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(54) APPARATUS FOR THE CONTROLLABLE REMOVAL OF ONE OR MORE PHASES FROM A LIQUID-LIQUID EXTRACTOR

(71) We, GESELLSCHAFT ZUR WIEDERAUFARBEITUNG VON KERNBRENNSTOFFEN mbH., of 7514 Eggenstein-Leopoldshafen 2, Postfach 220, Federal Republic of Germany; a German body corporate do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

The present invention is directed to an apparatus for the controllable removal of one or more phases from a liquid-liquid extractor, having a mixing chamber and a settling chamber for light and heavy phases, useful in the treatment of organic solvent wastes from radioactive systems.

Various systems are known for controlling the removal of one or more phases present in a settling chamber of a liquid-liquid extractor. These systems are generally susceptible to breakdown during their operation and, therefore, require systematic maintenance. Such systems are particularly not suitable for use in processing of radioactive substances wherein it is imperative that the system used is dependable and substantially maintenance free.

The various float or control valves, due to their susceptibility to malfunction, have not found use in controlling the removal of liquids from settling chambers in radioactive waste processing plants. The removal of the heavy liquid phase from liquid-liquid extractors in such processing plants is generally controlled by pneumatic syphoning systems. The drawback of these syphoning systems is that they tend to clog even when there is only a small amount of solid contaminants present and, further, they cannot be operated dependably in systems employing more than two phases. The removal of a light phase from a settling chamber by an overflow aided by means of incoming air is well known as discussed by Treybal, in *Reactor Handbook*, Volume II, page 455. Such a system, though, is restricted to the removal of only the light phase located at the top of the settling chamber and there is no regulation of the light and heavier phase interface position in the settling chamber.

The object of the present invention is to provide an apparatus for automatically regulating the phase interphase position of the light and heavier phases contained in the settling chamber and provides a control which is substantially maintenance free. The apparatus of the present invention is particularly useful in the treatment of organic wastes from radioactive processing systems and especially suitable in the phosphorus acid adduct treatment of such wastes.

According to the present invention there is provided a liquid-liquid extractor comprising a mixing chamber and a settling chamber for separation of light and heavy liquid phases, and including a tapering member connected to an opening in a wall of the settling chamber for removing a heavy phase from the settling chamber; a first tube having a first end connected to an opening in said tapering member and a second end extending, in use, to the level of a light phase; and a second tube connected to said first tube for introducing gas into said first tube to move liquid present in the first tube, in use, in an upwardly direction.

Preferably, the tapering member has its large end connected to the settling chamber and its small end connected to said first tube and said second tube has one end connected to said first tube at an intermediate position thereon. Preferably, the tapering member is connected to said settling chamber at a point remote from the mixing chamber and adjacent the phase to be removed.

The liquid-liquid extractor of the present invention preferably further comprises metering means connected to said second tube for regulation the flow of gas there-

through.

In one embodiment of the present invention, a bottom member of said tapering member is upwardly inclined with respect to the horizontal, the higher end of said inclined bottom member is connected to the first end of said first tube and said bottom member forms an integral inclined portion with a bottom member of the settling chamber. Alternatively, the tapering member may be connected in a downwardly inclined manner with respect to a bottom member of the settling chamber.

The liquid-liquid extractor of the resent invention may further include at least one staggered plate disposed in front of the large end of the tapering member. Furthermore, the liquid-liquid extractor may include an overflow vessel having a bottom member and a top member, said second end of said first tube terminating at an intermediate point in the overflow vessel, means for removing gas connected to said top member and means connected to the bottom member for removing a heavy phase from said overflow vessel.

The particular advantages of the apparatus according to the present invention are seen in the fact that a common discharge is provided for one or more of the heavy phases desired to be removed from a liquid-liquid extractor comprising a mixer-settler without there existing the danger of clogging of the apparatus or the depositing of sediment therein. The structure of the present invention is of simple design permitting ready, maintenance free regulation of the separation of the layers present based on the proportional properties of the phase liquids and is, therefore, self-stabilizing. Further, a particular advantage of the apparatus according to the present invention is its ability to permit the common extraction of a plurality of heavy phases as encountered, for exaple, during the adduct formation in the phosphorous acid adduct process. The low probability of clogging in the discharge conduit as a result of the presence of solid particles and the deposition of separating layers therein are further significant advantages of the apparatus.

The present invention will be further illustrated, by way of example, with reference to the accompanying drawing in which the single Figure is a partially broken away perspective view of a liquid-liquid extractor with a control apparatus.

The liquid-liquid extractor, including the control apparatus according to the present invention has a mixer-settler 1 which is divided into two chambers, 3 and 15, by a perforated metal sheet 2. In chamber 3 of the mixer-settler, a stirrer, including a stator housing 4 and a return conduit 5 are mounted in cover 6 which is over chamber 3. Conduits 7, 8, 9, and 10 represent conduits for the introduction of various liquids as well as for the purpose of decontamination and discharge as conventionally known. For example, in the phosphorus acid adduct treatment, conduit 7 is used to introduce a tributyl phosphate/dodecane mixture, conduit 8 is used to introduce H_3PO_4, conduit 9 is a decontamination connection and conduit 10 is a discharge conduit leading to chamber 3. The decontamination connection 9 is divided into two branch conduits 11 and 12, with conduit 11 extending into chamber 3 and conduit 12 leading to a funnel or tapering member 13.

The liquids entering the mixer chamber 3 are agitated by the stirrer contained therein to form small droplets of at least one of the liquids dispersed in other liquids in the conventional manner. The mixer chamber 3 should be of sufficient size to permit a residence time for the liquids to permit the desired diffusion transfer to occur. The liquid then pass into settling chamber 15 for separation of the lighter and heavier liquid phases present with the lightest phase taking the uppermost position. The bottom member 14 of the settling chamber 15 of mixer-settler 1 is preferably upwardly inclined with respect to the horizontal with its most elevated section being furthest away from chamber 3. The bottom 14 of the settler chamber 15 opens into a bottom portion 33 of funnel member 13 which is fastened to a side wall member 16 of the chamber 15. The bottom portion 33 of funnel member 13 is also upwardly inclined with members 33 and 14 forming an integral inclined member. The funnel member 13 is illustrated as a pyramidal tapering member but, it is realised that it may be of other forms, such as conical. The tapering design of member 13 provides additional phase separation.

Further, the funnel member 13 may be located at any other desired point depending on the location in chamber 15 of the one or the other liquid phase of phases which are to be extracted. For example, it is possible, in a two-phase system, to connect the funnel member 13 to an opening in bottom member 14 and to orient it downwardly.

Funnel member 13 opens at its smaller end into a vertical riser tube 21 which extends to a height equal to the level of the light phase contained in chamber 15 and, normally, to overflow opening 20.

Gas, in the form of air, is introduced into riser tube 21 by a gas inlet conduit 23 which is connected to tube 21 at an intermediate point thereon. The gas is supplied from a gas source (not shown) and its flow rate is regulated by a metering apparatus 22 in the form of a flowthrough meter. The introduction of air into riser tube 21 produces movement of the liquid which is dependent

on the quantity and rate of air introduced and the hydraulic prepressure formed by the pressure of the individual phases in mixer-settler 1. The amount of liquid in riser tube 21 above the gas inlet 23 without the introduction of air in relation to the conveying level above the air inlet is normally about 80 to 90% and consists of the monometric pressure of the total liquid in mixer-settler 1.

Plates 18 are disposed in a spaced and staggered manner in front of funnal member opening 17 to hold back the mixed phase. These plates have a calming effect and further provide separation of the lighter phase which may be present in funnel member 13 itself. The lightest phase, i.e. the uppermost phase in chamber 15 is able to flow off through an outlet 19 which is provided with a funnel overflow opening 20. The open end 26 of riser tube 21 terminates at an intermediate point within collecting vessel 25. An air exhaust line 28 is fastened to cover plate 27 of collecting vessel 25 and a discharge conduit 29 for the adduct and approximately 12M H_3PO_4 leads from the base plate 24 to the separator (not shown).

The self-regulation of the phase interface in relatonship to the proportional characteristic of the light and the heavy phase (e.g. phases 30 and 31) is based on the following principle:

If, for example, as a result of feeding in the heavy phase (concentrated phosphorus acid) the phase interface rises, the product of density times height increases and, thus, the hydrostatic pressure in tube 21 increases. With a constant amount of air bubbles in through gas feed tube 23, the heavy phase is conveyed to overflow vessel 25 until the position of the phase interface remains constant.

If the phase interface drops and the amount of air bubbled in remains the same, less of the heavy phase is extracted and the phase interface is stabilized at a lower level. The bubbling in of air also reduces the danger of clogging in that it prevents the caking together of mud or deposits in discharge conduit 29. Likewise, the device in the illustrated liquid-liquid extractor operates with three liquid phases of different densities and is capable of extracting the two heavy phases together. The lowest of the phase interfaces which then forms adjusts itself to the intake level of removal tube 21.

The illustrated apparatus which has length, width, and height dimensions of 900 mm × 300 mm × 600 mm is used with advantage in the phosphorus adduct process for the continuous adduct formation with concentrated phosphorus acid. The dodecane phase is then separated in settler 15 while phosphorus acid and adduct, in part, form viscous emulsions.

Tube 21 leaves the apparatus about 400 mm below the dodecane liquid surface 32 and has a connection for the air intake line 23 at a height of about 120 mm. The adduct (density 1.1 to 1.2 g/cm^3) and phosphorus acid (density 1.5 g/cm^3) can be easily extracted together with the bubbling in of air at a rate of 50 to 70 liters per hour. The position of the lowest of the phase interfaces depends on the rate of air used and remaining constant once an equilibrium has been reached.

It will be understood that the above description of the present invention is susceptible to various modifications, changes and adaptations, and the same are intended to be comprehended within the meaning and range of equivalents of the appended claims.

WHAT WE CLAIM IS:-

1. A liquid-liquid extractor comprising a mixing chamber and a settling chamber for separation of light and heavy liquid phases and including a tapering member connected to an opening in a wall of the settling chamber for removing a heavy phase from the settling chamber; a first tube having a first end connected to an opening in said tapering member and a second end extending, in use, to the level of a light phase; and a second tube connected to said first tube for introducing gas into said first tube to move liquid present in the first tube, in use, in an upwardly direction.

2. An apparatus as claimed in claim 1, wherein said tapering member has its large end connected to the settling chamber and its small end connected to said first tube and said second tube has one end connected to said first tube at an intermediate position thereon.

3. An apparatus as claimed in claim 2, wherein the tapering member is connected to said settling chamber at a point remote from the mixing chamber and adjacent the phase to be removed.

4. An apparatus as claimed in claim 1, 2 or 3, further comprising metering means connected to said second tube for regulating the flow of gas therethrough.

5. An apparatus as claimed in claim 3, wherein a bottom member of said tapering member is upwardly inclined with respect to the horizontal, the higher end of said inclined bottom member is connected to the first end of said first tube and said bottom member forms an integral inclined portion with a bottom member of the settling chamber.

6. An apparatus as claimed in claim 3, wherein said tapering member is connected in a downwardly inclined manner with respect to a bottom member of the settling chamber.

7. An apparatus as claimed in claim 2,

further comprising at least one staggered plate disposed in front of the large end of the tapering member.

8. An apparatus as claimed in claim 2, further comprising an overflow vessel having a bottom member and a top member, said second end of said first tube terminating at an intermediate point in the overflow vessel, means for removing gas connected to said top member, and means connected to the bottom member for removing a heavy phase from said overflow vessel.

9. A liquid-liquid extractor, substantially as hereinbefore described with reference to and as illustrated in the accompanying drawing.

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1564640 COMPLETE SPECIFICATION

1 SHEET

This drawing is a reproduction of the Original on a reduced scale