

Description

This invention relates to a continuous process for the production of dichlorohydrin. "Dichlorohydrin" is a term employed herein to designate the isomers 1,2-dichloro-3-hydroxypropane and 1,3-dichloro-2-hydroxypropane.

It is known to prepare an aqueous solution of dichlorohydrin by reacting in a reaction zone allyl chloride, water and chlorine in a dilute aqueous phase. U.S. patent specification 2,714,121 discloses producing halohydrins by using high dilution of, e.g., 250-400 volumes of water per volume of, e.g., a halosubstituted hydrocarbon in aqueous medium with subsequent addition of the halogen, and keeping the organic by-product phase dispersed as fine particles. U.S. patent specification 2,714,123 discloses producing an aqueous solution of dichlorohydrin in a series of reaction stages wherein substantially all of the water is fed to the first reaction stage and the other reactants are added in substantially equimolar proportions into each of the subsequent reaction stages.

The reaction zone effluent may be worked up in various ways to recover the dichlorohydrin therefrom, or may be processed further in an integrated process to convert the dichlorohydrin to derivatives such as epichlorohydrin and/or glycerine.

It is known, e.g. from Belgian patent specifications 614,890 and 614,891 that dichlorohydrin may be extracted from aqueous solution with organic solvents such as phosphate esters of aliphatic monohydric alcohols containing more than four carbon atoms, aryl phosphates, and liquid aliphatic alcohols and liquid ketones having 8 to 18 carbon atoms per molecule. U.S. patent specifications 4,620,911 and 4,665,240 disclose as further solvents for dichlorohydrin saturated aliphatic ethers and chlorinated hydrocarbons containing up to about 9 carbon atoms, including, e.g., carbon tetrachloride. The aforesaid U.S. patent specifications 4,620,911 and 4,665,240 employ solvent extraction together with membrane processes to reduce the amount of fresh water fed to the reaction zone of the process.

One disadvantage of the known processes is the formation of undesired by-products, which reduce the overall efficiency of the process and may complicate purification procedures of the desired product. Such conventional processes result in an aqueous effluent stream which contains minor amounts of organic impurities diluted in a substantial amount of water. Such effluent requires energy intensive treatment to reduce the amount of organic materials to levels acceptable to be passed to receiving bodies of water such as rivers, lakes and the like. Considerable savings could be effected if the amount of organics to be treated could be

significantly reduced.

A further disadvantage of the known processes is that polychlorinated alkane by-products are formed during the aqueous dichlorohydrin synthesis. When, as is often the case, it is desired to further convert the dichlorohydrin to epichlorohydrin (1,2-epoxy-3-chloropropane) in a subsequent step by the action of basic reagents, said by-products are dehydrochlorinated to form chloroaliphatic impurities which have volatility close to epichlorohydrin. These impurities, although removable by conventional fractional distillation procedures, require inordinate input of energy to achieve epichlorohydrin purity required for a number of demanding end-use applications. The present invention advantageously substantially removes the precursor impurities from the dichlorohydrin prior to conversion to epichlorohydrin and in a manner which is much more energy efficient than finishing of epichlorohydrin by conventional distillation.

A method has now been found to reduce the level of chloroaliphatic alkanes and chloroaliphatic ethers impurities in the dichlorohydrin product and to obtain said impurities in relatively concentrated form for further processing or disposal.

According to the invention there is provided a continuous process for the production of dichlorohydrin with reduced amounts of chloroaliphatic alkanes and chloroaliphatic ethers which comprises:

(a) reacting allyl chloride, water and chlorine in a reaction zone to form an aqueous mixture of dichlorohydrin, together with minor amounts of chloroaliphatic alkanes and chloroaliphatic ethers as impurities;

(b) contacting in an extraction zone said reaction zone effluent with from 1% to 8% by volume of a polyhalo-aliphatic or aliphatic hydrocarbon solvent, calculated on said effluent said solvent having a greater selectivity for said impurities than for dichlorohydrin, and an atmospheric boiling point from 40 °C to 105 °C, to obtain as extract an impurity-enriched solvent, and an aqueous phase containing a major amount of the dichlorohydrin;

(c) separating said extract from said aqueous phase;

(d) contacting in a water wash zone said separated extract from step (c) with water to obtain a water wash stream containing a major portion of dichlorohydrin present in the feed to step (d), and a washed extract stream containing less dichlorohydrin than said feed to step (d);

(e) recycling said water wash stream to at least one of step (a) and step (b);

(f) passing said washed extract stream from step (d) as feed to a distillation zone, and fractionat-

ing said feed by fractionation distillation to obtain an overhead stream containing impurities boiling at a temperature lower than the boiling temperature of said solvent, a residual fraction containing impurities boiling at a temperature higher than the boiling temperature of said solvent, and an intermediate stream comprising regenerated solvent containing fewer impurities than said feed; and

(g) recycling said regenerated solvent to step (b).

In the principal reaction, allyl chloride is converted to a mixture of the two isomers of glycerol dichlorohydrin by reaction with hypochlorous acid, HClO, which is readily formed when chlorine is dissolved in water. The chlorohydrination reaction takes place readily at temperatures in the range from 15 °C to 75 °C. The reaction typically results in the formation of undesired by-products such as, e.g., trichloropropane and tetrachloropropyl ether; such by-product formation may be aggravated by, e.g., the presence of allyl chloride in excess of its aqueous solubility. For maximum dichlorohydrin yield it is necessary to run the reaction at low concentrations of chloride ion and of chlorohydrin, i.e., with high water dilution. However, high water dilution may greatly increase the volume of aqueous effluent from the process which must be treated to remove tetrachloropropyl ethers and other by-products.

The chloroaliphatic alkane and chloroaliphatic ether impurities are extracted from the reaction zone effluent into a small amount of solvent having greater solvency at the extraction temperature for said impurities than for the dichlorohydrin. Inert organic solvents suitably employed to effectively remove said impurities from the aqueous glycerol dichlorohydrin solution are polyhalo-aliphatic and aliphatic hydrocarbons having an atmospheric boiling point from 40 °C to 105 °C and which are resistant to decomposition by a strong base such as slaked lime in aqueous solution at a temperature of 100 °C. A preferred solvent is carbon tetrachloride. The solvents include methylene chloride, methylene bromide, 1,1-dichloroethane, 1,1-dichloropropane, cyclohexane, and n-heptane. Particularly preferred are solvents having a density difference from water (either more dense or less dense than water) of at least 0.1 g/ml, and particularly a density difference of at least 0.25 g/ml when measured at a temperature of 50 °C. This density difference facilitates ready separation of the solvent from the aqueous phase.

The extraction is carried out using conventional extraction techniques, wherein the impurities are extracted into the solvent. The solvent is then separated from the remaining aqueous phase, following which the solvent may be regenerated as de-

scribed hereinbelow, and recycled to the extraction zone.

An illustrative embodiment of the invention will now be described with reference to the accompanying drawing which shows schematically a preferred assemblage adapted to the continuous manufacture of dichlorohydrin. The reaction zone may comprise one or more reaction stages in parallel or consecutive flow, comprised of stirred reactors, circulating loop reactors, vane disc turbine dispersers, sprayed towers or other equipment known to be suitable for chlorohydrin reactions.

Referring to the drawing, effluent from the reaction zone 10 is passed via conduit 12 to extraction zone 14 which may be any conventional liquid-liquid extractor such as an agitated vessel, jet mixer, perforated plate tower or rotating disc contactor. Preferred for its simplicity and ease of operation is an injection line 16 providing solvent just upstream of an orifice plate (not shown) to ensure good contacting of the solvent with the reaction zone effluent. Although counter-current extraction may be used, cocurrent flow with a very minor amount of solvent is suitable. The amount of solvent contacted with the aqueous phase should be in a solvent to aqueous phase volume ratio of from 1:100 to 8:100, with a volume ratio of 1.5:100 to 4.5:100 being preferred. Generally when cocurrent flow is employed it will be desirable to separate the mixture of aqueous phase and extraction solvent into the respective phases. As shown in the drawing the mixed phases are passed from extraction zone 14 via conduit 18 to phase separator 20 which may be any conventional phase separator such as a centrifugal separator or hydroclone. The aqueous phase containing a major amount of the dichlorohydrin and a significantly smaller amount of the chloroether and chloroaliphatic alkane impurities is removed from phase separator 20 via conduit 22 for concentration, or for chemical conversion into derivatives such as epichlorohydrin or glycerine.

The impurities-fat solvent, also referred to herein as separated extract, is passed from the separation zone 20 via conduit 24 to water wash zone 26. In water wash zone 26, which may comprise one or more separate stages the impurities-fat solvent is contacted with water supplied via conduit 28 to remove a substantial portion of any dichlorohydrin product that may be contained therein resulting in a washed extract stream and a water wash stream containing a major portion of dichlorohydrin present in the stream(s) fed to water wash zone 26. In a preferred mode the water wash stream removed from said wash zone 26 is recycled via conduit 30, valve 32 and conduit 34 to the reaction zone 10 and may displace a like amount of fresh water ordinarily supplied to said reaction zone. Alternately, the water from wash

zone 26 may be recycled to extraction zone 14 via conduit 30, valve 36 and conduit 38. Preferably the amount of water contacting the impurities-fat solvent in the washing zone is in a water to solvent volume ratio from 4:1 to 8:1 although greater or smaller amounts may be used.

From wash zone 26 the washed extract stream is passed via conduit 40 to solvent regeneration zone 42 for regeneration by fractional distillation.

Both the chloroether and polychloroaliphatic (such as 1,2,3-trichloropropane) impurities will be less volatile than the solvent chosen, which can therefore be removed as an overhead fraction via conduit 44, leaving both classes of impurities as a bottom residual fraction which can be removed via conduit 46 for further use or disposal. Typically there will also be monochloroaliphatic impurities arising from side reactions of the allyl chloride starting material and possibly some impurities, such as monochloropropanes, introduced with the allyl chloride charged to reaction zone 10. These lighter impurities have boiling points lower than the preferred solvents, such as carbon tetrachloride, employable in this process. Accordingly in a preferred embodiment the regenerated solvent is recovered as an intermediate fraction via conduit 16.

In order to compensate for minor solvent losses through the process, a small amount of solvent may be added via conduit 48 along with the recycle solvent to maintain the desired ratio of solvent to reaction zone effluent.

A preferred embodiment according to the invention further provides an integrated process wherein the aqueous dichlorohydrin product from phase separator 20 is converted to epichlorohydrin as shown in the lower part of the drawing and any solvent remaining in said aqueous phase is recovered and reused.

The aqueous dichlorohydrin phase from separator 20 is withdrawn via conduit 22, contacted with an aqueous base such as, e.g., slaked lime, sodium hydroxide or sodium carbonate supplied via conduit 50 to convert the dichlorohydrin to epichlorohydrin which is immediately stripped from the aqueous solution in stripping zone 54 with steam supplied via conduit 56. The stripped aqueous phase exits stripping zone 54 via conduit 58 for further treatment and disposal. The liquid resulting from condensed overhead vapour from stripping zone 54 forms two layers. The upper layer comprising substantially water with a small amount of epichlorohydrin is returned to a rectifying section in the top of stripping zone 54 as reflux. The lower layer comprising primarily epichlorohydrin together with a small amount of steam condensate is passed as an overhead stream via conduit 60 to distillation zone 62 for purification of the epichlorohydrin by fractional distillation. A small gaseous

vent stream 59, consisting essentially of fixed gases such as nitrogen which entered the system with the reactants fed to the reaction zone 10, together with some low boiling organic compounds such as acrolein, which may be generated in stripping zone 54 from by-products formed in the reaction zone 10 is removed for further treatment or disposal, such as combustive disposal in a conventional flare system.

Distillation zone 62 may comprise one column, but preferably is two or a plurality of columns (not shown) arranged to separate water, solvent and other lower boiling materials as an overhead fraction withdrawn via conduits 64 and 66, the desired epichlorohydrin as a very pure intermediate fraction withdrawn via conduit 68, and residual high-boiling impurities as a bottoms fraction, withdrawn via conduit 70. Said bottoms fraction in conduit 70 comprises primarily 1,2-dichlorohydrin which failed to react completely in stripping zone 54 together with small amounts of the chloroethers and trichloropropane which were not extracted in extraction zone 14. In a preferred embodiment most of the bottoms fraction in conduit 70 is recycled via conduits 71 and 72 to water wash zone 26 for recovery of the dichlorohydrin content, with small continuous or intermittent purge of said bottoms fraction via conduit 73 to waste, e.g., incineration. The purge is to prevent or control possible build-up in the system of the undesirable trace isomeric impurity of epichlorohydrin, 2-chloroallyl alcohol.

The overhead fraction in conduit 64 is partially condensed to recover any solvent and/or epichlorohydrin therein, with the uncondensed vapours passing to waste, e.g. combustive disposal via conduit 66 (similar to the disposal of gases via conduit 59). The condensed portion is passed via conduit 74 to reversion zone 76 for contact with chloride ion to convert any epichlorohydrin that may be present back to dichlorohydrin. Reversion zone 76 which may be any liquid-liquid contactor, is preferably a stirred vessel where the condensate may be contacted with, e.g. hydrochloric acid added via conduit 78. Although any chloride ion source could be employed in the reversion step, hydrochloric acid is most preferred as it is already present in the manufacturing process for dichlorohydrin and the acidity improves both the rate and selectivity to the desired dichlorohydrin. An aqueous system should be used in the reversion zone to prevent polymerization reaction which could form undesired high molecular weight polyols. Although hydrochloric acid is in principle already available in conduit 12 for the reversion reaction, use of a separate reversion zone enables better control and more selective reversion of epichlorohydrin back to dichlorohydrin. From reversion zone 76, the reverted condensate is recycled via conduits 80 and 72 to

wash zone 26 for recovery of the solvent and dichlorohydrin. In this preferred embodiment the epichlorohydrin is reverted back to the more water soluble dichlorohydrin before recycle of the stream to avoid contaminating the impurities-fat solvent in conduit 24 with epichlorohydrin. For some applications, e.g., where more than one stage is employed in the water wash zone, it may be desirable to convey the reversion zone product via a conduit (not shown) separate from the conduit carrying the bottoms fraction from the distillation zone to said water wash zone to enable contacting of the reversion zone product in a different stage of said water wash zone than is applied for contacting of said bottoms fraction with water in said water wash zone.

Claims

1. A continuous process for the production of dichlorohydrin with reduced amounts of chloroaliphatic alkanes and chloroaliphatic ethers, which comprises:

(a) reacting allyl chloride, water and chlorine in a reaction zone to form an aqueous mixture of dichlorohydrin, together with minor amounts of chloroaliphatic alkanes and chloroaliphatic ethers as impurities;

(b) contacting in an extraction zone said reaction zone effluent with from 1% to 8% by volume of a polyhalo-aliphatic or aliphatic hydrocarbon solvent, calculated on said effluent, said solvent having a greater selectivity for said impurities than for dichlorohydrin, and an atmospheric boiling point from 40 °C to 105 °C, to obtain as extract an impurity-enriched solvent, and an aqueous phase containing a major amount of the dichlorohydrin;

(c) separating said extract from said aqueous phase;

(d) contacting in a water wash zone said separated extract from step (c) with water to obtain a water wash stream containing a major portion of dichlorohydrin present in the feed to step (d), and a washed extract stream containing less dichlorohydrin than said feed to step (d);

(e) recycling said water wash stream to at least one of step (a) and step (b);

(f) passing said washed extract stream from step (d) as feed to a distillation zone, and fractionating said feed by fractionation distillation to obtain an overhead stream containing impurities boiling at a temperature lower than the boiling temperature of said solvent, a residual fraction containing impurities boiling at a temperature higher than

the boiling temperature of said solvent, and an intermediate stream comprising regenerated solvent containing fewer impurities than said feed; and

(g) recycling said regenerated solvent to step (b).

2. A process as claimed in claim 1 wherein said solvent is carbon tetrachloride.

3. A process as claimed in claim 1 wherein at least a portion of the water wash stream removed from the water wash zone of step (d) is recycled to the reaction zone.

4. A process as claimed in claim 1 wherein in step (d) the amount of water contacting said separated extract is in a water to solvent volume ratio from 4:1 to 8:1.

5. A process as claimed in claim 1 wherein in step (f) at least part of the residual fraction is recycled to the water wash zone of step (d).

6. A process for the production of epichlorohydrin comprising in addition to the steps (a) to (g) in claim 1 the steps of;

(h) contacting said aqueous phase from step (b) with a basic substance to obtain a basic aqueous phase and substantially simultaneously steam stripping said basic aqueous phase to convert said dichlorohydrin to epichlorohydrin;

(i) fractionating said epichlorohydrin by fractionation distillation to obtain an overhead fraction containing any solvent which had been entrained in said aqueous phase from step (b);

(j) contacting said overhead fraction with a source of chloride ion in aqueous solution to revert any epichlorohydrin therein to dichlorohydrin resulting in a reverted overhead fraction; and

(k) recycling said reverted overhead fraction to at least one of step (a), step (b), step (c) or said step (d).

7. A process as claimed in claim 6 wherein in step (k) said reverted fraction is recycled to the water wash zone of step (d).

Patentansprüche

1. Ein kontinuierliches Verfahren zur Herstellung von Dichlorhydrin mit verringerten Mengen an chloraliphatischen Alkanen und chloraliphatischen Ethern, das die folgenden Verfahrensstufen umfaßt:



wash zone 26 for recovery of the solvent and dichlorohydrin. In this preferred embodiment the epichlorohydrin is reverted back to the more water soluble dichlorohydrin before recycle of the stream to avoid contaminating the impurities-fat solvent in conduit 24 with epichlorohydrin. For some applications, e.g., where more than one stage is employed in the water wash zone, it may be desirable to convey the reversion zone product via a conduit (not shown) separate from the conduit carrying the bottoms fraction from the distillation zone to said water wash zone to enable contacting of the reversion zone product in a different stage of said water wash zone than is applied for contacting of said bottoms fraction with water in said water wash zone.

Claims

1. A continuous process for the production of dichlorohydrin with reduced amounts of chloroaliphatic alkanes and chloroaliphatic ethers, which comprises:
 - (a) reacting allyl chloride, water and chlorine in a reaction zone to form an aqueous mixture of dichlorohydrin, together with minor amounts of chloroaliphatic alkanes and chloroaliphatic ethers as impurities;
 - (b) contacting in an extraction zone said reaction zone effluent with from 1% to 8% by volume of a polyhalo-aliphatic or aliphatic hydrocarbon solvent, calculated on said effluent, said solvent having a greater selectivity for said impurities than for dichlorohydrin, and an atmospheric boiling point from 40 °C to 105 °C, to obtain as extract an impurity-enriched solvent, and an aqueous phase containing a major amount of the dichlorohydrin;
 - (c) separating said extract from said aqueous phase;
 - (d) contacting in a water wash zone said separated extract from step (c) with water to obtain a water wash stream containing a major portion of dichlorohydrin present in the feed to step (d), and a washed extract stream containing less dichlorohydrin than said feed to step (d);
 - (e) recycling said water wash stream to at least one of step (a) and step (b);
 - (f) passing said washed extract stream from step (d) as feed to a distillation zone, and fractionating said feed by fractionation distillation to obtain an overhead stream containing impurities boiling at a temperature lower than the boiling temperature of said solvent, a residual fraction containing impurities boiling at a temperature higher than

the boiling temperature of said solvent, and an intermediate stream comprising regenerated solvent containing fewer impurities than said feed; and

- (g) recycling said regenerated solvent to step (b).
2. A process as claimed in claim 1 wherein said solvent is carbon tetrachloride.
3. A process as claimed in claim 1 wherein at least a portion of the water wash stream removed from the water wash zone of step (d) is recycled to the reaction zone.
4. A process as claimed in claim 1 wherein in step (d) the amount of water contacting said separated extract is in a water to solvent volume ratio from 4:1 to 8:1.
5. A process as claimed in claim 1 wherein in step (f) at least part of the residual fraction is recycled to the water wash zone of step (d).
6. A process for the production of epichlorohydrin comprising in addition to the steps (a) to (g) in claim 1 the steps of;
 - (h) contacting said aqueous phase from step (b) with a basic substance to obtain a basic aqueous phase and substantially simultaneously steam stripping said basic aqueous phase to convert said dichlorohydrin to epichlorohydrin;
 - (i) fractionating said epichlorohydrin by fractionation distillation to obtain an overhead fraction containing any solvent which had been entrained in said aqueous phase from step (b);
 - (j) contacting said overhead fraction with a source of chloride ion in aqueous solution to revert any epichlorohydrin therein to dichlorohydrin resulting in a reverted overhead fraction; and
 - (k) recycling said reverted overhead fraction to at least one of step (a), step (b), step (c) or said step (d).
7. A process as claimed in claim 6 wherein in step (k) said reverted fraction is recycled to the water wash zone of step (d).

Patentansprüche

1. Ein kontinuierliches Verfahren zur Herstellung von Dichlorhydrin mit verringerten Mengen an chloraliphatischen Alkanen und chloraliphatischen Ethern, das die folgenden Verfahrensstufen umfaßt:



- (a) Umsetzen von Allylchlorid, Wasser und Chlor in einer Reaktionszone unter Bildung einer wäßrigen Mischung aus Dichlorhydrin mit geringen Mengen an chloraliphatischen Alkanen und chloraliphatischen Ethern als Verunreinigungen;
- (b) Kontaktieren des Abstroms aus besagter Reaktionszone mit 1 bis 8 Vol.-% eines polyhalogenaliphatischen Lösungsmittels oder eines aliphatischen Kohlenwasserstofflösungsmittels, berechnet auf besagten Abstrom, in einer Extraktionszone, wobei besagtes Lösungsmittel eine größere Selektivität für besagte Verunreinigungen als für Dichlorhydrin aufweist und einen Siedepunkt bei Atmosphärendruck im Bereich 40 bis 105 °C hat, um so als Extrakt ein an den Verunreinigungen angereichertes Lösungsmittel und eine wäßrige Phase zu erhalten, die einen größeren Anteil an Dichlorhydrin enthält;
- (c) Abtrennen des besagten Extrakts von besagter wäßriger Phase;
- (d) Kontaktieren des besagten abgetrennten Extrakts aus Stufe (c) in einer Wasserwaschzone mit Wasser, um so einen Wasserwaschstrom, der einen größeren Anteil des in der Zuspelung zu Stufe (d) vorhandenen Dichlorhydrins enthält, und einen gewaschenen Extraktstrom zu erhalten, der weniger Dichlorhydrin als die Zuspelung zu Stufe (d) enthält;
- (e) Rückführung des besagten Wasserwaschstroms im Kreislauf zu mindestens einer der Stufen (a) und (b);
- (f) Weiterleiten des besagten gewaschenen Extraktstroms aus Stufe (d) als Zuspelung zu einer Destillationszone und Fraktionieren besagter Zuspelung mittels fraktionierter Destillation unter Gewinnung eines Kopfstroms, der Verunreinigungen enthält, die bei einer Temperatur unterhalb der Siedetemperatur besagten Lösungsmittels siedend, sowie einer Rückstandsfraktion, die Verunreinigungen enthält, die bei einer Temperatur oberhalb der Siedetemperatur besagten Lösungsmittels siedend, und weiterhin eines mittleren Produktstroms, der regeneriertes Lösungsmittel mit einem geringeren Gehalt an Verunreinigungen als besagte Zuspelung enthält; und
- (g) Rückführen des regenerierten Lösungsmittels im Kreislauf in Stufe (b).
2. Ein Verfahren, wie in Anspruch 1 beansprucht, in dem das Lösungsmittel Tetrachlorkohlenstoff ist.
3. Ein Verfahren, wie in Anspruch 1 beansprucht, in dem mindestens ein Anteil des Wasserwaschstroms, der aus der Wasserwaschzone von Stufe (d) abgezogen wird, im Kreislauf in die Reaktionszone zurückgeführt wird.
4. Ein Verfahren, wie in Anspruch 1 beansprucht, in dem in Stufe (d) die Menge des mit besagtem abgetrennten Extrakt kontaktierten Wassers einem Volumenverhältnis von Wasser zu Lösungsmittel im Bereich von 4:1 bis 8:1 entspricht.
5. Ein Verfahren, wie in Anspruch 1 beansprucht, in dem in Stufe (f) mindestens eine Teilmenge der Rückstandsfraktion im Kreislauf in die Wasserwaschzone von Stufe (d) zurückgeführt wird.
6. Ein Verfahren zur Herstellung von Epichlorhydrin, umfassend zusätzlich zu den Stufen (a) bis (g) gemäß Anspruch 1 die nachstehenden Stufen:
- (h) Kontaktieren besagter wäßriger Phase aus Stufe (b) mit einer basischen Substanz unter Bildung einer basischen wäßrigen Phase und das praktisch gleichzeitig stattfindende Dampfabbstreifen besagter basischer wäßriger Phase zwecks Umwandlung von Dichlorhydrin in Epichlorhydrin;
- (i) Fraktionieren des besagten Epichlorhydrins mittels fraktionierter Destillation zum Erhalt einer Kopffraktion, die jegliches in besagter wäßriger Phase aus Stufe (b) mitgerissene Lösungsmittel enthält;
- (j) Kontaktieren besagter Kopffraktion mit einer Quelle für Chloridionen in wäßriger Lösung, um jegliches darin enthaltene Epichlorhydrin in Dichlorhydrin zurückzuverwandeln, wodurch eine umgewandelte Kopffraktion erhalten wird; und
- (k) Rückführen besagter umgewandelter Kopffraktion im Kreislauf in mindestens eine der Stufen (a), (b), (c) oder in besagte Stufe (d).
7. Ein Verfahren, wie in Anspruch 6 beansprucht, in dem in Stufe (k) die besagte umgewandelte Kopffraktion im Kreislauf in die Wasserwaschzone von Stufe (d) zurückgeführt wird.

Revendications

1. Un procédé continu pour la production de dichlorohydrine avec des quantités réduites d'alcane chloraliphatiques et d'éthers chloraliphatiques, qui comprend les étapes selon lesquelles :



- (a) on fait réagir du chlorure d'allyle, de l'eau et du chlore dans une zone de réaction pour former un mélange aqueux de dichlorohydrine, en même temps que des quantités mineures d'alcanes chloroaliphatiques et d'éthers chloroaliphatiques comme impuretés ;
- (b) on met en contact dans une zone d'extraction l'effluent de la zone de réaction avec 1 % à 8 % en volume d'un solvant hydrocarbure aliphatique ou hydrocarbure aliphatique polyhalogéné, par rapport à l'effluent, ce solvant ayant une plus grande sélectivité pour lesdites impuretés que pour la dichlorohydrine, et un point d'ébullition à la pression atmosphérique compris entre 40°C et 105°C, pour obtenir comme extrait un solvant enrichi en impuretés et une phase aqueuse contenant une quantité majeure de la dichlorohydrine ;
- (c) on sépare l'extrait de la phase aqueuse ;
- (d) on met en contact dans une zone de lavage à l'eau l'extrait séparé provenant de l'étape (c) avec de l'eau pour obtenir un courant de lavage à l'eau contenant une portion majeure de la dichlorohydrine présente dans la charge arrivant à l'étape (d), et un courant d'extrait lavé contenant moins de dichlorohydrine que la charge arrivant à l'étape (d) ;
- (e) on recycle le courant de lavage à l'eau à au moins une des étapes (a) et (b) ;
- (f) on fait passer le courant d'extrait lavé provenant de l'étape (d) comme charge pour une zone de distillation et on fractionne cette charge par distillation fractionnée pour obtenir un courant de tête contenant les impuretés bouillant à une température plus basse que la température d'ébullition du solvant, une fraction résiduelle contenant les impuretés bouillant à une température plus élevée que la température d'ébullition du solvant et un courant intermédiaire comprenant du solvant régénéré contenant moins d'impuretés que la charge ; et
- (g) on recycle le solvant régénéré à l'étape (b).
2. Un procédé selon la revendication 1, dans lequel le solvant est du tétrachlorure de carbone.
3. Un procédé selon la revendication 1, dans lequel au moins une portion du courant de lavage à l'eau provenant de la zone de lavage à l'eau de l'étape (d) est recyclée à la zone de réaction.
4. Un procédé selon la revendication m, dans lequel dans l'étape (d) la quantité d'eau mise en contact avec l'extrait séparé est telle que le rapport en volume de l'eau au solvant soit compris entre 4:1 et 8:1.
5. Un procédé selon la revendication 1, dans lequel dans l'étape (f) au moins une partie de la fraction résiduelle est recyclée à la zone de lavage à l'eau de l'étape (d).
6. Un procédé pour la production d'épichlorhydrine, comprenant, en plus des étapes (a) à (g) définies dans la revendication 1, les étapes selon lesquelles :
- (h) on met en contact la phase aqueuse provenant de l'étape (b) avec une substance basique de manière à obtenir une phase aqueuse basique et à peu près simultanément on effectue un strippage à la vapeur d'eau de cette phase aqueuse basique de manière à transformer la dichlorohydrine en épichlorhydrine ;
- (i) on fractionne l'épichlorhydrine par distillation fractionnée de manière à obtenir une fraction de tête contenant tout solvant qui a été entraîné dans la phase aqueuse provenant de l'étape (b) ;
- (j) on met en contact cette fraction de tête avec une source d'ion chlorure en solution aqueuse de manière à retransformer toute épichlorhydrine présente en dichlorohydrine, donnant une fraction de tête retransformée ; et
- (k) on recycle la fraction de tête retransformée à au moins une des étapes (a), (b), (c) et (d).
7. Un procédé selon la revendication 6, dans lequel dans l'étape (k) la fraction retransformée est recyclée à la zone de lavage à l'eau de l'étape (d).