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Description

The present invention is concerned with an improved process for preparing paraffin-sulfonic acids with a number of carbon atoms comprised within the range of from 10 to 20, as well as their salts. The process is based on the reaction of sulfo-oxidation of the concerned mixture of n-paraffins and on the removal from said reaction mixture of the therein formed sulfuric acid, which process is characterized by the addition of hydrogen peroxide to the reaction mixture before sulfuric acid is removed from it, and in that this removal of sulfuric acid is accomplished according to some particular treatments selected, alternatively to one another, from among such treatments as specified in the following.

From U.K. patent No. 1,194,699 to Hoechst it is known to prepare paraffin-sulfonic acids with a number of carbon atoms comprised within the range of from 10 to 20, by means of the sulfo-oxidation of n-paraffins with the same number of carbon atoms, with SO₂ and O₂ in the presence of water and using U.V. light in order to initiate said reaction.

The raw reaction product obtained from the sulfo-oxidation reactor is constituted by a mixture containing low percentages of paraffin-sulfonic acids, water, sulfuric acid, organic byproducts deriving from secondary reactions and, prevalently, by unreacted n-paraffins.

After a preliminary separation of a portion of sulfuric acid by heating to about 100°C, the raw reaction product is distilled at temperatures comprised within the range of from 60 to 180°C, with two phases being formed. The bottom phase is aqueous sulfuric acid, which is removed, and the top phase contains the unreacted n-paraffins and paraffin-sulfonic acids deprived of sulfuric acid.

The process disclosed in above said patent must mandatorily be carried out continuously, if one wants to avoid the formation of a product with colour characteristics which would render it unsuitable for the following uses.

From U.S. patent No. 4,696,097, it is known as well that the removal of sulfuric acid from the reaction mixture constituted by alkane-sulfonic acid, unreacted n-paraffins, SO₂, H₂O and H₂SO₄ can be improved by means of a process according to which SO₂ is initially vented and most H₂SO₄ is removed, according to U.K. patent No. 1,194,699, and to the so obtained concentrated mixture an alkali-metal compound (i.e., a salt, a peroxide or a hydroxide) is added: in that way, the residual sulfuric acid is transformed into bisulfate, which is removed.

However, the reported method, while is still affected by the drawback of a preliminary removal of most sulfuric acid, introduces an additional operating step with a solid body being formed, which could anyway cause problems, and, from the view point of the processes carried out batchwise, does not eliminate the drawback of the undesired formation of coloured useful product.

From Dutch patent application No. 6,812,646, it is known as well that light-coloured alkane-sulfonic acids, not containing sulfuric acid, can be prepared by means of a process according to which the reaction of sulfo-oxidation is carried first and the resulting solution is then heated at a temperature higher than 140°C, in the presence of H₂O₂ and under pressures higher than 405 kPa (4 atmospheres): this drastic treatment causes the nearly total dissociation of sulfuric acid and the formation of an aqueous solution composed by alkane-sulfonic acids, paraffinic hydrocarbons and a residual amount of sulfuric acid, which can be further removed by treatment under vacuum, at temperatures lower than 100°C.

The present Applicant has found now that paraffin-sulfonic acids with a number of carbon atoms comprised within the range of from 10 to 20, and their salts, free, or substantially free, from any residues of sulfuric acid, can be prepared without intermediate solid bodies being formed, and with no need of resorting to the use of drastic conditions of temperature and pressure: the product which is formed is endowed with all of the desired characteristics, in view of any possible successive applications.

In fact, the object of the instant invention is a process for preparing paraffin-sulfonic acids, and their salts, containing a number of carbon atoms comprised within the range of from 10 to 20, comprising the initial reaction of sulfo-oxidation of the concerned mixture of n-paraffins, the removal of unreacted n-paraffins from the reaction mixture, the removal of the excess of SO₂ from said mixture, and the removal from said reaction mixture of the therein formed sulfuric acid, with the end product being simultaneously recovered, characterized in that, after the removal of SO₂ from the reaction mixture hydrogen peroxide is added and the so obtained solution is submitted to one treatment selected from among the following treatments:

(a) addition of an alcohol with a number of carbon atoms comprised within the range of from 4 to 8;
(b) heating up to a temperature comprised within the range of from 50 to 150°C and vacuum distillation under a residual pressure comprised within the range of from 0.67 to 67 kPa (5 to 500 mmHg) until at least 60% of contained water is distilled off;
(c) addition of an either aliphatic or cycloaliphatic paraffin with a number of carbon atoms comprised within the range of from 6 to 8 and distillation of the resulting azetropic mixture at a temperature comprised within the range of from 20 to 110°C.

Hydrogen peroxide is added to the reaction mixture at a concentration comprised within the range of
from 40 to 120 volumes, in an amount comprised within the range of from 0.5 to 5% relatively to the above said mixture.

As said, the so obtained mixture can be submitted to a particular treatment in order to remove sulfuric acid formed in said reaction mixture, and recover the end product. As already seen, said treatment can be selected from among the following treatments:

(a) addition of an alcohol with a number of carbon atoms comprised within the range of from 4 to 8;,
(b) heating up to a temperature comprised within the range of from 50 to 150°C and vacuum distillation under a residual pressure comprised within the range of from 0.67 to 67 kPa (5 to 500 mmHg) until at least 60% of contained water is distilled off;
(c) addition of an either aliphatic or cycloaliphatic paraffin with a number of carbon atoms comprised within the range of from 8 to 15 and distillation of the resulting azeotropic mixture at a temperature comprised within the range of from 20 to 110°C.

In the first case, to the mixture an alcohol with a number of carbon atoms comprised within the range of from 4 to 8, preferably hexanol, is added, which causes the separation to take place of a heavy phase containing nearly all of H₂SO₄, which is thus removed, whilst the light phase is neutralized with alkali-metal hydroxides, with the salts of the paraffin-sulfonic acids being hence obtained after the removal of the n-paraffins still contained in the reaction mixture.

In the second case, after the addition of H₂O₂, the mixture is heated under vacuum to the temperature of 50-150°C, and is simultaneously submitted to a distillation under a residual pressure comprised within the range of from 0.67 to 67 kPa (5 to 500 mmHg), so as to remove not less than 60% of the water contained in the system.

After such a treatment, two phases are obtained, which are constituted as follows:

* light phase: containing the paraffin-sulfonic acids, n-paraffins, water and residual sulfuric acid;
* heavy-phase: constituted by a mixture of water and sulfuric acid, in which sulfuric acid has a concentration comprised within the range of from 25 to 70% as a function of the preceding distillation treatment.

The light phase of the above described double-phase system has a composition comprised within the following range:

* paraffin-sulfonic acids 35 - 41% by weight
* n-paraffins (C₁₀-C₂₀) 50 - 55% by weight
* sulfuric acid 2 - 7% by weight
* water 2 - 8% by weight

Such a phase is submitted to an extraction with supercritic CO₂, according to as disclosed in the European Patent n. 0261700 to the same Applicant's name, whose text is herein incorporated by reference in its entirety.

In particular, still with reference to the above cited European patent, the conditions of extraction of the above said phase with supercritic CO₂, result to be the following:

* extraction temperatures comprised within the range of from 32 to 80°C;
* extraction pressure comprised within the range of from 75 to 350 bar;
* weight ratio of supercritic CO₂, used for the extraction of the residual solution, to the paraffin-sulfonic acids contained in the reaction mixture, comprised within the range of from 1:1 to 50:1.

The mixture of paraffin-sulfonic acids resulting from this latter process can be generally neutralized in a way known from the prior art with any desired bases, with paraffin-sulfonates of any desired types being hence obtained.

In the (c) case, the solution obtained after the addition of H₂O₂ is submitted to a continuous treatment in order to remove water and sulfuric acid, according to which treatment an alifatic paraffin, with a number of carbon atoms comprised within the range of from 6 to 8, or a cycloaliphatic paraffin, is added, which forms an azeotropic mixture with the water contained in the above said mixture, said azeotropic mixture is heated up to a temperature comprised within the range of from 20 to 110°C, and is simultaneously submitted to an azeotropic distillation under a residual pressure comprised within the range of from 0.67 to 101 kPa (5 to 760 mmHg).

After such a treatment, two phases are obtained, which are constituted as follows:

* light phase: containing the paraffin-sulfonic acids, n-paraffins, residual water, residual sulfuric acid and the azeotropic mixture forming agent;
* heavy phase: constituted by a mixture of water and sulfuric acid, in which sulfuric acid has a concentration comprised within the range of from 25 to 70% as a function of the preceding azeotropic distillation treatment.

The light phase of the above described double-phase system, after the removal of the azeotropic mixture forming agent by distillation under vacuum, has a composition comprised within the following range:

* paraffin-sulfonic acids 40 - 55% by weight
* n-paraffins (C₁₀-C₂₀) 45 - 60% by weight
* sulfuric acid 0.5 - 4% by weight
* water 0.1 - 2% by weight

Such a phase is submitted to an extraction with supercritic, CO₂, according to as disclosed in already cited European Patent n. 261700 to the same Appli-
cant's name.

In particular, still with reference to the above cited European patent, the conditions of extraction of the above said phase with supercritic CO₂, result to be the following:

- extraction temperatures comprised within the range of from 32 to 80°C;
- extraction pressure comprised within the range of from 7.5 MPa to 35 MPa (75 to 350 bar);
- weight ratio of supercritic CO₂, used for the extraction of the residual solution, to the paraffin-sulfonic acids contained in the reaction mixture, comprised within the range of from 1:1 to 50:1.

The mixture of paraffin-sulfonic acids resulting from this latter process can be generally neutralized in a way known from the prior art, with any desired bases, with paraffin-sulfonates of any desired types being hence obtained.

In the following some examples are reported with the purpose of illustrating the same invention without limiting it.

Example No. 1

222.5 g of a raw reaction mixture of paraffin-sulfonic acids (from which decantable n-paraffins and SO₂ have been removed), obtained by means of the sulf-o-oxidation of (C₁₇-C₃₀)-n-paraffins, is treated in a flask of 500 ml of capacity with 2.3 g of H₂O₂ (80 volumes). The solution is heated to the temperature of 60-70°C, then 55.8 g of n-hexyl alcohol is added.

After phase decantation, 80.7 g of heavy phase and 192.2 g of light phase, containing the paraffin-sulfonic acids, n-hexyl alcohol and traces of H₂O and H₂SO₄ are separated.

Example No. 2

To 160 g of a raw reaction mixture of paraffin-sulfonic acids (from which decantable n-paraffins and SO₂ have been removed), obtained by means of the sulf-o-oxidation of (C₁₇-C₃₀)-n-paraffins, 1.9 g of H₂O₂ (80 volumes) is added. The solution is heated to the temperature of 60-70°C, then 40 g of n-hexyl alcohol is added.

The two phases are separated by centrifugation. The light phase, of 144.7 g, contains all of the paraffin-sulfonic acids, n-hexyl alcohol and traces of H₂O and H₂SO₄.

Example 3

A distillation equipment was used, which essentially consisted of a flask to which the mixture was charged, a condenser in order to cool the vapours, a content collecting vessel and a vacuum pump.

200 g of a raw reaction mixture having the following compositions:

- paraffin-sulfonic acids 22.4 %
- n-paraffins 31.3 %
- sulfuric acid 8.4 %
- water 35 %

was charged to a flask, to it 1 g of H₂O₂ (80 volumes) was added and the resulting mixture was stirred. Vacuum was applied to the flask, with a residual pressure of 4 kPa (30 mmHg) being obtained, and then the flask was heated, such as to cause 69.7 g of H₂O to distill off.

The pressure inside the flask was increased back up to atmospheric pressure, and 18.15 g of a heavy phase containing 67.45% of H₂SO₄ and 107 g of light phase were discharged. Said light phase had the following composition:

- paraffin-sulfonic acid 41 % by weight
- n-paraffins 54 % by weight
- sulfuric acid 2.3 % by weight
- water 2.6 % by weight

102.3 g of such a light phase was submitted to an extraction with supercritic CO₂.

The extraction was carried out at 45°C and 150 bar; the flow rate of CO₂ was kept constant; one hour later, the feed of CO₂ was interrupted and the refined product contained inside the extractor was discharged.

The analysis of such a product gave the following results:

- paraffin-sulfonic acid 88.85 % by weight
- n-paraffins 0.55 % by weight
- sulfuric acid 4.95 % by weight
- water 5.60 % by weight

Extracted n-paraffins are pure and can be recycled to the sulfo-oxidation reactor.

Example No. 4

1,000 g of raw reaction mixture (having the same composition as of the raw reaction mixture of Example No. 3) was charged to a flask and to it 9.5 g of H₂O₂ (80 volumes) was added.

The solution was heated up to its boiling point, was allowed to reflux for about 5 minutes and then was cooled down to 90°C.

Under these conditions, the solution separated into two phases: the heavy phase, of 153.4 g, was constituted by water and sulfuric acid at 25% by weight, and the light phase, of 854.6 g, had the following composition:

- paraffin-sulfonic acid 28.4 % by weight
- n-paraffins 39.1 % by weight
- sulfuric acid 5.5 % by weight
- water 27.7 % by weight

After phase separation, 497.7 g of the light phase was submitted to vacuum distillation in the same equipment as of Example No. 3.

Under a residual pressure of 2.67 kPa (20 mmHg)
and at the temperature of 80°C, 116.3 of water, equivalent to 84.36% of initially contained water, was distilled.

After distillation, the residual mixture separated into two phases: the heavy phase, of 33.9 g, contained 55.7% of sulfuric acid, and the light phase, of 349.3 g, had the following composition:
- paraffin-sulfonic acid 40.2% by weight
- n-paraffins 55.35% by weight
- sulfuric acid 2.2% by weight
- water 2.9% by weight

114.9 g of such a mixture was submitted to an extraction with supercritical CO₂.

The extraction was carried out at 45°C and 150 bar.

The end refined reaction product has the following composition:
- paraffin-sulfonic acid 88.05% by weight
- n-paraffins 0.25% by weight
- sulfuric acid 4.85% by weight
- water 6.30% by weight

Example No. 5

49.7 g of a raw solution of paraffin-sulfonic acids, obtained by means of the sulfo-oxidation of (C₁₀⁻C₂₀)-n-paraffins, deprived of decantable n-paraffins and of SO₂, and having the following composition:
- paraffin-sulfonic acids 28.1% by weight
- sulfuric acid 5.5% by weight
- water 26.6% by weight
- n-paraffins 39.8% by weight

and to which 0.5 g of H₂O₂ at 80 volumes were added, was charged to a flask of 0.5 litre of capacity and was treated with 251.7 g of n-heptane.

The solution was submitted to a distillation at a temperature comprised within the range of from 20 to 40°C and under a pressure comprised within the range of from 4 to 5.33 kPa (30 to 40 mmHg).

In that way, 10.12 g of water was distilled off, whilst n-heptane was recycled to the distillation flask.

Inside the flask the solution separated into two phases: the heavy phase, of 3.9 g, was constituted by H₂SO₄ at 55.8%, and the light phase, of 271 g, had the following composition:
- paraffin-sulfonic acids 5.30% by weight
- H₂SO₄ 0.18% by weight
- H₂O 0.70% by weight
- (C₁₀⁻C₂₀)-n-paraffins 7.15% by weight
- n-heptane 86.50% by weight

After distilling off all of n-heptane, said light phase was submitted to an extraction with supercritical CO₂.

The extraction was carried out at 45°C and 150 bar; the flow rate of CO₂ was kept constant.

One hour later, the feed of CO₂ was discontinued and the refined product contained inside the extractor was discharged.

The analysis of such a product gave the following results:
- paraffin-sulfonic acids 85.30% by weight
- H₂SO₄ 2.90% by weight
- H₂O 11.20% by weight
- n-paraffins 0.50% by weight

Extracted n-paraffins were pure and could be recycled to the sulfo-oxidation reactor.

Example No. 6

78.21 g of raw reaction mixture (deprived of decantable n-paraffins and of the excess of SO₂), containing the paraffin-sulfonic acids obtained by means of the sulfo-oxidation of (C₁₀⁻C₂₀)-n-paraffins and having the same composition as of Example No. 5, was charged to a flask of 1 litre of capacity, and to it 0.89 g of H₂O₂ (80 volumes) and 394.59 g of n-heptane were added.

The solution was submitted to a distillation at a temperature comprised within the range of from 85 to 104°C and under the atmospheric pressure.

16.4 g of water was distilled off, whilst n-heptane was recycled to the distillation flask.

Inside the flask two phases separated, in which the heavy phase, of 8.45 g, was constituted by an aqueous solution of sulfuric acid at 42.4%, and the light phase, of 447.8 g, had the following composition:
- paraffin-sulfonic acids 4.90% by weight
- H₂O 0.02% by weight
- H₂SO₄ 0.08% by weight
- (C₁₀⁻C₂₀)-n-paraffins 7.49% by weight
- n-heptane 87.50% by weight

After distilling off all of n-heptane under vacuum, said light phase was submitted to an extraction with supercritical CO₂.

The extraction was carried out at 45°C and 150 bar; the flow rate of CO₂ was kept constant.

After one hour, the feed of CO₂ was discontinued and the refined product contained inside the extractor was discharged.

The analysis of the obtained product gave the following results:
- paraffin-sulfonic acids 97.20% by weight
- H₂SO₄ 1.60% by weight
- H₂O 0.50% by weight
- n-paraffins 0.60% by weight

Extracted n-paraffins were pure and could be recycled to the sulfo-oxidation reactor.

Claims

1. Process for preparing paraffin-sulfonic acids containing a number of carbon atoms comprised within the range of from 10 to 20, as well as their
salties, which process comprises the initial reaction of sulfo-oxidation of the concerned mixture of n-paraffins, the removal of unreacted n-paraffins from the reaction mixture, the removal of the excess of SO₂ from said mixture, and the removal from said reaction mixture of the therein formed sulfuric acid, with the end product being simultaneously recovered, characterized in that, after the removal of SO₂, to the reaction mixture hydrogen peroxide is added and the so obtained solution is submitted to one treatment selected from among the following treatments:
(a) addition of an alcohol with a number of carbon atoms comprised within the range of from 4 to 8;
(b) heating up to a temperature comprised within the range of from 50 to 150°C and vacuum distillation under a residual pressure comprised within the range of from 0.67 to 67 kPa (5 to 500 mmHg) until at least 60% of contained water is distilled off;
(c) addition of an either aliphatic or cycloaliphatic paraffin with a number of carbon atoms comprised within the range of from 6 to 8 and distillation of the resulting azeotropic mixture to a temperature comprised within the range of from 20 to 110°C.

2. Process for preparing salts of paraffin-sulfonic acids according to claim 1, in which the alcohol used according to (a) preferably is hexanol.

3. Process for preparing paraffin-sulfonic acids according to claim 1, in which the residual solution, after the treatment according to (b), is submitted to an extraction with supercritic CO₂.

4. Process for preparing paraffin-sulfonic acids according to claim 1, in which the aliphatic paraffin, used according to (c) treatment, preferably is n-heptane.

5. Process for preparing paraffin-sulfonic acids according to claim 1, in which the cycloaliphatic paraffin, used according to the (c) treatment, preferably is cyclohexane.

6. Process for preparing paraffin-sulfonic acids according to claim 1, in which the residual solution, after the removal of the aqueous solution of H₂SO₄, in the treatment according to (c), is submitted to an extraction with supercritic CO₂.

Verfahren zur Herstellung von Paraffinsulfonsäuren mit einem Gehalt an 10 bis 20 Kohlenstoffatomen, sowie von ihren Salzen, welches Verfahren die Ausgangsreaktion der Sulfoxidation des entsprechenden Gemisches von n-Paraffinen, die Abtrennung der nich-umgesetzten n-Paraffine vom Reaktionsgemisch, die Abtrennung des SO₂-Überschusses von diesem Gemisch und die Abtrennung der dabei gebildeten Schwefelsäure aus diesem Reaktionsgemisch umfaßt, unter gleichzeitiger Gewinnung des Endproduktes, dadurch gekennzeichnet, daß nach der Abtrennung von SO₂ dem Reaktionsgemisch Wasserstoffperoxid zugesetzt wird und die solcherart erhaltene Lösung einer Behandlung unterworfen wird, die unter den folgenden Behandlungen ausgewählt ist:
(a) Zugabe eines Alkohols mit 4 bis 8 Kohlenstoffatomen;
(b) Erhitzen auf eine Temperatur im Bereich von 50 bis 150°C und Vakuumdestillation unter einem Restdruck im Bereich von 0.67 bis 67 kPa (5 bis 500 mmHg), bis wenigstens 60% des enthaltenen Wassers abdestilliert sind;
(c) Zugabe eines entweder aliphatischen oder cycloaliphatischen Paraffins mit 6 bis 8 Kohlenstoffatomen und Destillation des gebildeten azeotropen Gemisches bei einer Temperatur im Bereich von 20 bis 110°C.

2. Verfahren zur Herstellung von Salzen von Paraffinsulfonsäuren nach Anspruch 1, worin der gemäß (a) verwendete Alkohol vorzugsweise Hexanol ist.

3. Verfahren zur Herstellung von Paraffinsulfonsäuren nach Anspruch 1, worin die nach der Behandlung gemäß (b) verbleibende Lösung einer Extraktion mit überkritischem CO₂ unterworfen wird.

4. Verfahren zur Herstellung von Paraffinsulfonsäuren nach Anspruch 1, worin das gemäß Behandlung (c) verwendete aliphatische Paraffin vorzugsweise n-Heptan ist.

5. Verfahren zur Herstellung von Paraffinsulfonsäuren nach Anspruch 1, worin das gemäß Behandlung (c) verwendete cycloaliphatische Paraffin vorzugsweise Cyclohexan ist.

6. Verfahren zur Herstellung von Paraffinsulfonsäuren nach Anspruch 1, worin die in der Behandlung nach (c) nach der Abtrennung der wäßrigen H₂SO₄-Lösung verbleibende restliche Lösung einer Extraktion mit überkritischem CO₂ unterworfen wird.
Revendications

1. Procédé de préparation d'acides paraffine-sulfo-niques ayant un nombre d'atomes de carbone compris dans l'intervalle allant de 10 à 20, et de leurs sels, qui comprend la réaction initiale de sulfo-oxydation du mélange concerné de n-paraffines, l'élimination du mélange réactionnel des n-paraffines qui n'ont pas réagi, l'élimination de l'excès de SO₃ dudit mélange, et l'élimination du dit mélange réactionnel de l'acide sulfurique qui s'y est formé, le produit final étant simultanément récupéré, l'édit procédé étant caractérisé en ce que, après l'élimination de SO₃, on ajoute du peroxyde d'hydrogène au mélange réactionnel et on soumet la solution ainsi obtenue à un traitement choisi parmi les traitements suivants :
   (a) addition d'un alcool ayant un nombre d'atomes de carbone compris dans l'intervalle allant de 4 à 8,
   (b) chauffage jusqu'à une température comprise dans l'intervalle allant de 50 à 150°C et distillation sous vide, sous une pression résiduelle comprise dans l'intervalle allant de 0,67 à 67 kPa (5 à 500 mm de Hg), jusqu'à élimination par distillation d'au moins 60 % de l'eau contenue,
   (c) addition d'une paraffine aliphatique ou cycloaliphatique, ayant un nombre d'atomes de carbone compris dans l'intervalle allant de 6 à 8, et distillation du mélange azéotropique résultant à une température comprise dans l'intervalle allant de 20 à 110°C.

2. Procédé de préparation de sels d'acides paraffine-sulfoniques selon la revendication 1, dans lequel l'alcool utilisé conformément à (a) est de préférence de l'hexano 1.

3. Procédé de préparation d'acides paraffine-sulfoniques selon la revendication 1, dans lequel la solution résiduelle, après le traitement conformément à (b), est soumise à une extraction avec du CO₂ supercritique.

4. Procédé de préparation d'acides paraffine-sulfoniques selon la revendication 1, dans lequel la paraffine aliphatique, utilisée conformément au traitement (c), est de préférence du n-heptane.

5. Procédé de préparation d'acides paraffine-sulfoniques selon la revendication 1, dans lequel la paraffine cycloaliphatique, utilisée conformément au traitement (c), est de préférence du cyclohexane.

6. Procédé de préparation d'acides paraffine-sulfoniques selon la revendication 1, dans lequel la so-