RECOVERY OF VALUABLE CHEMICALS FROM INDUSTRY WASTE STREAM

Field of Invention

The present invention relates to recovery of valuable chemicals from industry waste stream, and more particularly relates to recovery of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid.

Background

The listing or discussion of a prior-published document in this specification should not necessarily be taken as an acknowledgement that the document is part of the state of the art or is common general knowledge.

As an important antioxidant agent, pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (IX1010) is used in polyolefins to stabilize the polymeric structure and to extend the lifetime of polyolefin products. IX1010 is produced via transesterification from methyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (Fig. 1, **A**, metilox) and pentaerythritol. The production lines produce metric tons of waste stream per day from tank cleaning. Such waste stream is a complex mixture, which usually contains a large amount of methanol (solvent), metilox (Fig. 1, **A**), pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (Fig. 1, **B,** IX1010), pentaerythritol tri(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (Fig. 1, **C**, triester), pentaerythritol di(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (Fig. 1, **D**, diester), pentaerythritol mono(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (Fig. 1, **E**, monoester), 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid (Fig. 1, **G**), 2,6-di-tert-butylphenol, 2,4-di-tert-butylphenol, methyl acrylate, iso-octanol, iso-propanol, and etc. It is difficult to isolate any single one of these structurally similar compounds from the waste stream by crystallization. While methanol can be recovered by distillation, the distillation process generates a large volume of viscous chemical residue that is currently incinerated.

The viscous chemical residue contains a number of valuable chemicals. However, due to the complex nature of the chemical residue, the only published processes that can recover valuable chemicals from the chemical residue involve harsh conditions, such as the recovery of metilox from the chemical residue by methanolysis (i.e. transesterification) and vacuum distillation at high temperatures of 150-180°C (e.g. CN1108236A, CN101007758B, CN103641715A and CN102060700A). However, this process is disadvantageous because

the distillation at 150-180°C requires a large amount of energy, increasing costs and environmental impact.

Therefore, there is a need to develop a process for the recovery of valuable chemicals from the chemical residue left over from the production of IX1010 under mild conditions and with a low energy consumption.

Summary of Invention

The current invention solves the problems associated with the prior art and discussed above. Thus, the invention enables the recovery of high value chemicals from the viscous chemical residue, under mild conditions and using aqueous solvents.

The invention therefore provides the following.

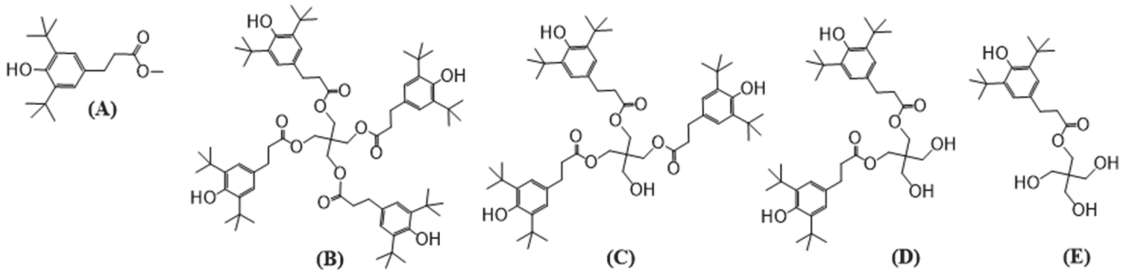
1. A process for recovering 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid from a stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid, which process comprises the steps of:

(i) hydrolysing the one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid in a basic solution to obtain a salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid;

(ii) acidifying the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid to obtain 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid.

2. The process according to Clause 1, wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid is a waste stream from a process for the production of pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (IX1010).

3. The process according to Clause 1 or 2, wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises one or more of the following esters (A) to (E):



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optionally wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises two or more of the esters (A) to (E),

more optionally wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises three or more of the esters (A) to (E),

further optionally wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises four or more of the esters (A) to (E),

such as wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises all of the esters (A) to (E).

4. The process according to any one of the preceding clauses, wherein the basic solution comprises a solvent selected from one or more of the group consisting of water, methanol, a mixture of methanol and water, a mixture of methanol and dichloromethane, a mixture of methanol and acetonitrile, and a mixture of methanol and tetrahydrofuran.

5. The process according to any one of the preceding clauses, wherein the basic solution comprises a base selected from one or more of the group consisting of potassium hydroxide, sodium hydroxide, potassium methoxide, sodium methoxide, potassium tert-butoxide, and sodium tert-butoxide.

6. The process according to Clause 5, wherein the base comprises sodium hydroxide.

7. The process according to any one of the preceding clauses, wherein step (i) is conducted at a temperature of from 10 to 150 °C, preferably from 20 to 80 °C.

8. The process according to any one of the preceding clauses, wherein hydrolysing the one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid in a basic solution is conducted for a reaction time of from 0.1 to 20 hours, preferably from 1 to 8 hours, more preferably from 4 to 6 hours.

9. The process according to any one of the preceding clauses, wherein the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid is a water-soluble salt,

optionally wherein the water-soluble salt has a solubility in water of at least 10 g per 100 g water at 25°C and 101 kPa.

10. The process according to any one of the preceding clauses, wherein step (i) further comprises extracting the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid in water, and removing insoluble impurities by filtration.

11. The process according to any one of the preceding clauses, wherein step (ii) comprises acidifying the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid with an acid selected from one or more of the group consisting of HCl, H_2SO_4, H_3PO_4, and acetic acid.

12. The process according to Clause 11, wherein the acid comprises HCl.

13. The process according to any one of the preceding clauses, wherein step (ii) is conducted at a reaction temperature of from 10 to 60 °C, preferably from 20 to 40 °C.

14. The process according to any one of the preceding clauses, wherein step (ii) is conducted for a reaction time of from 1 to 60 minutes, preferably from 10 to 30 minutes.

15. The process according to any one of the preceding clauses, further comprising a step: (iiia) purifying the 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid by washing the 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid with an alkane.

16. The process according to Clause 15, wherein the alkane is selected from one or more of the group consisting of pentane, hexane, heptane, octane, and cyclohexane.

17. The process according to Clause 15 or 16, wherein the alkane comprises hexane.

18. The process according to any one of Clauses 1 to 14, further comprising a step: (iiib) purifying the 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid by crystallisation in a solvent selected from one or more of the group consisting of methanol, dichloromethane, and acetonitrile.

Drawings

Fig. 1 depicts the steps in the recovery of **G** from a IX1010 waste stream.

Description

The invention provides a process for recovering 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid from a stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid, which process comprises the steps of:

(i) hydrolysing the one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid in a basic solution to obtain a salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid;

(ii) acidifying the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid to obtain 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid.

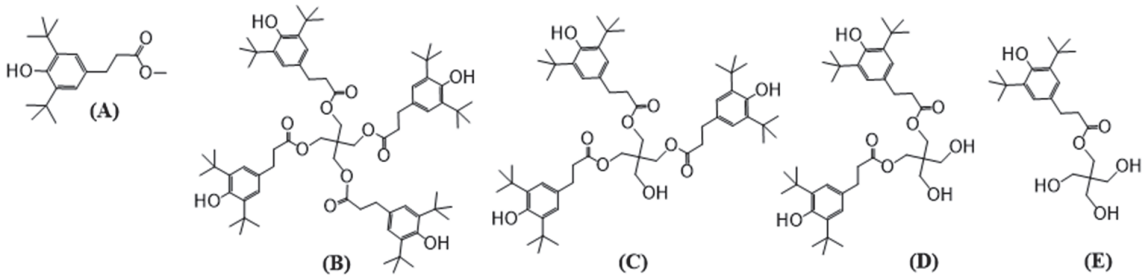
In embodiments herein, the word “comprising” may be interpreted as requiring the features mentioned, but not limiting the presence of other features. Alternatively, the word “comprising” may also relate to the situation where only the components/features listed are intended to be present (e.g. the word “comprising” may be replaced by the phrases “consists of” or “consists essentially of”). It is explicitly contemplated that both the broader and narrower interpretations can be applied to all aspects and embodiments of the present invention. In other words, the word “comprising” and synonyms thereof may be replaced by the phrase “consisting of” or the phrase “consists essentially of” or synonyms thereof and *vice versa*.

The phrase, “consists essentially of” and its pseudonyms may be interpreted herein to refer to a material where minor impurities may be present. For example, the material may be greater than or equal to 90% pure, such as greater than 95% pure, such as greater than 97% pure, such as greater than 99% pure, such as greater than 99.9% pure, such as greater than 99.99% pure, such as greater than 99.999% pure, such as 100% pure.

As used herein, the singular forms “a,” “an,” and “the” include plural referents unless the context clearly dictates otherwise. Thus, for example, reference to “a composition” includes mixtures of two or more such compositions, and the like.

The stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid may be any appropriate waste stream, and may particularly be a waste stream from a process for the production of pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (IX1010).

For example, the waste stream may comprise one or more of the following esters (A) to (E):



In some embodiments of the invention that may be mentioned herein, the waste stream may comprise two or more, three or more, four or more, or all five of, esters (A) to (E).

The hydrolysis step (i) involves hydrolysis of the one or more esters in a basic solution. In some embodiments of the invention that may be mentioned herein, the basic solution may comprise one or both of methanol and water, and may additionally comprise one or more other solvents. Thus, in some embodiments of the invention that may be mentioned herein, the basic solution may comprise a solvent selected from one or more of the group consisting of water, methanol, a mixture of methanol and water, a mixture of methanol and dichloromethane, a mixture of methanol and acetonitrile, and a mixture of methanol and tetrahydrofuran. As will be appreciated by a person skilled in the art, it may be preferable for the basic solution to comprise only water and/or methanol as the solvent (such as only methanol), to minimize the amount of organic solvent used in the process. It will be understood that any methanol used in the hydrolysis step may be recovered along with the other methanol present in the organic residue, such as by distillation.

The basic solution may comprise any appropriate base suitable for ester hydrolysis reactions. Thus, in some embodiments of the invention that may be mentioned herein, the basic solution may comprise a base selected from one or more of the group consisting of potassium hydroxide, sodium hydroxide, potassium methoxide, sodium methoxide, potassium tert-butoxide, and sodium tert-butoxide. A particular base that may be useful in the hydrolysis step is sodium hydroxide.

The hydrolysis step may be performed at any suitable temperature, such as from 10 to 150 °C, preferably from 20 to 80 °C. In order to reduce the total energy requirements of the process, the hydrolysis step may advantageously be performed at a temperature of from 20 to 80 °C, such as from 60 to 80 °C.

The hydrolysis step may be conducted for any suitable time period, such as from 0.1 to 20 hours. As will be appreciated by a person skilled in the art, the required reaction time may depend on the temperature. In some embodiments of the invention that may be mentioned herein, the hydrolysis step may be conducted for a reaction time of from 1 to 8 hours, such as from 4 to 6 hours.

After the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid has been subjected to a hydrolysis in basic solution, a salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid will be obtained. As will be appreciated by a person skilled in the art, the nature of the salt will depend on the base used. For example, if NaOH is used as the base then the obtained salt will be a sodium salt, while if KOH is used as the base then the obtained salt will be a potassium salt.

Preferably, the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid may be a water-soluble salt. For example, the water-soluble salt may have a solubility in water of at least 10 g of salt per 100 g water at 25°C and 101 kPa. The production of a highly water-soluble salt will facilitate the separation of the salt from water-insoluble impurities by a solvent extraction in water. This is advantageous from a cost and environmental perspective, since it avoids the need to use excess amounts of harmful organic solvents.

Therefore, in some embodiments of the invention that may be mentioned herein, step (i) may further comprise extracting the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid in water, and removing water-insoluble impurities by filtration.

The salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid may subsequently be reacted with an acid to obtain 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid. The process of the invention therefore comprises a step (ii) acidifying the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid to obtain 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid.

The acidification step may be conducted with any appropriate acid. Any acid that has a pK_a lower than that of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid may be reacted with the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid to provide 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid and a corresponding salt of the aforementioned acid having lower pK_a. In some embodiments of the invention that may be mentioned herein, the acid used in the acidification step may be selected from one or more of the group consisting of HCl, H_2SO_4, H_3PO_4, and acetic acid, such as HCl.

The acidification step may be conducted at any appropriate temperature, such as from 10 to 60 °C, preferably from 20 to 40 °C in order to reduce the energy requirements of the process.

The acidification step may be conducted for any appropriate time period, such as from 1 to 60 minutes, e.g. from 10 to 30 minutes.

The resulting 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid produced by the acidification step may be purified by any appropriate means. For example, in some embodiments of the invention that may be mentioned herein, the 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid may be purified by washing with an alkane, to remove organic impurities. Suitable alkanes for this washing step include one or more of the group consisting of pentane, hexane, heptane, octane, and cyclohexane (such as hexane). Another suitable purification step, which may be used in addition to, or instead of, the alkane washing step, is crystallisation. Thus, in some embodiments of the invention that may be mentioned herein, the 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid may be purified by crystallisation in a solvent selected from one or more of the group consisting of methanol, dichloromethane, and acetonitrile.

The invention is illustrated by the below Examples, which are not to be construed as limitative.

Examples

Materials

Deionized (DI) water was obtained from ELGA Purelab Option Water Purification System with DV25 Reservoir.
HCl (12 wt.%) was prepared from DI water and hydrochloric acid solution (36%) purchased from Sigma-Aldrich.
IX1010 waste stream was obtained from IX1010 production lines.
Potassium hydroxide (KOH): ≥85.0%, Merck KGaA.
Sodium hydroxyl (NaOH): ≥99.9%, Merck KGaA.
Chloroform-D (Deuterochloroform, CDCl3, 99.8% D): Cambridge Isotope Laboratories.
Hexane: ≥95%, Sigma-Aldrich.
Dichloromethane (DCM): ≥99.5%, Avantor Performance Materials.

Analytical techniques

Nuclear magnetic resonance (NMR) spectroscopy

^{1}H-NMR and ^{13}C-NMR spectroscopy were performed on a Jeol (Japan) 500MHz NMR spectrometer.

Example 1. Description of new procedure to recover G from chemical residue

Fig. 1 depicts a process for the recovery of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid, **G**, from a stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid (e.g. a IX1010 waste stream), including the following steps:

(1) hydrolysis of the one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid with a base (e.g. NaOH or KOH) in a solvent;
(2) recovery of the solvent;
(3) extraction of resulting residue with water to obtain a salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid;
(4) removal of water-insoluble impurities by filtration;
(5) acidification to produce water-insoluble **G**;
(6) isolation of **G** by filtration to remove water-soluble impurities; and
(7) purification by washing with hexane to remove hexane-soluble impurities.

As shown in Fig. 1, a waste stream comprising all of the [3-(3,5-di-tert-butyl-4-hydroxyphenyl) propanoate]-containing materials **A**, **B**, **C**, **D** and **E** may be hydrolysed with a base (e.g. NaOH or KOH) to form a (sodium or potassium) salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid which is soluble in water. Of course, the small amount of **G** present in the waste stream will also be converted to a (sodium or potassium) salt of **G**. The water-insoluble organic potion of the residue is removed by filtration. The aqueous phase is acidified with an acid that is a stronger acid than **G** (e.g. HCl or H_2SO_4), thus producing **G** [3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid] which will precipitate out from water together with any water-insoluble impurities. **G** is further purified by washing away organic impurities with an alkane such as pentane, hexane, octane, and/or cyclohexane.

The hydrolysis reaction can be carried out in various solvent systems, such as a mixture of methanol with water, a mixture of methanol with dichloromethane, a mixture of methanol with acetonitrile, a mixture of methanol with tetrahydrofuran, or use water only as reaction medium, or use methanol only as reaction medium.

Example 2. Production of G from the chemical residue after recovery of methanol

There are two main aspects in the isolation/recovery of **G** from IX1010 waste stream. The first is hydrolysis of all the [3-(3,5-di-tert-butyl-4-hydroxyphenyl) propanoate]-containing esters **A**, **B**, **C**, **D** and **E**. Other ester impurities (such as methyl acrylate) can also be hydrolyzed. The second is isolation and purification of **G** from the complex mixture. In general, the experimental operations can be conducted in two ways. In (A) below, the IX1010 waste solution may be hydrolysed directly, whereas in (B) it may be evaporated to form a residue that may be re-dissolved before hydrolysis.

(A) The original IX1010 waste solution can be used to run hydrolysis directly. In this case, the required amount of base (e.g. NaOH or KOH) will be added into the IX1010 waste solution, and then refluxed for several hours. The solvent (e.g. methanol) will be recovered by distillation. The dark brown residue will be extracted with DI water. The obtained aqueous solution will be acidified (e.g. with HCl) to produce crude product **G**, which will be purified by washing with an alkane (e.g. hexane). The white power product is isolated by filtration and characterized with ^{1}H-NMR and ^{13}C-NMR. The results show that the white powder is **G** [3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid].

(B) The original IX1010 waste solution will be distilled to recover methanol. The brown residue will be dissolved into a solvent (e.g. a mixture of dichloromethane and methanol (9:1 = v:v)). Then, a base (e.g. NaOH or KOH) will be added to run hydrolysis at room temperature for several hours under stirring. After dichloromethane and methanol are recovered by distillation, the brown residue will be extracted with DI water, and the obtained aqueous solution will be acidified (e.g. with HCl) to produce crude product **G**. Finally, **G** will also be purified by washing with an alkane (e.g. hexane).

In these procedures, there are three steps that remove three kinds of impurities: (1) water-insoluble impurities, (2) water soluble impurities, and (3) alkane soluble impurities. Therefore, the process of the invention can produce pure **G** as a valuable chemical from IX1010 waste stream.

Example 3. Recovery of methanol from IX1010 waste stream

To a 250 mL round-bottom flask (RBF), IX1010 waste solution (100 g, dark brown solution) was added. Colorless methanol was distilled out on a rotary evaporator at 40 °C under a reduced pressure of 130 mbar. After all the methanol was distilled out, a brown residue (44 g) was obtained, which is 44 wt% in the IX1010 waste solution. The methanol percentage is 56%

(56 g). The obtained methanol was analyzed with ^{1}H-NMR and ^{13}C-NMR. The results showed that the recovered methanol contains a trace amount of impurities, thus the purity is good enough for reuse in industry.

^{1}H-NMR of recovered methanol ($CDCl_3$, 500MHz, pm): 3.31 (3H, s, OC***H***$_3$), 3.74 (1H, s, O***H***). ^{13}C-NMR of recovered methanol ($CDCl_3$, 125MHz, pm): 49.92.

Example 4. Recover G from the original IX1010 waste solution

To a 250 mL RBF, IX1010 waste solution (100 g) and NaOH (7.5 g) were added. The mixture was refluxed for 4 hours under stirring. The solvent (methanol) was then recovered by distillation, leaving a wet brown residue, which was extracted with water (50 mL × 3). The aqueous solution was acidified with HCl (12 wt% aqueous solution) till pH = 2, which produced crude **G** with pale brown color. The pale brown **G** was isolated by filtration and washed with hexane (10 mL × 3), affording a white powder product. The white powder was dried in an oven at 60 °C. Yield = 10 g. ^{1}H-NMR and ^{13}C-NMR characterizations showed that the white powder product is **G** [3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid].

^{1}H-NMR of **G** ($CDCl_3$, 500MHz, ppm): 1.44 (18H, s, C***H***$_3$), 2.66 (2H, t, $J_{C\text{-}H}$=8Hz, C***H***$_2$), 2.89 (2H, t, $J_{C\text{-}H}$=8Hz, C***H***$_2$), 5.10 (1H, s, O***H***), 7.01 (2H, s, Ph-***H***).

^{13}C-NMR of **G** ($CDCl_3$, 125MHz, ppm): 30.44, 30.81, 34.46, 36.31, 124.95, 130.88, 136.12, 152.39, 179.33.

Example 5. Recover G from the brown residue of IX1010 waste solution

To a 250 mL RBF, chemical residue (44 g, obtained from Example 3), NaOH (7.5 g), and a mixture of dichloromethane with methanol (150 mL, 9:1 = v:v) were added. The mixture was stirred at room temperature for 6 hours. After dichloromethane and methanol were recovered by distillation, a wet brown residue was generated. The wet brown residue was extracted with water (50 mL × 3). The aqueous solution was acidified with HCl (12 wt% aqueous solution) till pH = 2, which produced crude **G** with pale brown color. The pale brown **G** was isolated by filtration and washed with hexane (10 mL × 3), affording a white powder product. The white powder was dried in an oven at 60 °C. Yield = 11 g. ^{1}H-NMR and ^{13}C-NMR characterizations showed that the white powder product is **G** [3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid].

Here, the inventors have developed a new procedure comprising (step-1) hydrolysis of viscous chemical residue, (step-2) acidification of hydrolyzed aqueous solution, and (step-3) isolation and purification of valuable chemical 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid (**G**). The recovery of purified 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid was confirmed with ^{1}H-NMR and ^{13}C-NMR.

Claims

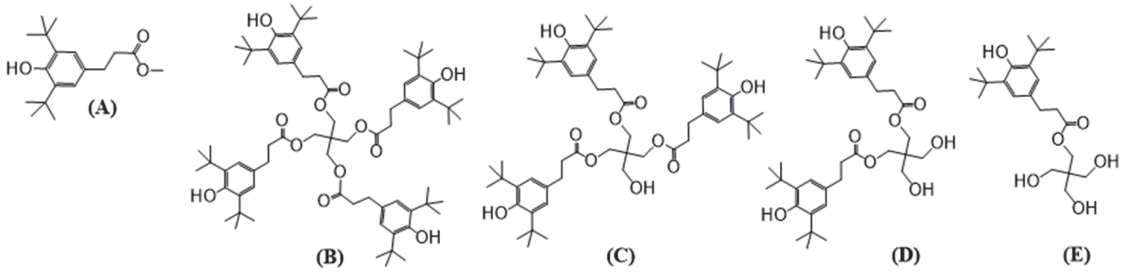
1. A process for recovering 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid from a stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid, which process comprises the steps of:

(i) hydrolysing the one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid in a basic solution to obtain a salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid;

(ii) acidifying the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid to obtain 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid.

2. The process according to Claim 1, wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid is a waste stream from a process for the production of pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (IX1010).

3. The process according to Claim 1 or 2, wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises one or more of the following esters (A) to (E):



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optionally wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises two or more of the esters (A) to (E),

more optionally wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises three or more of the esters (A) to (E),

further optionally wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises four or more of the esters (A) to (E),

such as wherein the stream comprising one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid comprises all of the esters (A) to (E).

4. The process according to any one of the preceding claims, wherein the basic solution comprises a solvent selected from one or more of the group consisting of water, methanol, a

mixture of methanol and water, a mixture of methanol and dichloromethane, a mixture of methanol and acetonitrile, and a mixture of methanol and tetrahydrofuran.

5. The process according to any one of the preceding claims, wherein the basic solution comprises a base selected from one or more of the group consisting of potassium hydroxide, sodium hydroxide, potassium methoxide, sodium methoxide, potassium tert-butoxide, and sodium tert-butoxide.

6. The process according to Claim 5, wherein the base comprises sodium hydroxide.

7. The process according to any one of the preceding claims, wherein step (i) is conducted at a temperature of from 10 to 150 °C, preferably from 20 to 80 °C.

8. The process according to any one of the preceding claims, wherein hydrolysing the one or more esters of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid in a basic solution is conducted for a reaction time of from 0.1 to 20 hours, preferably from 1 to 8 hours, more preferably from 4 to 6 hours.

9. The process according to any one of the preceding claims, wherein the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid is a water-soluble salt,

optionally wherein the water-soluble salt has a solubility in water of at least 10 g per 100 g water at 25°C and 101 kPa.

10. The process according to any one of the preceding claims, wherein step (i) further comprises extracting the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid in water, and removing insoluble impurities by filtration.

11. The process according to any one of the preceding claims, wherein step (ii) comprises acidifying the salt of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid with an acid selected from one or more of the group consisting of HCl, H_2SO_4, H_3PO_4, and acetic acid.

12. The process according to Claim 11, wherein the acid comprises HCl.

13. The process according to any one of the preceding claims, wherein step (ii) is conducted at a reaction temperature of from 10 to 60 °C, preferably from 20 to 40 °C.

14. The process according to any one of the preceding claims, wherein step (ii) is conducted for a reaction time of from 1 to 60 minutes, preferably from 10 to 30 minutes.

15. The process according to any one of the preceding claims, further comprising a step:
(iiia) purifying the 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid by washing the 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid with an alkane.

16. The process according to Claim 15, wherein the alkane is selected from one or more of the group consisting of pentane, hexane, heptane, octane, and cyclohexane.

17. The process according to Claim 15 or 16, wherein the alkane comprises hexane.

18. The process according to any one of Claims 1 to 14, further comprising a step:
(iiib) purifying the 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid by crystallisation in a solvent selected from one or more of the group consisting of methanol, dichloromethane, and acetonitrile.