partitions **2**, and horizontally spaced whereby gas such as air may escape from any chamber to the higher chamber, or liquid may be drained to a lower chamber. The partitions may be slightly warped or grooved in the vicinity of the opening of the by-pass conduits to provide for the complete withdrawal of gas or liquid. The end mixing chambers are provided with draining conduits **12** and **13**, provided with valves **14** and **15**.

An inlet connection **16** communicates with the bottom of the uppermost mixing chamber **3**, and is adapted to supply a relatively heavier liquid to the apparatus from a source, not shown, under a suitable pressure. It is preferably arranged to enter the mixing chamber radially inwardly, and to extend inwardly from the periphery of the chamber, as shown, its inner end being provided with an opening to discharge the material upwardly. A similar inlet connection **17**, adapted to supply the relatively lighter fluid under suitable pressure, which may be either a gas or a liquid, communicates with the upper level of the lowermost mixing chamber **6** extending radially inwardly and discharging downwardly.

Elongated horizontal settling tubes **18**, **19**, **20** and **21** communicate at their inlet ends with the mixing chambers **3**, **4**, **5** and **6**, respectively. The inlet ends of these tubes may be inclined to the radius of the chamber to the ends of the tubes to enter the mixing chambers at the periphery thereof and substantially tangentially thereto in a direction opposed to the direction of the peripheral motion of the paddles, as shown, whereby the peripheral momentum of the emulsion within the mixing chambers aids in the flow through the tubes. The settling tubes extend along an arc of a circle exteriorly of the column and terminate in straight sections, which are connected to T fittings **22**, **23**, **24** and **25**, which are connected to upwardly and downwardly extending vertical conduits, these conduits being in free and open communication with the settling tubes. The downwardly extending conduits **26**, **27** and **28** from the T fittings **22**, **23** and **24**, are connected to the lower levels of the mixing chambers **4**, **5** and **6**, respectively, and the upwardly extending conduits **29**, **30** and **31** from the T fittings **23**, **24** and **25** are connected to the upper levels of the mixing chambers **3**, **4** and **5**, respectively, extending radially inwardly and discharging in the directions of the mid-levels of the respective chambers, similar to the inlets **16** and **17**. The conduits **16**, **26**, **27** and **28** may be provided with small holes at their lower portions in the chambers or with external petcocks to permit drainage when the apparatus is to be emptied. The downwardly extending conduit **32** from the T fitting **25** and the upwardly extending conduit **33** from the T fitting **22** are adapted to discharge the heavy and light fluids, respectively, and are provided with valves **34** and **35**. Any suitable device for regulating the operation of these valves may be employed. According to the preferred embodiment one of these valves is actuated by a liquid level controller, and the other by a pressure controller. Thus, the valve **34** may be actuated by a liquid level controller **36**, operable to cause only heavier liquid to flow downwardly through the vertical conduit **32**, and the valve **35** may be activated by a pressure controller **37**, arranged to maintain a predetermined pressure in the system. Each T-fitting may be provided with a sight glass **38** for determining the interface.

The arrangement of the conduits is indicated diagrammatically in Figure 3.

The apparatus may, for example, be used for the extraction of lubricating oil with furfural as the selective solvent. When used for this purpose, the operation is as follows: The apparatus being empty, except for air, the outlet valves **34** and **35** are closed, the by-pass valves **11** and the valve **14** are opened, and the fluids are introduced through the conduits **17** and **18**, until the column **1** and all settling tubes and conduits are filled and air has been expelled. The valves **11** and **14** are then closed, the paddles are set in operation, and the valves **34** and **35** permitted to operate normally, i. e., to open when the level controllers indicate that a lighter phase is present in the conduit **33** and a heavier phase in the conduit **32**. When this condition has been established, heavy and light fluids are continuously fed into the column through the conduits **18** and **17**. The rates of flow being regulated with the air of the sight glasses **38** so as to cause only lighter phases to flow upwardly through the conduits **29**, **30** and **33**, and only heavier phases to flow through the conduits **26**, **27** and **28**; the flow through the conduits **31** and **33** will be automatically regulated by the level controller **36**.

Alternatively, the apparatus may be filled completely with but one of the fluids, the by-pass and drain valves closed, the stirrers set into operation, and the other fluid introduced. When a sufficient quantity of the second fluid has been introduced to cause phase separation in one or more settling tubes both fluids may be fed continuously into the device, and countercurrent flow will be established automatically; the rates of flow, upon reaching equilibrium, are regulated as described above.

The intimately mixed fluids leave the mixing chambers at the intermediate levels, and flow through the horizontal settling tubes, wherein the mixture separates into two phases. These phases are separated at the T-fittings, the lighter fluid flowing upwardly, and the lower fluid downwardly from these fittings.

The inlets **16** and **17**, and the vertical conduits from the T-fittings, as well as the settling tubes, need not be arranged as shown in Figures 1 to 3, but may enter the mixing chambers at any desired levels. Thus, the conduits supplying material to the mixing chamber may enter the latter at the same level, and the conduit supplying the heavier liquid may even be located higher than that supplying the lighter fluid. This arrangement is illustrated in Figure 4 which is similar to Figure 3, and illustrates the levels at which the conduits are connected to the mixing vessels, primed reference characters in Figure 4 corresponding to similar un-primed characters in Figure 3. Since in the arrangement shown in Figure 4 the upwardly extending conduits feed into the bottom of the adjacent higher chamber, and the downwardly extending conduits feed into the top of the adjacent lower chamber, it is desirable to provide traps **39** therein, to prevent the drainage of heavy fluid to the lower chambers, and the drainage of

lighter fluid to the upper chambers when, for any reason, the mixing paddles are stopped.

In the arrangement shown in Figure 4 the pressure differential between adjacent stages, i. e., the pressure drop in the settling tubes and conduits (disregarding the head impressed by the peripheral momentum of the mixture, due to the tangential arrangement of the settling tubes) is about one third of that of Figures 1 to 3, so that, with the same sizes and lengths of conduits, a lower rate of flow will be obtained. In general, lowering the level of the discharge end of the conduits supplying the lighter fluid to the mixing chambers, and raising that of the conduits supplying the heavier fluid decreases the pressure differential or driving force which causes the fluids to assume countercurrent flow. Thus, for conduits and settling tubes of a given resistance or hydraulic constant, the maximum rate of flow is obtainable in the arrangement of Figures 1 to 3. Since this rate of flow can be reduced by employing smaller conduits having a greater flow resistance, or by means of throttle valves in the vertical conduits, and since it does not require traps to prevent the drainage of the fluids when the operation of the mixing paddles is interrupted, the construction shown in Figures 1 to 3 is the preferred embodiment.

The mixing paddles need not be mounted on the same shaft, but may be arranged to be rotatable at different speeds. Moreover, the paddles need not be mounted on a vertical shaft. Thus, as illustrated in Figure 5, each mixing chamber may be provided with a mixing paddle 40, mounted in a horizontal shaft 41, and operated driving means 42, such as a motor, the speed of which may be controlled independently of the driving means for the other mixing chambers. In this manner the degree of mixing in each chamber can be regulated to that necessary to obtain the necessary intermixing of the fluids, and limited to that required for proper phase separation in the settling tubes. The settling tubes in the case of Figure 1 need not be connected to the mixing chambers tangentially thereto, since regardless of the direction of the inlet, the action of the agitators will produce a mixture having a density intermediate to those of the two fluids; the existence of such a mixture in the mixing chambers, and of relatively higher and heavier fluids in the vertical conduits will cause countercurrent flow. Since the hydraulic head induced by the tangential arrangement of the settling tubes according to Figure 1 is practically never exactly the same in all stages, and since it is frequently desirable to maintain the same pressure differential between the termini of all of the settling tubes, the arrangement according to Figure 5, or a radial connection of the settling tubes to the mixing chambers in the case of Figure 1 may be desirable. Whether or not the peripheral momentum of the mixture in the mixing chambers aids the flow, the flow between stages is caused solely by the pressure differential induced by the mixing within the chambers and by the difference between the specific gravities of the fluids. It will be seen that the apparatus requires no pumps or valves for the countercurrent flow of the fluids between the stages thereof, so that the device can be easily controlled and regulated. Any number of stages may be employed.

The expression "in free communication", as used in the claims, is intended to designate a connection which is not provided with pumping means, whereby a fluid may flow therethrough entirely by means of the pressure drop imposed by the fluids in the system.

I claim as my invention:

1. An apparatus for countercurrent contact treatment of a liquid with fluid of a lower specific gravity than said liquid and at least partially immiscible therewith, comprising a series of mixing chambers located at progressively different levels, mixing means within said mixing chambers, an elongated settling tube for each of said mixing chambers in free communication at its inlet end with its mixing chamber, a generally downwardly extending conduit for each settling tube except the lowermost in free communication with its settling tube at a point remote from its said inlet end and with a lower mixing chamber, a generally upwardly extending conduit for each settling tube except the uppermost in free communication with the settling tube at a point remote from its said inlet end and with a higher mixing chamber, means for supplying said liquid to a relatively higher mixing chamber, means for supplying said fluid to a relatively lower mixing chamber, means for withdrawing contacted fluid from a settling tube connected to a relatively higher mixing chamber, whereby said fluid flows through a plurality of mixing chambers and settling tubes in a generally upwardly direction countercurrently to said liquid, said flow being caused solely by the pressure differential between the inlets of the settling tubes and the outlets of said conduits, impressed by the mixing in the mixing chambers and the difference in specific gravity between the fluid and the liquid.

2. An apparatus for countercurrent contact treatment of a liquid with fluid of a lower specific gravity than said liquid and at least partially immiscible therewith, comprising a series of mixing chambers located at progressively different levels, mixing means within said mixing chambers, an elongated settling tube for each of said mixing chambers in free communication at its inlet end with its mixing chamber at an intermediate level in said chamber, a generally downwardly extending conduit for each settling tube except the lowermost in free communication with its settling tube at a point remote from its said inlet and with the lower adjacent mixing chamber near the lower level thereof, a generally upwardly extending conduit for each settling tube except the uppermost in free communication with its settling tube at a point remote from its said inlet and with the higher adjacent mixing chamber near the upper level thereof, means for supplying said liquid to a relatively higher chamber, means for supplying said fluid to a relatively lower chamber, means for withdrawing contacted liquid from a settling tube connected to a relatively higher chamber, and means for withdrawing contacted fluid from a settling tube connected to a relatively higher mixing chamber, whereby said liquid flows through a plurality of mixing chambers and settling tubes in a generally downward direction and said fluid flows through a plurality of mixing chambers and settling tubes in a generally upward direction countercurrently to said liquid, said flow being caused solely by the pressure differential between the inlets of the settling tubes and the outlets of said conduits impressed by the mixing in the mixing chambers and the difference in specific gravity between the fluid and the liquid.

3. The apparatus according to claim 1 in which

the mixing means comprises blades rotatable about a vertical axis, and the inlet of each settling tube opens into the mixing chamber in a direction substantially opposed to the direction of motion of the periphery of the blades in the chamber.

4. The apparatus according to claim 1 in which the means for withdrawing contacted liquid comprises a valve and a level control arranged and constructed to actuate said valve to cause the withdrawal of substantially all of the said liquid from the settling tube connected thereto, and to permit the flow of substantially all of said fluid from said settling tube upwardly therefrom, and the means for withdrawing contacted fluid comprises a valve arranged and constructed to maintain a predetermined pressure within the apparatus.

5. An apparatus for countercurrent contact treatment of a liquid with a fluid of lower specific gravity than said liquid and at least partially immiscible therewith, comprising a vertical tower provided with partitions dividing the same into a plurality of superimposed mixing chambers, mixing means within said chambers, an elongated substantially horizontal settling tube for each of said mixing chambers having its inlet end in communication with its mixing chamber and disposed peripherally about a major portion of the circumference of the mixing chamber, a pair of generally upwardly and downwardly extending conduits for each settling tube in communication with the outlet end of said tube, each upwardly extending conduit except that connected to the uppermost settling tube being in communication with the higher adjacent mixing chamber, and each downwardly extending conduit except that connected to the lowermost settling tube being in communication with the lower adjacent mixing chamber, means for supplying said liquid to a relatively higher chamber, and means for supplying said fluid to a relatively lower chamber, whereby said liquid flows through a plurality of mixing chambers and settling tubes in a generally downward direction, and said fluid flows through a plurality of mixing chambers and settling tubes in a generally upward direction countercurrently to said liquid.

6. An apparatus for countercurrent contact treatment of a liquid with a fluid of lower specific gravity than said liquid and at least partially immiscible therewith, comprising a vertical tower provided with partitions dividing the same into a plurality of superimposed mixing chambers, mixing means within said chambers, an elongated substantially horizontal settling tube for each of said mixing chambers having its inlet end in free communication with its mixing chamber at an intermediate level thereof and disposed peripherally about a major portion of the circumference of the mixing chamber, a pair of generally upwardly and downwardly extending conduits for each settling tube in free communication with the outlet end of said tube, each upwardly extending conduit except that connected to the uppermost settling tube being in free communication with the higher adjacent mixing chamber near the upper level thereof, and each downwardly extending conduit except that in communication with the lowermost settling tube being in free communication with the lower adjacent mixing chamber near the lower level thereof, means for supplying said liquid to a relatively higher chamber, and means for supplying said fluid to a relatively lower chamber, whereby said liquid flows through a plurality of mixing chambers and settling tubes in a generally downward direction and said fluid flows through a plurality of mixing chambers and settling tubes in a generally upward direction countercurrently to said liquid, said flow being caused solely by the pressure differential between the inlet of the settling tubes and the outlets of said conduits impressed by the mixing in the mixing chambers and the difference in specific gravity between the fluid and the liquid.

7. The apparatus according to claim 6 in which the mixing means comprises blades rotatable about a vertical axis, and the inlet of each settling tube opens into the mixing chamber in a direction substantially opposed to the direction of motion of the periphery of the blades in the chamber.

8. The apparatus according to claim 6 in which the mixing means are driven by power means arranged and constructed to permit speed of each mixer to be adjusted independently of the other mixers.

9. The apparatus according to claim 6 in which the mixing means comprises blades rotatable about a horizontal axis.

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