a valuable educt and as such difficult to handle due to its tendency to decompose spontaneously.

WO-A 2005/000827 discloses the regeneration of a titanium silicalite catalyst following a process for the continuous expoxidation of propene with hydrogen peroxide. The catalyst is periodically regenerated with a methanol solvent at a temperature of at least 100 °C. Is indicated above, methanol is a valuable organic compound which requires an expensive and time consuming recovery. Also, after regeneration, the epoxidation has to be restarted at a higher temperature compared with fresh catalyst in the WO-A 2005/000827.

WO-A 2007/013739 discloses the regeneration of a titanium containing molecular sieve wherein, after a pretreatment of the spent catalyst with water or alcohol, the thus pretreated catalyst is brought into contact with a mixture which comprises hydrogen peroxide, water, and alcohol. This process thus includes two mandatory and subsequent steps in which the spent catalyst is brought into contact with two different solution.

US 2003/0187284 A1 discloses a method for producing an epoxide in the presence of a zeolite catalyst and the regeneration of the catalyst by treating it with a solution comprising an acid with a pKa value of less than 6.

US 2012/142950 A1 discloses a continuous process for the production of propylene oxide comprising reacting propene with hydrogen peroxide in methanolic solution in the presence of a titanium silicalite-1 catalyst to obtain propylene oxide.

WO 2011/115234 A1 discloses a method for regenerating titanosilicate catalysts.

US 2004/058798 A1 discloses a method for regeneration of titanium-containing silicone oxide catalysts by heating the used catalysts at a temperature of at least 400 °C in the presence of an oxygen-containing gas stream.

US 5 916 835 A discloses a method of regenerating used non-zeolitic heterogeneous catalysts.

Therefore, it was an object of the embodiments disclosed herein to provide a simple and cost-effective process for regeneration of a catalyst comprising a titanium containing zeolite as catalytically active material used in an epoxidation of olefins. It was a further object of the embodiments disclosed herein to provide a regenerated catalyst comprising a titanium containing zeolite as catalytically active material which may be readily reused in the catalysis of the epoxidation of olefins.

Thus, the present disclosure relates to a process for the regeneration of a catalyst comprising a titanium containing zeolite as catalytically active material, said catalyst having been used in a process for the preparation of an olefin oxide comprising

(i) providing a mixture comprising an organic solvent, an olefin, an epoxidation agent and an at least partially dissolved potassium comprising salt;

(ii) subjecting the mixture provided in (i) in a reactor to epoxidation conditions in the presence of the catalyst, obtaining a mixture comprising the organic solvent and the olefin oxide, and obtaining the catalyst having a potassium salt deposited thereon;

said process for the regeneration comprising

(a) separating the mixture obtained from (ii) from the catalyst;

(b) washing the catalyst obtained from (a) with a liquid aqueous system;

(c) optionally drying the catalyst obtained from (b) in a gas stream comprising an inert gas at a temperature of less than 300 °C;

(d) calcining the catalyst obtained from (b) or (c) in a gas stream comprising oxygen at a temperature of at least 300 °C.

In an aspect, there is provided a process for the regeneration of a catalyst comprising a titanium containing zeolite as catalytically active material, said catalyst having been used in a process for the preparation of an olefin oxide comprising

(i) providing a mixture comprising an organic solvent, an olefin, an epoxidation agent and an at least partially dissolved potassium comprising salt;

(ii) subjecting the mixture provided in (i) in a reactor to epoxidation conditions in the presence of the catalyst, obtaining a mixture comprising the organic solvent and the olefin oxide, and obtaining the catalyst having a potassium salt deposited thereon;

said process for the regeneration comprising

(a) separating the mixture obtained from (ii) from the catalyst;

(b) washing the catalyst obtained from (a) with a liquid aqueous system which comprises less than 0.1 wt.-% of the compounds with a pKa value of 8 or less;

(c) optionally drying the catalyst obtained from (b) in a gas stream comprising an inert gas at a temperature of less than 300 °C;

(d) calcining the catalyst obtained from (b) or (c) in a gas stream comprising oxygen at a temperature of at least 300 °C.

Surprisingly, according to the regeneration process of the present disclosure, which comprises washing spent catalyst comprising a titanium containing zeolite with a liquid aqueous system, combined with optional drying, and further calcining, a regenerated catalyst comprising a titanium containing zeolite with excellent catalytic properties is obtained which may be readily reused, for example in the process for the preparation of an olefin oxide.

In this respect, the inventors found out that after subjecting the spent catalyst comprising a titanium containing zeolite as catalytically active material to the regeneration of the present disclosure, its activity and selectivity is over a long term comparable with the activity of the respective fresh catalyst comprising a titanium containing zeolite. Such a favorable result may be obtained after performing only one cycle of regeneration steps (a) to (d).

Further, it was surprisingly found that even repeated cycles of steps (a) to (b) did not affect the activity and selectivity of the catalyst comprising a titanium containing zeolite as catalytically

active material in an unfavorable way. The regeneration according to steps (a) to (b) of the present disclosure has thus proven to be a mild process to which the same catalytic material may be submitted repeatedly, since no deteriorating effect on the catalytic activity and thus, presumably, on the zeolitic structure have been observed after several repetitions of steps (a) to (b).

Step (a)

The first regeneration step (a) requires separating the reaction mixture resulting from the epoxidation reaction of an olefin from the catalyst which comprises a titanium containing zeolite as catalytically active material.

This separation of the reaction mixture form the spent catalyst comprising a titanium containing zeolite can be achieved in any suitable manner, such as pumping, draining, decanting, filtrating, and the like. Preferably, in case the epoxidation reaction is carried out in batch mode, it is preferred to separate the mixture obtained from (ii) from the spent catalyst by filtration. In case the epoxidation reaction is carried out in continuous mode, it is preferred to separate the mixture obtained from (ii) from the spent catalyst by stopping to subject the mixture provided in (i) to epoxidation conditions according to (ii) and subject the spent catalyst to the regeneration step

Claims

1. A process for the regeneration of a catalyst comprising a titanium containing zeolite as catalytically active material, said catalyst having been used in a process for the preparation of an olefin oxide comprising
   (i) providing a mixture comprising an organic solvent, an olefin, an epoxidation agent and an at least partially dissolved potassium comprising salt;
   (ii) subjecting the mixture provided in (i) in a reactor to epoxidation conditions in the presence of the catalyst, obtaining a mixture comprising the organic solvent and the olefin oxide, and obtaining the catalyst having a potassium salt deposited thereon;
   said process for the regeneration comprising
   (a) separating the mixture obtained from (ii) from the catalyst;
   (b) washing the catalyst obtained from (a) with a liquid aqueous system which comprises less than 0.1 wt.-% of the compounds with a pKa value of 8 or less;
   (c) optionally drying the catalyst obtained from (b) in a gas stream comprising an inert gas at a temperature of less than 300 °C;
   (d) calcining the catalyst obtained from (b) or (c) in a gas stream comprising oxygen at a temperature of at least 300 °C.

2. The process of claim 1, wherein the liquid aqueous system used in (b) contains at least 75 weight-% water, based on the total weight of the liquid aqueous system.

3. The process of claim 1, wherein the washing (b) is performed at a pressure in a range of from 0.8 to 1.5 bar, and a temperature in a range of from 40 to 90 °C.

4. The process of claim 1, wherein the washing (b) is performed until a potassium content of the liquid aqueous system after having been contacted with the catalyst is at most 1000 weight-ppm.

5. The process of claim 1, wherein the washing (b) is performed until a potassium content of the liquid aqueous system after having been contacted with the catalyst relative to the potassium content of the liquid aqueous system before having been contacted with the catalyst is at most 333 : 1.

6. The process of claim 1, wherein the process comprises the drying (c) and at least 90 volume-% of the gas stream comprising the inert gas consist of at least one inert gas selected from the group consisting of nitrogen, helium, and argon.

7. The process of claim 1, wherein the process comprises the drying (c), which is performed until a water content of the gas stream comprising the inert gas after having been contacted with the catalyst relative to a water content of the gas stream comprising the inert gas before having been contacted with the catalyst is at most 1.10 : 1.

8. The process of claim 1, wherein the process comprises the drying (c), and after (c), the dried catalyst is heated to the calcination temperature according to (d) with a rate in the range of from 0.5 to 5 K/min.

9. The process of claim 1, wherein the catalyst obtained from (d) has a potassium content of at most 0.5 weight-%

based on a total weight of the catalyst and determined via elemental analysis.

10. The process of claim 1, wherein the mixture provided in (i) has a potassium content with a molar range of potassium relative to the epoxidation agent comprised in the mixture in a range of from $10x10^{-6}$:1 to $1500x10^{-6}$:1.

11. The process of claim 1, wherein the at least partially dissolved potassium comprising salt in (i) is selected from the group consisting of an inorganic potassium salt, an organic potassium salt, and a combination thereof.

12. The process of claim 1, wherein the at least partially dissolved potassium comprising salt in (i) is selected from the group consisting of at least one inorganic potassium salt selected from the group consisting of potassium hydroxide, a potassium halide, potassium nitrate, potassium sulfate, potassium hydrogen sulfate, potassium perchlorate, potassium dihydrogen phosphate, dipotassium hydrogen phosphate, potassium phosphate, a potassium pyrophosphate and a potassium etidronate, at least one organic potassium salt selected from the group consisting of a potassium salt of an aliphatic saturated monocarboxylic acid, potassium carbonate, and potassium hydrogen carbonate, and a combination of the at least one inorganic potassium salt and the at least one organic potassium salt.

13. The process of claim 1, wherein the titanium containing zeolite has an MFI framework structure, an MEL framework structure, an MWW framework structure, an MWW-type framework structure, an ITQ framework structure, a BEA framework structure, a MOR framework structure, or a mixed structure of two or more thereof.

14. The process of claim 1, wherein the titanium containing zeolite comprises at least one of Al, B, Zr, V, Nb, Ta, Cr, Mo, W, Mn, Fe, Co, Ni, Zn, Ga, Ge, In, Sn, Pb, Pd, Pt, and Au.

15. The process of claim 1, wherein the titanium containing zeolite is an aluminum-free zeolitic material of MWW or MWW-type framework structure comprising titanium.

16. The process of claim 1, wherein the catalyst comprising the titanium containing zeolite is a micropowder.

17. The process of claim 16, wherein the molding further comprises at least one binder.

18. The process of claim 1, wherein the process for the regeneration is carried out in the reactor in which the mixture provided in (i) is subjected to epoxidation conditions according to (ii).

19. The process of claim 1, further comprising employing the catalyst obtained from (d) in an olefin epoxidation process comprising

(i') providing a mixture comprising an organic solvent, an olefin, an epoxidation agent and an at least partially dissolved potassium comprising salt;

(ii') subjecting the mixture provided in (i') in a reactor to epoxidation conditions in the presence of the catalyst obtained from (d), obtaining a mixture comprising the organic solvent and the olefin oxide.