Apparatus for the separation of non-miscible liquids.

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References cited:
US-A- 3 213 157
US-A- 3 527 348
US-A- 4 579 998

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Description

This invention relates generally to method and apparatus for handling fluids. In one aspect it relates to apparatus for fluid handling in an alkylation process. In another aspect it relates to a method for reducing spillage of acidic material in the event of a leak in an alkylation process.

Background of the Invention

It is common practice in the petroleum industry to produce high octane motor fuel by alkylation an isoparaffin with an olefin in the presence of a catalyst which preferably is liquid hydrofluoric acid or hydrogen fluoride (HF). Such a process is commonly known as an HF alkylation process or merely an alkylation process. US-A-4579998 discloses a continuous method and apparatus for contacting olefin with paraffin in the presence of HF catalyst to produce alkylate. Product effluent from the riser reactors enters a common setting vessel. In such a known apparatus the effluent from the alkylation reactor containing hydrocarbon and acid, is usually passed to a generally vertical arranged settler vessel at an intermediate point along the length of the settler vessel. A hydrocarbon phase is separated from an acid phase in the settler vessel, with the hydrocarbon phase contained in the upper portion of the settler vessel and the acid phase contained in a lower portion of the settler vessel. Accordingly, a liquid-liquid interface between the acid phase and hydrocarbon phase is formed within the settler vessel. As used herein, the liquid-liquid interface is located at a point along the height of the settler vessel where the acid concentration of the material in the settler vessel is greater by a predetermined amount than the acid concentration in the alkylate product supplied to the settler from the reactor. The hydrocarbon phase is fractionated to separate low boiling hydrocarbons from the alkylate product while the acid phase is cooled and recycled to the alkylation reactor for reuse in the alkylation process. As necessary, acid catalyst can be withdrawn from the system for purification. The purified acid catalyst and, as necessary, additional fresh acid is returned to the alkylation reactor.

It is known to improve the economics of an alkylation process by employing two or more alkylation reactors and passing the individual alkylation product streams to a common settler vessel, thereby forming a common pool of acid catalyst in the lower portion of the common settler vessel. Acid catalyst is then withdrawn from the common pool and passed in individual streams to the respective alkylation reactors. While an alkylation system employing multiple reactors and a common acid catalyst pool is effective for reducing equipment cost while maintaining a desired hydrocarbon/catalyst ratio for each reactor, and is also effective for increasing production of high quality gasoline boiling range materials, the system presents certain safety considerations. For example, with a common acid catalyst pool, a leak in one reactor could result in spillage of the entire catalyst pool which supplies the multiple reactors.

Acid catalyst fluid handling systems associated with alkylation processes are designed with due concern for providing a non-leaking catalyst fluid handling system. In order to provide greater safety, however, it is desirable to reduce, as much as possible, the spillage that would occur in the event of a leak affecting the liquid acid catalyst.

Accordingly, it is an object of this invention to improve safety in operating an alkylation process. A further object of this invention is to increase the safety of a petroleum refining process and the apparatus employed therein.

Another object of this invention is to provide apparatus and method for reducing the spillage of acid catalyst in the invent of a leak in the acid catalyst handling system associated with an alkylation process.

In accordance with this invention an apparatus comprising a vertically extending settler vessel and a method for handling fluids by means of this apparatus as defined in claim 1 and claim 3, respectively, are provided. Preferred embodiments are defined in the dependent claims.

Summary of the Invention

In accordance with one aspect of this invention, multiple chambers are provided in the bottom of a common acid settler vessel to contain the acid catalyst. The number of chambers provided at least corresponds to the number of respective reactors which supply alkylation product to the common settler vessel. Separate acid return streams for each reactor are also provided so that a leak in one reactor, or its associated acid cooler, would result in liquid spillage of no more than the amount of the acid catalyst in the chamber of the common settler vessel associated with that reactor.

In accordance with another aspect of this invention the common settler vessel is operated with both liquid and gaseous hydrocarbon phases at lower pressures, so that the leak rate resulting from a leak in a reactor would be minimized.

In a preferred embodiment of this invention a common settler vessel is provided with at least one baffle extending from wall to wall at the bottom of the common settler vessel and upwardly into close proximity, or above, the interface between the liquid acid catalyst phase and the liquid hydrocarbon phase in the common settler vessel. The baffle
separates the lower portion of the settler vessel into two or more chambers and prevents liquid communication between the quantities of liquid acid catalyst which are contained within the chambers and are to be recycled to the respective reactors. A separate outlet is provided for each chamber in the bottom of the common settler vessel for return of the acid catalyst to the respective reactor and associated cooler.

Should the acid catalyst level lower in one chamber due to leakage or spillage, liquid hydrocarbon from the correspondingly lowered hydrocarbon phase thereabove displaces the acid catalyst volume lost and thus provides a liquid seal for the acid catalyst in the non-leaking chambers. Also another material which is less dense and of higher boiling point than the acid catalyst, could be injected to replace the spilled acid catalyst and provide the liquid seal.

In a preferred embodiment of the present invention, four reactors, each having an associated cooler, are utilized. Two riser reactors are positioned on two opposite sides of a vertically oriented settler vessel. Alkylate product streams of the two reactors on one side of the settler are combined at or above the elevation of the settler inlet, and enter the settler in a combined stream. In a similar manner the alkylate product of the two reactors on the other side of the settler vessel are combined and enter the settler vessel in a single stream. Four separate streams are provided for returning the acid catalyst from the settler to four corresponding riser reactors via four corresponding acid catalyst coolers.

Additional objects and advantages of the invention will be apparent from the following detailed description of the preferred embodiment of the invention as illustrated by the drawings in which:

FIG. 1 is a diagrammatic elevation of riser reactors, a settler vessel and coolers provided in an arrangement suitable for carrying out the invention.

FIG. 2 is a side elevation taken along line 2-2 of FIG. 1.

FIG. 3 is a side elevation taken along lines 3-3 of FIG. 1.

FIG. 4 is a cross sectional view taken along line 4-4 of FIG. 1.

Detailed Description of the Preferred Embodiment

In the following discussion, parts which appear in more than one of the drawing figures shall be referred to by the same reference numeral in each of the drawing figures in which the part appears. Referring now to the drawings and in particular to FIGS. 1, 2 and 3, four upwardly extending tubular reactors, referred to hereinafter as riser reactors, are designated by the reference characters 10, 11, 12 and 13 and are in open communication at the tops thereof with a generally vertically disposed settler vessel 14 via conduits 16, 17, 18 and 19. The settler vessel 14 defines a vertically extending separation zone therewithin having a lower portion, an upper portion and an intermediate portion. The settler vessel 14 provides means for separating a mixture containing a heavier liquid and a lighter liquid. Effluent alkylate, together with acid catalyst, is introduced into a lower portion of the settler 14 from the outlets of reactors 10, 11, 12 and 13 through conduits 16, 17, 18 and 19. Although four reactors are illustrated in FIGS. 1-4, any number of reactors can be used in the practice of the invention.

The lower ends of riser reactors 10, 11, 12 and 13 are in open fluid communication with coolers 20, 21, 22 and 23, respectively. Hydrocarbon feed is provided via conduit 24 to coolers 20, 21, 22 and 23 along with additional fresh acid where cooled recycled or rerun acid catalyst is picked up to form a hydrocarbon and acid catalyst mixture. The hydrocarbon and acid catalyst mixture is dispersed upwardly with high velocity through the coolers 20, 21, 22 and 23 and into the corresponding inlets of riser reactors 10, 11, 12 and 13, respectively.

At the bottom of common settler vessel 14, outlet conduits 26, 27, 28 and 29, which extend downwardly from settler vessel 14, are provided for the withdrawal of liquid acid catalyst for recycle. Conduits 26, 27, 28 and 29 are connected at the lower ends thereof with coolers 20, 21, 22 and 23, respectively via corresponding conduits 30, 31, 32 and 33. At an intermediate point along the length of the settler vessel 14 an outlet conduit 34 is provided for the removal of the separated liquid hydrocarbon product.

In operation, a liquid hydrocarbon feed mixture comprising a mixture of an alkylating agent, such as a low boiling olefin, e.g. butylene, and an alkylatable hydrocarbon, such as a low boiling isoparaffin, e.g. isobutane, is introduced through conduit 24, as well as fresh makeup acid catalyst. The feed mixture is dispersed at high velocity in the shells of coolers 20, 21, 22 and 23 which contain cooled liquid acid catalyst, thus inducing acid catalyst circulation into the hydrocarbon feed mixture by density difference between the settled acid 36 from the settler 14 and the fresh makeup acid catalyst dispersed with the hydrocarbon feed. In this manner acid catalyst is picked up by the flow action of the liquid hydrocarbon feed mixture. Hydrocarbon feed mixture and cooled recycled acid catalyst pass through the reactors 10, 11, 12 and 13 in co-current flow which results in formation of higher molecular weight hydrocarbon material or alkylate of increased octane value, as is well known.
in the art.

Reaction effluent, containing alkylate (i.e. hydrocarbon product), catalyst and unreacted feed hydrocarbon, passes from reactors 10, 11, 12 and 13 and enters settler vessel 14 through conduit 16, 17, 18 and 19. Within settler vessel 14, the effluent from reactors 10, 11, 12 and 13 separates into a lower liquid acid phase and an upper liquid hydrocarbon phase. In accordance with the invention, however, settler vessel 14 is preferably operated with both liquid and gaseous hydrocarbon phases, as illustrated in FIG. 1.

A liquid-liquid interface is formed at a point 36 in settler vessel 14. The interface occurs at the level between the lower heavy acid phase 38 and the lighter hydrocarbon phase 40. As used herein the interface 36 is considered to be the point along the vertical length of the chamber of the common settler vessel 14 where the acid concentration of the material settled in the lower portion of settler vessel 14 is equal to, or greater by a predetermined amount, than the acid concentration in the reactor effluent material supplied to the common settler vessel 14, through conduits 17 and 19.

As most clearly illustrated in FIG. 4, the lower portion of common settler vessel 14 is provided with baffles 50 and 52 which divide the lower portion of vessel 14 into four chambers 54, 56, 58, and 60. As illustrated most clearly in FIGS. 1, 2 and 3, the baffles 50 and 52 extend from the bottom of the settler vessel 14, at least into close proximity to the interface level 36. Thus, acid catalyst supplied to settler vessel 14 from a pair of riser reactors on one side of the settler vessel 14, and which descends mostly along the walls of settler vessel 14, is for the most part returned to the respective pair of riser reactors.

An alkylation unit such as shown in the drawings may be operated such that the interface level 36 is different in the various chambers 54, 56, 58 and 60. The baffles 50 and 52 therefore may extend upwardly to near proximity of the interface level in one chamber while extending significantly above the interface level in an adjacent chamber. Accordingly, the baffles 50 and 52 may be any desired height and may extend significantly into the hydrocarbon settling zone 40 if desired. The requirement for the baffles 50 and 52 is that they extend to a height sufficient to assure that the chambers 54, 56, 58 and 60 contain substantially all of the acid catalyst in the lower portion of settler vessel 14.

The acid catalyst is withdrawn from the chambers 54, 56, 58 and 60 through outlets 62, 64, 66 and 68, respectively, illustrated in FIG. 4, which outlets are provided in the bottom of common settler vessel 14 and are connected in fluid flow communication with conduits 28, 26, 27 and 29, respectively, and the acid catalyst is recycled to the riser reactors via the coolers and interconnecting conduits.

The invention is not dependent upon specific reaction conditions, or reactants, as these are conventional and well known in the art. It is, however, as previously stated, desired to operate the common settler 14 less than liquid full and at a low pressure, so as to minimize the leakage rate in the event of a failure.

For reasons of brevity, conventional auxiliary equipment such as pumps, additional feed lines, additional heat exchangers, measurement and control devices, etc. have not been included in the above description as they play no part in the explanation of the invention.

The invention is thus broadly applicable to containing heavy liquid in a settler vessel. Various modifications of this invention, such as providing additional chambers for the containment of the acid catalyst, can be made in view of the foregoing disclosure and the appended claims. Such variations and modifications are within the scope of the present invention as claimed.

Claims

1. An apparatus comprising a vertically extending settler vessel (14), having at least one baffle (50, 52) extending wall to wall ad upwardly from the bottom of the vessel to form a plurality of chambers (54, 56, 58, 60), where each chamber is provided with an outlet conduit (26, 27, 28, 29) extending from the bottom of each chamber and a inlet conduit (16, 17, 18, 19) proximate to the upper extension of said baffles, said apparatus further comprising a plurality of reactors (10, 11, 12, 13) connected to the settler vessel (14) by means of the inlet conduits, and an outlet conduit (34) connected to said vessel (14) at an intermediate point above said inlet conduits.

2. Apparatus of claim 1, wherein each of said outlet conduits (26, 27, 28, 29) from said plurality of chambers (54, 56, 58, 60) communicate with coolers (20, 21, 22, 23) associated with each of said plurality of riser reactors (10, 11, 12, 13).

3. A method of operating the apparatus of claim 1 or 2 comprising:

(a) introducing a mixture containing a heavier liquid and a lighter liquid formed in said plurality of reactors (10, 11, 12, 13) through said inlet conduits into said settler vessel (14),
(b) allowing said mixture to separate to form a liquid-liquid interface, where a major portion of said heavier liquid is contained in said plurality of chambers (54, 56, 58, 60).

4. Method of claim 3, further comprising
   (c) operating the settler vessel (14) less than liquid full to form a gaseous phase in a upper portion of said vessel.

5. Method of claim 3 or 4, wherein said reactors (10, 11, 12, 13) are provided as riser reactors for alkylation of olefins with isoparaffins in the presence of a hydrogen fluoride catalyst.

6. Method of claim 5, wherein said heavier liquid comprises a liquid hydrogen fluoride catalyst and said lighter liquid comprises a liquid hydrocarbon product.

7. Method of claim 6, wherein the separated hydrocarbon fluoride catalyst discharged from the outlets of the plurality of chambers is partially recycled to the riser reactors (10, 11, 12, 13) via the coolers (20, 21, 22, 23).

Patentansprüche

1. Vorrichtung, umfassend einen sich vertikal erstreckenden Absetzbehälter (14) mit mindestens einer Trennwand (50, 52), die sich von Wand zu Wand und nach oben vom Boden des Behälters unter Bildung einer Mehrzahl von Kammern (54, 56, 58, 60) erstreckt, wobei jede Kammer mit einer Einlaßrohrleitung (26, 27, 28, 29), die sich vom Boden jeder Kammer erstreckt, und einer Einlaßrohrleitung (16, 17, 18, 19) in der Nähe der oberen Ausdehnung der Trennwände versehen ist, wobei die Vorrichtung ferner eine Mehrzahl von Reaktoren (10, 11, 12, 13) umfaßt, die mit dem Absetzbehälter (14) mittels der Einlaßrohrleitungen (16, 17, 18, 19) und einer Auslaßrohrleitung (34) verbunden sind, die mit dem Behälter (14) an einem Zwischenpunkt oberhalb der Einlaßrohrleitungen verbunden ist.

2. Vorrichtung nach Anspruch 1, wobei jede der Auslaßrohrleitungen (26, 27, 28, 29) von der Mehrzahl der Kammern (54, 56, 58, 60) mit Kühlern (20, 21, 22, 23) in Verbindung steht, die mit jedem der Mehrzahl der Steigrohrreaktoren (10, 11, 12, 13) verbunden sind.

3. Verfahren zum Betrieb der Vorrichtung nach Anspruch 1 oder 2, umfassend:
   (a) Einführen eines Gemisches, das eine schwerere Flüssigkeit und eine leichtere Flüssigkeit umfaßt und in der Mehrzahl der Reaktoren (10, 11, 12, 13) gebildet wurde, durch die Einlaßrohrleitungen (16, 17, 18, 19) in den Absetzbehälter (14);
   (b) Ermöglichen des Trennens des Gemisches unter Bildung einer Flüssigkeits-Flüssigkeits-Grenzschicht, wobei der Hauptteil der schwereren Flüssigkeit in der Mehrzahl der Kammern (54, 56, 58, 60) enthalten ist.

4. Verfahren nach Anspruch 3, ferner umfassend
   (c) Betreiben des Absetzbehälters (14), der weniger als vollständig mit Flüssigkeit gefüllt ist, unter Bildung einer gastförmigen Phase im oberen Abschnitt des Behälters.

5. Verfahren nach Anspruch 3 oder 4, wobei die Reaktoren (10, 11, 12, 13) als Steigrohrreaktoren zur Alkylierung von Olefinen mit Isoparaffinen in Gegenwart eines Fluorwasserstoffkatalysators bereitgestellt werden.

6. Verfahren nach Anspruch 5, wobei die schwere Flüssigkeit einen flüssigen Fluorwasserstoffkatalysator und die leichtere Flüssigkeit ein flüssiges Kohlenwasserstoffprodukt umfaßt.

7. Verfahren nach Anspruch 6, wobei der abgetrennte Fluorwasserstoffkatalysator, der aus den Auslässen der Mehrzahl der Kammern abgelassen wird, teilweise in die Steigrohrreaktoren (10, 11, 12, 13) über die Kühlern (20, 21, 22, 23) zurückgeführt wird.

Revendications

1. Un dispositif comprenant un caisson décanteur s'étendant verticalement (14), comprenant au moins une cloison (50, 52) s'étendant d'une paroi à l'autre et dirigée vers le haut depuis le bas du caisson de manière à former une pluralité de chambres (54, 56, 58, 60), chacune des chambres étant pourvue d'une conduite de sortie (26, 27, 28, 29) s'étendant depuis le bas de chacune des chambres et d'une conduite d'admission (16, 17, 18, 19) située à proximité du prolongement supérieur de ces cloisons,
   ce dispositif comprenant en outre une pluralité de réacteurs (10, 11, 12, 13) reliés au caisson décanteur (14) au moyen des conduites d'admission (16, 17, 18, 19) et une conduite de sortie (34) reliée à ce caisson (14) en un point intermédiaire au-dessus des conduites d'admission.

2. Dispositif de la revendication 1, dans lequel chacune des conduites de sortie (26, 27, 28, 29) provenant de ladite pluralité de chambres
(54, 56, 58, 60) communique avec des refroidisseurs (20, 21, 22, 23) associés à chacun des réacteurs à colonne montante (10, 11, 12, 13) de ladite pluralité.

3. Un procédé de mise en œuvre du dispositif de la revendication 1 ou 2, comprenant les étapes consistant à :
   a) introduire un mélange contenant un liquide lourd et un liquide léger formés dans ladite pluralité de réacteurs (10, 11, 12, 13) par l'intermédiaire des conduites d'admission (16, 17, 18, 19) jusque dans le caisson décanteur (14);
   b) permettre à ce mélange de se séparer de manière à former une interface liquide-liquide, une majeure partie du liquide lourd étant contenue dans ladite pluralité de chambres (54, 56, 58, 60).

4. Procédé de la revendication 3, comprenant en outre l'étape consistant à :
   c) mettre en oeuvre le caisson décanteur (14) au-dessous d'un remplissage complet de liquide, afin de former une phase gazeuse dans une partie supérieure de ce caisson.

5. Procédé de la revendication 3 ou 4, dans lequel lesdits réacteurs (10, 11, 12, 13) sont prévus en tant que réacteurs à colonne montante pour alkyler des oléfines avec des isoparaaffines en présence de fluorure d'hydrogène comme catalyseur.

6. Procédé de la revendication 5, dans lequel le liquide lourd comporte un catalyseur liquide au fluorure d'hydrogène et le liquide léger comporte un produit d'hydrocarbures liquide.

7. Procédé de la revendication 6, dans lequel le fluorure d'hydrogène séparé formant catalyseur rejeté par les sorties de la pluralité de chambres est partiellement recyclé vers les réacteurs à colonne montante (10, 11, 12, 13) via les refroidisseurs (20, 21, 22, 23).