PATENT SPECIFICATION

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1566

PROVISIONAL SPECIFICATION

Improvements in and Apparatus for the Manufacture and Production of Soaps from Oxidation Products of Non-Aromatic Hydrocarbons

I, GEORGE WILLIAM JOHNSON, a British Subject, of 47, Lincoln's Inn Fields, in the County of London, Gentleman, do hereby declare the nature of this invention (which has been communicated to me from abroad by I. G. Farbenindustrie Aktiengesellschaft, of Frankfort-on-Main, Germany, a Joint Stock Company organised under the Laws of Germany) to be as follows:—

In the oxidation of solid or liquid non-aromatic hydrocarbons with air or other gases containing oxygen there is formed, as is well known, a mixture of acid and neutral oxidation products from which fatty acids or soaps may be obtained. The fatty acids or soaps have, however, the drawback that they have an unpleasant characteristic odour which considerably impairs their capacity for being used for example as domestic or toilet soaps.

My foreign correspondents have now found that considerably better soaps can be obtained from the oxidation products of solid or liquid non-aromatic hydrocarbons by subjecting the saponified oxidation products to a treatment with steam. In this way the substances causing the unpleasant odour are almost completely removed and practically odourless soaps are obtained.

Oxidation products of solid or liquid non-aromatic hydrocarbons suitable for the process according to this invention are for example those of hard or soft paraffin wax, Diesel oils, cracking products of petroleums and the like or hydrogenation products of tars and the like. These are obtained in known manner by oxidation in the liquid phase at temperatures for example of from 100° to 160° Centigrade, if necessary while using catalysts, such as manganese compounds.

The removal of the unsaponifiable constituents, as for example the non-oxidised hydrocarbons, alcohols, ketones and the like, contained in the oxidation products may be effected, before or after the treatment with steam, by known methods, as for example by extraction with benzine. For the purpose of preliminary purification, the oxidation products may be treated with water, acids or organic solvents before the saponification, whereby for example water-soluble or readily soluble oxidation products of low boiling point are removed. The process may be carried out while using alkali salts of the acid oxidation products in aqueous solution or of alkaline earth or heavy metal salts in aqueous suspension or also in an anhydrous, used form. Mixtures of salts, as for example those containing sodium, potassium and magnesium salts, may also be subjected to the treatment with steam.

Ordinary, wet or superheated steam may be used according to this invention; it is advantageous to work according to the counter-current principle and the process may be carried out for example in columns provided with Raschig rings or bell platforms.

The amount of steam to be used according to this invention depends mainly on the amount of odorous substances present in the oxidation products. Generally speaking an amount of steam of from about 0.5 to 3 times the weight of the soap solution used is sufficient; it may readily be determined in each case by a small preliminary test. The temperature to be used depends on the nature of the oxidation products and the amount of odorous substances; temperatures below the distillation temperature of the unsaponifiable oxidation products are sufficient. Generally speaking temperatures between 100° and 200° Centigrade, advantageously between 150° and 200° Centigrade, are used.

For example if an oxidation product which still contains unsaponifiable constituents is to be worked up, the unsaponifiable constituents may be first removed by blowing in steam for a short time at temperatures at which the unsaponifiable constituents distil off, the blowing in of steam then being continued at lower temperatures, as for example from 150° to 200° Centigrade, until the odorous substances have been removed.

[Price 1/-]

The procedure may, however, also be reversed by first removing the odorous substances by blowing in steam for a long period at comparatively low temperatures, as for example from 150° to 200° Centigrade and then distilling off the unsaponifiable constituents by treatment for a short time at higher temperature.

In the case of alkali salts of the oxidation products, which frequently foam injuriously at atmospheric pressure, it is advantageous to carry out the treatment with steam under pressure and at elevated temperature, as for example between 140° and 300° Centigrade. The alkali salts of the oxidation products may be bleached if desired in known manner, as for example with hydrogen peroxide or sodium hypochlorite, after the treatment with steam. The odorless and colourless soaps thus obtained may be directly worked up into industrial finished products, such as domestic soaps or soap powders.

The process according to this invention may be carried out for example very advantageously by heating the soap solutions or the soap suspensions under pressure to high temperatures, as for example from 250° to 300° Centigrade, releasing the pressure on the hot solutions with the simultaneous evaporation of a part of the solvent water in a vessel in which there is then carried out a treatment with steam, preferably according to the counter-current principle. This method of working may be carried out for example in an apparatus such as is shewn diagrammatically in the accompanying drawing, but the invention is not restricted to the particular apparatus shewn.

Referring to the drawing, the oxidation product saponified with soda and freed from unsaponifiable constituents by extraction with benzine is led with a water content of about 60 per cent by means of a pump 1 through a preheater 2 in which it is heated under pressure to from 250° to 300° Centigrade. The hot soap solution then passes through a valve 3 with partial release of pressure and while being sprayed into a tower 4 in the pressure-release chamber 5 of which a part of the water is evaporated; this evaporated water passes through the conduit 6 to a condenser 7 from which it is withdrawn through a valve 8. The soap solution which is still at a temperature of about 150° Centigrade and which is under a pressure of from about 5 to 6 atmospheres, trickles down in the tower 4 over the Raschig ring filling 9 and collects in a sump 12. Steam is blown in simultaneously in counter-current to the trickling soap solution through a valve 11 and a distributing device 10; this carries away the odorous substances which have been only incompletely removed by the spraying of the soap solution. The odorless soap is withdrawn from the valve 13 after releasing the pressure to atmospheric pressure.

When the extraction of the oxidation products for the purpose of removing the unsaponifiable constituents has been carried out with benzine, alcohols of low molecular weight having been added in known manner for the avoidance of the formation of emulsions, the recovery of the organic solvents may be combined with the said purification of the soap solution under pressure. In this case it is preferable, however to carry out the spraying of the soap solution and the treatment with steam in two separate apparatus in order to avoid too great a dilution of the organic solvents.

The process according to this invention offers in particular the advantage that not only is a complete removal of the odorous substances effected but that there is practically no loss of initial materials.

The following Example will further illustrate the nature of this invention but the invention is not restricted to this Example.

EXAMPLE.

An oxidation product of hard paraffin wax containing 40 per cent of acid compounds (obtained by leading air into the fused paraffin wax at 115° Centigrade in the presence of a manganese catalyst) is washed with an equal volume of water at from 70° to 80° Centigrade and then saponified by treatment for 2 hours with 20 per cent soda solution under pressure at 200° Centigrade. After adding about 30 per cent of a 40 per cent aqueous isopropyl alcohol, the unsaponifiable constituents are removed from the saponification product by extraction with petroleum ether; the organic solvent is recovered from the soap solution by distillation.

The soap solution thus obtained, which contains about 35 per cent of fatty acids, is then heated to 160° Centigrade under pressure and treated with steam in counter-current in a tower filled with Raschig rings. The effluent vapours, which contain the odorous substances, are condensed. The soap treated with steam is then bleached with hydrogen peroxide. By evaporating and drying, a practically colourless product is obtained which may be worked up into soap powder.

Dated this 21st day of September, 1936.

J. Y. & G. W. JOHNSON,
47, Lincoln's Inn Fields,
London, W.C.2,
Agents.

COMPLETE SPECIFICATION

Improvements in and Apparatus for the Manufacture and Production of Soaps from Oxidation Products of Non-Aromatic Hydrocarbons

I, GEORGE WILLIAM JOHNSON, a British Subject, of 47, Lincoln's Inn Fields, in the County of London, Gentleman, do hereby declare the nature of this invention (which has been communicated to me from abroad by I. G. Farbenindustrie Aktiengesellschaft, of Frankfort-on-Main, Germany, a Joint Stock Company organised under the Laws of Germany) and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

In the oxidation of solid or liquid non-aromatic hydrocarbons with air or other gases containing oxygen there is formed, as is well known, a mixture of acid and neutral oxidation products from which fatty acids or soaps may be obtained. The fatty acids or soaps have, however, the drawback that they have an unpleasant characteristic odour which considerably impairs their capacity for being used for example as domestic or toilet soaps. It is known (see specification No. 396,769) to remove the unsaponifiable constituents from the saponified oxidation products by a treatment with steam at temperatures up to 310° Centigrade.

My foreign correspondents have now found that considerably better soaps can be obtained from the oxidation products of solid or liquid non-aromatic hydrocarbons obtained by oxidation with air or other gases containing oxygen by subjecting the saponified oxidation products to a treatment with steam at a temperature below that necessary for distilling off the higher boiling unsaponifiable constituents for such a length of time that the unpleasant odour is removed. The unsaponified constituents are removed after such treatment or still better prior to it. In this way the substances causing the unpleasant odour are almost completely removed and practically odourless soaps are obtained.

Oxidation products of solid or liquid non-aromatic hydrocarbons suitable for the process according to this invention are for example those of hard or soft paraffin wax, Diesel oils, cracking products of petroleums and the like or hydrogenation products of tars and the like. These are obtained in known manner by oxidation in the liquid phase at temperatures for example of from 100° to 160° Centigrade, if necessary while using catalysts such as manganese compounds, with air or other gases containing oxygen.

The treatment with steam of the oxidation products so obtained may be carried out while using alkali salts of the acid oxidation products in aqueous solution or of alkaline earth or heavy metal salts in aqueous suspension or also in an anhydrous, fused form. Mixtures of salts, as for example those containing sodium, potassium and magnesium salts, may also be subjected to the treatment with steam.

Ordinary, wet or superheated steam may be used according to this invention; it is advantageous to work according to the counter-current principle and the process may be carried out for example in columns provided with Raschig rings or bell platforms.

The amount of steam to be used according to this invention depends mainly on the amount of odorous substances present in the oxidation products. Generally speaking an amount of steam of from about 0.5 to 3 times the weight of the soap solution used is sufficient; it may readily be determined in each case by a small preliminary test. The temperature to be used depends on the nature of the oxidation products and the amount of odorous substances; temperatures below the distillation temperature of the unsaponifiable oxidation products are sufficient. Generally speaking temperatures between 80° and 200° Centigrade, advantageously between 150° and 200° Centigrade, are used.

The removal of the unsaponifiable constituents, as for example the non-oxidised hydrocarbons, alcohols, ketones and the like, contained in the oxidation products may be effected, before or after the treatment with steam, by known methods, as for example by extraction with benzine. For the purpose of preliminary purification, the oxidation products may be treated with water, acids or organic solvents before the saponification, whereby for example water-soluble or readily

soluble oxidation products of low boiling point are removed.

For example if an oxidation product which still contains unsaponifiable constituents is to be worked up, the unsaponifiable constituents may be first removed by blowing in steam for a short time (insufficient to remove much of the odorous substances) at temperatures at which the unsaponifiable constituents distil off, the blowing in of steam then being continued at lower temperatures, as for example from 150° to 200° Centigrade, until the odorous substances have been removed. The procedure may, however, also be reversed by first removing the odorous substances by blowing in steam for a long period at comparatively low temperatures, as for example from 150° to 200° Centigrade and then distilling off the unsaponifiable constituents by treatment for a short time at higher temperature.

In the case of alkali salts of the oxidation products, which frequently foam injuriously at atmospheric pressure, it is advantageous to carry out the treatment with steam under pressure and at elevated temperature, as for example between 140° and 300° Centigrade. The alkali salts of the oxidation products may be bleached if desired in known manner, as for example with hydrogen peroxide or sodium hypochlorite, after the treatment with steam. The odorless and colourless soaps thus obtained may be directly worked up into industrial finished products, such as domestic soaps or soap powders.

The process according to this invention may be carried out for example very advantageously by heating the soap solutions or the soap suspensions under pressure to high temperatures, as for example from 250° to 300° Centigrade, releasing the pressure on the hot solutions with the simultaneous evaporation of a part of the solvent water in a vessel in which there is then carried out a treatment with steam, preferably according to the counter-current principle. This method of working may be carried out for example in an apparatus such as is shewn diagrammatically in the drawing accompanying the Provisional Specification, but the invention is not restricted to the particular apparatus shewn.

Referring to the drawing, the oxidation product saponified with soda and freed from unsaponifiable constituents by extraction with benzine is led with a water content of about 60 per cent by means of a pump 1 through a preheater 2 in which it is heated under pressure to from 250° to 300° Centigrade. The hot soap solution then passes through a valve 3 with partial release of pressure and while being sprayed into a tower 4 in the pressure-release chamber 5 of which a part of the water is evaporated; this evaporated water passes through the conduit 6 to a condenser 7 from which it is withdrawn through a valve 8. The soap solution which is still at a temperature of about 150° Centigrade and which is under a pressure of from about 5 to 6 atmospheres, trickles down in the tower 4 over the Raschig ring filling 9 and collects in a sump 12. Steam is blown in simultaneously in counter-current to the trickling soap solution through a valve 11 and a distributing device 10; this carries away the odorous substances which have been only incompletely removed by the spraying of the soap solution. The odorless soap is withdrawn with the valve 13 after releasing the pressure to atmospheric pressure.

When the extraction of the oxidation products for the purpose of removing the unsaponifiable constituents has been carried out with benzine, alcohols of low molecular weight having been added in known manner for the avoidance of the formation of emulsions, the recovery of the organic solvents may be combined with the said purification of the soap solution under pressure. In this case it is preferable, however, to carry out the spraying of the soap solution and the treatment with steam in two separate apparatus in order to avoid too great a dilution of the organic solvents.

The process according to this invention offers in particular the advantage that not only is a complete removal of the odorous substances effected but that there is practically no loss of initial materials.

The following Examples will further illustrate how the said invention may be carried out in practice but the invention is not restricted to these Examples. The parts are by weight.

EXAMPLE 1.

An oxidation product of hard paraffin wax containing 40 per cent of acid compounds (obtained by leading air into the fused paraffin wax at 115° Centigrade in the presence of a manganese catalyst) is washed with an equal volume of water at from 70° to 80° Centigrade and then saponified by treatment for 2 hours with 20 per cent soda solution under pressure at 200° Centigrade. After adding about 30 per cent of a 40 per cent aqueous isopropyl alcohol, the unsapomifiable constituents are removed from the saponification product by extraction with petroleum ether; the organic solvent is recovered from the soap solution by dis-

tillation.

The soap solution thus obtained, which contains about 35 per cent of fatty acids, is then heated to 160° Centigrade under pressure and treated with steam in counter-current in a tower filled with Raschig rings. The effluent vapours, which contain the odorous substances, are condensed. The soap treated with steam is then bleached with hydrogen peroxide. By evaporating and drying, a practically colourless product is obtained which may be worked up into soap powder.

EXAMPLE 2.

A mixture consisting of 40 parts of crude scale wax and 60 parts of unsaponifiable constituents obtained from an oxidation product of crude scale wax is oxidised at 115° Centigrade in the presence of 0.15 per cent potassium permanganate with air until the oxidation product has an acid value of 68. The oxidation product is then saponified with a 15 per cent aqueous soda solution at 230° Centigrade under superatmospheric pressure for about 1 hour and subsequently the mixture is diluted with water and ethyl alcohol in such a manner that a 20 per cent soap solution is obtained. This solution is subjected to extraction with a benzine fraction boiling between 80° and 120° Centigrade in a tower provided with filler bodies in counter-current. The ratio by volume of the benzine to the soap-solution is chosen in a manner sufficiently high for practically completely removing the unsaponified constituents from the soap solution.

The benzine fraction is then separated from the soap solution which contains small amounts of benzine and practically the whole of the alcohol employed. The soap solution is heated up to 250° Centigrade under superatmospheric pressure and then while partially releasing the pressure led through a valve into the upper part of a tower in which a part of the water and the solvents are distilled off. The concentrated soap solution flows downwardly over the Raschig rings contained in the lower part of the tower whereby it is simultaneously treated in counter-current with steam. By this treatment the undesired odorous substances are removed from the soap solution. From this soap solution, soap powders or pure fatty acids may be prepared in any known manner. The products thus obtained also have no undesired odour. The mixture of benzine, alcohol and water which is obtained by condensing the vapours can be subjected to a fractionated distillation whereby the benzine and alcohol can be recovered.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. A process for the manufacture and production of soaps free from unpleasant odour from oxidation products of solid or liquid non-aromatic hydrocarbons obtained by oxidation with air or other gases containing oxygen by subjecting the saponified oxidation products to a treatment with steam at a temperature below that necessary for distilling off the higher boiling unsaponifiable constituents for such a length of time that the unpleasant odour is removed.

2. The process for the manufacture and production of soaps free from unpleasant odour from oxidation products of solid or liquid non-aromatic hydrocarbons as herein particularly described and ascertained.

3. The process for the manufacture and production of soaps free from unpleasant odour from oxidation products of solid or liquid non-aromatic hydrocarbons substantially as described in the foregoing Examples.

4. Soaps free from unpleasant odour when obtained according to the process particularly described and ascertained.

Dated the 9th day of September, 1937.

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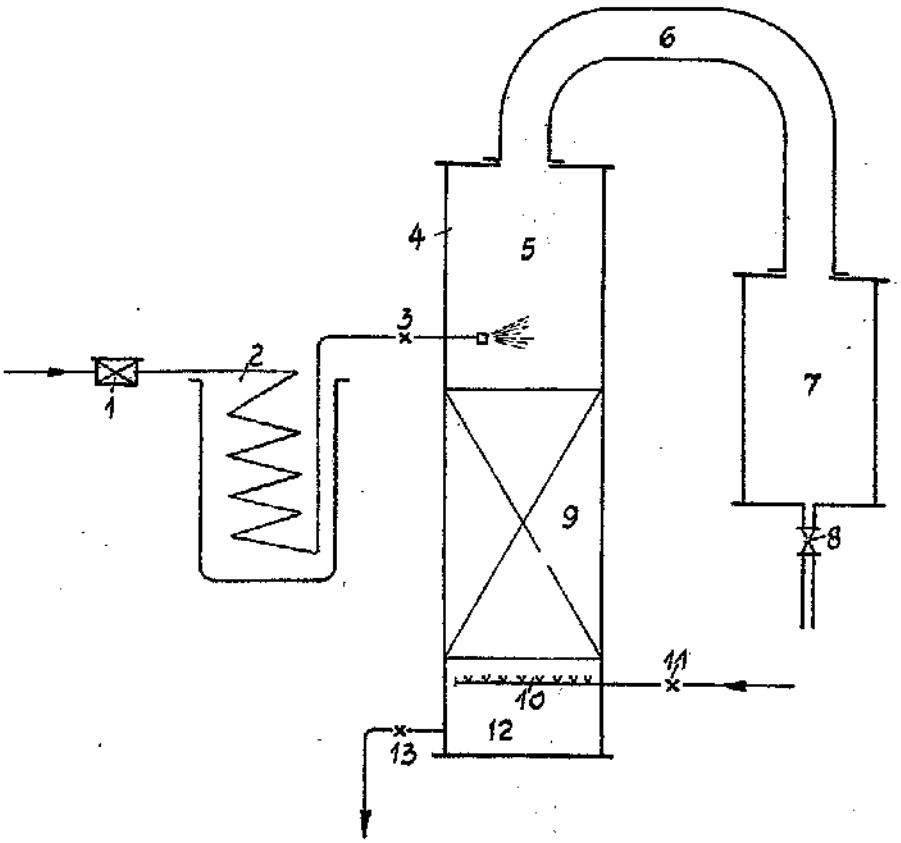

[This Drawing is a reproduction of the Original on a reduced scale.]

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