corporation of titanium, wherein after hydrothermal incorporation of titanium, the titanium-containing zeolitic material is separated from its mother liquor by filtration.

According to the prior art, the crystallization time of the titanium-containing zeolitic material is usually comparatively high. Further, separation of the zeolitic material via filtration usually increases the overall duration of the preparation process. Therefore, it was an object of the present disclosure to provide a process for the preparation of a titanium containing zeolitic material having MWW framework structure which is better suitable in particular for the industrial-scale preparation.

Surprisingly, it was found that employing a specific amount of template compound during the hydrothermal incorporation of titanium in a deboronated material having MWW framework structure allows to decrease the crystallization time and thus leads to an advantageous process. Yet further, it was found that this specific amount of template compound is comparatively low compared to the respective amount as described in the prior art. Therefore, the process according to the disclosure not only allows to decrease the crystallization time but also allows to decrease the relative amount of template compound, thus rendering the overall process even more advantageous. Further it was found that in order to obtain the titanium containing zeolitic material having MWW framework structure, it is not necessary to subject the mother liquor obtained according to the process of the present disclosure containing said material to filtration, and that is possible to subject said mother liquor to a direct spray-drying. Thus, a process is provided in which the separation of the titanium containing zeolitic material having MWW framework structure from its mother liquor and drying said material is combined in one single step, which combination of steps also renders the overall process more advantageous.

Yet further, it was found that according to a preferred process of providing a deboronated zeolitic material having MWW framework structure, the overall process can be designed even more advantageous if prior to the filtration of the deboronated zeolitic material having MWW framework structure, an pH adjustment of the suspension containing the zeolitic material having MWW framework structure is carried out, which acid treatment improves the filtration characteristics of the zeolitic material having MWW framework structure and thus renders the overall process even more advantageous.

Also surprisingly, it was found that the novel and advantageous process also results in a novel zeolitic material having MWW framework structure which, compared to known zeolitic materials having MWW framework structure, is characterized by different physical parameters and exhibits improved catalytic properties.

Therefore, the present disclosure relates to a process for preparing a titanium-containing zeolitic material having an MWW framework structure comprising

(i) providing a zeolitic material having an MWW framework structure comprising SiO_2 and B_2O_3, wherein the molar ratio B_2O_3 : SiO_2 is at most 0.02 : 1 and wherein at least 99 weight-% of the MWW framework structure consist of B_2O_3 and SiO_2;

(ii) incorporating titanium into the zeolitic material provided in (i) comprising

(ii.1) preparing an aqueous synthesis mixture containing the zeolitic material provided in (i), an MWW template compound and a titanium source, wherein the molar ratio of the MWW template compound relative to Si, calculated as SiO_2 and contained in the zeolitic material provided in (i), is in the range of from 0.5 : 1 to 1.4 : 1;

(ii.2) hydrothermally synthesizing a titanium-containing zeolitic material having an MWW framework structure from the aqueous synthesis mixture prepared in (ii.1), obtaining a mother liquor comprising the titanium-containing zeolitic material having an MWW framework structure;

(iii) spray-drying the mother liquor obtained from (ii.2) comprising the titanium-containing zeolitic material having an MWW framework structure.

In an aspect, there is provided a process for preparing a titanium-comprising zeolitic material having an MWW framework structure comprising

(i) providing a zeolitic material having an MWW framework structure comprising SiO_2 and B_2O_3, wherein a molar ratio B_2O_3 : SiO_2 is at most 0.02 : 1 and wherein at least 99 weight-% of the MWW framework structure consist of B_2O_3 and SiO_2;

(ii) incorporating titanium into the zeolitic material provided in (i) comprising

(ii.1) preparing an aqueous synthesis mixture comprising the zeolitic material provided in (i), an MWW template compound and a titanium source, wherein a molar ratio of the MWW template compound relative to Si, calculated as SiO_2 and comprised in the zeolitic material provided in (i), is in a range of from 0.5 : 1 to 1.4: 1;

(ii.2) hydrothermally synthesizing a titanium-comprising zeolitic material having an MWW framework structure from the aqueous synthesis mixture prepared in (ii.1), obtaining a mother liquor comprising the titanium-comprising zeolitic material having an MWW framework structure;

(iii) spray-drying the mother liquor obtained from (ii.2) comprising the titanium-comprising zeolitic material having an MWW framework structure to obtain a spray-dried titanium-comprising zeolitic material having an MWW framework structure.

In another aspect, there is provided a spray powder obtained by the process as defined above.

Further, the present disclosure relates to a spray powder comprising a titanium-containing zeolitic material having an MWW framework structure, wherein at least 99

weight-% of the framework structure consist of titanium, silicon, and oxygen, and wherein at least 99 weight-% of the spray powder consist of the titanium-containing zeolitic material having an MWW framework structure, wherein the lattice parameter c of the titanium-containing zeolitic material having an MWW framework structure is (25.2 ± 0.2) Angstrom, as determined via XRD, and wherein preferably, the UV/VIS spectrum of the titanium-containing zeolitic material having an MWW framework structure has a band with a maximum in the range of from 200 to 220 nm, and has no band with a maximum in the range of at least 250 nm.

In yet another aspect, there is provided a spray powder, comprising a titanium-comprising zeolitic material having an MWW framework structure, wherein at least 99 weight-% of the framework structure consist of titanium, silicon, and oxygen, and wherein at least 99 weight-% of the spray powder consist of the titanium-comprising zeolitic material having an MWW framework structure, wherein a lattice parameter c of the titanium-comprising zeolitic material having an MWW framework structure is (25.2 ± 0.2) Angstrom, as determined via XRD, and wherein a UV/VIS spectrum of the titanium-comprising zeolitic material having an MWW framework structure has a band with a maximum in a range of from 200 to 220 nm, and has no band with a maximum in a range of at least 250 nm.

In yet another aspect, there is provided a molding, comprising shaping and, optionally, post-treating, the spray powder as defined above.

Short description of the Figures

Fig. 1 shows the X-ray diffraction pattern (copper K alpha radiation) of the spray powder obtained according to Example 1. On the x axis, the degree values (2 Theta) are shown, on the y axis, the intensity (Lin (Counts)). The measurement was performed on a D8 Advance series 2 diffractometer from Bruker AXS. The diffractometer was configured with an opening of the divergence aperture of 0.1 ° and a Lynxeye detector.

Fig. 2 shows the UV/VIS spectrum of the spray powder obtained according to Example 1. The measurement was performed on a Perkin Elmer Lambda 900 spectrophotometer with 150 mm integrating sphere. Spectralon white reflectance standards are used as reference. On the x axis, the wavelength values (nanometer) are shown.

Claims

1. A process for preparing a titanium-comprising zeolitic material having an MWW framework structure comprising
   (i) providing a zeolitic material having an MWW framework structure comprising SiO_2 and B_2O_3, wherein a molar ratio B_2O_3 : SiO_2 is at most 0.02 : 1 and wherein at least 99 weight-% of the MWW framework structure consist of B_2O_3 and SiO_2;
   (ii) incorporating titanium into the zeolitic material provided in (i) comprising
      (ii.1) preparing an aqueous synthesis mixture comprising the zeolitic material provided in (i), an MWW template compound and a titanium source, wherein a molar ratio of the MWW template compound relative to Si, calculated as SiO_2 and comprised in the zeolitic material provided in (i), is in a range of from 0.5 : 1 to 1.4: 1;
      (ii.2) hydrothermally synthesizing a titanium-comprising zeolitic material having an MWW framework structure from the aqueous synthesis mixture prepared in (ii.1), obtaining a mother liquor comprising the titanium-comprising zeolitic material having an MWW framework structure;
   (iii) spray-drying the mother liquor obtained from (ii.2) comprising the titanium-comprising zeolitic material having an MWW framework structure to obtain a spray-dried titanium-comprising zeolitic material having an MWW framework structure.

2. The process of claim 1, wherein in (i), the zeolitic material having an MWW framework structure is provided by a process comprising
   (a) hydrothermally synthesizing a B-MWW precursor comprising SiO_2 and B_2O_3 with a molar ratio B_2O_3 : SiO_2 of greater than 0.02 : 1 from a synthesis mixture comprising a silicon source, a boron source, and an MWW template compound, obtaining the B-MWW precursor in a mother liquor;
   (b) separating the B-MWW precursor from the mother liquor to obtain a separated B-MWW precursor, and calcining the separated B-MWW precursor, obtaining a B-MWW;
   (c) deboronating the B-MWW obtained from (b) by treating the B-MWW with a liquid solvent system obtaining the zeolitic material having an MWW framework structure and a molar ratio B_2O_3 : SiO_2 of at most 0.02 : 1, and at least partially separating the zeolitic material from the liquid solvent system.

3. The process of claim 2, wherein (c) comprises drying the zeolitic material having an MWW framework structure and a molar ratio B_2O_3 : SiO_2 of at most 0.02 : 1.

4. The process of claim 2, wherein the separated zeolitic material having an MWW framework structure and a molar ratio B_2O_3 : SiO_2 of at most 0.02 : 1 obtained from (c) is not subjected to calcination prior to (ii).

5. The process of claim 2, wherein in (c), the zeolitic material having an MWW framework structure and a molar ratio B_2O_3 : SiO_2 of at most 0.02 : 1 is obtained in a form of particles having a Dv10 value in a range of from 1 to 10 micrometer, a Dv50 value in a range of from 5 to 50 micrometer, and a Dv90 value in a range of from 12 to 200 micrometer.

6. The process of claim 1, wherein the molar ratio B_2O_3 : SiO_2 of the zeolitic material having an MWW framework structure provided in (i) is at most 0.01 : 1, wherein at least 99.5 weight-%of the MWW framework structure consist of B_2O_3 and SiO_2.

7. The process of claim 1, wherein in (ii.1), the MWW template compound is selected from the group consisting of piperidine, hexamethylene imine, N,N,N,N',N',N'-hexamethyl-1,5-pentanediammonium ion, 1,4-bis(N-methylpyrrolidini-um)butane, octyltrimethylammonium hydroxide, heptyltrimethylammonium hydroxide, hexyltrimethylammonium hydroxide, and a mixture of two or more thereof, and the titanium source is selected from the group consisting of tetrabutylorthotitanate, tetraisopropylorthotitanate, tetra-ethylorthotitanate, titanium dioxide, titanium tetrachloride, titanium tert-butoxide, and a mixture of two or more thereof.

8. The process of claim 1, wherein in the aqueous synthesis mixture in (ii.1), a molar ratio of Ti, calculated as TiO_2 and comprised in the titanium source, relative to Si, calculated as SiO_2 and comprised in the zeolitic material having a molar ratio B_2O_3 : SiO_2 of at most 0.02 : 1, is in a range of from 0.005 : 1 to 0.1 : 1, a molar ratio of H_2O relative to Si, calculated as SiO_2 and comprised in the zeolitic material having a molar ratio B_2O_3 : SiO_2 of at most 0.02 : 1, is in a range of from 8 : 1 to 20 : 1, and a molar ratio of the MWW template compound relative to Si, calculated as SiO_2 and comprised in the zeolitic material provided in (i), is in a range of from 0.5 : 1 to 1.7 : 1.

9. The process of claim 1, wherein in (ii.2), the hydrothermal synthesizing is carried out at a temperature in a range of from 80 to 250 °C, and for a period in a range of from 10 to 100 h.

10. The process of claim 1, wherein neither during (ii.2), nor after (ii.2) and before (iii), the titanium-comprising zeolitic material having an MWW framework structure is separated from the mother liquor.

11. The process of claim 1, wherein the mother liquor subjected to (iii) comprising the titanium-comprising zeolitic material having an MWW framework structure has a solids content, in a range of from 5 to 25 weight-%, based on a total weight of the mother liquor comprising the titanium-comprising zeolitic material.

12. The process of claim 1, wherein during spray-drying in (iii), a drying gas inlet temperature is in a range of from 200 to 700 °C, and a drying gas outlet temperature is in a range of from 70 to 190°C.

13. The process of claim 1, wherein the zeolitic material having an MWW framework structure obtained from (iii) has a Si content in a range of from 30 to 40 weight-%, calculated as elemental Si, a total organic carbon content (TOC) in a range of from 0 to 14 weight-%, and a Ti content of from 2.1 to 2.8 weight-%, calculated as elemental titanium, in each case based on a total weight of the zeolitic material, and wherein in (iii), the zeolitic material having an MWW framework structure is obtained in a form of particles having a Dv10 value in a range of from 1 to 10 micrometer, a Dv50 value in a range of from 5 to 50 micrometer, and a Dv90 value in a range of from 12 to 200 micrometer.

14. The process of claim 1, further comprising
(iv) treating the titanium-comprising zeolitic material having an MWW framework structure obtained from (iii) with an aqueous solution having a pH of at most 5.

15. The process of claim 14, wherein after (iii) and before (iv), the spray-dried titanium-comprising zeolitic material having an MWW framework structure obtained from (iii) is not subjected to calcination.

16. The process of claim 14, wherein in (iv), the aqueous solution has a pH in a range of from 0 to 5.

17. The process of claim 14, wherein in (iv), the titanium-comprising zeolitic material having an MWW framework structure is treated with the aqueous solution at a temperature in a range of from 50 to 175 °C.

18. The process of claim 14, further comprising
(v) separating the titanium-comprising zeolitic material having an MWW framework structure obtained from (iv) from the aqueous solution to obtain a separated titanium-comprising zeolitic material having an MWW framework.

19. The process of claim 18, wherein (v) comprises drying the separated titanium-comprising zeolitic material having an MWW framework structure.

20. The process of claim 18, further comprising
(vi) preparing a suspension and subjecting the suspension to spray-drying.

21. The process of claim 20, further comprising

(vii) calcining the titanium-comprising zeolitic material having an MWW framework structure obtained from (iv) .

22. The process of claim 21, further comprising

(viii) shaping the titanium-comprising zeolitic material having an MWW framework structure obtained in (iv) or (v) or (vi) or (vii), obtaining a molding;

(ix) drying and/or calcining the molding obtained in (viii);

(x) subjecting the molding obtained in (viii) or (ix), to a water-treatment, wherein the water-treatment comprises treating the molding with liquid water in an autoclave under autogenous pressure at a temperature in a range of from 100 to 200 °C to obtain a water-treated molding;

(xi) drying and/or calcining the water-treated molding.

23. A spray powder obtained by the process according to claim 1.

24. A spray powder, comprising a titanium-comprising zeolitic material having an MWW framework structure, wherein at least 99 weight-% of the framework structure consist of titanium, silicon, and oxygen, and wherein at least 99 weight-% of the spray powder consist of the titanium-comprising zeolitic material having an MWW framework structure, wherein a lattice parameter c of the titanium-comprising zeolitic material having an MWW framework structure is (25.2 ± 0.2) Angstrom, as determined via XRD, and wherein a UV/VIS spectrum of the titanium-comprising zeolitic material having an MWW framework structure has a band with a maximum in a range of from 200 to 220 nm, and has no band with a maximum in a range of at least 250 nm.

25. The spray powder of claim 24, wherein a crystallinity of the titanium-comprising zeolitic material having an MWW framework structure, as determined via XRD, is at least (77 ± 5) %.

26. The spray powder of claim 24, having a Ti content, calculated as elemental Ti, in a range of from 1.5 to 2.0 weight-%, and a Si content, calculated as elemental Si, in a range of from 44 to 48 weight-%, in each case based on a total weight of the spray powder, having a total organic carbon content (TOC) of at most 0.1 weight-%, based on the total weight of the spray powder, and having a boron content, calculated as elemental boron, of at most 0.1 weight-%, based on the total weight of the spray powder.

27. The spray powder of claim 24, wherein particles of the spray powder have a Dv10 value in a range of from 1 to 10 micrometer, a Dv50 value in a range of from 5 to 50 micrometer, and a Dv90 value in a range of from 12 to 200 micrometer, and comprising macropores having an average pore diameter (4V/A) in a range of from more than 50 nanometer, as determined by Hg porosimetry according to DIN 66133.

28. A molding, comprising shaping and, optionally, post-treating, the spray powder of claim 24.