

ISOPRENE SEPARATION AND RECOVERY METHOD AND ISOPRENE SEPARATION AND RECOVERY APPARATUS

TECHNICAL FIELD

5 **[0001]** The present disclosure relates to an isoprene separation and recovery
method and an isoprene separation and recovery apparatus, and, in particular,
relates to an isoprene separation and recovery method and an isoprene
separation and recovery apparatus that can reduce the amount of polymer of
isoprene that is produced during separation and recovery of isoprene from a
10 mixed fluid containing isoprene.

BACKGROUND

[0002] Methods using extractive distillation are known as methods for
separation and recovery of high-purity isoprene from a mixed fluid such as a
15 C5 fraction produced as a by-product in the manufacture of ethylene through
steam cracking or other high-temperature treatment of hydrocarbons.

[0003] In one specific example of a method for separation and recovery of
isoprene from a mixed fluid such as a C5 fraction using extractive distillation,
impurities having a similar boiling point to isoprene, such as pentane and
20 pentene, are separated from the mixed fluid containing isoprene in order to
separate and recover high-purity isoprene (for example, refer to Patent
Literature (PTL) 1).

[0004] This isoprene separation and recovery method using extractive
distillation can separate and remove impurities having a lower boiling point
25 than isoprene (for example, 1,4-pentadiene and 2-butyne) and impurities
having a higher boiling point than isoprene (for example, piperylene) that act
as a hinderance in a polymerization reaction for producing isoprene synthetic
rubber or the like.

30 CITATION LIST

Patent Literature

[0005] PTL 1: JP S47-41323 A

SUMMARY

(Technical Problem)

[0006] However, in the isoprene separation and recovery method using extractive distillation described above, it has not been possible to inhibit production of polymer of isoprene, which may act as a cause of apparatus staining.

[0007] Accordingly, an objective of the present disclosure is to provide an isoprene separation and recovery method and an isoprene separation and recovery apparatus that can reduce the amount of polymer of isoprene that is produced during separation and recovery of isoprene from a mixed fluid containing isoprene.

(Solution to Problem)

[0008] The present disclosure aims to advantageously solve the problem set forth above by disclosing an isoprene separation and recovery method for separating and recovering isoprene from a mixed fluid containing isoprene, comprising: an extractive distillation step of performing extractive distillation of the mixed fluid to obtain a fraction containing an impurity having lower solubility than isoprene in an extractant used in the extractive distillation and a fraction containing crude isoprene; and a dividing wall distillation step of performing dividing wall distillation of the fraction containing crude isoprene to obtain a fraction containing an impurity having a higher boiling point than isoprene, a fraction containing an impurity having a lower boiling point than isoprene, and a fraction containing high-purity isoprene. Performing dividing wall distillation of the fraction containing crude isoprene in this manner can reduce heat load and can reduce the amount of polymer of isoprene that is produced during separation and recovery of isoprene from the mixed fluid containing isoprene. As a consequence, it is possible to reduce the frequency of polymer cleaning, which it is necessary to perform while operation of an isoprene separation and recovery apparatus is suspended.

Note that the term “high-purity isoprene” as used in the present disclosure refers to “isoprene of higher concentration than isoprene concentration in crude isoprene”. The “isoprene concentration” referred to above can be measured by gas chromatography.

Moreover, the term “dividing wall distillation” as used in the present disclosure refers to the separation of a material that is to be distilled into three

or more (preferably three) fractions through a single distillation operation. This dividing wall distillation can be implemented, for example, using a vertical dividing wall column or the like.

[0009] In the presently disclosed isoprene separation and recovery method,
5 the extractant used in the extractive distillation step is preferably an amide compound, and more preferably dimethylformamide. The use of an amide compound as an extractant, and particularly of dimethylformamide, enables efficient implementation of extractive distillation.

[0010] The presently disclosed isoprene separation and recovery method
10 preferably further comprises a second extractive distillation step of performing further extractive distillation of the fraction containing high-purity isoprene to obtain a fraction containing an impurity having higher solubility than isoprene in an extractant used in the extractive distillation and a fraction containing even higher purity isoprene. Through further extractive
15 distillation (second extractive distillation) of the fraction containing high-purity isoprene, highly soluble substances (impurities having higher solubility than isoprene in the extractant) can be separated so as to obtain even higher purity isoprene. Moreover, by performing further extractive distillation (second extractive distillation) after dividing wall distillation,
20 even higher purity isoprene can efficiently be obtained because cyclopentadiene, which is a highly soluble substance, has already been separated through the dividing wall distillation.

[0011] In the presently disclosed isoprene separation and recovery method,
the extractant used in the second extractive distillation step is preferably an
25 amide compound, and more preferably dimethylformamide. The use of an amide compound as an extractant, and particularly of dimethylformamide, enables efficient implementation of extractive distillation.

[0012] In the presently disclosed isoprene separation and recovery method,
the fraction (mixed fluid) containing an impurity having a lower boiling point
30 than isoprene preferably has an isoprene concentration of not less than 5 mass% and not more than 90 mass%. When this mixed fluid has an isoprene concentration of not less than 5 mass% and not more than 90 mass%, high-purity isoprene can efficiently be separated and recovered from the mixed fluid.

[0013] In the presently disclosed isoprene separation and recovery method, the dividing wall distillation in the dividing wall distillation step is preferably performed in the presence of not less than 1 mass ppm and not more than 100 mass ppm of a polymerization inhibitor relative to the crude isoprene. By performing the dividing wall distillation in the presence of the amount of polymerization inhibitor set forth above, the occurrence of a polymerization reaction during the dividing wall distillation can be inhibited, and the occurrence of staining or blocking in a dividing wall column in which the dividing wall distillation is carried out can be prevented.

10 [0014] In the presently disclosed isoprene separation and recovery method, it is preferable that the dividing wall distillation in the dividing wall distillation step is performed in a dividing wall column, and a polymerization inhibitor is fed from a vertical direction upper part of the dividing wall column. By feeding the polymerization inhibitor from the vertical direction upper part of the dividing wall column, the polymerization inhibitor can be distributed throughout the entire dividing wall column.

Note that the term "vertical direction upper part of a dividing wall column" as used in the present disclosure refers to "an upper part corresponding to not less than 70% and not more than 100% of the vertical direction height of the dividing wall column".

20 [0015] In the presently disclosed isoprene separation and recovery method, the fraction containing high-purity isoprene is preferably a gas. When the fraction containing high-purity isoprene is a gas, this can prevent inclusion of polymerization inhibitor in the high-purity isoprene. As a consequence, the high-purity isoprene can advantageously be used in polymerization to obtain polyisoprene or the like.

[0016] The presently disclosed isoprene separation and recovery method preferably further comprises a step of removing a polymerization inhibitor from the fraction containing high-purity isoprene. By removing polymerization inhibitor from the fraction containing high-purity isoprene, the high-purity isoprene can advantageously be used in polymerization to obtain polyisoprene or the like.

30 [0017] Moreover, the present disclosure aims to advantageously solve the problem set forth above by disclosing an isoprene separation and recovery

apparatus comprising: an extractive distillation section that includes a feeding port for feeding of a mixed fluid containing isoprene and that performs extractive distillation of the mixed fluid to obtain a fraction containing an impurity having lower solubility than isoprene in an extractant used in the
5 extractive distillation and a fraction containing crude isoprene; and a dividing wall distillation section that includes a feeding port for feeding of the fraction containing crude isoprene and that performs dividing wall distillation of the fraction containing crude isoprene to obtain a fraction containing an impurity having a higher boiling point than isoprene, a fraction containing an impurity
10 having a lower boiling point than isoprene, and a fraction containing high-purity isoprene. Performing dividing wall distillation of the fraction containing crude isoprene can reduce heat load and can reduce the amount of polymer of isoprene that is produced during separation and recovery of isoprene from the mixed fluid containing isoprene. As a consequence, it is
15 possible to reduce the frequency of polymer cleaning, which it is necessary to perform while operation of the isoprene separation and recovery apparatus is suspended.

[0018] The presently disclosed isoprene separation and recovery apparatus preferably further comprises a second extractive distillation section that
20 includes a feeding port for feeding the fraction containing high-purity isoprene and that performs further extractive distillation of the fraction containing high-purity isoprene to obtain a fraction containing an impurity having higher solubility than isoprene in an extractant used in the extractive distillation and a fraction containing even higher purity isoprene. Through
25 further extractive distillation of the fraction containing high-purity isoprene, highly soluble substances (impurities having higher solubility than isoprene in the extractant) can efficiently be separated so as to obtain even higher purity isoprene.

(Advantageous Effect)

30 **[0019]** According to the present disclosure, it is possible to reduce the amount of polymer of isoprene that is produced during separation and recovery of isoprene from a mixed fluid containing isoprene.

BRIEF DESCRIPTION OF THE DRAWINGS

[0020] In the accompanying drawings:

FIG. 1 schematically illustrates configuration of one example of a presently disclosed isoprene separation and recovery apparatus; and

FIG. 2 schematically illustrates configuration of one example of a conventional isoprene separation and recovery apparatus.

DETAILED DESCRIPTION

[0021] The following provides a detailed description of embodiments of the present disclosure.

10 The presently disclosed isoprene separation and recovery method can be used in separation and recovery of isoprene from an isoprene-containing mixed fluid feedstock, such as a C5 fraction. Moreover, the presently disclosed isoprene separation and recovery apparatus can suitably be used, for example, in separation and recovery of isoprene by the presently disclosed
15 isoprene separation and recovery method.

[0022] (Isoprene separation and recovery method)

The presently disclosed isoprene separation and recovery method is a method for separating and recovering isoprene from a mixed fluid containing isoprene. The presently disclosed isoprene separation and recovery method
20 includes: a step of performing extractive distillation of the mixed fluid containing isoprene to obtain a fraction containing impurities having lower solubility than isoprene in an extractant used in the extractive distillation and a fraction containing crude isoprene (extractive distillation step); and a step of performing dividing wall distillation of the fraction containing crude isoprene,
25 optionally in the presence of a polymerization inhibitor, to obtain a fraction containing impurities having a higher boiling point than isoprene, a fraction containing impurities having a lower boiling point than isoprene, and a fraction containing high-purity isoprene (dividing wall distillation step), and may optionally further include: a step of performing further extractive
30 distillation of the fraction containing high-purity isoprene to obtain a fraction containing impurities having higher solubility than isoprene in an extractant used in the extractive distillation and a fraction containing even higher purity isoprene (second extractive distillation step); and a step of removing polymerization inhibitor used in the dividing wall distillation from the

fraction containing high-purity isoprene (removal step).

[0023] By performing dividing wall distillation of the fraction containing crude isoprene in the presently disclosed isoprene separation and recovery method, heat load can be reduced, and the amount of polymer of isoprene that is produced during separation and recovery of isoprene from the mixed fluid containing isoprene can be reduced. As a consequence, it is possible to reduce the frequency of polymer cleaning, which it is necessary to perform while operation of an isoprene separation and recovery apparatus is suspended.

[0024] Moreover, by performing dividing wall distillation in the presence of a polymerization inhibitor in the dividing wall distillation step of the presently disclosed isoprene separation and recovery method, it is possible to inhibit the occurrence of a polymerization reaction during dividing wall distillation. In general, a polymerization reaction tends to readily occur during dividing wall distillation because dividing wall distillation is implemented with a comparatively high operating pressure and temperature compared to normal distillation. However, by performing the dividing wall distillation in the presence of a polymerization inhibitor, the occurrence of a polymerization reaction during dividing wall distillation can be inhibited, and the occurrence of staining or blocking of an apparatus used to perform the dividing wall distillation (for example, a dividing wall column) can be prevented.

[0025] Furthermore, by removing polymerization inhibitor used in dividing wall distillation from the fraction containing high-purity isoprene, inclusion of polymerization inhibitor in the high-purity isoprene can be prevented. More specifically, when polymerization of high-purity isoprene is to be carried out, the polymerization is inhibited if polymerization inhibitor is contained in the high-purity isoprene. However, by removing the polymerization inhibitor used in dividing wall distillation, it is possible to prevent polymerization from being inhibited when polymerization of high-purity isoprene is to be carried out.

[0026] Moreover, by withdrawing the fraction containing high-purity isoprene as a gas in the dividing wall distillation step of the presently disclosed isoprene separation and recovery method, inclusion of polymerization inhibitor in the high-purity isoprene can be prevented. More specifically, when polymerization of high-purity isoprene is to be carried out,

the polymerization is inhibited if polymerization inhibitor is contained in the high-purity isoprene. However, by withdrawing the fraction containing high-purity isoprene as a gas, it is possible to prevent the inclusion of polymerization inhibitor in the high-purity isoprene and also prevent inhibition of polymerization when polymerization of the high-purity isoprene is to be carried out.

[0027] <Mixed fluid containing isoprene>

The mixed fluid preferably contains 1 mass% or more of isoprene. When the isoprene content of the mixed fluid is 1 mass% or more, isoprene can efficiently be separated and recovered from the mixed fluid.

The isoprene content in the mixed fluid is normally not less than 5 mass% and not more than 30 mass%. Besides isoprene, the mixed fluid may further contain n-pentane, 1,4-pentadiene, 2-butyne, piperylene, cyclopentadiene, and the like.

[0028] <Extractive distillation step>

In the extractive distillation step of the presently disclosed mixed fluid separation and recovery method, extractive distillation of a mixed fluid containing isoprene is performed to separate, from the mixed fluid, impurities of different solubility to isoprene in an extractant used in the extractive distillation, and to obtain a fraction containing crude isoprene.

[0029] [Extractive distillation]

FIG. 1 schematically illustrates configuration of one example of an isoprene separation and recovery apparatus that can implement the presently disclosed isoprene separation and recovery method.

In FIG. 1, an extractive distillation section 10 includes, for example, a first extractive distillation column 12 that performs extractive distillation of an isoprene-containing mixed fluid feedstock, such as a C5 fraction, to separate the mixed fluid feedstock into a fraction (C) and an extract (D), and a stripping column 13 that removes solvent from the extract (D).

[0030] In the first extractive distillation column 12, a solvent is fed from a higher level (vertical direction upper side) relative to a feeding level of the isoprene-containing mixed fluid feedstock (for example, a C5 fraction), and extractive distillation is performed so as to distil a fraction (C) containing n-pentane (boiling point: 36.1°C) and the like that are less soluble than

isoprene in the solvent from the top of the column and drain an extract (D) containing isoprene, 1,4-pentadiene, 2-butyne, piperylene, cyclopentadiene, and the like from the bottom of the column.

[0031] The solvent that is fed into the first extractive distillation column 12 may be a known solvent that is used in extractive distillation of a C5 fraction, such as any of the solvents described in WO 99/051552 A1, for example. Of these solvents, an amide compound is preferable, and dimethylformamide is more preferable. The use of an amide compound as an extractant, and particularly of dimethylformamide, enables efficient implementation of extractive distillation.

[0032] In the stripping column 13, solvent is drained from the bottom of the column and a fraction (X) containing isoprene, 1,4-pentadiene, 2-butyne, piperylene, cyclopentadiene, and the like (fraction containing crude isoprene) is distilled from the top of the column. Note that solvent recovered by the stripping column 13 may optionally be reused in the first extractive distillation column 12, the subsequently described second extractive distillation column 15, or the like.

[0033] <Dividing wall distillation step>

In the dividing wall distillation step of the presently disclosed isoprene separation and recovery method, dividing wall distillation of the fraction containing crude isoprene is performed to separate impurities having a higher boiling point than isoprene and impurities having a lower boiling point than isoprene from the fraction containing crude isoprene to thereby obtain a fraction containing high-purity isoprene. Performing dividing wall distillation of the fraction containing crude isoprene in this manner can reduce heat load and can reduce the amount of polymer of isoprene that is produced during separation and recovery of isoprene from the mixed fluid containing isoprene compared to a situation in which removal of high boiling point components by distillation and removal of low boiling point components by distillation are implemented separately in two stages. As a consequence, it is possible to reduce the frequency of polymer cleaning, which it is necessary to perform while operation of an isoprene separation and recovery apparatus is suspended.

[0034] [Dividing wall distillation]

The dividing wall distillation can obtain a fraction containing impurities having a higher boiling point than isoprene, a fraction containing impurities having a lower boiling point than isoprene, and a fraction containing high-purity isoprene by using a dividing wall column to discharge
5 high boiling point components (fraction containing impurities having a higher boiling point than isoprene) from the bottom of the column, discharge middle boiling point components from a central region of the column, and discharge low boiling point components (fraction containing impurities having a lower boiling point than isoprene) from the top of the column as described in JP
10 2016-524522 A, for example.

The isoprene concentration of the fraction containing impurities having a lower boiling point than isoprene (i.e., the isoprene concentration at the top of a dividing wall column 21) is preferably not less than 5 mass% and not more than 90 mass%, and more preferably not less than 10 mass% and not
15 more than 70 mass%. When the isoprene concentration of the fraction containing impurities having a lower boiling point than isoprene is not less than 5 mass% and not more than 90 mass% as set forth above, mixing of high boiling point components (impurities having a higher boiling point than isoprene) and low boiling point components (impurities having a lower
20 boiling point than isoprene) into the middle boiling point components can be prevented, and high boiling point components and low boiling point components can efficiently be separated.

Note that the isoprene concentration of the fraction containing impurities having a lower boiling point than isoprene is normally not less than
25 15 mass% and not more than 25 mass%.

Moreover, the fraction containing impurities having a higher boiling point than isoprene preferably contains 3 mass% or more of piperylene, which is a useful component.

[0035] -Dividing wall column-

30 The dividing wall column may, for example, be a vertical dividing wall column in which a dividing wall is disposed in a longitudinal direction of the column such as described in JP 2016-524522 A, for example. The dividing wall divides the inside of the column into, for example, a feeding section (left side of dividing wall), a removal section (right side of dividing wall), an

upper joined column section (rectifying section), and a lower joined column section (stripping section).

5 A feeding port (feeding inlet) for feeding of the fraction containing crude isoprene that is to be subjected to dividing wall distillation is normally disposed in a central region of the feeding section (i.e., between an upper region and a lower region of the feeding section). One or more further feeding ports (feeding inlets) may be disposed in the central region of the feeding section.

10 In general, one or more side-stream withdrawing sections are disposed in a central region of the removal section (i.e., between an upper region and a lower region of the removal section). One or more further side-stream outlets may be disposed in the central region of the removal section.

15 Compared to in a conventional case in which a high boiling distillation column and a low boiling distillation column are directly connected (for example, a case illustrated in FIG. 2 described further below), the use of a dividing wall column such as described above can provide roughly 30% cost reduction and allow reduction of the residence time of the processing target substance, and enables dividing wall distillation of a mixed fluid (separation and removal of high boiling point components and low boiling point components from the fraction containing crude isoprene) with low heat load.

20 **[0036]** The following describes a case in which dividing wall distillation is performed using a dividing wall distillation section composed of a dividing wall column with reference to the drawings.

25 In FIG. 1, a dividing wall distillation section 20 includes, for example, a dividing wall column 21 that performs dividing wall distillation of the fraction (X) containing crude isoprene that has been distilled from the top of the stripping column 13.

30 The dividing wall column 21, for example, distills a fraction (E) containing low boiling point impurities (for example, 1,4-pentadiene (boiling point: 26°C) and 2-butyne (boiling point: 27°C)) that were contained in the fraction (X) obtained in the extractive distillation section 10 from the top of the column, drains a fraction (F) containing high boiling point impurities (for example, piperylene (boiling point: 42°C)) that were contained in the fraction

(X) from the bottom of the column, and distills a fraction (G) in which isoprene (boiling point: 34.1°C) is enriched as high-purity isoprene from a central region of the column.

5 [0037] In general, a polymerization reaction tends to readily occur during dividing wall distillation because dividing wall distillation is implemented with a comparatively high operating pressure and temperature compared to normal distillation. Although a polymerization reaction readily occurs inside the dividing wall column 21, the occurrence of a polymerization reaction during dividing wall distillation can be inhibited and the occurrence of
10 staining or blocking of the dividing wall column 21 can be prevented by performing the dividing wall distillation in the presence of a polymerization inhibitor.

[0038] -Polymerization inhibitor-

Specific examples of polymerization inhibitors that may be used
15 include, but are not specifically limited to, 4-tert-butylcatechol (TBC) and diethylhydroxylamine (DEHA).

[0039] Although no specific limitations are placed on the amount of polymerization inhibitor that is present during dividing wall distillation, the amount is preferably 1 mass ppm or more relative to crude isoprene from a
20 viewpoint of inhibiting a polymerization reaction during dividing wall distillation and is preferably 100 mass ppm or less relative to crude isoprene from a viewpoint of reducing the polymerization inhibitor content in high-purity isoprene.

[0040] The location at which the polymerization inhibitor is fed is not
25 specifically limited so long as the polymerization inhibitor is present during dividing wall distillation. For example, the polymerization inhibitor may be fed directly into the dividing wall column 21 or may be fed into a pipe directly preceding the dividing wall column 21. The polymerization inhibitor can be distributed throughout the entire dividing wall column 21 by feeding the
30 polymerization inhibitor at a vertical direction upper part that is at a higher level (vertical direction upper side) relative to a feeding level of the fraction (X) containing crude isoprene in the dividing wall column 21.

[0041] Also note that by withdrawing the fraction containing high-purity isoprene from the dividing wall column 21 as a gas in the dividing wall

distillation section 20, inclusion of polymerization inhibitor in the high-purity isoprene can be prevented. More specifically, when polymerization of high-purity isoprene is to be carried out, the polymerization is inhibited if polymerization inhibitor is contained in the high-purity isoprene. However, by withdrawing the fraction containing high-purity isoprene from the dividing wall column 21 as a gas, it is possible to prevent the inclusion of polymerization inhibitor in the high-purity isoprene and also prevent inhibition of polymerization when polymerization of the high-purity isoprene is to be carried out.

10 **[0042]** <Second extractive distillation step>

In the presently disclosed isoprene separation and recovery method, it is preferable that further extractive distillation (second extractive distillation) of the fraction containing high-purity isoprene is performed. By carrying out further extractive distillation (second extractive distillation) of the fraction containing high-purity isoprene in this manner, a fraction containing even higher purity isoprene can be obtained.

Note that with regard to cyclopentadiene, which is a highly soluble substance in an extractant, reducing the cyclopentadiene concentration of the fraction containing high-purity isoprene in advance in the dividing wall distillation section enables efficient production of even higher purity isoprene through the second extractive distillation.

20 **[0043]** [Second extractive distillation]

In FIG. 1, a second extractive distillation section 30 includes a second extractive distillation column 15 that performs further extractive distillation (second extractive distillation) of the fraction (G) containing high-purity isoprene to separate the fraction (G) into a fraction (A) and an extract (B), and a stripping column 16 that removes solvent from the extract.

[0044] In the second extractive distillation column 15, a solvent is fed from a higher level (vertical direction upper side) relative to a feeding level of the fraction (G) containing high-purity isoprene that has been distilled from the dividing wall column 21 and further extractive distillation (second extractive distillation) of the fraction (G) containing high-purity isoprene is performed to distil a fraction (A) containing even higher purity isoprene from the top of the column and drain an extract (B) containing substances that are more

soluble than isoprene in the solvent (for example, cyclopentadiene and the like) from the bottom of the column. Moreover, in the stripping column 16, solvent is drained from the bottom of the column and substances that are more soluble than isoprene are distilled from the top of the column. Note that solvent recovered by the stripping column 16 may optionally be reused in the first extractive distillation column 12, the second extractive distillation column 15, or the like.

[0045] The solvent that is fed into the second extractive distillation column 15 may be a known solvent that is used in extractive distillation of a C5 fraction, such as any of the solvents described in WO 99/051552 A1, for example. Of these solvents, an amide compound is preferable, and dimethylformamide is more preferable. The use of an amide compound as an extractant, and particularly of dimethylformamide, enables efficient implementation of other extractive distillation.

[0046] <Removal step>

In the presently disclosed isoprene separation and recovery method, polymerization inhibitor used in dividing wall distillation in the dividing wall column 21 is removed from the fraction containing high-purity isoprene. By removing polymerization inhibitor used in dividing wall distillation in the dividing wall column 21 from the fraction containing high-purity isoprene in this manner, inclusion of polymerization inhibitor in the high-purity isoprene can be prevented. More specifically, when polymerization of high-purity isoprene is to be carried out, the polymerization is inhibited if polymerization inhibitor is contained in the high-purity isoprene. However, by removing the polymerization inhibitor used in dividing wall distillation in the dividing wall column 21 from the fraction containing high-purity isoprene, it is possible to prevent polymerization from being inhibited when polymerization of the high-purity isoprene is to be carried out.

Examples of methods by which the polymerization inhibitor may be removed include, but are not specifically limited to, a method in which the fraction containing high-purity isoprene is washed using water and a method in which the polymerization inhibitor is separated by distillation.

[0047] (Isoprene separation and recovery apparatus)

The presently disclosed isoprene separation and recovery apparatus

can suitably be used in separation and recovery of isoprene by the isoprene separation and recovery method set forth above. One example of the presently disclosed isoprene separation and recovery apparatus may, for example, have a configuration such as illustrated in FIG. 1.

5 **[0048]** A separation and recovery apparatus 100 illustrated in FIG. 1 includes an extractive distillation section 10 that performs extractive distillation of a mixed fluid used as a feedstock to obtain a fraction (X) containing crude isoprene, a dividing wall distillation section 20 that performs dividing wall distillation of the fraction (X) obtained in the extractive distillation section 10
10 to obtain a fraction (E) containing impurities having a lower boiling point than isoprene, a fraction (F) containing impurities having a higher boiling point than isoprene, and high-purity isoprene in the form of a fraction (G) in which high-purity isoprene is enriched, and a second extractive distillation section 30 that performs further extractive distillation of the fraction (G) in
15 which isoprene is enriched to obtain a fraction containing even higher purity isoprene. The mixed fluid used in the separation and recovery apparatus 100 may, for example, be a C5 fraction that is produced as a by-product in the manufacture of ethylene through steam cracking or other high-temperature treatment of hydrocarbons.

20 **[0049]** <Extractive distillation section>

 In FIG. 1, the extractive distillation section 10 includes, for example, a first extractive distillation column 12 that includes a feeding port for feeding an isoprene-containing mixed fluid feedstock, such as a C5 fraction, and that performs extractive distillation (first extractive distillation) of the mixed fluid
25 feedstock fed via the feeding port to separate the mixed fluid feedstock into a fraction (C) and an extract (D).

[0050] In the first extractive distillation column 12, a solvent is fed from a higher level (vertical direction upper side) relative to a feeding level of the mixed fluid and extractive distillation (first extractive distillation) of the
30 mixed fluid is performed to distil a fraction (C) containing pentane, pentene, and the like that are less soluble than isoprene in the solvent from the top of the column and drain an extract (D) containing isoprene, 1,4-pentadiene, 2-butyne, piperylene, cyclopentadiene, and the like from the bottom of the column.

[0051] The solvent that is fed into the first extractive distillation column 12 may be a known solvent that is used in extractive distillation of a C5 fraction, such as any of the solvents described in WO 99/051552 A1, for example. Of these solvents, an amide compound is preferable, and dimethylformamide is more preferable. The use of an amide compound as an extractant, and particularly of dimethylformamide, enables efficient implementation of extractive distillation.

[0052] In a stripping column 13, solvent is drained from the bottom of the column and a fraction (X) containing isoprene, 1,4-pentadiene, 2-butyne, piperylene, cyclopentadiene, and the like (fraction containing crude isoprene) is distilled from the top of the column. Note that solvent recovered by the stripping column 13 may optionally be reused in the first extractive distillation column 12, a second extractive distillation column 15, or the like.

[0053] <Dividing wall distillation section>

In FIG. 1, the dividing wall distillation section 20 includes, for example, a dividing wall column 21 that includes a feeding port for feeding the fraction (X) (fraction containing crude isoprene) obtained by removing solvent from the extract (D) and that performs dividing wall distillation of the fraction (X) fed via the feeding port (dividing wall distillation).

The dividing wall column 21, for example, distils a fraction (E) containing low boiling point impurities such as 1,4-pentadiene and 2-butyne from the top of the column, drains a fraction (F) containing high boiling point impurities such as piperylene from the bottom of the column, and distils a fraction (G) containing high-purity isoprene from a central region of the column.

[0054] <Second extractive distillation section>

In FIG. 1, the second extractive distillation section 30 includes a second extractive distillation column 15 that performs further extractive distillation (second extractive distillation) of the fraction (G) containing high-purity isoprene to separate the fraction (G) into a fraction (A) (fraction containing even higher purity isoprene) and an extract (B), and a stripping column 16 that removes solvent from the extract.

[0055] In the second extractive distillation column 15, a solvent is fed from a higher level (vertical direction upper side) relative to a feeding level of the

fraction (G) containing high-purity isoprene that has been distilled from the dividing wall column 21 and further extractive distillation (second extractive distillation) of the fraction (G) containing high-purity isoprene is performed to distil a fraction (A) containing even higher purity isoprene from the top of the column and drain an extract (B) containing substances that are more soluble than isoprene in the solvent (for example, cyclopentadiene and the like) from the bottom of the column. Moreover, in the stripping column 16, solvent is drained from the bottom of the column and substances that are more soluble than isoprene are distilled from the top of the column. Note that solvent recovered by the stripping column 16 may optionally be reused in the first extractive distillation column 12, the second extractive distillation column 15, or the like.

[0056] The solvent that is fed into the second extractive distillation column 15 may be a known solvent that is used in extractive distillation of a C5 fraction, such as any of the solvents described in WO 99/051552 A1, for example. Of these solvents, an amide compound is preferable, and dimethylformamide is more preferable. The use of an amide compound as an extractant, and particularly of dimethylformamide, enables efficient implementation of other extractive distillation.

[0057] Although the presently disclosed isoprene separation and recovery apparatus has been described using one example, the presently disclosed isoprene separation and recovery apparatus is not limited to the example described above. Moreover, the configurations of the extractive distillation section 10, the dividing wall distillation section 20, and the other extractive distillation section 30 are not limited to the configurations described above and may be changed to any configurations that enables separation and recovery of isoprene.

EXAMPLES

[0058] The following provides a more detailed description of the present disclosure through examples. However, the present disclosure is not limited to these examples.

[0059] (Example 1)

Separation and recovery of isoprene was performed using the

separation and recovery apparatus 100 illustrated in FIG. 1. More specifically, isoprene was separated and recovered from a mixed fluid formed from a C5 fraction that had been produced as a by-product in the manufacture of ethylene by steam cracking or other high-temperature treatment of hydrocarbons. The amount of isoprene in the mixed fluid formed from the C5 fraction was 20 mass%.

The compositions of the mixed fluid (C5 fraction), fraction (C), fraction (E), fraction (F), and fraction (G) were measured under the following conditions using a gas chromatograph (7890A produced by Agilent Technologies, Inc.). The results are shown in Table 1.

- Gas chromatograph: Agilent® (Agilent is a registered trademark in Japan, other countries, or both) 7890A (produced by Agilent Technologies, Inc.)
- Column: Agilent 19091P-S33, 30.0 m × 250 μm × 5.00 μm
- Column temperature: 35°C × 2.5 mins ⇒ heating at 5°C/min ⇒ 100°C ⇒ heating at 10°C/min ⇒ 180°C × 10 mins
- Injection temperature: 200°C
- Detector temperature: 200°C
- Carrier gas: Helium
- Split ratio: 200/1
- Detector: FID

[0060]

Table 1

	Mixed fluid (C5 fraction)	Fraction (C)	Fraction (E)	Fraction (F)	Fraction (G)
Pentane, pentene, etc. [mass%]	50	99	-	1	0.4
Cyclopentadiene [mass%]	2	-	-	3	0.4
1,4-Pentadiene [mass%]	2	-	40	-	-
2-Butyne [mass%]	1	-	40	-	-
Piperylene [mass%]	15	-	-	60	-
Isoprene [mass%]	20	1	20	1	99.2
Others [mass%]	10	-	-	35	-

[0061] (Comparative Example 1)

Separation and recovery of isoprene was performed in the same way as in Example 1 with the exception that a separation and recovery apparatus 200 illustrated in FIG. 2 was used instead of the separation and recovery apparatus illustrated in FIG. 1 in Example 1.

Note that the separation and recovery apparatus 200 is the same with the exception that the dividing wall distillation section 20 in FIG. 1 is replaced by an impurity removal section 40.

[0062] <Impurity removal section>

5 The impurity removal section 40 includes a low boiling point substance removal column 41 that removes impurities having a lower boiling point than isoprene that are contained in the fraction (X) and a high boiling point substance removal column 42 that removes impurities having a higher boiling point than isoprene that are contained in the fraction (X).

10 **[0063]** In the low boiling point substance removal column 41, a fraction (E) containing impurities having a lower boiling point than isoprene is distilled from the top of the column and a bottom liquid in which isoprene is enriched is drained from the bottom of the column.

[0064] Moreover, in the high boiling point substance removal column 42, a
15 fraction (G) in which isoprene is further enriched is distilled from the top of the column and a fraction (F) containing impurities having a higher boiling point than isoprene is drained from the bottom of the column.

[0065] When operation was performed continuously for 1 year in Example 1 and Comparative Example 1 and then the inside of the distillation columns
20 was inspected by eye, the amount of polymer of isoprene that had been produced was less in Example 1 than in Comparative Example 1.

INDUSTRIAL APPLICABILITY

[0066] According to the presently disclosed isoprene separation and recovery
25 method and isoprene separation and recovery apparatus, by performing dividing wall distillation, it is possible to reduce heat load and reduce the amount of polymer of isoprene that is produced, and it is also possible to reduce the frequency of polymer cleaning, which it is necessary to perform with operation of the isoprene separation and recovery apparatus suspended.

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REFERENCE SIGNS LIST

- [0067]** 10 extractive distillation section
 12 first extractive distillation column
 13 stripping column

	15	second extractive distillation column
	16	stripping column
	20	dividing wall distillation section
	21	dividing wall column
5	30	second extractive distillation section
	40	impurity removal section
	41	low boiling point substance removal column
	42	high boiling point substance removal column
	100	separation and recovery apparatus
10	200	separation and recovery apparatus

CLAIMS

1. An isoprene separation and recovery method for separating and recovering isoprene from a mixed fluid containing isoprene, comprising:

5 an extractive distillation step of performing extractive distillation of the mixed fluid to obtain a fraction containing an impurity having lower solubility than isoprene in an extractant used in the extractive distillation and a fraction containing crude isoprene; and

10 a dividing wall distillation step of performing dividing wall distillation of the fraction containing crude isoprene to obtain a fraction (F) containing an impurity having a higher boiling point than isoprene, a fraction (E) containing an impurity having a lower boiling point than isoprene, and a fraction (G) containing high-purity isoprene, wherein

15 the mixed fluid further contains at least one selected from the group consisting of pentane, pentene, cyclopentadiene, 1,4-pentadiene, 2-butyne, and piperylene,

 the extractant used in the extractive distillation step is an amide compound,

 the dividing wall distillation in the dividing wall distillation step is performed in a dividing wall column,

20 the dividing wall column distils the fraction (E) from the top of the column, drains the fraction (F) from the bottom of the column, and distils the fraction (G) from a central region of the column,

 the dividing wall column has a dividing wall disposed in a longitudinal direction inside,

25 the dividing wall divides the inside of the dividing wall column into a feeding section on one side of the dividing wall in the direction of thickness, a removal section on the other side of the dividing wall in the direction of thickness, an upper joined column section, and a lower joined column section, and

 a feeding port for feeding of the fraction containing crude isoprene is disposed in a central region between an upper region and a lower region of the feeding section.

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2. The isoprene separation and recovery method according to claim 1, wherein the amide compound is dimethylformamide.

3. The isoprene separation and recovery method according to claim 1 or 2, further comprising a second extractive distillation step of performing further extractive distillation of the fraction (G) containing high-purity isoprene to obtain a fraction
5 containing an impurity having higher solubility than isoprene in an extractant used in the extractive distillation and a fraction containing even higher purity isoprene.

4. The isoprene separation and recovery method according to claim 3, wherein the extractant used in the second extractive distillation step is an amide compound.
10

5. The isoprene separation and recovery method according to claim 4, wherein the amide compound used as an extractant in the second extractive distillation step is dimethylformamide.

15 6. The isoprene separation and recovery method according to any one of claims 1 to 5, wherein the fraction (E) containing the impurity having a lower boiling point than isoprene has an isoprene concentration of not less than 5 mass% and not more than 90 mass%.

20 7. The isoprene separation and recovery method according to any one of claims 1 to 6, wherein the dividing wall distillation in the dividing wall distillation step is performed in the presence of not less than 1 mass ppm and not more than 100 mass ppm of a polymerization inhibitor relative to the crude isoprene.

25 8. The isoprene separation and recovery method according to any one of claims 1 to 7, wherein a polymerization inhibitor is fed from a vertical direction upper part of the dividing wall column.

9. The isoprene separation and recovery method according to any one of claims
30 1 to 8, wherein the fraction (G) containing high-purity isoprene is a gas.

10. The isoprene separation and recovery method according to any one of claims 1 to 8, further comprising a step of removing a polymerization inhibitor from the fraction (G) containing high-purity isoprene.

5 11. An isoprene separation and recovery apparatus comprising:

an extractive distillation section that includes a feeding port for feeding of a mixed fluid containing isoprene and that performs extractive distillation of the mixed fluid by using an amide compound as an extractant to obtain a fraction containing an impurity having lower solubility than isoprene in an extractant used in the extractive
10 distillation and a fraction containing crude isoprene; and

a dividing wall distillation section that includes a dividing wall column including a feeding port for feeding of the fraction containing crude isoprene and that performs dividing wall distillation of the fraction containing crude isoprene to obtain a fraction (F) containing an impurity having a higher boiling point than isoprene, a
15 fraction (E) containing an impurity having a lower boiling point than isoprene, and a fraction (G) containing high-purity isoprene, wherein

the mixed fluid further contains at least one selected from the group consisting of pentane, pentene, cyclopentadiene, 1,4-pentadiene, 2-butyne, and piperylene,

the dividing wall column distils the fraction (E) from the top of the column,
20 drains the fraction (F) from the bottom of the column, and distils the fraction (G) from a central region of the column,

the dividing wall column has a dividing wall disposed in a longitudinal direction inside,

the dividing wall divides the inside of the dividing wall column into a feeding
25 section on one side of the dividing wall in the direction of thickness, a removal section on the other side of the dividing wall in the direction of thickness, an upper joined column section, and a lower joined column section, and

a feeding port for feeding of the fraction containing crude isoprene is disposed in a central region between an upper region and a lower region of the feeding section.

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12. The isoprene separation and recovery apparatus according to claim 11, further comprising a second extractive distillation section that includes a feeding port

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for feeding the fraction containing high-purity isoprene and that performs further extractive distillation of the fraction containing high-purity isoprene to obtain a fraction containing an impurity having higher solubility than isoprene in an extractant used in the extractive distillation and a fraction containing even higher purity isoprene.