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(72) Inventeurs/Inventors:
DEVER, JOHN P., US;
HOFFMAN, WILLIAM C., US;
MCCAIN, JAMES H., US
(73) Propriétaire/Owner:
DOW TECHNOLOGY INVESTMENTS LLC, US
(74) Agent: SMART & BIGGAR

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(54) Title: PROCESS FOR PROCESSING ETHYLENE OXIDE STREAMS CONTAINING NOX OR ORGANIC NITROGEN COMPOUNDS

(57) **Abrégé/Abstract:**

Crude aqueous alkylene oxide streams are converted to the corresponding alkylene glycol. The crude alkylene oxide stream contains NO_x or organic nitrogen compounds. The hydrolysis reaction is conducted in the presence of water-soluble reducing agents, of which an alkali metal bisulfite is a preferred type.



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(74) Agent: RUSZALA, Lois, K.; The Dow Chemical Company, Intellectual Property, P.O. Box 1967, Midland, MI 48641-1967 (US).

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(71) Applicant (*for all designated States except US*): DOW TECHNOLOGY INVESTMENTS LLC [US/US]; 2040 Dow Center, Midland, MI 48674 (US).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): DEVER, John, P. [US/US]; 31 Acadia Street, Kenner, LA 70065 (US). HOFFMAN, William, C. [US/US]; 572 Roxalana Hills Drive, Dunbar, WV 25064 (US). JAMES, McCain, H. [US/US]; 1987 Parkwood Road, Charleston, WV 25314 (US).

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(54) Title: PROCESS FOR PROCESSING ETHYLENE OXIDE STREAMS CONTAINING NOX OR ORGANIC NITROGEN COMPOUNDS

(57) Abstract: Crude aqueous alkylene oxide streams are converted to the corresponding alkylene glycol. The crude alkylene oxide stream contains NOx or organic nitrogen compounds. The hydrolysis reaction is conducted in the presence of water-soluble reducing agents, of which an alkali metal bisulfite is a preferred type.



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PROCESS FOR PROCESSING ETHYLENE OXIDE STREAMS CONTAINING NO_x OR
ORGANIC NITROGEN COMPOUNDS

FIELD OF THE INVENTION

5 The present invention relates to methods for producing an alkylene glycol by reaction of an alkylene oxide and water. The invention more specifically relates to methods for reducing the amounts of certain types of impurities and/or by-products that can be generated during the alkylene glycol-forming reaction.

BACKGROUND OF THE INVENTION

10 1,2-alkylene glycols are manufactured by heating a mixture of the corresponding alkylene oxide and water to an elevated temperature at which the water will react at the site of the epoxide group to form vicinal hydroxyls. Thus, ethylene oxide and water react to form 1,2-ethylene glycol and propylene oxide and water react to form 1,2-propylene glycol.

 Various unwanted side reactions can occur during this process. Carbonyl
15 compounds often form via various mechanisms. For example, the alkylene glycol can oxidize to form the corresponding aldehyde plus a molecule of water. Other impurities present in the system can react to form color bodies which must be removed from the product. The impurities can also form solids that need to be removed. Impurities can also cause unwanted foaming of the reaction mixture.

20 These problems may occur at various stages of the alkylene glycol manufacturing process, but occur most frequently at points in the process in which the reaction mixture and/or alkylene glycol product are exposed to high temperature conditions.

 For example, temperatures inside an ethylene glycol reactor, where ethylene oxide and water react to form the glycol product, are typically in the order of 150°C or more.
25 Impurities present in the reactant streams often form a variety of colored, solid and/or foam-producing by-products under those conditions. The problem may be particularly acute in cases in which one or more reactant streams contain NO_x compounds and/or organic nitrogen compounds such as nitromethane, 2-nitroethanol and nitroethylene. NO_x compounds are often present because they (or a precursor such as ammonia) are added into
30 the ethylene oxide manufacturing process as an inhibitor or for other reasons, as described for example in U. S. Patent Nos. 4,822,900 and 6,511,938, particularly when the ethylene oxide catalyst includes an alkali metal nitrate component. These NO_x compounds can react

with organic species in the stream to form organic nitrogen compounds in the process stream. When the NO_x or organic nitrogen compounds are carried downstream into an ethylene glycol reactor, the amount of by-products that are formed can be significant.

Often, a special purge unit is operated to remove NO_x, organic nitrogen compounds and
5 other impurities from the main process stream before it enters the ethylene glycol reactor.

The purge unit produces a purge stream that contains most of the NO_x and organic nitrogen compounds. The ethylene oxide values in these purge streams can constitute a significant proportion of the output of the manufacturing plant. To recover these values, the purge stream is processed into ethylene glycol in a smaller system, separate from the main
10 reactor(s) where the bulk of the ethylene glycol is produced, so the problem with impurities can be dealt with apart from the main product stream. Discoloration, solids formation and foaming may be particularly acute in these purge stream treatment units.

These problems can also occur in other process waste streams that contain little or no ethylene oxide or ethylene glycol values. The process streams may be stripped at
15 elevated temperature to recover water, for example, and impurities in those streams can cause unwanted foaming or solids formation as described before.

One way of treating alkylene glycols to remove aldehydes is to contact the mixture with a bisulfite compound. For example, U. S. Patent No. 6,187,973 describes a method for removing aldehydes from ethylene glycol by contacting the ethylene glycol with a bisulfite-
20 treated anion exchange resin. Canadian Patent No. 1,330,350 describes adding bisulfite ions to an ethylene glycol mixture, followed by contacting the mixture with an anion exchange resin in the hydroxyl form, to remove aldehydes. JP 53-029292 describes a process for absorbing aldehydes from a gas stream, in which the stream is contacted with an activated carbon that is impregnated with a sulphite or acid sulfite salt. SU 1498752 (abstract)
25 describes a method of purifying ethylene glycol with a first reagent mixture that contains sodium hypochlorite, bromine, p-chlorobenzenesulfonic acid dichloramine or chlorosuccinamide, and then treating the solution with a solution of sodium bisulfite. These processes all focus on removal methods rather than methods for reducing aldehyde (or other by-product) generation in the first instance. Research Disclosure 465117 (Kenneth
30 Mason Publications, Ltd., January 2003) describes adding a reactant such as a sulphite to certain EO/EG process streams for impurity conversion. Bisulfite ions also have been added

into processes for producing ethylene glycol from ethylene oxide, carbon dioxide and water via an ethylene carbonate intermediate.

What is needed is a process by which reduced levels of undesired by-products, such as aldehydes, colorants, solids and/or foaming agents, are produced in an alkylene glycol production process.

SUMMARY OF THE INVENTION

In one aspect, this invention is a method comprising subjecting a reaction mixture containing an alkylene oxide, water and at least one NO_x or organic nitrogen compound to reaction conditions including an elevated temperature sufficient to convert at least a portion of the alkylene oxide to the corresponding alkylene glycol by reaction with water, wherein the reaction mixture further contains from 1 part per billion (ppb) to 20% by weight of the reaction mixture of a water-soluble reducing agent. Examples of suitable reducing agents include an alkali metal bisulfite, alkali metal sulfite or combination of two or more thereof. Applicants have found that the presence of the water-soluble reducing agent in many cases decreases the amounts of undesired side-reactions that occur when the reaction mixture is at the elevated temperature conditions. The method of this aspect of the invention tends to produce fewer colored impurities, fewer solid impurities and/or fewer foam-forming impurities than when the reducing agent is not present. As a result, subsequent purification processes are simplified and less expensive. Problems with plugging that can occur when solids are produced are avoided or reduced. The need to employ foam abatement measures is also reduced or eliminated. The formation of carbonyl compounds such as aldehydes can also be suppressed in this aspect of the invention, thus reducing or eliminating yield losses and downstream purification expenses that accompany the production of the carbonyl compounds.

In another aspect, this invention is a process that comprises removing a purge stream from an alkylene oxide production unit, the purge stream containing at least water, ethylene oxide and one or more NO_x and/or organic nitrogen compounds, and subjecting the purge stream to conditions including an elevated temperature sufficient to convert at least a portion of the ethylene oxide to ethylene glycol, wherein from about 1 part per billion to 20% by weight of a water-soluble reducing agent, based on the combined weight of purge stream and water-soluble reducing agent, is added to the purge stream prior to or during the time the purge stream is subjected to said elevated temperature.

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In another aspect, this invention is a process that comprises removing a purge stream from an ethylene oxide production unit, the purge stream containing at least water, ethylene glycol and/or ethylene oxide and one or more NO_x or organic nitrogen compounds, and subjecting the purge stream to conditions including an elevated temperature sufficient to recover water wherein from about 0.001 to 20% by weight of a water-soluble reducing agent, based on the combined weight of purge stream and water-soluble reducing agent, is added to the purge stream prior to or during the time the purge stream is subjected to said elevated temperature.

In another aspect, this invention relates to a method comprising subjecting a reaction mixture containing an alkylene oxide, water and at least one NO_x or organic nitrogen compound to reaction conditions including an elevated temperature sufficient to convert at least a portion of the alkylene oxide to the corresponding alkylene glycol, wherein the reaction mixture further contains from 1 part per billion to 20% by weight of a water-soluble reducing agent, wherein the reducing agent is a water-soluble sulfite, bisulfite or phosphite compound, hydroxylamine, or a mixture of two or more thereof.

In another aspect, this invention relates to a process that comprises removing a purge stream from an ethylene oxide production unit, the purge stream containing at least water, ethylene oxide and one or more NO_x or organic nitrogen compounds, and subjecting the purge stream to conditions including an elevated temperature sufficient to convert at least a portion of the ethylene oxide to ethylene glycol, wherein from about 0.001 to 20% by weight of a water-soluble reducing agent, based on the combined weight of purge stream and water-soluble reducing agent, is added to the purge stream prior to or during the time the purge stream is subjected to said elevated temperature and wherein the reducing agent is a water-soluble sulfite, bisulfite, or phosphite compound, hydroxylamine, or a mixture of two or more thereof.

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DETAILED DESCRIPTION OF THE INVENTION

In this invention, a water-soluble reducing agent is present in a reaction mixture that contains at least an alkylene oxide, water and at least one NO_x or organic nitrogen compound when the reaction mixture is subjected to conditions sufficient to convert all or
5 part of the alkylene oxide to the corresponding alkylene glycol.

The alkylene oxide is a 1,2-alkylene oxide such as ethylene oxide, propylene oxide, 1,2-butylene oxide, 1,2-hexene oxide and the like. The corresponding alkylene glycol is a vicinal dihydroxy alkane, such as 1,2-ethylene glycol, 1,2-propylene glycol, 1,2-butylene glycol, 1,2-hexane glycol and the like. The alkylene glycol of most interest by far is 1,2-
10 ethylene glycol.

The process stream contains, in addition to the alkylene oxide, at least one NO_x compound or organic nitrogen compound, or both. NO_x compounds (or precursors thereto) may be added into the alkylene oxide production process for various purposes. For example, these materials have been used as inhibitors as described in US. Patent No.
15 2,279,469. NO_x compounds are also used as co-promoters when certain alkene oxidation catalysts are used in the alkylene oxide-forming reaction, as described in U. S. Patent No. 6,511,938. For example, NO_x compounds are often fed into an ethylene oxide reactor when a silver alkene oxidation catalyst containing an alkali metal nitrate promoter is used. NO_x compounds include NO , NO_2 , N_2O_3 , N_2O_4 , N_2O_5 species, as well as nitrite or nitrate ion,
20 which is formed when these NO_x compounds become dissolved in water.

Organic nitrogen compounds that are present are often reaction products of one or more of the foregoing NO_x compounds with organic species, particularly alkenes, which are present in the process stream. They can also be formed by reaction of ammonia (which is sometimes introduced as a precursor to NO_x formation) with the organic compounds,

followed by oxidation. The predominant organic nitrogen compounds are nitro compounds such as nitromethane, 1-nitroethanol or nitroethylene, but other types of nitrogen-containing organic species can be formed and be present as well.

The NO_x compounds and organic nitrogen compounds are in most cases soluble in water or condensed alkylene oxide, or a mixture thereof. The process stream is generally a liquid under the conditions of the reaction to form the alkylene glycol. It preferably contains water as the primary component. Water may constitute from 50 to 99.5% of the weight of the process stream. A preferred water content is from 80 to 99% by weight and a more preferred water content is from 90 to 99% by weight. The process stream in most cases will contain an alkylene oxide and/or alkylene glycol, particularly ethylene oxide and/or ethylene glycol. The concentrations in the process stream may be in the range of 0.5 to 50%, preferably from 1 to 20% and more preferably from 1 to 10% by weight.

The concentration of NO_x and organic nitrogen compounds together may range from 0.1 to 20,000 parts per million, and is more typically from 1 to 2000 parts per million.

When NO_x compounds are introduced to the ethylene oxide reactor of an integrated ethylene oxide/ethylene glycol production facility, it is often preferred to remove the NO_x compounds and any organic nitrogen compounds that may form from the process streams before the main process stream is introduced into the ethylene glycol reaction unit. Some or all of the NO_x and organic nitrogen compounds are often removed as a purge stream which also contains significant levels of ethylene oxide. This purge steam can be obtained, for example, from a cooler in which water is condensed from an ethylene oxide-containing process stream or from an absorption unit or recovery column, in which the ethylene oxide-containing process stream is contacted with water to absorb ethylene oxide from the gaseous reaction mixture. The purge stream therefore will typically contain water in addition to ethylene oxide and NO_x and organic nitrogen compounds. The ethylene oxide content in such a purge stream often ranges from 0.25 to 20% by weight of the stream, and more often is from 0.5 to 5% of the weight of the stream. The total amount of ethylene oxide in the purge stream may constitute as much as 15% of the total ethylene oxide values produced in the process. More typically, from 0.5 to 10% of the ethylene oxide values are contained in the purge stream. A purge stream may contain up to 20,000 parts per million of NO_x and organic nitrogen compounds combined, and more typically contains from 5 to 2000 parts per million thereof.

The ethylene oxide values in such a purge stream are recovered by subjecting the purge stream to conditions such that they are converted to glycols. A glycol reactor, separate from the main glycol reactor, is generally used to accomplish this. Because the streams that are processed in this separate glycol reactor contain high levels of NO_x, organic nitrogen compounds and other impurities, the problems of color, solid formation and foaming tend to be particularly acute here. Carbonyl compounds can also form in such purge stream glycol units.

A process stream can also be formed by condensing crude alkylene oxide and adding water to the desired water and ethylene oxide contents. NO_x and organic nitrogen compounds in that case are carried over from the alkylene oxide-forming reaction in the crude alkylene oxide.

In this invention, the formation of these various types of byproducts is suppressed through the presence of the reducing agent in the process stream.

The reducing agent is water-soluble. It should not react significantly, under the conditions of the process, with any alkylene oxide or alkylene glycol that is present, although some reaction can be tolerated if yield losses are not too high. Suitable reducing agents include, for example, water-soluble sulfite, bisulfite, phosphite compounds, as well as hydroxylamine. Water-soluble sulfite and bisulfite salts are preferred. Suitable alkali metal sulfite and bisulfite salts include sodium sulfite, sodium bisulfite, potassium sulfite, potassium bisulfite, cesium sulfite, cesium bisulfite, lithium sulfite and lithium bisulfite. The sodium and potassium sulfites and bisulfites are preferred.

The reducing agent is generally benign to the overall process, and often can be introduced either into the process unit where it is needed, or at some upstream point from which it is carried through the process to the unit operations described before. However, the reducing agent is preferably not present in an upstream ethylene oxide reactor in which process streams are in the presence of the alkene oxidation catalyst. The reducing agent is therefore added downstream of the alkylene oxide reactor in an integrated process, such as in an ethylene oxide absorption column (where ethylene oxide is removed from process gasses through absorption into water), into a purge stream such as is described before, or into other equipment intermediate to the alkylene oxide reactor and alkylene oxide absorber.

The reducing agent can be added in any convenient manner. It is typically preferred to add it as a solution in water or other component of the process stream (such as the

alkylene glycol, for example). This can avoid introducing the reducing agent in the form of a solid, and the greater volume of a solution can make metering more accurate and easier to control, particularly when low levels of the reducing agent are added.

5 Under certain circumstances, the reducing agent may be generated *in situ*, by adding an appropriate precursor material. For example, sulfurous acid, sulfur dioxide, an organic ester of sulfurous acid, an addition product of bisulfite or sulfite with an organic material, or an alkali metal salt thereof can be added to a process stream having a pH of greater than 7, to form sulfite or bisulfite ions *in situ*.

10 The amount of reducing agent can range widely. The reducing agent can constitute from as little as 1 part per billion or as much as 20%, based on the weight of the process stream being treated. The amount that is added may be adjusted in response to the amount of NO_x and organic nitrogen compounds present in the process stream. Generally, excess amounts over what are needed are not harmful, although they can add unnecessary expense. When treating purge streams as described before, which may contain higher levels of NO_x or organic nitrogen compounds, a preferred level is from 100 parts per million to 3 weight
15 percent and a more preferred level is from 500 ppm to 3 weight percent. The reducing agent can be added continuously or intermittently as needed to maintain effective levels.

The process stream is subjected to conditions sufficient to cause the alkylene oxide to react with water in the process stream to form the alkylene glycol. Suitable conditions for
20 reacting an alkylene oxide with water to form the corresponding alkylene glycol are described, for example, in U. S. Patent Nos. 4,822,926, 3,922,314, or 6,514,388. Suitable conditions for operating an integrated ethylene oxide/ethylene glycol process are described, for example, in U. S. Patent No. 6,437,199.. Conditions for converting ethylene oxide values contained in a purge stream are described in U. S. Patent No. 4,822,926. The
25 conditions described therein are generally suitable for use with this invention.

Typically, those conditions will include an elevated temperature, such as from 100 to 210°C, especially from 150 to 190°C. The reaction conditions also will typically include a superatmospheric pressure, such as from 200 psig to 500 psig (379-3448 kPa) or more. Water is usually present in stoichiometric excess, relative to ethylene oxide. From 1 to 15
30 moles of water may be present per mole of alkylene oxide in the starting reaction mixture. The reaction may be catalyzed. Suitable catalysts for the reaction of alkylene oxides to the corresponding glycols are described in U. S. Patent No. 5,260,495.

The reaction to form the alkylene glycol is one of the corresponding alkylene oxide with water. The reaction of this process is not believed to proceed through an alkylene carbonate intermediate which can be formed in the presence of carbon dioxide. Carbon dioxide, if present at all, is preferably present in small quantities, such as less than 0.1 mole per mole of alkylene oxide in the process stream.

It is not normally necessary to make further adjustments to the process, other than supplying an effective amount of the reducing agent to the appropriate process stream. Conditions for the alkylene glycol-forming reaction and subsequent processing of the product stream can be operated in the same manner as when the reducing agent is absent.

The presence of the reducing agent has been found to reduce the levels of various types of impurities. Different types of impurities tend to form in various unit operations. These include carbonyl compounds such as aldehydes, and may include other impurities such as colorants, solids and foam-foamers. When solid impurities are formed in the absence of the reducing agent, it has been found that the addition of effective amounts of the reducing agent can reduce the solids formation by 70% or more, often by as much as 98%. The formation of carbonyl compounds is often reduced by 25% or more, often by 35% or more.

The following examples are provided to illustrate the invention, but are not intended to limit the scope thereof. All parts and percentages are by weight unless otherwise indicated.

Comparative Run A

Water containing approximately 2 wt% ethylene oxide condensed from the outlet of an ethylene oxide reactor is fed to a coiled eighteen-foot (5.5 meter) long by ¼ inch (6.25 mm) diameter stainless steel tubular reactor. The reaction mixture contains ethylene oxide, dissolved NO_x and dissolved organic nitrogen compounds. Conditions in the tubular reactor include 300 psig (2068 kPa) operating pressure and an operating temperature of 180°C. Residence time is 10 minutes. Ethylene oxide contained in the feed is converted to ethylene glycol in the tubular reactor. The process stream exiting the reactor is dark and foamy. Solids are isolated from the exiting process stream by filtering through a tarred, fine mesh filter and measuring the weight gain on the filter as the process steam is passed through it. The process stream contains 116 parts per million of suspended solids.

Example 1

Comparative Run A is repeated, except that 0.14% by weight sodium bisulfite is added to the process stream entering the tubular reactor. The outlet fluid is dark and foamy, but contains only 7 ppm of suspended solids. This represents a 94% reduction in solids compared to Comparative Run A.

Example 2

Comparative Run A is repeated, except that 0.42% by weight sodium bisulfite is added to the process stream entering the tubular reactor. The outlet fluid is much lighter in color than that of Comparative Run A and little foam is generated. The outlet fluid contains only 20 ppm of suspended solids, which represents a 82% reduction in solids compared to Comparative Run A.

Example 3

Comparative Run A is repeated, except that 1.25% by weight sodium bisulfite is added to the process stream entering the tubular reactor. The outlet fluid is much lighter in color than that of Comparative Run A and little foam is generated. The outlet fluid contains only 15 ppm of suspended solids, which represents an 87% reduction in solids compared to Comparative Run A.

Comparative Run B

Comparative Run A is repeated using another batch of water containing ethylene oxide condensed from an ethylene oxide reactor outlet. The process stream exiting the tubular reactor contains 42 ppm of solids.

Example 4

Comparative Run B is repeated, except that 1.8% by weight of a solution containing 11 ppm of sodium hydroxymethane sulfonate is added to the process stream entering the tubular reactor. The pH of the condensed water is >7. Under these conditions, the sodium hydroxymethane sulfonate salt is believed to form sodium sulfite and/or sodium bisulfite salts. The outlet fluid contains essentially no suspended solids.

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CLAIMS:

1. A method comprising subjecting a reaction mixture containing an alkylene oxide, water and at least one NO_x or organic nitrogen compound to reaction conditions including an elevated temperature sufficient to convert at least a portion of the alkylene oxide to the corresponding alkylene glycol, wherein the reaction mixture further contains from 1 part per billion to 20% by weight of a water-soluble reducing agent, wherein the reducing agent is a water-soluble sulfite, bisulfite or phosphite compound, hydroxylamine, or a mixture of two or more thereof.
2. The method of claim 1 wherein the alkylene oxide is ethylene oxide and the corresponding alkylene glycol is ethylene glycol.
3. The method of claim 1 or 2 wherein the reducing agent is sodium sulfite, sodium bisulfite, potassium sulfite, potassium bisulfite, cesium sulfite, cesium bisulfite, lithium sulfite, lithium bisulfite or a mixture of two or more thereof.
4. The method of any one of claims 1 to 3 wherein the reaction mixture contains from 500 ppm to 3% by weight of the reducing agent.
5. A process that comprises removing a purge stream from an ethylene oxide production unit, the purge stream containing at least water, ethylene oxide and one or more NO_x or organic nitrogen compounds, and subjecting the purge stream to conditions including an elevated temperature sufficient to convert at least a portion of the ethylene oxide to ethylene glycol, wherein from about 0.001 to 20% by weight of a water-soluble reducing agent, based on the combined weight of purge stream and water-soluble reducing agent, is added to the purge stream prior to or during the time the purge stream is subjected to said elevated temperature and wherein the reducing agent is a water-soluble sulfite, bisulfite, or phosphite compound, hydroxylamine, or a mixture of two or more thereof.

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6. The process of claim 5 wherein the reducing agent is sodium sulfite, sodium bisulfite, potassium sulfite, potassium bisulfite, cesium sulfite, cesium bisulfite, lithium sulfite, lithium bisulfite or a mixture of two or more thereof.

7. The process of claim 5 or 6 wherein the reaction mixture contains from
5 500 ppm to 3% by weight of the reducing agent.