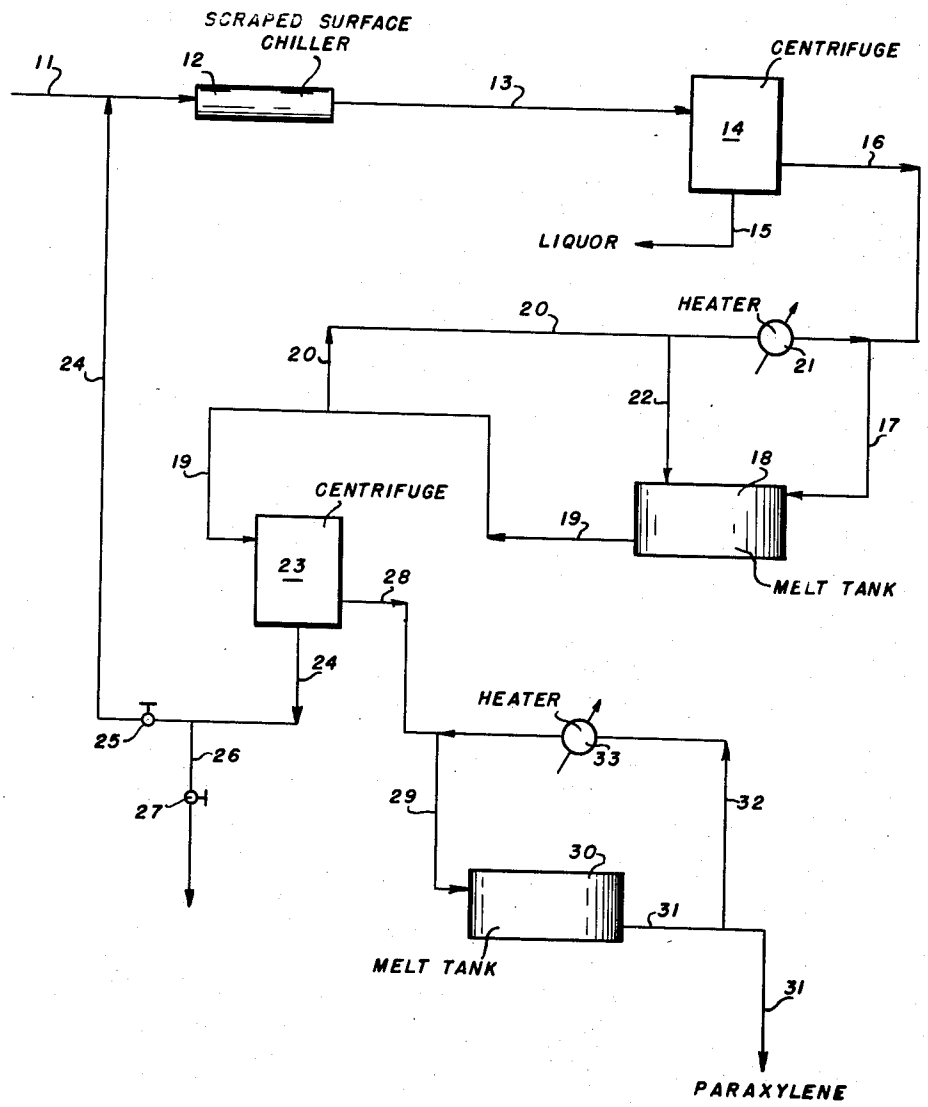
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RECOVERY OF PURE PARAXYLENE

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RECOVERY OF PURE PARAXYLENE

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The present invention is concerned with a process for separating paraxylene of over 90% purity from a mixture of it with at least one other isomeric xylene wherein the mixture contains a minor portion of paraxylene. In this particular invention the mixture is subjected to cooling to the first eutectic temperature of paraxylene and another constituent of the mixture to form a first slurry; the slurry is subjected to centrifugal action for a time sufficient to form a first cake comprising a major portion of paraxylene and a first mother liquor. In accordance with the present invention, the first cake is heated to a temperature in the range between −30° and 40° F. to form, by partial melting, a second slurry containing paraxylene crystals in an amount in the range between 30% and 85% by weight of said second slurry. This second slurry is then centrifuged for a time sufficient to form a second cake having a paraxylene content in excess of 90% and a second mother liquor, the second mother liquor being recycled or admixed at least in part with the said mixture and substantially pure paraxylene is recovered from the second cake.

The invention will be further illustrated by reference to the drawing in which the single figure is a flow diagram of a preferred mode thereof.

Referring now to the drawing, numeral 11 designates a charge line through which a mixture containing paraxylene with other isomeric xylenes is introduced into the system. A typical analysis of a suitable feed stock in the practice of the present invention is as follows:

Component	Volume Percent by Infrared Analysis
Toluene	2.0
Ethylbenzene	19.2
Paraxylene	15.7
Metaxylene	42.2
Orthoxylene	20.9

A feed stock having a composition as indicated above is discharged by line 11 into a chiller, such as a scraped surface chiller, indicated by numeral 12, wherein the temperature of the feed mixture is reduced to a temperature in the range of −90° and −110° F. If desired, the chilled mixture may be held in a suitable holding tank or other facility, not shown, for a time, such as about 30 minutes, sufficient to allow the crystals in the slurry formed by the chilling operation to attain a form conducive to give best results. However, it may be desirable to omit the holding time since good

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results may be obtained without holding the crystals at the low temperature. In any event, the slurry at a temperature in the range between −90° and −110° F. is discharged from chiller 12 by line 13 and introduced thereby into a first centrifuge 14 which may be a centrifuge of the basket type. This centrifuge may operate at a rate sufficient to impose on the slurry a centrifugal force from about 400 to 1000 times gravity. However, usually it will be desirable to employ a centrifugal force above about 500 times gravity. Satisfactory results may be obtained at centrifugal forces of about 750 times gravity. By virtue of the operation of centrifuge 14 a first cake and a first mother liquor is formed. The first mother liquor may be discharged from centrifuge 14 by line 15 for further handling as may be desired while the cake comprising substantially paraxylene crystals, with some mother liquor adhering thereto, is discharged from centrifuge 14 by line 16 which connects to branch line 17 and thereby discharges the cake into a melt tank 18. Melt tank 18 is held at a sufficient temperature to cause partial melting of the crystals from the cake discharged from centrifuge 14. It is desirable to melt from 15 to 70% of the cake from centrifuge 14 to provide a second slurry containing paraxylene crystals in the range from 30% to 85% by weight of said slurry. The second slurry formed in melt tank 18 is withdrawn therefrom by line 19 and a portion of the second slurry is recycled to melt tank 18 by line 20 containing heater 21 which introduces a sufficient amount of heat into the slurry to allow proper functioning of melt tank 18 and also to form the second slurry. In order to insure proper control of the temperature in melt tank 18, a portion of the slurry in line 20 may be discharged directly into melt tank 18 through line 22 which will allow a portion of the slurry to by-pass heater 21. It will be noted that line 20 connects into line 17 with line 16 and allows recycling of the slurry to tank 18.

The slurry containing paraxylene crystals from melt tank 18 is discharged by line 19 into second centrifuge 23. The slurry introduced into second centrifuge 23 is at a temperature in the range between −30° and −40° F., the temperature at which the slurry is formed in melt tank 18. Centrifuge 23, like centrifuge 14, may be of the basket type and may also operate at rates sufficient to allow the formation of a second cake and a second mother liquor. Centrifugal forces in the range from 400 to 1000 times gravity may also be employed in centrifuge 23.

The second mother liquor separated in centri-

fuge 23 is discharged therefrom by line 24 controlled by valve 25 which connects into line 11. Thus the mother liquor which contains a substantial amount of paraxylene may be recycled to chiller 12 to allow substantially complete recovery of the paraxylene in the total feed. It may be desirable at times to recycle only a portion of the mother liquor and to withdraw the unrecycled portion. Provision is, therefore, made by line 26 controlled by valve 27 to withdraw mother liquor from the system as may be desired.

The second cake formed in centrifuge 23 is discharged therefrom by line 28 and introduced by line 29, which connects thereto, into a second melt tank 30 which is operated at a temperature above about 55° F. wherein the paraxylene crystals are melted to allow recovery of liquid paraxylene from tank 30 by line 31. In order to introduce heat into the paraxylene crystals withdrawn by line 28 from centrifuge 23 a portion of the paraxylene in line 31 may be recycled by line 32 through heater 33 for admixture with the paraxylene crystals in line 28 and for re-introduction into tank 30 by line 29.

It will be seen from the foregoing description taken with the drawing that a simple, commercially adaptable process is provided wherein substantially pure paraxylene of 90% by weight and better purity may be recovered from a mixture of paraxylene containing only minor portions of it.

The chilling temperature employed in the first stage is a function of the composition of the feed thereto since it is desired to obtain the maximum precipitation of paraxylene crystals without precipitation of other contaminating crystals. This is achieved by lowering the temperature to just above the eutectic point of paraxylene with the next most easily precipitatable component. As an example, with a feed containing 20 mole per cent paraxylene, 50 mole per cent methaxylene and 30 mole per cent orthoxylene, the methaxylene in such a concentration is soluble at −93° F. and the orthoxylene at −90° F. Thus, by chilling the feed to about −82° F. paraxylene crystals may be separated until 65% of the amount originally present may be recovered and the concentration of paraxylene in the residual liquor is 8 mole per cent. For a composition of the feed stock such as given above, the temperature in the first stage will be in the range between −90° and −110° F.

In order to illustrate the beneficial aspects of the present invention, 100 lbs. of a slurry containing paraxylene at about −100° F. is introduced into a centrifuge similar to centrifuge 14. This slurry comprises 10 lbs. of paraxylene crystals, 5 lbs. of paraxylene liquid and 85 lbs. of isomeric xylenes which may be termed diluent. The equivalent of centrifuge 14 is operated at a rate sufficient to apply a centrifugal force of about 750 times of gravity to form a mother liquor and a cake. The mother liquor contains no paraxylene crystals, 4.9 lbs of paraxylene liquid and 83.34 lbs of diluent. On the other hand, the cake contains 10 lbs. of paraxylene crystals, .10 lb. of paraxylene liquid and 1.66 lbs. of diluent liquid for a total of 11.76 lbs. This total of 11.76 lbs. is heated to a temperature in the range of −30° to 40° F. to form a second slurry which is charged to a centrifuge equivalent to second centrifuge 23 wherein a second mother liquor corresponding to 4.7 lbs. is separated. This second mother liquor contains 3.34 lbs. of paraxylene liquid and 1.34 lbs. of diluent while the second cake which is

formed in the second centifuge liquid and 0.30 lb. of diluent, making a total of 7.06 lbs. of product recovered. Therefore, this product has a purity of 96% paraxylene.

The feature of the present invention is particularly the heating of the first cake from the first centrifuge to a temperature in the range between −30° and 40° F. to form a second slurry which contains from 30% to 85% by weight of the slurry of paraxylene crystals. By virtue of the formation of the second slurry in which paraxylene crystals and paraxylene diluent are formed, it is possible to recover by centrifuging in the second stage substantially pure paraxylene while the recycling process allows ultimate recovery of substantial yields of pure paraxylene. It is desirable to recycle the second mothor liquor in an amount in the range of 20% to 250% of the purified paraxylene recovered from the second centrifuge stage. A preferred range of recycle is from 150% to 175%. The formation of the second slurry in the first melt tank by partially melting the first cake is dependent on the concentration of the paraxylene remaining in the liquid recycled and that which is required for a given product purity. Stating this otherwise, the first cake is melted to that extent required to give a product having the desired purity with a given amount of recycle of the mother liquor separated from the second cake. For example, in the 20% recycle roughly 16.7% of the cake would be melted, and in the 250% recycle, approximately 71.5% of the cake would be melted to form the second slurry.

One of the benefits of the present invention is the reduction in the time during which centrifugation takes place. Ordinarily in one stage processes in which substantially pure paraxylene is recovered, it is necessary to operate a centrifuge for long periods of time to obtain substantially pure paraxylene of over 90% purity. In the present invention instead of operating the centrifuge in the one stage for 30 mins. or more, the total time of the centrifuging operation is reduced to no more than 10 mins. and usually to around 2 to 4 mins. Thus the first centrifuge is equivalent to centrifuge 14. This centrifuge would operate for no more than 10 mins. and usually no more than 2 mins. Similar times would obtain in centrifuge 23. For example, centrifuge 23 may be operated with 2 mins. centrifuging time at 32° F. to form a cake of 93.5% paraxylene. With 2 mins. time of operation and at 40° F. a cake of 95% purity of paraxylene would be produced. If the centrifuging time is increased to the maximum of about 10 mins. the corresponding temperature for a given purity may be lowered to a lower temperature in the range given. As the centrifuging time is increased the temperature may be decreased.

While the present invention has been described with relationship to scraped surface chillers and basket centrifuges, it will be apparent that other chilling devices and other separation devices may be used. For example, it may be desirable to use different types of centrifuges or to use filters and other types of chillers such as are available on the market.

The nature and objects of the present invention having been completely described and illustrated, what I wish to claim as new and useful and to secure by Letters Patent is:

1. A process for separating paraxylene of over 90% purity from a mixture of it with at least one other isomeric xylene wherein the mixture

contains a minor portion of paraxylene which comprises cooling said mixture to a temperature no lower than its eutectic point to form a first slurry of paraxylene crystals in a mother liquor, centrifuging said first slurry for no more than two minutes and sufficient to separate a first cake comprising a major portion of paraxylene and a first mother liquor, heating the first cake to a temperature in the range between —30° and 40° F. to form a second slurry containing paraxylene crystals in an amount in the range between 30% and 85% by weight of said slurry, centrifuging the second slurry for a time sufficient to separate a second cake having a paraxylene content in excess of 90% and a second mother liquor the time of centrifuging the second slurry plus the time for centrifuging the first slurry being no greater than a total of ten minutes, admixing at least a portion of the second mother liquor with the feed mixture, and recovering substantially pure paraxylene from said second cake.

2. A process for separating paraxylene of over 90% purity from a mixture of it with at least one other isomeric xylene wherein the mixture contains a minor portion of paraxylene which comprises cooling said mixture to a temperature in the range between —90° and —110° F. to form a first slurry of paraxylene crystals in a mother liquor, centrifuging said first slurry for no more than two minutes and sufficient to form a first cake comprising a major portion of paraxylene and a first mother liquor, heating the first cake to a temperature in the range between —30° and 40° F. to form a second slurry containing paraxylene crystals in an amount in the range between 30% and 85% by weight of said slurry, centrifuging the second slurry for a time sufficient to form a second cake having a paraxylene content in excess of 90% and a second mother liquor the time of centrifuging the second slurry plus the time for centrifuging the first slurry being no greater than a total of ten minutes, recycling the second mother liquor for admixture with said feed mixture in an amount in the range between 20% and 250% by weight based on said second cake, and recovering substantially pure paraxylene from said second cake.

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