Comparative Example No 2:

In another plant for the oxidation of cyclohexane to cyclohexyl hydroperoxide comprising a cascade of n bubble column type reactors with a co-current supply of oxidizing gas (air) and reaction medium to be oxidized, a stream of hot cyclohexane was supplied to the first reactor of the column at a temperature of 185°C. The stream of hot cyclohexane contained 0.20% by weight of cyclohexyl hydroperoxide, cyclohexanol and cyclohexanone.

The oxidation reaction was carried out without a catalyst. The liquid reaction medium leaving one reactor was supplied to the next reactor in the cascade. The gaseous phases or oxidation gases from each reactor were recovered and condensed, some of them after mixing.

The temperature in the last reactor n of the cascade was 179°C. In order to maintain the temperature at this value, "cold" cyclohexane, at 70°C, was supplied, as a complement to the reaction medium, to the last three reactors n-2, n-1 and n of the cascade. The stream of "cold" cyclohexane supplied to each reactor represented 2.7%, 3.1% and 2.7% by weight of the stream of "hot" cyclohexane supplied to the first reactor of the cascade, i.e. 8.5% in total.

At the outlet from the last oxidation reactor, a portion of the unreacted cyclohexane was separated from the reaction mixture for recycling to the oxidation step. The reaction mixture was then washed with water and treated with a catalyst in order to transform the cyclohexyl hydroperoxide into cyclohexanol and cyclohexanone. The cyclohexane was then separated by distillation, and the impure cyclohexanol – cyclohexanone mixture was purified by distillation.

The oxidation gases were mixed and condensed in a condenser train. The liquid fraction recovered was flashed. The organic phase recovered after water separation was recycled to the cyclohexane supplied to the first reactor of the cascade before the cyclohexane heating step.

Example 2, in accordance with the invention:

The process of Example 2 was carried out in the plant described in comparative Example 2, using the same reagents.

The oxidation gases or gaseous phases from the reactors were mixed and condensed. The least volatile fraction of these condensates, recovered at a temperature of 125°C, representing 8.1% of the flow of the hot cyclohexane supplied to the first reactor, was recycled to the reaction medium entering the reactor n-2. The remainder of the condensates was treated in a manner similar to that described in the comparative

Claims

1. Process for producing alkyl hydroperoxide by oxidizing a saturated hydrocarbon in the liquid phase with an oxygen-containing gas, the reaction being carried out in a plurality of successive steps, the hydrocarbon being supplied at least to the first step at a temperature in the range 100°C to 250°C, at least the last step of the reaction being cooled to maintain and control the temperature in said step at a level which is less than or equal to that of the first step, characterized in that each step of the reaction comprises a liquid phase and a gaseous phase forming a gaseous overhead in said step, said gaseous phase forming the overhead being extracted and evacuated to be at least partially condensed, and in that the condensate recovered from the gaseous phase of one step is recycled to one of said oxidation steps, wherein the concentration of oxidized products in the supplied reaction medium is at least equal to the concentration of oxidized products in the condensate or condensates to be recycled.

2. Process according to Claim 1, characterized in that the condensate recycled to one step is obtained by mixing condensates or gaseous phases deriving from at least two oxidation steps.

3. Process according to Claim 1 or Claim 2, characterized in that the gaseous phase recovered at the head of one reaction step is partially condensed and the condensate obtained is recycled to the supply to the preceding step.

4. Process according to one of Claims 1 to 3, characterized in that the gaseous phase recovered at the head of the last reaction step is partially condensed and the condensate obtained is recycled to the supply to the preceding step.

5. Process according to one of the preceding claims, characterized in that the saturated hydrocarbon is a cyclic hydrocarbon.

6. Process according to Claim 5, characterized in that the saturated hydrocarbon is selected from the group consisting of cyclohexane, cyclooctane, cyclododecane and decalin.

7. Process according to Claim 6, characterized in that the saturated hydrocarbon is cyclohexane.

8. Process according to one of the preceding claims, characterized in that after partial condensation of the gaseous phases forming the overhead of each step, the uncondensed fraction is condensed completely, then said condensate obtained is flashed in order to obtain an aqueous liquid fraction and an organic fraction, said organic fraction being recycled to the first oxidation step.

9. Process according to one of the preceding claims, characterized in that the oxidation is carried out in a plant comprising a plurality of oxidation reactors mounted in series or as a cascade, each reactor forming an oxidation step.

10. Process according to Claim 9, characterized in that the reactors are of the bubble column type.

11. Process according to one of Claims 1 to 8, characterized in that oxidation is carried out in a plant comprising as the oxidation reactor a partitioned bubble column, each compartment forming an oxidation step.

Comparative Example No 2:

In another plant for the oxidation of cyclohexane to cyclohexyl hydroperoxide comprising a cascade of n bubble column type reactors with a co-current supply of oxidizing gas (air) and reaction medium to be oxidized, a stream of hot cyclohexane was supplied to the first reactor of the column at a temperature of 185°C. The stream of hot cyclohexane contained 0.20% by weight of cyclohexyl hydroperoxide, cyclohexanol and cyclohexanone.

The oxidation reaction was carried out without a catalyst. The liquid reaction medium leaving one reactor was supplied to the next reactor in the cascade. The gaseous phases or oxidation gases from each reactor were recovered and condensed, some of them after mixing.

The temperature in the last reactor n of the cascade was 179°C. In order to maintain the temperature at this value, "cold" cyclohexane, at 70°C, was supplied, as a complement to the reaction medium, to the last three reactors n-2, n-1 and n of the cascade. The stream of "cold" cyclohexane supplied to each reactor represented 2.7%, 3.1% and 2.7% by weight of the stream of "hot" cyclohexane supplied to the first reactor of the cascade, i.e. 8.5% in total.

At the outlet from the last oxidation reactor, a portion of the unreacted cyclohexane was separated from the reaction mixture for recycling to the oxidation step. The reaction mixture was then washed with water and treated with a catalyst in order to transform the cyclohexyl hydroperoxide into cyclohexanol and cyclohexanone. The cyclohexane was then separated by distillation, and the impure cyclohexanol – cyclohexanone mixture was purified by distillation.

The oxidation gases were mixed and condensed in a condenser train. The liquid fraction recovered was flashed. The organic phase recovered after water separation was recycled to the cyclohexane supplied to the first reactor of the cascade before the cyclohexane heating step.

Example 2, in accordance with the invention:

The process of Example 2 was carried out in the plant described in comparative Example 2, using the same reagents.

The oxidation gases or gaseous phases from the first n-1 reactors were mixed and condensed. The least volatile fraction of these condensates, recovered at a temperature of 125°C, representing 8.1% of the flow of the hot cyclohexane supplied to the first reactor, was recycled to the reaction medium entering the reactor n-2. The remainder of the condensates was treated in a manner similar to that described in the comparative

Claims

Claim 1
Process for producing alkyl hydroperoxide by oxidizing a saturated hydrocarbon in the liquid phase with an oxygen-containing gas, the reaction being carried out in a plurality of successive steps, the hydrocarbon being supplied at least to the first step at a temperature in the range 100°C to 250°C, at least the last step of the reaction being cooled to maintain and control the temperature in said step at a level which is less than or equal to that of the first step,
• characterized in that each step of the reaction comprises a liquid phase and a gaseous phase forming a gaseous overhead in said step, said gaseous phase forming the overhead being extracted and evacuated to be at least partially condensed, and in that the condensate recovered from the gaseous phase of one step is recycled to one of said oxidation steps, wherein the concentration of oxidized products in the supplied reaction medium is equal to or greater than the concentration of oxidized products in the condensate or condensates to be recycled, and
• further characterized in that the condensate or condensates obtained are recycled directly into at least one step wherein the concentration of oxidized products in the reaction medium supplied to said step is equal to or greater than the concentration of oxidized products contained in the condensate or condensates to be recycled.

Claim 2
Process according to claim 1, characterized in that the condensate recycled to one step is obtained by mixing condensates or gaseous phases deriving from at least two oxidation steps.

Claim 3
Process according to claim 1 or claim 2, characterized in that the gaseous phase recovered at the head of one reaction step is partially condensed and the condensate obtained is recycled to the supply to the preceding step.

Claim 4
Process according to claim 1 or claim 2, characterized in that the gaseous phase recovered at the head of the last reaction step is partially condensed and the condensate obtained is recycled to the supply to the preceding step.

Claim 5
Process according to claim 1 or claim 2, characterized in that the saturated hydrocarbon is a cyclic hydrocarbon.

Claim 6
Process according to claim 5, characterized in that the saturated hydrocarbon is selected from the group consisting of cyclohexane, cyclooctane, cyclododecane and decalin.

Claim 7
Process according to claim 6, characterized in that the saturated hydrocarbon is cyclohexane.

Claim 8
Process according to claim 1 or claim 2, characterized in that after partial condensation of the gaseous phases forming the overhead of each step, the uncondensed fraction is condensed completely, then said condensate obtained is flashed in order to obtain an aqueous liquid fraction and an organic fraction, said organic fraction being recycled to the first oxidation step.

Claim 9
Process according to claim 1 or claim 2, characterized in that the oxidation is carried out in a plant comprising a plurality of oxidation reactors mounted in series or as a cascade, each reactor forming an oxidation step.

Claim 10.
Process according to claim 9, characterized in that the reactors are of the bubble column type.

Claim 11
Process according to claim 1 or claim 2, characterized in that oxidation is carried out in a plant comprising as the oxidation reactor a partitioned bubble column, each compartment forming an oxidation step.