

PATENT SPECIFICATION

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(54) HYDROCARBON SEPARATION PROCESS

(71) We, UNIVERSAL OIL PRODUCTS COMPANY, 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, 60016, 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 field of art to which the invention pertains is hydrocarbon separation. More specifically this invention relates to a simulated moving bed separation process in which a fixed mass of adsorbent and a varying concentration of components of a fluid stream contact each other in a countercurrent flow fashion.

The prior art contains references to countercurrent separation operations using a fixed bed of adsorbent in which a fluid inlet is shifted though the adsorbent to simulate a moving bed. Thus there is flow of adsorbent with respect to fluid circulating in the process. Specifically the prior art discloses the basic concept of a simulated countercurrent solid-fluid contacting process employing a fixed bed of solid adsorbent having moving input and output streams which allows a segregation of zones in which separate functions are taking place in order to separate a feed into a raffinate product and an extract product. It is also known to use a rectification zone, located between the feed inlet and the extract outlet, in which a sweeping agent is passed into the process to push raffinate material, trapped in the void spaces between adsorbent particles, back into an adsorption zone. This prevents raffinate material from passing from the adsorption zone into the desorption zone, which would contaminate extract product with raffinate material. The sweeping agent used is selected to be easily separable from the feed stock by distillation.

The prior art has also disclosed the re-

cycling of a raffinate material to a second desorption zone to remove desorbent from the adsorbent passing to an adsorption zone. The purpose of recycling raffinate is to totally remove desorbent from the adsorbent before it has the opportunity to contact feed components passing into the adsorption zone via a charge stream. Thus the recycled raffinate material is passed into an adsorption zone at a location between feed stock introduction and raffinate withdrawal points. The method of the present invention allows an adsorption zone to be operated by charging solely feed to the adsorption zone while removing a raffinate stream from that zone. There is no recycling of raffinate material to the adsorption zone of the invention herein presented.

The process of the present invention relates to a simulated moving bed countercurrent processing scheme in which zones are shifted through the adsorbent to allow various portions of the adsorbent to function as adsorption and desorption zones. A purification stream containing extract material is passed into the rectification zone (1) to displace from the interstitial spaces in the adsorbent bed any raffinate materials carried into this zone, (2) to remove feed contaminants adsorbed by the adsorbent, and (3) to substantially reduce the quantity of desorbent which normally surrounds the adsorbent particles in this zone when there is no purification stream used. Since in normal operations the material removed from the desorption zone is passed into the rectification zone, desorbent material can contact the adsorbent in that zone. The adsorbent present in the zone located between the adsorption and desorption zones contains much adsorbed extract yet the fluid surrounding the adsorbent contains relatively little extract. The presence of desorbent reduces the ability of the adsorbent to selectively remove this small quantity of extract material from the fluid surrounding the adsorbent. To overcome this problem, the present invention

provides a purification stream, containing a relatively high percentage of extract material, which is passed into the rectification zone to replace the desorbent which, in prior art processes, would enter this zone.

The extract passed into the intermediate zone may be either the extract first removed from the desorption zone, since this material is almost pure extract with little desorbent present, or from extract recovered in an extract stream fractionator.

It is an object of the present invention to pass a portion of a relatively pure extract stream into a rectification zone to allow raffinate present in that zone (both adsorbed by the adsorbent and within the voids) to be displaced from that zone by extract. It is another object of the present invention to pass extract material into the intermediate zone to reduce the quantity of desorbent in this zone.

Accordingly, the present invention provides a simulated moving bed process for separating a hydrocarbon feed stream into extract and raffinate with an adsorbent selective for said extract, said process comprising the steps of: (a) contacting said feed with a bed of adsorbent in a first adsorption zone and selectively retaining extract and a portion of raffinate, and (b) contacting at least a portion of adsorbent from step (a) with a desorbent and displacing from said adsorbent a stream comprising extract, and including the improvement which comprises contacting at least a portion of the adsorbent from step (a) with a purification stream comprising substantially desorbent-free extract before said adsorbent from step (a) is contacted with said desorbent in step (b) of the process.

Either the extract or raffinate streams may contain much or only a little desorbent and still be considered extract and raffinate streams.

For example, when the feed to the hydrocarbon separation process of the present invention is a mixture of C_6 aromatic hydrocarbons, para-xylene (p-x) may be selectively retained by the adsorbent and would be considered extract, whether almost pure p-x or p-x with a desorbent. In this example, the raffinate would be meta-xylene, ortho-xylene and ethylbenzene (m-x, o-x and EB), whether pure or diluted with desorbent. Similarly, the purification stream would be substantially pure p-x, though a small amount of desorbent may be present.

The "selective volume" of the adsorbent is the volume of the adsorbent which absorbs extract from the feed. The "non-selective volume" is the volume of the adsorbent which does not selectively absorb extract from the feed. This volume includes both cavities of the adsorbent which contains no adsorptive sites and the interstitial volume of the adsorbent particles.

When adsorbent "passes" into an opera-

tional zone its selective and non-selective volumes carry fluid into that zone. The non-selective volume determines the amount of fluid which should be added to this zone to displace the fluid present in the non-selective volume. If the fluid added does not flush out the non-selective volume, there is a net entrainment of liquid into the zone by the adsorbent. This entrainment is undesirable because the entrained liquid frequently contains raffinate.

The selective volume of the adsorbent can, in certain instances, adsorb raffinate. There is competition between extract and raffinate for adsorptive sites in the adsorbent. If a large quantity of raffinate is present in the void volume, and very little extract is in the void volume, some raffinate may be adsorbed by the adsorbent.

Three separate operational zones are needed for the process of the present invention although an optional fourth zone may also be used. In the adsorption zone (zone 1) adsorbent contacts the feed and adsorbs an extract material from the feed stock. Non-retained components of the feed are withdrawn from zone 1 as a raffinate stream. The raffinate composition varies from essentially pure desorbent, present either in the adsorbent's selective or non-selective volume from a previous desorption step, to essentially 100% raffinate material. Zone 1 may be defined as the adsorbent located between the feed inlet and the raffinate outlet. The general flow in zone 1 is considered to be a down-stream direction when proceeding from the feed to the raffinate streams.

Immediately upstream of zone 1 is zone 2 which is a purification zone. The boundaries of zone 2 are defined by the extract outlet and the feed inlet. The essential operations taking place in this zone are: (1) the displacement from the non-selective volume of any raffinate material carried into zone 2 by the shifting of adsorbent into this zone; and, (2) the desorption of any raffinate material adsorbed within the selective volume of the adsorbent. Purification is effected with extract. The flow in zone 2 is toward the downstream boundary, the feed stream inlet.

Immediately upstream of zone 2 is zone 3, the desorption zone. A desorbent is charged to this zone to displace extract adsorbed upon the adsorbent during a previous contact with feed. Zone 3 is defined by its upstream boundary, the desorbent inlet, and its downstream boundary, the extract outlet. The direction of fluid flow in zone 3 is the same as in zones 1 and 2.

In some instances a fourth zone may also be used. This zone, if used, is located immediately upstream of zone 3. Zone 4 conserves the amount of desorbent needed in the desorption step, because raffinate removed from zone 1 is passed into zone 4 to displace de-

sorbent from that zone into the desorption zone 3. Zone 4 contains enough adsorbent to prevent raffinate from passing out of zone 1 into zone 3 via zone 4. If raffinate from zone 1 entered zone 3, the extract removed from zone 3 would be contaminated. If the fourth zone is not used, raffinate withdrawal rate is carefully monitored to prevent flow of raffinate material from zone 1 into zone 3. The raffinate stream withdrawn from zone 1 will also be fractionated to separate desorbent from raffinate and at least a portion of the fractionated desorbent will be sent to zone 4. This step requires additional fractionation facilities and results in the inefficient use of desorbent material.

In adsorptive-separation processes, an important factor is the selectivity of the adsorbent for one component when compared to another component. The selectivity (B) is defined by the following equation:

$$\text{Selectivity} = B_{x/y} = \frac{\left(\frac{x}{y}\right)}{\left(\frac{X}{Y}\right)}$$

where B represents the selectivity, x and y represents the volume % of components being compared, a and u represent the adsorbed and unadsorbed phases. The selectivity is measured at equilibrium conditions, i.e., when the feed passed over a bed of adsorbent does not change in composition after contacting the bed of adsorbent.

In adsorptive-separation processes the separation of aromatics such as xylenes or diethylbenzene can be effected with crystalline aluminosilicate adsorbents. Common crystalline aluminosilicates which can effectively separate aromatics are the synthetically prepared type X and Y zeolites.

The type X and Y zeolite adsorbents preferably contain at their exchangeable cationic sites cations selected from potassium, rubidium, cesium, barium, copper, silver, lithium, sodium, beryllium, magnesium, calcium, strontium, cadmium, cobalt, nickel, manganese and zinc or combinations thereof.

The desorbent used in the process of this invention should be easily separable from the feed. In desorbing the extract, both desorbent and extract are removed from the adsorbent together. Separation of these two materials is required to increase the purity of the extract. Thus desorbents of a different boiling range than the feed are preferred to allow fractionation to be used to separate extract from desorbent and allow reuse of the desorbent in the process. When the desired product is a xylene isomer, desorbents which can be used

include benzene, toluene, ethers, alcohols, chlorinated cyclics, cyclic dienes and the ketones. Xylenes can be used as desorbents for diethylbenzene feed stocks while diethylbenzene can be used as desorbents for xylene feed stocks.

Both liquid and vapor phase operation is possible, though liquid phase operation is preferred because of the lower temperature requirements and slightly improved selectivities resulting from lower temperatures. Temperatures which can be used in adsorption of a xylene isomer include about 40 to 250° C. Pressures may range from about 1 to 35 atm. Desorption conditions include the same range of temperatures and pressures as used for adsorption.

The present invention is especially useful in processing feed streams comprising at least two components selected from the diethylbenzenes or xylenes including ethylbenzene with the possible inclusion of portions of straight and branched-chain paraffins, cycloparaffins and aromatics including benzene, toluene, naphthalenes, and similar materials. When separating aromatic isomers, it is preferred to use feed streams containing about 80 to 100 volume % aromatics.

The attached drawing shows one specific manner of operating the process of this invention. In the attached drawing there are four separate operating zones including an optional fourth zone. The drawing represents a fixed bed or series of fixed beds of adsorbent having input and output streams connected to the individual beds. The liquid flow in the beds is upward and the adsorbent remains stationary. A countercurrent flow of fluid, with respect to the solid adsorbent, is simulated by shifting the individual input and output streams after each period of operation.

Some of the zones 1, 2, 3 and 4 may require more adsorbent than other zones or all may have the same amount. Three basic zones are required to effect a separation: an adsorption zone, a purification zone and a desorption zone. An optional fourth zone prevents contamination of the extract with raffinate from zone 1. Terminal zones 1 and 4 are connected by lines 10 and 11 which allow fluid from zone 1 to enter zone 4, or zone 3 if optional zone 4 is not used. New fluid flow is in an upward direction, but in some instances a zone may be operated to allow flow of fluid, for a short time, in a direction opposite to the net flow of fluid. Input and output streams 5, 6, 7, 8 and 9 are shifted, in the same direction as the net fluid flow, after each cycle of operation. Normally the shifting of the input and output streams along the fixed bed of adsorbent is done simultaneously and in the same distance along the bed of adsorbent. In other instances, two or more zonal functions may take place in

the adsorbent between two input and output streams before the input and output streams are shifted.

In the drawing, zone 1 is an adsorption zone defined by the feed line 6 and the raffinate line 5, connected to zone 1 via line 11. The purification zone, zone 2, is defined by the extract line 8 and feed line 6. The desorption zone, zone 3, is defined by the desorbent inlet 9 and extract line 8. The optional zone 4 is located between the raffinate line 5 and the desorbent line 9.

Pumps and valves are not shown. A pump may be located on one or more of the lines to induce an overall flow. The systems hydraulic characteristics may be designed to impose a flow in one direction through the system, or flow directing devices such as check valves may be located between the zones to impose an overall flow in one direction.

Feed enters the process via line 6. Since the overall flow is upward, any material from zone 2 in line 12 enters zone 1. Extract, and some raffinate, is adsorbed within the selective volume of the adsorbent. The material which is adsorbed frequently must desorb desorbent already present in the selective volume of the adsorbent. It is possible to have desorbent-free adsorbent by allowing essentially pure raffinate from zone 1 to contact adsorbent in zone 4 to displace desorbent from adsorbent which in the next cycle will be part of the adsorption zone.

As feed enters zone 1 an equal volume of raffinate is displaced from zone 1 into line 11. A portion or all of the raffinate in line 11 may be recovered via line 5 with the remainder sent via line 10 into either zone 3 or zone 4, if used.

The recycle stream is passed into the purification zone 2 via line 7. Line 7 may be anywhere in zone 2, but it is preferred that line 7 be located so that there is some adsorbent located between line 7 and line 6.

Zone 2 removes raffinate from adsorbent. When the adsorbent passes into the purification zone from the adsorption zone it generally contains some raffinate in both its selective and non-selective volume. The raffinate is removed from the adsorbent by contact with the extract or purification stream. In the prior art process, the adsorbent would contact a mixture of extract and a substantial amount of desorbent, and this mixture would remove raffinate from the adsorbent. A portion of extract also enters purification zone 2 from zone 3 via line 13. The purification stream of the present invention allows removal of raffinate from the adsorbent while substantially reducing or totally eliminating the quantity of desorbent which enters the purification zone. Elimination or substantial reduction of desorbent in the purification zone enhances the

adsorbent's ability to adsorb extract from the fluid surrounding the adsorbent.

Fluid flow in zone 2 may be regulated by controlling the quantity of purification material entering via line 7, the material entering via line 13, or the amount of material leaving via line 12.

Zone 3, immediately upstream of zone 2 with respect to the principal fluid flow in zone 2, allows recovery of extract adsorbed by the adsorbent in zones 1 and 2. Desorbent enters zone 3 via lines 9 and 14 and desorbs extract from the selective volume of the adsorbent. Extract is recovered from zone 3 via lines 13 and 8. Fluid flow in zone 3 is therefore from line 9 towards line 8. The adsorbent in zone 3, especially at the upstream end near line 9, contains essentially pure desorbent in both its non-selective and selective volumes.

Zone 4 can be used to both reduce the desorbent requirement of the process and prevent the contamination of extract with raffinate. When zone 4 is used, raffinate not removed via line 5 will enter zone 4 via lines 10 and 11 to displace desorbent from the non-selective volume of the adsorbent. Desorbent will simultaneously be displaced from zone 4 via line 14 into zone 3. The displaced desorbent from zone 4 tends to reduce the requirement for fresh desorbent from line 9.

When zone 4 is not used, the material which leaves zone 1 via line 11 and which by-passes line 5 should be raffinate free. The initial fluid withdrawn from zone 1 contains a very high concentration of desorbent and may safely be passed from zone 1 to zone 3. As this fluid contains little or no raffinate, there will be diminished or stopped withdrawal of fluid via line 5. When the fluid withdrawn from zone 1 contains an appreciable quantity of raffinate, flow to zone 3 is stopped and the raffinate is withdrawn via line 5. While raffinate is withdrawn via line 5, desorbent may be passed into zone 3 via line 9.

The material initially taken out via line 8 is almost pure extract. This extract may be totally withdrawn from the process via line 8, or a part of the extract may enter zone 2 via line 13 to flush the adsorbent in zone 2. After a period of time there is an appreciable quantity of desorbent present in the extract withdrawn from zone 3 via line 13. This material can then be withdrawn from the process via line 8. Thereafter, line 13, which connects line 8 to zone 2, can be blocked off and an external purification stream of extract can enter zone 2 via line 7. Extract withdrawn from zone 3 via line 8 may also be fractionated to separate desorbent from extract, with a portion of the extract then recycled via line 7 to zone 2. Thus extract may pass into zone 2 via line 13, with the extract stream passing out of line 8 essen-

tially stopped, or if appreciable desorbent is present, an external recycle purification stream may be used in zone 2 or some combination of both schemes. The overall effects of the recycle stream are twofold:

1) reduction of the adverse influence of desorbent on selectivity of the adsorbent, especially where there are relatively small quantities of extract present in the non-selective volume, and it is desired to adsorb the last trace portions of extract material; and

2) passage of a purification stream into zone 2 displaces from the downstream portion of this zone raffinate which is present in the non-selective volume of the adsorbent. This raffinate is displaced into zone 1 via line 12.

A cyclic advancement of the input and output streams through the fixed bed can be accomplished with a manifold in which the valves are operated in a sequential manner to shift input and output streams in the same direction as the overall fluid flow and thus simulate countercurrent flow of fluid with respect to adsorbents. A rotating disc valve in which the input and output streams are connected to the valve and the lines through which inlet and outlet streams pass are advanced in the same direction through the bed. Both the manifold arrangement and rotating disc valve are known in the art.

Adsorbent is, in effect, shifted during operation, in order to allow the adsorbent to remove extract from the feed in an adsorption zone and to recover the extract in a desorption zone. It may also be desirable to purge the void volume of the adsorbent as it, in effect, moves from zone to zone. This will prevent contamination of fluid in the new zone by fluid from the old zone.

It is desirable in many instances to have one or more input or output streams in a zone which remain dormant during the operations taking place in that zone. There may be one or more lines connected to the adsorbent in the same manner as one of the operating input or output streams so that when a period of operations is completed the input and output streams are simply shifted to the next dormant line in a downstream direction. Thus shifting the input and output streams permits constant production of raffinate and extract along with the constant use of feed and desorbent streams and the purification recycle stream.

In this example comparisons were made between fixed bed countercurrent processes with and without purification streams.

The apparatus used in these experiments consisted of a column containing 24 individual beds of adsorbent connected in series. Transfer taps between the beds allowed material to enter or leave the process.

The entire apparatus contained approxi-

mately 42.7 l (11.3 gal or U.S. gallons) of crystalline aluminosilicate adsorbent. The sieves contain about 6.2 l (1.64 gal) of selective volume or pore volume and about 25.4 (6.71 gal) of non-selective volume or void volume.

All experiments were conducted at about 177° C (350° F) and a pressure of about 14.6 atm (200 psig). The feed was a xylene mixture containing about 14.4 wt % p-x, 35.1 wt % m-x, 18.1 wt % o-x, 31.9 wt % BB, 0.4 wt % isopropyl benzene, and 0.1 wt % butylbenzene.

The adsorbent used in all experiments was a 13 X type zeolite which had been ion-exchanged with both barium and potassium. The adsorbent, prior to its use in the experiments was dried at an elevated temperature to reduce the water content of the zeolite.

The desorbent used in the experiments was a mixture of DEB (diethylbenzene) isomers consisting of 60.4 LV% m-DEB (liquid volume % meta-diethylbenzene), 7.4 LV% o-DEB (ortho-diethylbenzene), 25.6 LV% p-DEB (para-diethylbenzene) and 6.6 LV% BB (butylbenzene). The sole purpose of these experiments was to determine the advantages of a purification recycle stream. No attempt was made to determine optimum operating parameters with respect to temperatures, pressures, adsorbent, types of desorbents utilized, or other feeds that may be processed.

The apparatus used contained four separate operating zones.

A particular description of the apparatus will now be given, proceeding in a downstream direction from the raffinate outlet, line 5, into zone 4. Zone 4 contains four beds connected in series. At the downstream boundary of zone 4, line 9, is the desorbent inlet. Next is zone 3 containing six adsorbent beds in series. At the downstream boundary of zone 3, line 3, is the extract outlet. Next is zone 2 containing 8 adsorbent beds. The purification inlet, line 7, is one bed removed from line 8. Downstream of line 7 are six adsorbent beds. Downstream of these six adsorbent beds is a flush inlet, not shown, followed by one additional bed downstream of the flush inlet. Thus zone 2 contains eight beds, one bed between lines 7 and 8, six beds between line 7 and the flush inlet, and one bed between the flush inlet and line 6. The flush stream removed feed components from the line through which feed had passed after the feed had been shifted to its new location downstream. This prevented the extract from being contaminated with raffinate. Zones 1 and 4 were connected by a line 10 which contained a pump which circulated fluid from zone 1 to zone 4. Check valves between the adsorbent beds may also be used to insure that net fluid flow through the bed is in one direction.

In the operations in which no purification

stream was used, line 7, was omitted from the process and the remaining input and output streams were as described above.

- 5 In all the experiments conducted in this example the total cycle of operations around the 24 adsorbent beds required about $\frac{1}{2}$ hour,

or 1.25 minutes of operation between each shift of inlet and outlet streams.

An important factor is the reflux ratio in various zones of operation. The reflux ratio in any zone is defined by the following equation:

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$$\text{Reflux Ratio} = \frac{\text{Liquid Entering Zone—Non-Selective Volume of Adsorbent Entering Zone}}{\text{Selective Volume of Adsorbent Entering Zone}}$$

- 15 Where the reflux ratio is 0 the volume of liquid entering a zone is exactly equal to the non-selective volume of the adsorbent entering that zone. If the reflux ratio is positive, the net liquid entering the zone exceeds the void volume of the adsorbent entering the zone. Thus the liquid entering the zone will purge any liquid entrained in the non-selective volume of the adsorbent entering the zone. If the reflux ratio is a negative number, the volume of liquid brought into the zone in the non-selective volume of the adsorbent ex-

ceeds the liquid flow rate into that zone. Thus liquid in the non-selective volume of the adsorbent will not be totally removed from the adsorbent.

In most instances, the reflux ratio of the zones was positive to effectively purge the adsorbent.

Three tests were conducted to show the effects of using external reflux (purification stream). The basic operating conditions and test results are shown in the following Table:

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TABLE

Experiments	1	2	3
Reflux Ratios			
Zone 2	1.2	1.2	0.85
Zone 3	2.4	2.4	2.0
Zone 4	-0.1	-0.1	-0.1
Flow Rates, liters per minute at 15°C (gph @ 60°F)			
Feed	10.4 (2.74)	10.4 (2.74)	10.4 (2.74)
Desorbent	32.2 (8.52)	32.2 (8.52)	27.3 (7.22)
Purification	(0)	.95(0.25)	1.70(0.45)
Extract Purity, wt. %	96.7	99.2	99.6
Efficiency of Extraction, %	84.4	84.8	85.0

¹Defined as the ratio of extract in the extract stream over the extract in both the raffinate and extract streams.

- 40 Experiments 2 and 3, in which the purification stream was used, increased the purity of the extract material from about 96.7% up to about 99.2—99.6% purity. The three experiments were all operated at the same basic operating conditions with an extraction efficiency of about 84 to 85%, which is quite high enough for commercial operations.

- 45 In Experiment 3 less desorbent was used

than in Experiments 1 and 2. The reduced desorbent flow into zone 3 reduced the zone 3 reflux ratio, and reduced the volume of liquid bypassing extract stream 8, which resulted in a slight reduction of the reflux ratio in zone 2. The improved results for experiment 3 are due to the increase in purification stream flow, or external reflux, and not to the changes in zonal reflux ratios.

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The increase in extract purity was dramatic. Test 1 extract contained 825% of the impurities contained in Test 3 extract (3.3 wt% impurities vs. 0.4 wt % impurities). Similar beneficial results were observed, though to a lesser degree, in Test 2.

An embodiment of this process relates to an aromatic separation process in which at least three distinct zones effect adsorption, purification and desorption of an extract material from a feed stock wherein a purification stream which comprises extract material is passed to a process in order to allow recovery of substantially all the extract material from the feed.

WHAT WE CLAIM IS:—

1. A simulated moving bed process for separating a hydrocarbon feed stream into extract and raffinate with an adsorbent selective for said extract, said process comprising the steps of:

(a) contacting said feed with a bed of adsorbent in a first adsorption zone and selectively retaining extract and a portion of raffinate, and

(b) contacting at least a portion of adsorbent from step (a) with a desorbent and displacing from said adsorbent a stream comprising extract, and including the improvement which comprises contacting at least a portion of the adsorbent from step (a) with a purification stream comprising substantially desorbent-free extract before said adsorbent from step (a) is contacted with said desorbent in step (b) of the process.

2. A process as claimed in Claim 1, wherein the purification stream comprising substantially desorbent-free extract is obtained by fractionating a stream comprising extract and desorbent to separate extract from desorbent.

3. A process as claimed in Claim 1 or 2, wherein a portion of the extract displaced from said adsorbent in step (b) contains only a minor portion of desorbent and the purification stream is at least a portion of said portion of the extract displaced from said adsorbent in step (b).

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

5. A process as claimed in Claim 4, wherein the feed comprises mixed xylenes, para-xylene is the extract and the other xylenes are raffinate.

6. A process as claimed in Claim 5, wherein the desorbent is selected from benzene, toluene, ethylbenzene and diethylbenzene.

7. A process for the separation of a hydrocarbon feed stream into extract and raffinate with an adsorbent selective for said extract as compared to said raffinate, said process comprising:

(i) introducing said feed into the first zone of at least three zones of adsorbent, said three zones connected in series with means

between adjacent zones to provide cyclic fluid flow through said adsorbent zones, and selectively adsorbing on said adsorbent said extract and a portion of said raffinate, said first zone being defined as the adsorbent located between the feed inlet and the raffinate outlet;

(ii) withdrawing raffinate from said first zone downstream from said feed inlet with respect to the feed flow in said first zone;

(iii) recovering at least a portion of said raffinate;

(iv) introducing a desorbent into a third zone of adsorbent located upstream from said first zone with respect to feed flow in said first zone and desorbing therefrom extract;

(v) withdrawing extract from said third zone downstream from said desorbent inlet with respect to the fluid flow in said third zone;

(vi) recovering at least a portion of said extract;

(vii) introducing a purification stream of substantially desorbent-free extract into a second zone of adsorbent located upstream of said first zone with respect to fluid flow in said first zone and downstream of said third zone with respect to fluid flow in said third zone, and thereby desorbing raffinate from said adsorbent and displacing raffinate from the interstitial void spaces of said adsorbent; and

(viii) periodically advancing in a downstream direction with respect to fluid flow in zone 1 through said adsorbent the feed inlet, raffinate outlet, desorbent inlet, extract outlet and purification stream inlet thereby shifting said zones through said adsorbent.

8. A process as claimed in Claim 7, wherein the feed stream comprises aromatic hydrocarbons.

9. A process as claimed in Claim 8, wherein the feed stream comprises mixed xylenes, and para-xylene is the extract and the other xylenes are raffinate.

10. A process as claimed in Claims 8 or 9 wherein the desorbent is a hydrocarbon having a different number of carbon atoms per molecule as compared to the extract and is selected from benzene, toluene, xylene, ethylbenzene and diethylbenzene.

11. A process as claimed in any of Claims 8 to 10 wherein the adsorbent is selected from the type X and Y structured crystalline aluminosilicates.

12. A process as claimed in any of Claims 8 to 11 wherein a fourth zone is located between zone 1 and zone 3 and wherein at least a portion of the raffinate displaced from zone 1 enters zone 4 and therein displaces desorbent from zone 4 into zone 3.

13. A hydrocarbon separation process as claimed in Claim 1 and substantially as herein

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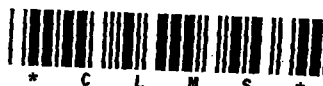
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beneficial results were observed, though to a lesser degree, in Test 2. An embodiment of this process relates to an aromatic separation process in which at least three distinct zones effect adsorption, purification and desorption of an extract material from a feed stock wherein a purification stream which comprises extract material is passed to a process in order to allow recovery of substantially all the extract material from the feed.

WHAT WE CLAIM IS:—

1. A simulated moving bed process for separating a hydrocarbon feed stream into extract and raffinate with an adsorbent selective for said extract, said process comprising the steps of:

(a) contacting said feed with a bed of adsorbent in a first adsorption zone and selectively retaining extract and a portion of raffinate, and

(b) contacting at least a portion of adsorbent from step (a) with a desorbent and displacing from said adsorbent a stream comprising extract, and including the improvement which comprises contacting at least a portion of the adsorbent from step (a) with a purification stream comprising substantially desorbent-free extract before said adsorbent from step (a) is contacted with said desorbent in step (b) of the process.

2. A process as claimed in Claim 1, wherein the purification stream comprising substantially desorbent-free extract is obtained by fractionating a stream comprising extract and desorbent to separate extract from desorbent.

3. A process as claimed in Claim 1 or 2, wherein a portion of the extract displaced from said adsorbent in step (b) contains only a minor portion of desorbent and the purification stream is at least a portion of said portion of the extract displaced from said adsorbent in step (b).

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

5. A process as claimed in Claim 4, wherein the feed comprises mixed xylenes, para-xylene is the extract and the other xylenes are raffinate.

6. A process as claimed in Claim 5, wherein the desorbent is selected from benzene, toluene, ethylbenzene and diethylbenzene.

7. A process for the separation of a hydrocarbon feed stream into extract and raffinate with an adsorbent selective for said extract as compared to said raffinate, said process comprising:

(i) introducing said feed into the first zone of at least three zones of adsorbent, said three zones connected in series with means

between adjacent zones to provide cyclic fluid flow through said adsorbent zones, and selectively adsorbing on said adsorbent said extract and a portion of said raffinate, said first zone being defined as the adsorbent located between the feed inlet and the raffinate outlet;

(ii) withdrawing raffinate from said first zone downstream from said feed inlet with respect to the feed flow in said first zone;

(iii) recovering at least a portion of said raffinate;

(iv) introducing a desorbent into a third zone of adsorbent located upstream from said first zone with respect to feed flow in said first zone and desorbing therefrom extract;

(v) withdrawing extract from said third zone downstream from said desorbent inlet with respect to the fluid flow in said third zone;

(vi) recovering at least a portion of said extract;

(vii) introducing a purification stream of substantially desorbent-free extract into a second zone of adsorbent located upstream of said first zone with respect to fluid flow in said first zone and downstream of said third zone with respect to fluid flow in said third zone, and thereby desorbing raffinate from said adsorbent and displacing raffinate from the interstitial void spaces of said adsorbent; and

(viii) periodically advancing in a downstream direction with respect to fluid flow in zone 1 through said adsorbent the feed inlet, raffinate outlet, desorbent inlet, extract outlet and purification stream inlet thereby shifting said zones through said adsorbent.

8. A process as claimed in Claim 7, wherein the feed stream comprises aromatic hydrocarbons.

9. A process as claimed in Claim 8, wherein the feed stream comprises mixed xylenes, and para-xylene is the extract and the other xylenes are raffinate.

10. A process as claimed in Claims 8 or 9 wherein the desorbent is a hydrocarbon having a different number of carbon atoms per molecule as compared to the extract and is selected from benzene, toluene, xylene, ethylbenzene and diethylbenzene.

11. A process as claimed in any of Claims 8 to 10 wherein the adsorbent is selected from the type X and Y structured crystalline aluminosilicates.

12. A process as claimed in any of Claims 8 to 11 wherein a fourth zone is located between zone 1 and zone 3 and wherein at least a portion of the raffinate displaced from zone 1 enters zone 4 and therein displaces desorbent from zone 4 into zone 3.

13. A hydrocarbon separation process as claimed in Claim 1 and substantially as herein

before described with reference to the accompanying drawing.

14. Hydrocarbons wherever separated by a process as claimed in any one of Claims 1 to 13.

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