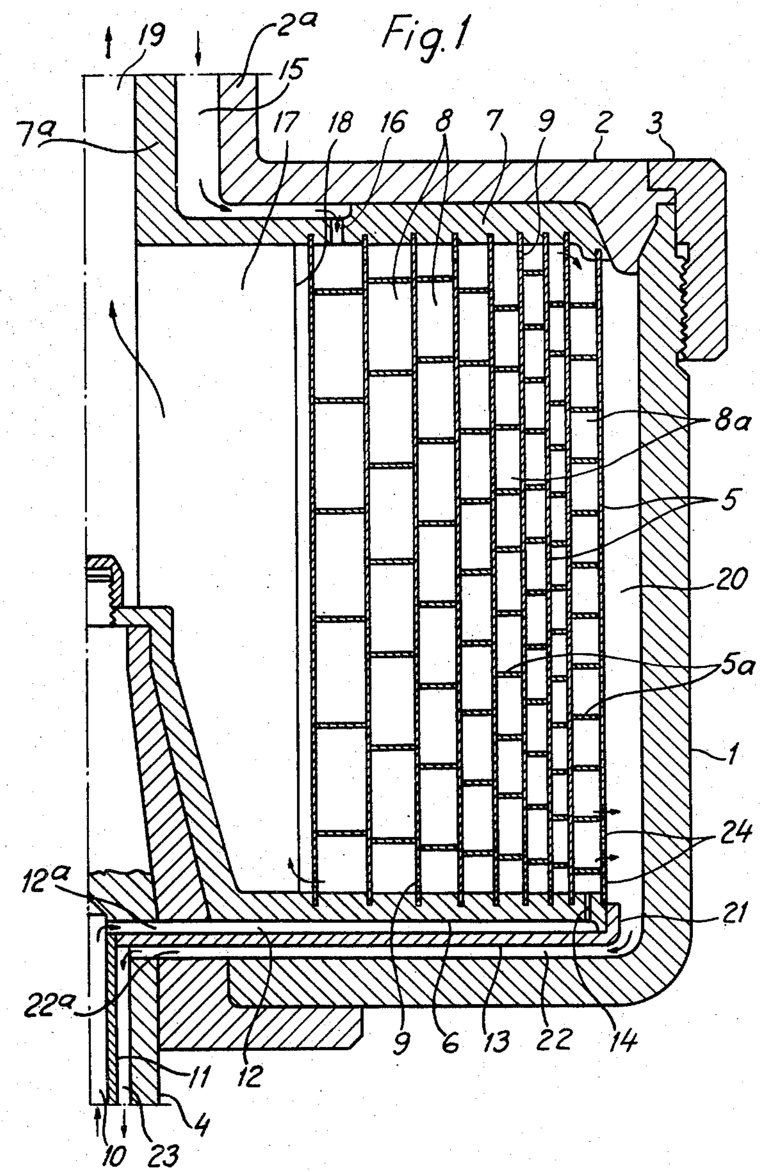
**June 15, 1965** F. T. E. PALMQVIST **3,189,266**

CENTRIFUGAL COUNTERCURRENT EXTRACTION APPARATUS

Filed April 25, 1962 2 Sheets-Sheet 1

Fig.1

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CENTRIFUGAL COUNTERCURRENT EXTRACTION APPARATUS

Filed April 25, 1962 2 Sheets-Sheet 2

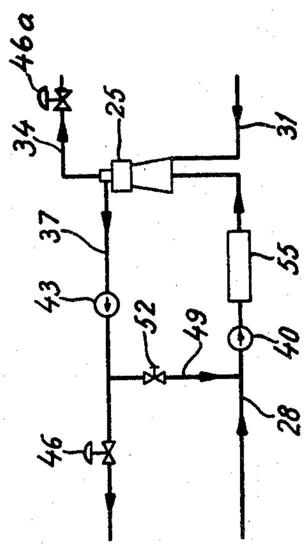

Fig. 3

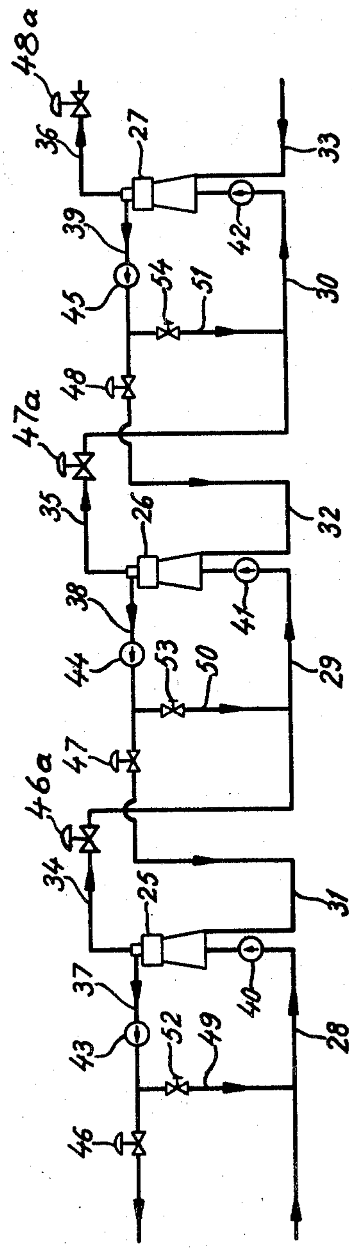

Fig. 2

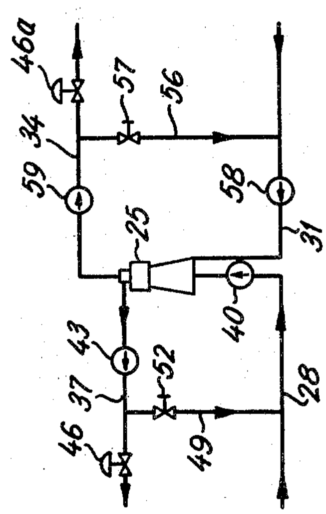

Fig. 4

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CENTRIFUGAL COUNTERCURRENT EXTRACTION APPARATUS

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3 Claims. (Cl. 233—15)

The present invention relates to centrifugal extraction with countercurrent flow of the two components involved, and it has particular reference to an improved apparatus for this purpose. An extractor of this general type is disclosed, for example in U.S. Patent No. 3,108,953, dated October 29, 1963.

The principal object of the present invention is to provide a more effective extraction apparatus than those known heretofore.

According to the invention, part of one component discharging from the centrifugal extractor is supplied to the inlet of the other component into the extractor. This has several advantages. By such recirculation of part of the one component to the inlet side of the extractor, the other component is subjected to an extraction with an increased quantity of extraction agent. Depending upon the degree of recirculation, this quantity can be a multiple of the quantity of extraction agent supplied to the extraction process. Through this arrangement the extraction method itself is made more effective. As a result, it is possible to reduce the quantity of extraction agent used in the process and/or the number of or the size of the extractors used in the process. Also, due to the reduction of the quantity of extraction agent used, the percentage of extract in the extraction agent is increased. This leads in turn to reduced costs for the recovery of the extract from the extraction agent. When washing soap, for example, with an aqueous solution of an electrolyte, a reduced consumption of electrolyte solution not only entails a reduction of the electrolyte consumption but also, through the increased percentage of glycerol in the electrolyte solution, results in a reduction of the consumption of evaporation energy for the recovery of the glycerol.

The extraction efficiency can be further improved by passing the recirculated component, on its way to the extractor inlet, through a mixer together with the other component supplied to the extractor. This mixer can be a centrifugal pump and thus serve to feed the component mixture into the extractor against a counterpressure. The mixer or centrifugal pump thereby effects an intimate contact between the two components and thus an improved extraction.

The apparatus of the invention can be used to advantage in washing soap with an aqueous solution of an electrolyte. In this case, part of the electrolyte solution discharging from the extractor is supplied to the soap inlet of the extractor. In addition to the advantages mentioned earlier, this use has the advantage that the soap mass, which is of a high viscosity even when heated, has its viscosity appreciably reduced and its specific gravity increased through the dilution with recirculated electrolyte solution. This means that the pumping of the supply of soap to the extractor is facilitated in two respects. First, the necessary pumping force is reduced through the acquired reduction of the flow resistance of the soap mass. Secondly, a lower pumping pressure is required to convey the soap mass, which corresponds to the light component in the extraction process, outward to its inlet into the extraction chamber proper (this inlet being located on a relatively large radius) since its dilution with electrolyte solution has caused the soap mass to become heavier.



In a centrifugal extractor of the countercurrent type made according to the invention, a pipe connects the outlet of the extractor for the one component with the extractor inlet for the other component. Preferably, openings are provided close to the inlet of this latter component into the extraction chamber, these openings conducting the recirculated component directly to its outlet from the extraction chamber. In this way, the recirculated component will not place any significant load on the extraction process in the extration chamber proper.

The invention is described more in detail below, reference being made to the accompanying drawings in which:

FIG. 1 is a vertical sectional view of the right-hand half of a preferred embodiment of the centrifugal extractor rotor;

FIG. 2 is a schematic view of an installation having three such extractor rotors connected in series, and

FIGS. 3 and 4 are schematic views of other installations embodying the extractor rotor.

Referring to FIG. 1, the extractor rotor comprises a hollow rotor body 1 having a cover 2, which is fastened to the body 1 by means of a threaded locking ring 3. The rotor body is supported and driven by a vertical hollow spindle 4, although the spindle and rotor body may be arranged to rotate around a horizontal axis. Concentric cylinders 5 are inserted in the interior of the rotor body between its end walls 6 and 7, the radial spacing between these cylinders being reduced toward the periphery of the rotor. The annular interspaces 8 formed by the cylinders 5 communicate with each other by means of holes 9 situated at their ends.

A light liquid is fed into the rotor body 1 or locus of centrifugal force through an axial channel 10 which is formed by the interior of a tube 11 mounted concentrically in the hollow spindle 4. The light liquid from channel 10 enters a chamber 12 between the end wall 6 and an annular disc 13 and is conveyed outwardly, by means of radially extending vanes 12*a* in this chamber, to openings 14 in the end wall 6. These openings lead into the outermost one of the interspaces 8 between the cylinders 5. Heavy liquid is fed in through a channel 15, which is annular in cross section and is formed by cylindrical concentric flanges 2*a* and 7*a* on the cover 2 and the end wall 7, and is delivered through channel 15 to the inmost one of the cylinder interspaces 8 by way of openings 16 provided in the end wall 7. The two liquids now flow through the successive interpsaces 8 in countercurrent to each other, the light liquid forming an inner cylindrical layer and the heavy liquid forming an outer cylindrical layer in each interspace 8, due to the centrifugal force. The liquids flow through the holes 9 in opposite directions to each other so that the light liquid travels radially inward from one interspace to the next one while the heavy liquid flows radially outward from one interspace to the next. The light liquid accumulates in a central space 17 within the innermost cylinder 5 and is discharged through a channel 19 by means of radial vanes 18 provided in the space 17. A clean centrifugal separation of the light liquid takes place in the innermost one of the interspaces 8 as well as in the space 17 and this separation effect can be improved, if necessary, by substituting a set of conventional conical discs (not shown) for the vanes 18.

The throughflowing heavy liquid is centrifugally separated from light liquid in the outermost interspace 8 and in the space 20 situated between the outermost cylinder and the periphery of the rotor. Thereafter, it flows over the outer edge 21 of the disc 13 and through the annular space 22 between disc 13 and the rotor bottom, finally discharging through annular channel 23 formed by the spindle 4 and the tube 11. The annular space 22 may be provided with radial vanes 22*a* which convey the liquid inward toward the spindle 4. The inlets 10 and 15 as well as the outlets 19 and 23 can be hermetically sealed from atmosphere by connecting them to their corresponding stationary pipes through conventional sealing means. Thus, the liquids can be fed into the rotor by pumps which produce the desired pressure in the inlet pipes. In the outermost cylinder 5 there are radial openings 24 located in the vicinity of the end wall 6 or the openings 14.

In order to intensify the extraction, a prolonged contact between the liquids is effected by inserting spiral metal ribbons 5*a* in the cylinder interspaces 8. These ribbons spiral in screw fashion around the rotor axis from the end wall 6 to the end wall 7 and are of the same width as the corresponding interspaces 8. Due to the fact that the centrifugal separating action on the liquids is increased toward the periphery of the rotor, it is necessary, in the outer interspaces 8, to reduce the cross-sectional area of the screw-shaped channels 8*a* formed by the ribbons and thus increase the flow velocity of the liquids, if an equally intense mixing between the liquids and consequently an equally good extraction is to be obtained in all interspaces. Due to such increase in the liquid velocities the forces tending to mix the liquids are increased, whereby the increased separating effect is counteracted. The cross-sectional areas of the spiral channels 8*a* can be reduced by reducing the radial spacing between the cylinders 5 and also by reducing the pitch of the screw-shaped ribbons 5*a*. A spiral ribbon (not shown) may be inserted in the space 20 in the same way as the ribbons 5*a* in the interspaces 8.

The throughflow holes 9 in cylinders 5 are staggered to effect flow through the interspaces 8 in series. Thus, as shown, alternate cylinders have their holes 9 near the bottom while the others have their holes 9 near the top.

Three extractors are shown connected in series in FIG. 2. These extractors may have open inlets and outlets but are here shown to be of a hermetically closed type, for example, as shown in FIG. 1. They are hermetically connected to each other by means of pipe lines which conduct the two extraction components in countercurrent to each other through the extractor series. In the following, the two extraction components are assumed to be soap and an aqueous solution of an electrolyte.

In FIG. 2, the three extractors are designated 25, 26 and 27 and have soap supply pipes 28, 29 and 30, respectively, and electrolyte supply pipes 31, 32 and 33, respectively. The extractors also have soap discharge pipes 34, 35 and 36, respectively, and electrolyte discharge pipes 37, 38 and 39, respectively. The soap supply pipes are provided with centrifugal pumps 40, 41 and 42, respectively, which also form mixing zones. The electrolyte discharge pipes are provided with centrifugal pumps 43, 44 and 45, respectively, and with valves 46, 47 and 48, respectively, which keep the pressure constant on the pressure side of the respective pumps last mentioned. Valves 46*a*, 47*a* and 48*a* maintain a constant pressure in the soap outlet pipes of the extractors. In thus connecting the extractors together in a hermetically closed system, the principle has been used which is disclosed in U.S. Patent No. 2,963,219, dated December 6, 1960. Branch pipes 49, 50 and 51, respectively, connect the electrolyte discharge pipes of the extractors to the corresponding soap supply pipes, and these branch pipes have valves 52, 53 and 54, respectively, for controlling the circulation rate of the electrolyte solution recirculated from each extractor outlet to the extractor inlet.

The plant of FIG. 2 operates in the following way:

Soap is fed by the pump 40 through the pipe line 28 from a saponification plant into the extractor 25 against the counterpressure prevailing in it. Electrolyte solution is fed through a pipe line 31 into the same extractor. (If there were only one extractor, the electrolyte solution would be fresh, but in the case shown in FIG. 2 the electrolyte solution supplied to extractor 25 has already been used, according to the countercurrent principle, in the two following extractors for washing of soap.) Soap leaves the extractor 25 through the pipe line 34 and is delivered to the following extractor 26, and electrolyte solution leaves the extractor 25 through the pipe line 37 and is delivered to a plant (not shown) for the recovery of glycerol. A desired quantity of electrolyte solution is recirculated to the extractor 25 through the pipe line 49 and the soap supply pipe line 28. In this way the soap enters the extractor through the channel 10 (FIG. 1) strongly diluted and intimately mixed with electrolyte solution and flows through the holes 14 into the outermost one of the interspaces 8. This interspace, in order to be able to receive the large volume which the diluted soap constitutes, is wider than the next inner extraction zone or interspace 8. Most of the electrolyte solution is separated immediately from the soap in this outermost space 8 and flows outward through the holes 24 into the space 20 and thence inward through the space 22. In this way, the normal extraction process in the other parts of the extractor is not disturbed; but the extraction starts with a soap which is more washed than is the case without any recirculation of electrolyte from the extractor outlet.

The process which has been described as taking place in the extractor 25 is repeated in the extractors 26 and 27, as will be seen from FIG. 2. This means that fresh electrolyte solution (for example, a solution of common salt) is supplied through the pipe line 33 and is discharged, enriched with glycerol, as so called spent lye through the pipe line 37 and that the soap mass to be washed is supplied through the pipe line 28 and is discharged as washed through the pipe line 36.

The system shown in FIG. 3 comprises a single extractor 25 and a container 55 inserted in the soap pipe line 28. In this container diffusion between the two mixed components is utilized so as to improve the extraction process. The diluted soap stays in the container 55 for the time desired for the diffusion. In view of the diffusion, the pump 40 is preferably located, calculated in the flow direction, before the container 55 so that the soap enters this container well mixed with the electrolyte solution.

In the foregoing, it has been presupposed that the recirculated component (electrolyte solution) is the heavier one of the components leaving the extractor. Of course, in another use of the apparatus the recirculated component can be the lighter one without departing from the principle of the invention.

The system of FIG. 4 has a double return flow causing an intensified extraction, as compared with a single return flow. As shown, this system has a single extractor 25. The additional return pipe is shown at 56 and is provided with a valve 57 having a function similar to that of valve 52. Pumps 58 and 59 correspond in their function to the pumps 40 and 43.

*Example 1*

Vanillin was extracted from sulphite waste liquor by means of toluene without recirculation of the latter to the inlet of the extractor. More exactly, 1000 parts by weight of a waste liquor containing 0.38% of vanillin was contacted with 200 parts by weight of toluene in an extractor according to FIG. 1, in countercurrent and at a temperature of 70° C. The toluene leaving the extractor contained 1.69% of vanillin, and the waste liquor leaving the extractor contained 0.04% of vanillin. Thus, the toluene had extracted from the waste liquor 89% of the content of vanillin of said liquor. This result corresponds to 2.0 theoretical extraction steps according to the definition found in the book "Liquid-Liquid Extraction" by L. Alders, the second edition, 1959.

*Example 2*

The preceding example was modified in that the extraction was carried out while recirculating toluene, discharging from the extractor, to the inlet of the waste liquor into the extractor. The recirculated quantity of toluene was three times as large as the quantity discharged from the extraction process. The discharged quantity of toluene contained 1.80% of vanillin, and the discharged waste liquor contained 0.02% of vanillin. Thus, the toluene had extracted from the waste liquor 94.5% of the content of vanillin of said liquor. The extraction yield was improved through the recirculation of toluene to such a degree that the yield corresponded to 2.8 theoretical extraction steps.

*Example 3*

Phenol was extracted from phenol-containing gas works water by means of benzene without recirculation of the latter to the inlet of the extractor. More exactly, 1000 parts by weight of gas works water containing 0.40% of phenol were contacted with 600 parts by weight of benzene in an extractor according to FIG. 1, in countercurrent and at a temperature of 50° C. The benzene leaving the extractor contained 0.59% of phenol, and the water leaving the extractor contained 0.05% of phenol. Thus, the benzene had extracted from the water 88% of the content of phenol of the latter. This result corresponds to 3.0 theoretical extraction steps.

*Example 4*

Example 3 was modified in that the extraction was carried out while recirculating benzene, leaving the extractor, to the water inlet of the extractor. The recirculated quantity of benzene was 1.7 times as large as the quantity discharged from the extraction process. The discharged quantity of benzene contained 0.61% of phenol, and the discharged water contained 0.03% of phenol. Thus, the benzene had extracted from the water 92% of the content of phenol of the latter. Through the recirculation of benzene the extraction yield was improved to such a degree that it corresponded to 3.8 theoretical extraction steps.

*Example 5*

Aromatic and unsaturated aliphatic hydrocarbons, which are characterized by a low content of hydrogen, were extracted from shale oil by means of furfural without recirculation of the so-called extract phase to the inlet of the extractor. The extract phase consisted of a solution of the aromatic and unsaturated aliphatic hydrocarbons in the furfural. More exactly, 1000 parts by volume of shale oil with a specific gravity of 0.959 and an iodine number of 92.2 were contacted with 1185 parts by volume of furfural in an extractor according to FIG. 1, in countercurrent and at the temperature of 0° C. The extract phase, 1787 parts by volume, leaving the extractor consisted of 1153 parts by volume of furfural and 634 parts by volume of oil; and the raffinate phase, 398 parts by volume, leaving the extractor consisted of 366 parts by volume of oil and 32 parts by volume of furfural. After the furfural had been distilled off from the raffinate phase, a refined oil was obtained having a specific gravity of 0.911 and an iodine number of 64.5. Thus, the refined oil had a lower specific gravity and a lower iodine number than the crude oil, which means that the raffinate is more rich in hydrogen and has a higher value.

*Example 6*

Example 5 was modified in that the extraction was carried out while recirculating the extract phase leaving the extractor to the inlet of the crude oil into the extractor. The recirculated quantity of extract phase was as large as the quantity discharged from the extraction process. The extract phase, 1880 parts by volume, leaving the extractor consisted of 1160 parts by volume of furfural and 720 parts by volume of oil; and the raffinate phase, 305 parts by volume, leaving the extractor consisted of 280 parts by volume of oil and 25 parts by volume of furfural. After the furfural had been distilled off from the raffinate phase, a refined oil was obtained having a specific gravity of 0.901 and an iodine number of 57.7.



Thus, the quality of the refined oil was appreciably better than that in the preceding example.

Usual mineral oil (petroleum) is refined in the same way as has been stated above for shale oil, the operation conditions, of course, being adjusted to the kind of the oil.

*Example 7*

Soap mass coming from the saponification step of a plant for continuous soap making was washed in two extractors connected in series in the manner shown in FIG. 2. However, no recirculation of the washing liquid took place. More exactly, 1527 parts by weight of soap mass containing 100 parts by weight of glycerol were contacted with 881 parts by weight of a 12% solution of common salt, in countercurrent and at a temperature of 95° C. The spent lye or salt solution (903 parts by weight) leaving the extractors contained 95 parts by weight of glycerol, and the soap (1505 parts by weight) leaving the extractors contained 5 parts by weight of glycerol. Thus, the salt solution had extracted from the soap mass 95% of the content of glycerol of the latter.

*Example 8*

Example 7 was modified in that the washing was carried out while recirculating spent lye, leaving each extractor, to the inlet of the soap mass into the respective extractor. If it is assumed that the washing was carried out in the two left-hand extraction steps of FIG. 2, a quantity of spent lye, being twice as large as that which was discharged to the next step or was discharged from the washing process, was recirculated through each of the pipes **50** and **49**. The quantities of salt solution and soap eventually discharged from the washing process were practically the same as those in Example 7. The quantity of salt solution discharged from the washing process contained 98 parts by weight of glycerol, and the quantity of soap discharged from the same process contained 2 parts by weight of glycerol. Thus, the salt solution had extracted from the soap mass 98% of the content of glycerol of the latter.

I claim:

**1.** A centrifugal extractor comprising a hollow rotor rotatable about an axis, annular partition means in the rotor forming therewith a group of annular zones generally concentric to said axis and interconnected in series, said zones being disposed in adjacent intersurrounding relation and including innermost and outermost extraction zones constituting final extraction zones and also including intermediate extraction zones and a separating zone adjacent one of said final extraction zones but remote from said intermediate zones, said separating zone communicating with the adjacent part of said one final extraction zone to provide the sole source of liquid supply to the separating zone, the rotor having a first outlet leading directly from said separating zone and a second and separate outlet leading from the other of said final extraction zones, the rotor also having a first separate inlet leading to said other final extraction zone and a second separate inlet leading directly to said one final extraction zone, means external of the rotor for supplying fresh feed to said first inlet, and means external of the rotor for feeding a mixture of heaviest and lightest liquid components to said second inlet, said feeding means including means for mixing with fresh feed to said second inlet some of the discharge from said first outlet in the same composition in which it is dishcarged from said first outlet, said mixing means including a return pipe leading directly from said first outlet to feed one of said components and a separate pipe to initially feed the other of said components.

**2.** A centrifugal extractor according to claim **1**, in which said separating zone is adjacent said innermost extraction zone.

**3.** A centrifugal extractor according to claim **1**, in which said separating zone is adjacent said outermost extraction zone.


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