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(72) Inventor: STEVE ARTHUR GEWARTOWSKI

(19) THE PATENT OFFICE LONDON

(54) TEMPERATURE CONTROL OF INTEGRATED FRACTIONATION AND CLAYTREATING OF HYDROCARBONS

(71) We, UOP INC, a corporation organized under the laws of the State of Delaware United States of America, of Ten UOP Plaza, Algonquin & Mt. Prospect Roads, Des Plaines, Illinois, United States of America, 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 relates to an integrated process for the fractionation and clay treating of a hydrocarbon feed stream. The invention more specifically relates to the control of the temperature of the inlet stream to the clay treating zone.

The operations of fractionation and clay treatment of hydrocarbons are both well deveoped and are widely practiced in the petroleum and petrochemical industries. It is normally the heavier hydrocarbons, that is those having six or more carbon atoms per molecule, which are subjected to clay treating rather than lighter hydrocarbons. This segregation is often accomplished by fractional distillation. A common flow scheme therefore consists of the passage of a reaction zone effluent stream into a stripping column followed by passage of the bottoms stream of the stripping column through a clay treating zone. This is illustrated in United States Patent 3,754,045. This reference teaches the passage of the stripping column bottoms directly into the clay treating zone and is concerned with the hydrodealkylation of toluene. The effluent of the clay treating zone is passed into a benzene column. United States Patent 2,733,286 illustrates the clay treatment of a benzene-rich intermediate fraction derived from a thermally cracked heavy naphtha or gas oil. The effluent of this clay treating zone is then passed into a fractionation column used to remove the heavy polymers formed by the polymerization of olefins and diolefins in the clay treating zone.

Another of the common flow schemes utilizing distillation followed by clay treating is shown in United States Patent 2,775,632. In this process a naphtha ia subjected to a reforming operation and the reformate is separated by liquid-liquid extraction to produce a stream rich in aromatic hydrocarbons. This stream is dried, typically by distillation, clay treated and then distilled for the removal of polymers. The result is a nitration grade aromatic product. This reference teaches the use of a temperature of from 275°F. to 375°F. and a pressure sufficient to maintain liquid phase conditions in the clay treating zone. A similar process is described in United States Patent 2,744,942. Previously cited U.S. Patent 2,733,286 increases the range of suitable clay treating temperatures to 250°F. to 400°F., and U.S. Patent 3,835,037 lowers the bottom limit of the range to about 203°F. to 257°F.

The preferred configuration of the operations upstream of the clay treating zone is shown in United States Patent 3,285,986. This reference describes the treatment of the effluent of a thermal hydro-dealkylation zone used to produce benzene from various alkylbenzenes. This effluent stream is first partially condensed and then separated into liquid and vapor phase portions. The liquid phase portion is passed into a stripping column which removes light hydrocarbons overhead to produce a bottoms stream of C_6-plus liquid. The conditions taught for use in the stripping column include a kettle temperature of about 447°F. at a pressure of about 300 psia. Previously cited U.S. Patent 2,778,863 teaches operation of the column directly upstream of a clay treating zone at a pressure of about 100 psig. and a bottom temperature of 380°F. The prior art stripping-clay treating sequence is also illustrated in United States Patent 3,291,850.

In all the prior art systems the bottoms stream from the stripping column is heated or cooled as needed as an entity before being introduced into the clay treating zone.

We have found that good results may be achieved in a temperature control if the bottoms stream from the stripping column is bifurcated into two streams, and one of these streams is cooled and then remixed with the other to adjust the clay treating zone inlet temperature.

The invention thus provides a method of controlling the inlet temperature of a stream of hydrocarbons fed to a clay treating zone from a fractionation column.

According to the invention there is provided an integrated process for the fractionation of a hydrocarbon feed stream and clay treating (as hereinafter defined) of a fractionation effluent stream with control of the temperature of the clay treating inlet stream, which process comprises the steps of:

(a) passing a hydrocarbon feed stream which comprises C_2-C_8 hydrocarbons into a fractionation column operated at effective fractionation conditions including a bottom temperature above a maximum desired inlet temperature of a downstream clay treating zone, effecting the removal of C_2-C_5 hydrocarbons from the hydrocarbon feed stream by fractionation and producing a fractionation column effluent stream having a first temperature which is above an instantaneous preselected inlet temperature for the clay treating zone;

(b) cooling the fractionation column effluent stream to a second temperature by dividing the fractionation column effluent stream into a first portion and a second portion, cooling the second portion by indirect heat exchange against the hydrocarbon feed stream and then recombining the first portion and the second portion of the fractionation column effluent stream;

(c) measuring the resulting second temperature of the fractionation column effluent stream, and comparing the second temperature with the instantaneous preselected inlet temperature for the clay treating zone;

(d) adjusting the relative flow rates of the first portion and the second portion of the fractionation column effluent stream in a manner which changes the second temperature of the fractionation column effluent stream to the instantaneous preselected inlet temperature for the clay treating zone; and

(e) passing the resulting cooled fractionation column effluent stream through the clay treating zone at clay treating conditions including an inlet temperature within the range of from 210°F to 425°F.

Preferably, the effluent stream from the fractionation column is the bottoms product of a stripping column.

A system for use according to the invention for stripping and clay treating a hydrocarbon feed stream comprises indirect heat exchange means located on the transfer line carrying a feed stream to a stripping column, means for bifurcating the bottoms stream of the stripping column into two portions, a control valve for regulating the relative flow rate of these two portions, means to direct one portion of the bottoms stream through the heat exchange means, commingling means to then admix the two portions, and temperature measuring and control means which determines the temperature of the recombined bottoms stream and generates a signal representative of the difference between this temperature and the instantaneous desired inlet temperature of the clay treating zone for transmission to the control valve.

Briefly stated, the essential features are the steps of removing a stream from the stripping column while it is operated with a bottom temperature in excess of the maximum desired inlet temperature of the clay treating zone, cooling this stream by first dividing it into two portions, next cooling one portion by heat exchange against the feed stream to the stripping column and then recombining the two portions, measuring the resulting lower temperature of the recombined stream and comparing the measured temperature to the then desired inlet temperature of the clay treating zone, and adjusting the relative flow rates of the first portion and the second portion of the stream in a manner which changes the measured temperature to the desired inlet temperature. The recombined stream is then passed into the clay treating zone. The desired inlet temperature of the clay treating zone may then be gradually increased during the useful life of the clay until some maximum temperature is reached.

The accompanying drawing depicts a preferred embodiment of the invention. Referring to the drawing a mixture of toluene and hydrogen is charged through line 1 and heat exchanged against a reaction zone effluent stream in a feed-effluent heat exchanger 2. It is then further heated by a furnace not shown and passed through a hydrodealkylation reaction zone 3. The effluent of this reaction zone is passed through the heat exchanger 2 and into a vapor-liquid separator 5 via transfer line 4. A hydrogen-rich recycle gas stream is removed from the separator in line 6, and a condensate stream containing benzene and toluene is removed in line 7. The condensate stream is heated in heat exchangers 8 and 9 and then passed into a stripping column 10. This column is operated with a bottom temperature which is maintained above the maximum desired inlet temperature of the clay treating zone 18 by a reboiler system which is not shown. An overhead stream containing essentially all of the C_1-C_5 hydrocarbons in the condensate stream and a very minor amount of benzene is removed from the stripping column in line 11.

A bottoms stream comprising benzene and toluene is removed from the stripping column in line 12 and is divided into a first portion which passes through line 14 at a rate controlled by valve 15 and a second portion which passes through line 13. The rate of flow of the bottoms stream in transfer line 12 is preferably controlled through use of a valve means 16 located downstream of heat exchanger 8 and operated in response to a level control system 25 in the bottom of the stripping column. The second portion of the bottoms stream is cooled in heat exchanger 9 by exchange against the condensate stream and is then admixed with the first portion of the bottoms stream. This recombined bottoms stream is carried to the clay treater 18 by line 17. The temperature of this stream is monitored by a temperature sensor and control means 24 which generates a signal carried by means 23 to the valve 15. This signal is based on a comparison of the actual temperature of the cooled bottoms stream to the then desired clay treater inlet temperature which is performed by means 24. The bottoms stream emerges from the clay treater as an effluent stream in line 19. This effluent stream is first cooled by heat exchange against the condensate stream and is then passed into a benzene column 20. This column separates the effluent stream into an overhead stream carried in line 21, a benzene sidecut stream removed through line 26 and a bottoms stream comprising toluene and polymers from the clay treating operation removed in line 22.

Required subsystems and assemblies such as valves, control systems, reboilers, condensers and fractionator internals have been omitted from the drawing for the purposes of simplicity and clarity. This description of one preferred embodiment is not intended to limit the scope of the invention. As indicated below, various modifications to the control system, reaction system and fractionation train may be made while still retaining the operational characteristics of the invention.

Clay treating is used to remove impurities from hydrocarbons in wide variety of processes in the petroleum and petrochemical industries. The materials which are beneficially affected by this operation range from lube oils and benzene to pharmaceutical products and normal paraffins. One of the most common reasons for clay treating these materials is the removal of olefinic materials, sometimes called color bodies, in order to meet various quality specifications. As used herein the term olefinic compound of olefinic material is intended to refer to both mono and diolefins. Olefinic materials may be objectionable in aromatic hydrocarbons at even very low concentrations of about a few parts per million. Their presence is readily shown by the well known acid wash color test. Removal of these olefinic materials is, for instance, necessary for the manufacture of nitration grade aromatics including benzene, toluene and xylenes. Clay treating is also used to reduce the bromine index of various hydrocarbon products. This beneficial utility of clay treating is further demonstrated by the references wich were previously cited.

The term "clay treating" is used herein to refer to the passage of a liquid phase hydrocarbon stream through a fixed bed of contact material which possesses the capability of polmerizing or oligomerizing olefinic compounds present in the hydrocarbon stream, and the term "clay treating zone" has a corresponding meaning. Preferably the contact material is an acidic aluminosilicate. It may be either a naturally occurring material, such as bauxite or mordenite clay, or a synthetic material and may comprise alumina, silica, magnesia or zirconia or some other compound which exhibits similar properties. Several types of clay are available commercially and are suitable for use herein. Typical of these are Filtrol 24, Filtrol 25 and Filtrol 62 produced by the Filtrol Corporation, Attapulgus clay and Tonsil clay.

Clay treating is now conducted over a wide temperature range generally extending from 203 °F. to 425°F. or more, although the inlet temperature of the hydrocarbon stream is required by the invention to be from 210°F to 425°F. The exact temperature utilized in the clay treating zone is dependent on at least three separate factors. The first of these is the minimum temperature which is required for the contact material to function properly. This temperature is known to increase in a positive relation to the quantity of hydrocarbons which have been treated per unit mass of contact material. The minimum required temperature is therefore affected by the prior use of the clay. A second factor is the particular type of contact material which is being used. This is related to the minimum required temperature, but is an independent factor since individual contact materials exhibit differing degrees of selectivity and other properties, such as useful life, which may be taken into account. For instance, at the same level of color body removal activity two different clays may have varying degrees of catalytic activity for undesired reactions as described below. Finally, the optimum clay treating temperature will be dependent on intrinsic and extrinsic qualities of the hydrocarbon stream being treated. These qualities include the rate of flow of the hydrocarbon stream and the concentration of olefinic compounds in it. The individual hydrocarbon species which is being treated will also be a material factor. For instance, the optimum temperature for use with normal paraffins may well differ from that for use with aromatics, and individual aromatics will also differ in the maximum temperature which may be imposed. For example, relatively stable benzene can normally be subjected to higher temperatures without adverse consequences than alkylaromatics such as ethylbenzene.

The preference for specific clays and the criticality of temperature is illustrated by the following experimental results of various clay treating tests. The purpose of the tests was to compare the clays for the desired ability to polmerize olefinic compounds and for the undesired tendency to cause the transalkylation of aromatic hydrocarbons. These tests were conducted at a pressure of 500 psig., a liquid hourly space velocity of 1.0 and with upward liquid flow. The variables examined were three clay types and temperature. In one test Filtrol 24 clay was used to treat C_8 aromatic fraction. This clay exhibited an excellent ability to reduce the bromine index of the aromatics from a value of 1070 to a normal value of from 3 to 7. It was found that the activity of this clay to promote transalkylation, as measured by the increase, in weight percent, of benzene and toluene, was directly related to temperature and increased from about 0.4% at 300°F. to about 2.0% at 448°F. However, in continuous operation at 425°F. the transalkylation rate decreased from about 1.8% at 46 hours to about 1.0% at about 122 hours of operation. To further investigate the transalkylation properties of this clay a feedstock containing 93.6 wt.% diethylbenzene and 0.2 wt.% triethylbenzene was also treated under these same conditions. The sum of the ethylbenzene and the triethylbenzene in the clay treater effluent was about 4.7 wt.% at about 324°F. and increased to about 26.4 wt.% at 376°F.

A similar series of tests was performed using a Tonsil clay at the same conditions. This clay also demonstrated excellent olefin removal capabilities by reducing the bromine index of the effluent to a value of about 5 or less. The extent of the accompanying transalkylation was surprisingly low however when compared to the Filtrol clay. For instance, the weight percent of benzene and toluene in the effluent due to the clay treating was below 0.1 at temeratures of 350°F. and below and reached only 0.9 at 450°F. When charging the diethylbenzene feedstock, the effluent contained about 1.4 wt.% ethylbenzene and triethylbenzene at 325°F. and a maximum of 9.7 wt.% at 376°F.

Attapulgas clay was also tested with the C_8 aromatic feedstock. This clay produced an effluent having a bromine index of about 59 at 325°F. and of about 47 at 425°F. Its activity for olefin removal was therefore measurably less, but these same tests indicate its activity for the undesirable transalkylation reaction was also lower. The analysis showed only a trace of benzene and toluene indicating that essentially no transalkylation occurred.

This test data indicates the importance of using the proper clay treater inlet temperature and of being able to control and adjust this temperature during the life of the clay. The probability of fluctuations in the operation of the fractionation column which is producing the stream fed to the clay treater require the use of a control system which is not dependent on the control system of the fractionation column. Furthermore, if the clay treater is operated at a higher temperature than the upstream fractionator it is necessary to heat the clay treater feed stream to this elevated temperature. This requires the use of a separate heater and heat source, an undesirable requirement. It is therefore an objective of the present invention to provide a method for controlling at a preselected temperature a hydrocarbon stream which has been removed from a fractionation column and is being passed into a clay treater.

The present invention involves, inter alia, operating the fractionation column at an increased pressure which requires the use of a bottoms temperature greater than the maximum clay treater inlet temperature which will be used. This high bottoms temperature is preferably maintained constant by a control system regulating the operation of the reboiler. The next step in the method is to cool the fractionation column bottoms stream by the unique operation of bifurcating the bottoms stream and then heat exchanging one portion of it against the relatively cool material being charged into the fractionation column. The amount of this portion will vary with such changeable factors as the instantaneous preselected clay treater inlet temperature, the temperature of the feed to the fractionation column, and the temperature and flow rate of the bottoms stream itself. For instance, as the activity of the clay decreases and the preselected temperature is gradually increased to compensate, the flow rate of the cooled portion of the bottoms stream will be adjusted downward to raise the clay treater inlet temperature.

Although the fractionate column could be operated at a temperature which would not require cooling of the bottoms stream when operating the clay treater at its maximum desired inlet temperature we choose to maintain a higher temperature in the column such that some cooling of the bottoms stream is required at all times. This makes control of the temperature more stable and allows room for adjustment. Specifically, it is preferred that the bottom temperature of the column is set at least 15°F. above the maximum foreseeable desired inlet temperature for the clay treater. Furthermore, it is preferred that at all times at least 5-10 wt.% of the bottoms stream is split off for heat exchange. The maximum amount of the bottoms stream which is split off and cooled is preferably less than 50 wt.% but may be higher.

While this description is given both above and below in terms of passing a stripping column bottoms stream through a clay treater, it is not intended to limit the scope of the invention to the use only of such a bottoms stream. The method according to th invention may also be applied to other stripping column effluent streams, such as side-cut streams, which are withdrawn at a temperature above the clay treater inlet temperature. It may also be applied to the bottom streams of fractionation colums not properly referred to as stripping columns. The design and other operational aspects of the fractionation column may be as is customary and established in the art.

After being cooled, the split-off portion of the bottoms stream is admixed with the uncooled portion of the bottoms stream and the temperature of the resulting stream is measured. This temperature is compared to the clay treater inlet temperature which is desired at this point in time, and the control valve which is regulating the division of the bottoms stream is then adjusted as needed to control the relative rates of flow of the two portions and hence to bring the measured temperature to the desired temperature. The control valve, heat exchanger, and temperature sensing and control means used for this may be any of the conventional devices known to those skilled in the art. All of the control system components are preferably utilized as shown in the drawing, i.e. the rate of the bottoms stream division is preferably set by a control valve in the transfer line carrying the uncooled portion of the bottoms stream, and the rate of flow of the total bottoms stream is regulated by a level control means which signals a control valve in the transfer line carrying the clay treating zone effluent from the first heat exchange means. Other systems may be used to practice the method of the invention. For instance, the flow rate of the total bottoms stream may be regulated by a valve means in line 12 or the temperature of the resulting admixture could be determined by temperature measurements of both portions of the bottoms stream. Other possible variations include using a temperature measured within the clay treating zone or the temperature of the clay treating zone effluent to control the cooling of the bottoms stream.

The clay treating zone may be of any type and configuration which is effective in achieving the desired degree of purification. It may utilize either upward or downward flow, downward flow being preferred. The pressure in the clay treating zone should in general be sufficient to maintain liquid phase conditions. This will normally be a pressure of from 50 to 500 psig. or higher. Preferably the pressure is set about 50 psig. higher than the vapor pressure of the hydrocarbons at the inlet temperature of the zone. This inlet temperature is within the range of from 210°F. to 425°F. Clay treating may be performed over a broad range of liquid hourly space velocities. This variable is often set by the desired on-stream life of the clay and may range from 0.5 or lower to about 10. Preferred are liquid hourly space velocities of from 1.0 to 4.0 depending on the material being treated. Two separate clay treater vessels may be used on an alternating basis to provide continuous operation. The effluent of the clay treating zone is preferably heat exchanged with the condensate stream being charged to the fractionation column. This recovers a sizable amount of heat and reduces the column's reboiler duty.

The invention is one of general application. That is, it may be applied to processes which differ in either their upstream or downstream configuration from that illustrated in the drawing. The upstream reaction zone may perform a different purpose, such as the catalytic reforming of a naphtha or the isomerization of alkyl-aromatic hydrocarbons, including xylenes (in which case p-xylene will be present in the feed stream), instead of vapor phase hydro-dealkylation. The invention may also be utilized as part of a feed pretreatment operation which is upstream of a reaction zone.

There are two other advantages to the high temperature fractionation operation of the invention. First, the recovery of heavier components, such as benzene, in the bottoms stream of a stripping column is greater than at a lower pressure and temperature. Second, the overhead vapors of the column are at higher temperatures which allow more efficient utilization of their heat content. For instance, they can be used to generate steam or as a heating fluid for the reboiler of another column. While these two advantages to high temperature operation have been known for some time, the synergy of their benefits with those of the invention overcome the disadvantages of high temperature operation.

The invention is further illustrated by the following Example based on a catalytic dealkylation unit designed for the production of benzene from toluene and heavier aromatics.

Example

At the beginning of the run the desired inlet temperature to the clay treating zone is 350 °F., and the end-of-run desired inlet temperature is 420°F. The reaction zone effluent is cooled to form a net condensate stream which is separated from the remaining vapors. This condensate stream is produced at a rate of about 4,706 BPD (barrels per day) and is depressured to about 260 psig. to form the mixed-phase feed stream to a stripping column. This stream enters the first heat exchanger at a temperature of about 100°F. It is therein raised in temperature to about 182°F. by heat exchange against the clay treating zone effluent stream. Heat exchange in the second exchanger against a portion of the stripping column bottoms further heats this stream to about 288°F., and it is then passed into the stripping column at the top tray. The stripping column is operated with an overhead vapor temperature of 300°F. at a pressure of about 250 psig. About 34.8 moles per hour of gases are vented from the column's overhead receier. Of ttis about 66 mole percent is methane, about 20 mole percent is ethane and about 12 mole percent is hydrogen. The stripping column is operated with a bottom liquid temperature of about 437°F. The net bottoms stream has a flow rate of about 4,567 BPD and is cooled to 350°F. by heat exchanging a split-off portion of it against the feed to the stripping column as described above and then recombining the portions.

Passage of the bottoms stream through the clay treating zone produces a clay treating zone effluent stream hhaving a temperature of about 340°F. and a pressure of about 230 psig. This effluent stream is cooled to approximately 272°F. by heat exchange against the reaction zone condensate stream. It is then passed into a benzene column operated to produce an overhead vapor having a temperature of about 190°F. at a pressure of about 6 psig. A net benzene stream of about 3,379 BPD is removed as a sidecut. The net bottoms stream contains about 41 BPD of heavy aromatics which are rejected and about 1147 BPD of aromatics which are recycled to the reaction zone.

When operating at end-of-run conditions, the reaction zone condensate stream is heated to about 267°F. in the first heat exchanger by the clay treating zone effluent. Passage through the second heat exchanger again raises its temperature to about 288°F. prior to passage into the stripping column, which is operated at the same conditions. The stripping column bottoms stream is removed at the same temperature of about 437°F., but is only cooled to 420°F. by the heat exchange of a smaller portion against the condensate stream. The clay treater effluent emerges at a temperature of about 410°F., and it is then cooled by heat exchange against the condensate stream down to the benzene column feed temperature of about 272°F.

WHAT WE CLAIM IS:

1. An integrated process for the fractionation of a hydrocarbon feed stream and clay treating (as hereinbefore defined) of a fractionation effluent stream with control of the temperature of the clay treating inlet stream, which process comprises the steps of:

(a) passing a hydrocarbon feed stream which comprises C_2-C_8 hydrocarbons into a fractionation column operated at effective fractionation conditions including a bottom temperature above a maximum desired inlet temperature of a downstream clay treating zone, effecting the removal of C_2-C_5 hydrocarbons from the hydrocarbon feed stream by fractionation and producing a fractionation column effluent stream having a first temperature which is above an instantaneous preselected inlet temperature for the clay treating zone;

(b) cooling the fractionation column effluent stream to a second temperature by dividing the fractionation column effluent stream into a first portion and a second portion, cooling the second portion by indirect heat exchange against the hydrocarbon feed stream, and then recombining the first portion and the second portion of the fractionation column effluent stream;

(c) measuring the resulting second temperature of the fractionation column effluent stream, and comparing the second temperature with the instantaneous preselected inlet temperature for the clay treating zone;

(d) adjusting the relative flow rates of the first portion and the second portion of the fractionation column effluent stream in a manner which changes the secnd temperature of the fractionation column effluent stream to the instantaneous preselected inlet temperature for the clay treating zone; and

(e) passing the resulting cooled fractionation column effluent stream through the clay treating zone at clay treating conditions including an inlet temperature within the range of from 210°F to 425°F.

2. A process as claimed in Claim 1 wherein the fractionation column effluent stream is the bottoms stream of the fractionation column.

3. A process as claimed in Claim 1 or 2 wherein after passage through the clay treating zone the fractonation column effluent stream is heat exchanged against the hydrocarbon feed stream to effect the heating of the hydrocarbon feed stream at a point prior to the heat exchange of step (b)

4. A process as claimed in any of Claims 1 to 3 wherein the hydrocarbon feed stream comprises aromatic hydrocarbons,

5. A process as claimed in in any of Claims 1 to 4 wherein the hydrocarbon feed stream results from the partial condensation and vapor-liquid separation of a vapor phase hydrodealkylation reaction zone effluent stream.

6. A method as claimed in in any of Claims 1 to 4 wherein the hydrocarbon feed stream results from an effluent stream of an alkylaromatic hydrocarbon isomerization zone and comprises paraxylene.

7. A method as claimed in Claim 1 carried out substantially as hereinbefore described with reference to the accompanying drawing.

8. A method as claimed in Claim 1 carried out substantially as described in the foregoing specific Example.

J.Y & G.W. JOHNSON.
Furnival House.
14-18 High Holborn.
London WC1V 6DE.
Chartered Patent Agents.
Agents for the Applicants.

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