27129

38290

27129

A PROCESS AND AN APPARATUS FOR THE SEPARATION OF PROPYLENE GLYCOL

ABSTRACT

The invention relates to a process for the separation of propylene glycol from a mixture of low-boiling fatty alcohols and propylene glycol and to an apparatus for carrying out this process. To carry out this process particularly economically, it is proposed that propylene glycol be extracted from the mixture with water.

27123

This invention relates to a process for the separation of propylene glycol from a mixture of low-boiling fatty alcohols and propylene glycol. The invention also relates to an apparatus for carrying out this process.

The term "low-boiling fatty alcohols" applies to fatty alcohols of which the boiling points are close to the boiling point of propylene glycol. Because of this property, the propylene glycol cannot be separated by rectification.

The problem addressed by the invention is to provide a process for the separation of propylene glycol from a mixture of low-boiling fatty alcohols and propylene glycol which is economical and provides for the substantially complete removal of propylene glycol from fatty alcohols. A solution to this problem was only found after extensive tests.

According to the invention, the problem as stated above is solved in a process of the type mentioned at the beginning in that propylene glycol is extracted from the mixture with water. In the apparatus for carrying out the process, this problem is solved in accordance with the invention by at least one extraction column and a following rectification column connected thereto for the separation of water

27129

from propylene glycol.

To produce substantially anhydrous propylene glycol from the extract, the propylene glycol/water mixture, it is proposed in accordance with the invention that substantially anhydrous propylene glycol be separated off from the propylene glycol/water mixture by rectification. By virtue of very favorable equilibrium data in the water/propylene glycol system, three to five theoretical stages of a rectification column are sufficient to obtain from the extract a product which - starting from an extract of 35 to 45% by weight propylene glycol and 55 to 65% by weight water - contains 97 to 99% by weight propylene glycol.

Accordingly, for the apparatus for the production of substantially anhydrous propylene glycol from a fatty alcohol/propylene glycol mixture, it is proposed in accordance with the invention that this apparatus comprise at least one extraction column and at least one following rectification column connected thereto.

The process and the apparatus according to the invention are used in particular in the working up of the reaction mixture formed in the direct hydrogenation of glyceride oils. For hydrogenation

27129

conversions of 97 to 99%, this reaction mixture predominantly contains fatty alcohols commensurate with the C-chain distribution of the glyceride oil used and propylene glycol commensurate with the proportion of bound glycerol and also low-boiling fractions. The object of a working-up process in this regard is to produce fatty alcohols free from propylene glycol and also anhydrous propylene glycol. Tests have shown that it is of particular advantage in this case initially to fractionate the reaction mixture so that propylene glycol and low-boiling fatty alcohols are separated off as first runnings.

The hydrogenation effluent is fractionated into the following boiling cuts:

I. water/low-boiling fractions

II. first runnings: C_6/C_{10} fatty alcohols and propylene glycol

III. main fraction: C_{12}/C_{18} fatty alcohols.

Analysis of a sample of the main fraction reveals C_{12}/C_{18} fatty alcohols according to specification which are eminently suitable, for example, for sulfatization. This does not apply to the first runnings which have to be further worked up on account of the propylene glycol therein. The extraction-

27129

based working up mentioned above is proposed for this purpose. Although extraction of the entire fatty alcohol after the separation of water/low-boiling fractions is possible in theory, it is disadvantageous on economic grounds.

To ensure a high concentration of propylene glycol in the extract and, hence, to achieve low further processing costs, for example in the production of substantially anhydrous propylene glycol, it is proposed that extraction of the propylene glycol take place in apparatus comprising several theoretical separation stages. It is also of advantage to carry out extraction of the propylene glycol/water mixture continuously, particularly in countercurrent.

Extraction is carried out particularly effectively in at least one sieve-plate extraction column.

It is also of advantage to use at least one pulsed extraction column for extraction.

To obtain propylene glycol of high quality in regard to color, odor and composition, it is proposed that the substantially anhydrous propylene glycol obtained from the rectification step be treated with active carbon or optionally distilled overhead in another column. In the latter case, a purity of 99.69%, an acid value AV below 0.01, a

27129

saponification value SV of 0.15 and a water content of 0.16% are obtained.

In the apparatus according to the invention, the extraction column advantageously comprises several stages.

In a particularly economical embodiment, the extraction column comprises fittings, more particularly sieve plates.

In addition, the extraction column is advantageously a pulsed column.

In one particularly advantageous embodiment, the apparatus according to the invention comprises at least one other rectification column which is connected at its first runnings exit to the entrance of the extraction column. With this other rectification column, it is possible for example to fractionate the hydrogenation effluent into water and low-boiling fractions, the first runnings and the main fraction so that only the first runnings are further processed in the remaining part of the apparatus according to the invention.

One example of embodiment of the invention is described in detail in the following with reference to the accompanying drawing.

The drawing is a flow chart of an apparatus

27129

according to the invention for working up the hydrogenation effluent from the direct hydrogenation of glyceride oils.

Gas chromatographic analysis of the hydrogenation effluent produced the following values: 84% by weight fatty alcohols, 0.2% by weight hydrocarbons, 10.5% by weight propylene glycol, 1% by weight i- and n-propanol and 2.8% by weight water and traces of methanol and ethanol.

The overall process for working up the hydrogenation effluent consists of three steps, namely:

1. fractionation of the hydrogenation effluent
2. extraction of the propylene glycol from the first runnings of fractionation in the extraction apparatus
3. rectification of the extract from the extraction apparatus and subsequent treatment of the product with active carbon.

The hydrogenation effluent (1) was fractionated into three boiling cuts in a rectification column (2). The proportions by weight of the three fractions were correspondingly 5%, 22% and 73%. Analysis of the main fraction (3) produced the following values: C_{10}: 0.8%, C_{12}: 51%, C_{14}: 21.7%, C_{16}: 11.2%, C_{18}: 14.9%, C_{20}: 0.1%, hydrocarbons: 0.04%, water: 0.06%.

Water and low-boiling fractions were separated off at (5) at the head of the column. The first runnings (4) (fatty alcohols/propylene glycol) were extracted in a pulsating sieve-plate extraction column (6). The extraction column consisted of 40 sieve plates (free cross-section 14%) at intervals of 100 mm. The nominal width of the column was 100 mm. The pulsation unit (7) operates with a pulsation stroke of 15 mm and at a pulsation frequency of 1.5 to 1.6 1/s.

The first runnings (4) were delivered to the lower part and the extractant (8), water, to the upper part of the column (6). The throughputs of the two phases were as follows: first runnings: 90 to 100 dm^3/h, water: 40 to 50 dm^3/h, water being selected as the continuous phase on the basis of extensive preliminary tests. Colourless liquids were obtained as the raffinate (9) and extract (10). The raffinate (9) contains 95 to 97% by weight fatty alcohols, 3 to 4% by weight water and no propylene glycol.

The extract (10) consists of 35 to 45% by weight propylene glycol, 55 to 65% by weight water and traces of the above-mentioned low-boiling fractions.

The raffinate (9) is collected in the vessel

(12). The extract from the vessel (11) is brought to the operating temperature (approx. 100°C) in the heat exchanger (13) and delivered to the rectification column (14). A packed rectification column (Kuhni Rombopack, height: 4 m, diameter: 316 mm) was used. It was operated at normal pressure (1 bar) with a reflux ratio of 0.5. The extract throughput was approximately 70 kg/h which - for the reflux ratio of 0.5 mentioned above - necessitated an energy input in the evaporator (19) of the column of approximately 40 to 47 kW. The throughputs of the two head and sump products amounted correspondingly to 42 kg/h and 28 kg/h. The slight yellowish coloration of the sump product (16) (97 to 99% by weight propylene glycol) and its faint odor could be completely removed without difficulty by subsequent bleaching (17) with active carbon (for example NORIT CA1 or BRILLONIT; active carbon consumption: approx. 1 to 2 g/100 g).

The drawing also shows the column sump at (15) and a condenser at (18).

2773924

27129

NEW CLAIMS:

1. A process for separating propylene glycol from a mixture of C_6-C_{10} low-boiling fatty alcohols and propylene glycol comprising the steps of: (a) extracting said mixture with water to produce a water-propylene glycol mixture and a water-fatty alcohol mixture; and (b) fractionating said water-propylene glycol mixture to produce a distillate that contains at least about 95% by weight propylene glycol.

2. The process of Claim 1 wherein said mixture of low-boiling fatty alcohols and propylene glycol is a first cut from the fractionating of a hydrogenation affluent from the direct hydrogenation of glyceride oils.

3. The process of Claim 1 wherein said distillate from step (b) is distilled to produce a second distillate that contains at least 99% by weight propylene glycol.

4. The process of Claim 1 further comprising the step of treating the distillate from step (b) with activated carbon for a sufficient time to remove color and odor from said distillate.

5. The process of Claim 1 wherein step (a)

is carried out continuously.

6. The process of Claim 5 wherein step (a) is carried out counter-currently.

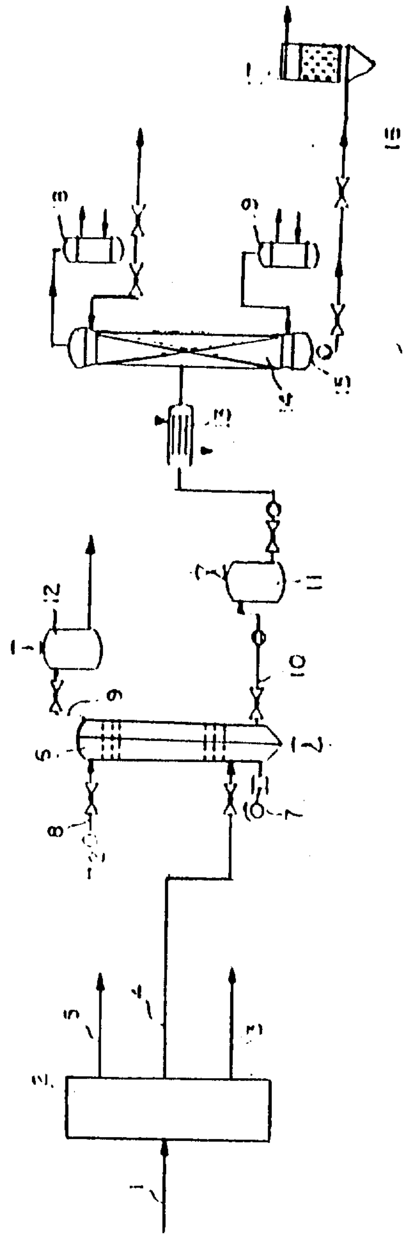
7. The process of Claim 2 wherein in step (a) the water-propylene glycol mixture contains from about 35% to about 45% by weight of propylene glycol and from about 55% to about 65% by weight of water.

8. The process of Claim 2 wherein in step (b) the distillate contains from about 97% to about 99% by weight of propylene glycol.

9. The process of Claim 1 wherein step (a) is carried out continuously and counter-currently and the water-propylene glycol mixture contains from about 35% to about 45% by weight of propylene glycol and from about 55% to about 65% by weight of water, and wherein in step (b) the distillate contains from about 97% to about 99% by weight of propylene glycol.

DR. FRANK-JOSEF CARDUCK
DR. LUTZ JEROMIN
DR. GERD GOBEL
Inventors

Patented MAR 16 1993 **Patent No. 27129**



Dr. FRANZ - JOSEF CARDUCK
Dr. LUTZ JEROMIN
Dr. GERD GOBEL
Inventors
BITO, LOZADA, ORTEGA y CASTILLO
By:
John G. Borges
Attorney