It is known to use hydrogen peroxide in the presence of a heterogeneous catalyst to convert an olefin into an oxirane, more particularly to convert propylene into propylene oxide (1,2-epoxypropane) by reaction with hydrogen peroxide. Such a process is generally performed in huge plants which consume huge amounts of hydrogen peroxide. Like many other (catalytic) organic oxidation processes, it requires pure reagents with no risk of process precipitation, i.e. a metal content of the order of 10 ppb. In the case of propylene oxide manufacture, it has namely been observed that if metals are present, like Fe (coming from the material of construction of the apparatus used) or Al (coming from the catalyst) and even in low amounts of between 50 and 200 ppb, these react with the reactives fed to the reactor and lead to the precipitation of insolubles which in turn leads to filter clogging.

Patent applications EP 529723 and EP 965562 in the name of the Applicant describe a process for manufacturing a purified aqueous hydrogen peroxide solution, in which a crude aqueous hydrogen peroxide solution is subjected to a washing operation with at least one organic solvent. In such a process, washing is intended to remove from the peroxide, working solution residuals, i.e. quinones and their degradation products. The solvents are generally those of the AO process in question used normally for dissolving the quinones in order to make working solution (the solvents being generally circulated from crude washing to main loop). The purpose of this traditional crude washing is to reduce TOC (organic matter) in the peroxide, and it has no effect on inorganic impurities (Al, Fe etc.).

On the other hand, various techniques (membrane filtration, distillation etc) are available when ultrapure peroxides are needed, but they are either too costly or not suitable for high volume (>100000 t per year) applications.

The purpose of the present invention is hence to provide an improved process for the preparation of hydrogen peroxide, in particular a new method for producing hydrogen peroxide with a low concentration of easily precipitating metals (Al, Fe, Cr...) which is not expensive and is suitable for high volumes.

In one aspect, ~~The~~ the present ~~invention~~ disclosure therefore concerns a process for manufacturing a purified aqueous hydrogen peroxide solution, in which a crude aqueous hydrogen peroxide solution is subjected to a washing operation with at least one organic solvent, and wherein an organophosphorus chelating agent is added to the organic solvent.

The regeneration treatments mentioned above consist, for example, in subjecting the solvent to one or more extractions, and to one or more treatments with a chemical reagent which is compatible with the organic solvent, in order to remove the impurities. The solvent regeneration is preferably done using an aqueous acidic solution for instance an aqueous nitric acid solution (HNO3). This solvent regeneration is in fact also an extraction, the acid transforming the organic metal complexes in inorganic salts (nitrates in the case of nitric acid) of said metals which are water soluble and hence are extracted from the solvent (organic phase) in an aqueous phase namely consisting of water in which the metal salts are dissolved. Hence, this regeneration can be done using classical industrial extraction equipment like a column, a centrifugal decanter etc.

Besides the nature of the chelating agent and of the solvent, several other parameters may be adapted in order to optimize the process of the invention, namely: the pH, the amount of chelating agent, the ratio peroxide to solvent and the temperature. Preferably, the pH is acidic because this limits hydrogen peroxide decomposition and also, the solubility of the chelating agent in said hydrogen peroxide.

Figure 4 attached shows a bloc diagram of a preferred embodiment of the invention, according to which a crude hydrogen peroxide aqueous solution (1) is submitted to a washing operation (first extraction step 2) with an organic solvent to which an organophosphorus chelating agent has been added, and which actually is a mixture of fresh extraction solvent (3) and of regenerated solvent (8). This washing operation is in fact an extraction, the organic phase (solvent + chelating agent) being first mixed with the crude hydrogen peroxide solution in order to be able of forming complexes with the metals it contains and thereafter, two phases are separated to provide respectively a purified aqueous hydrogen peroxide solution (4) and an organic phase charged with metal complexes (5). The latter is then at least partly regenerated in a second extraction step (6) using an aqueous HNO3 solution (7) as explained above. At the issue of this regeneration step (6), there is provided an aqueous phase comprising metal nitrates (9) and a purified extraction solvent of which part (8) is recycled to the first extraction step (washing operation 2) and part (10) is removed to balance the process.

In another aspect, the~~The~~ present ~~invention~~ disclosure also relates to a process for manufacturing propylene oxide (1,2-epoxypropane) by reaction of propylene with hydrogen peroxide , said process using a hydrogen peroxide purified with a process as described above.

CLAIMS

1. Process for manufacturing a purified aqueous hydrogen peroxide solution, in which a crude aqueous hydrogen peroxide solution is subjected to a washing operation with at least one organic solvent, and wherein an organophosphorus chelating agent is added to the organic solvent.

2. Process according to claim 1, wherein the crude aqueous hydrogen peroxide solution is washed several times i.e. at least two times consecutively.

3. Process according to claim 1 or 2, said process comprising the following steps:
a) hydrogenation, in the presence of a catalyst, of a working solution comprising at least one organic solvent and at least one alkylanthraquinone to obtain the corresponding alkylanthrahydroquinone,
b) separation of the hydrogenated working solution comprising the alkylanthrahydroquinone from the catalyst,
c) oxidation of the recovered hydrogenated working solution from step b) to form hydrogen peroxide,
d) separation, from the working solution, of said hydrogen peroxide during and/or subsequently to said oxidation step, preferably with an aqueous medium, and
e) recycling of the recovered working solution to step a)
wherein the crude aqueous hydrogen peroxide solution separated in step d) is subjected to the washing operation.

4. Process according to claim 3, wherein the organic solvent of the alkylanthraquinone is ~~Solvesso® 150~~aromatic hydrocarbon solvent and/or diisobutycarbinol (DiBC), preferably a mixture of both.

5. Process according to claim 3 or 4, wherein the organic solvent used for the washing operation is a part of the working solution.

6. Process according to claim 5, wherein the organic solvent used for the washing operation is recycled into the working solution.

7. Process according to claim 3 or 4, wherein the organic solvent used for the washing operation is not part of the working solution and is not recycled to it.

8. Process according to claim 7, wherein the nature of the solvent used for the washing operation is chosen so that it improves the metal extraction by the organophosphorus chelating agent.

9. Process according to claim 8, wherein the organic solvent used for the washing operation comprises an organophosphorus compound different from the organophosphorus chelating agent, for instance chosen from alkyl-phosphates or alkyl-phosphonates.

10. Process according to claim 9, wherein when the organophosphorus chelating agent is di-(2-ethylhexyl) phosphoric acid (DEHPA or HDEHP) and wherein the organic solvent used for the washing operation comprises TOP (trioctylphosphate) or TEHP (triethylhexylphosphate).

11. Process for manufacturing propylene oxide (1,2-epoxypropane) by reaction of propylene with hydrogen peroxide, said process using a hydrogen peroxide purified by a process according to any of the preceding claims.

~~12. Process for manufacturing propylene oxide (1,2-epoxypropane) by reaction of propylene with hydrogen peroxide, said process using a hydrogen peroxide solution containing less than 100 ppb of Al and/or Fe.~~

~~13. Process according to claim 12, wherein the hydrogen peroxide solution contains less than 100 ppb of Cr.~~