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(54) **PROCESS FOR THE PRODUCTION OF AN AEROGEL MATERIAL**

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ABSTRACT

A process for the production of an aerogel material comprises the following steps:

- a) preparation of a silicon oxide sol in an alcoholic solvent mixture;
- b) triggering of the gelation of the sol by adding base, whereby a gel is formed, and optionally aging of the gel;
- c) hydrophobicization of the optionally aged gel;
- d) removal of the solvent mixture by subcritical drying, whereby the aerogel material is formed.

The silicon oxide sol formed in step a) comprises at least one acid-catalytically activatable hydrophobicization agent, wherein the volume fraction of the hydrophobicization agent in the sol is 5 to 60%. The hydrophobicization in step c) is induced as a result of release or addition of at least one hydrophobicization catalyst acting in combination with the hydrophobicization agent.

PROCESS FOR THE PRODUCTION OF AN AEROGEL MATERIAL

[0001] This application claims priority from PCT application No. PCT/EP2014/066213 filed Jul. 28, 2014 which claims priority from European application No. EP 13179185.7 filed on Aug. 2, 2013, the disclosures of which are incorporated herein by reference.

TECHNICAL FIELD

[0002] The invention relates to a process for the production of an aerogel material. Furthermore, the invention relates to an insulant panel formed from an aerogel material and to a precursor product for producing an aerogel material.

PRIOR ART

[0003] Aerogels find increased application in building technologies as highly insulating insulation materials. Numerous methods for their preparation are known. With increasing industrialization of these materials since the turn of the millennium, the manufacturing processes used therefor have continuously been simplified. Basically, one can distinguish between water glass (sodium silicate) and alkoxide-based compounds such as tetraethylorthosilicate (TEOS) and tetramethylorthosilicate (TMOS) as silicate donors which significantly affect the further synthesis steps. The principally used hydrophobicizing agents or silylation reagents are hexamethyldisilazane (HMDS), trimethylchlorosilane (TMS) and hexamethyldisiloxane (HMDSO).

[0004] The critical step in the production of aerogel materials is the drying of a wet gel. In earlier times it was customary to exclusively use supercritical drying, i.e. drying from a supercritical fluid (typically lower alcohols, later also CO₂). By using solvent drying (uncritical drying) of hydrophobic gels, materials can be produced with virtually identical properties as the supercritically dried aerogels. According to the classic definition, these used to be called xerogels, a term that is still being used also today for aerogels dried from solvents. Hereinafter, the definition based on the material's properties (density < 0.20 g/cm³, porosity > 85%, voids size 20 to 80 nm) is also used for uncritically dried materials, and these are also termed as aerogels.

[0005] The main cost contributors for these insulation materials, which presently are still comparatively expensive, are the costs due to the laborious type of processing, solvent, solvent losses and concomitant VOC release, as well as raw materials and hydrophobicization agents. New developments relating to production processes aim at savings in all of the above mentioned items, with the following development steps having been achieved as of today.

[0006] An early patent document WO 1995/006617 A1 describes the use of water glass as an inexpensive silicate donor. The formation of the silicate gel is carried out by neutralization of a water glass solution with acid to reach a pH value in the range of 7.5 to 11 and subsequent removal of the sodium ions present in the gel by washing with water. A solvent exchange from the aqueous void fluid to alcohol allows for drying from supercritical alcohol by heating in an autoclave. This process is very time-consuming and energy-intensive. Moreover, operating with supercritical alcohol is a significant safety risk due to the high pressures and temperatures used in combination with the flammability of the alcohol.

[0007] An important breakthrough was achieved by means of a simplified drying at atmospheric pressure starting from solvent-containing, hydrophobicized gels. This method has been described in the scientific literature, for example by Anderson, Scherer and their coworkers [J. Non. Cryst. Solids, 1995, 186, 104-112]. The process became rapidly known and spurred new processes for silicate aerogel production. The patent U.S. Pat. No. 5,565,142 describes such a production process.

[0008] WO 1998/005591 A1 relates to a production process for organically modified permanently hydrophobic aerogels. Like in the case of WO 1995/006617 A1 the SiO₂ gel is formed starting from a water glass solvent by means of neutralization with acid or, after formation of a silica sol, by ion exchange and subsequent addition of a base. The pH value during the gelation typically lies in the range between 4 and 8. The wet gel is washed with an organic solvent until the water content is below 5%, and then hydrophobicized. Drying under atmospheric pressure by evaporation of the solvent leaves the aerogel material as granulate material.

[0009] WO 2012/044052 A2 is concerned with the preparation of optically transparent and non-transparent SiO₂ aerogel in granulated form. For this purpose a water glass sol is injected into an alcoholic phase, whereby the gel is formed therein in the form of a granulate material. The gel is further exchanged with alcohol and hydrophobicized by means of a silylating agent. Subsequently, the gel is dried under atmospheric pressure or reduced pressure. The process allows producing an aerogel granulate material with a significantly lower time consumption, but a significant disadvantage consists in the washing with ethanol, which is required in order to remove the water from the hydrogel phase. The processing of the water alcohol mixture requires large amounts of energy, which is a great disadvantage of this process for a large-scale industrial production.

[0010] As a central issue for the present invention one should mention hydrophobicization of alcogels in a mixture comprising a hydrophobicization agent and a hydrophobicization catalyst (HCl):

[0011] WO2013/053951 A1 describes the formation of silicate aerogels and xerogels starting from an alkoxide-based sol. The latter is gelled by adding base and aged under refluxing in alcohol. The gel is then hydrophobicized in a solution comprising a hydrophobicization agent, preferably HMDSO, and HCl as a catalyst and subcritically dried also under refluxing. Various forms of drying are described in detail, and their impact on the quality of the materials thus obtained is explained. A central point to be mentioned is the composition of the void fluid before drying, which is a mixture of alcohol and hydrophobicization agent A. A disadvantage in the process according to WO2013/053951 A1 is that the hydrophobicization agent and, optionally, a corresponding catalyst are added only after the gelation and aging of the gel to form a solid. Particularly in the case of bulky charges the hydrophobicization agent can penetrate into the interior of the gels only in a diffusion-limited manner, i.e. gradually, so that depending on the thickness of the material the hydrophobicization step takes a long time.

SUMMARY OF THE INVENTION

[0012] It is an object of the invention to provide an improved process for the production of aerogel materials.

Further objects are to provide an improved insulant panel and a ready-to-use precursor product for producing an aerogel material.

[0013] These objects are achieved according to the present invention by the production process, by the insulant panel and by the precursor product.

[0014] The invention's process for the production of an aerogel material comprises the following steps:

[0015] a) preparation of a silicon oxide sol in an alcoholic solvent mixture;

[0016] b) triggering of the gelation of the sol by adding base, whereby a gel is formed, and optionally aging of the gel;

[0017] c) hydrophobicization of the optionally aged gel;

[0018] d) removal of the solvent mixture by subcritical drying, whereby the aerogel material is formed;

wherein the silicon oxide sol formed in step a) comprises at least one acid-catalytically activatable hydrophobicization agent, where the volume fraction of the hydrophobicization agent in the sol is 5 to 60%, and wherein the hydrophobicization in step c) is induced as a result of activation or addition of at least one hydrophobicization catalyst acting in combination with the hydrophobicization agent.

[0019] Preferred embodiments will henceforth be described.

[0020] The invention's process allows for a substantially simpler production of silicate-aerogel materials as compared to the presently known processes. By virtue of the fact that hydrophobicization is an acid-catalyzed process, i.e. is catalyzed by H^+ or H_3O^+ ions, respectively, the gelation process that occurs under slightly basic conditions and the hydrophobicization process that occurs under acidic conditions can be carried out in one and the same organogel well-separated from each other temporally. A further advantage of the invention's process is the significantly reduced solvent consumption. In particular, it is possible with the invention's process to limit the solvent amount used for the production of an aerogel to a maximum of 1.1 times the gel volume. According to prior art, typically more than 2 times the gel volume is needed.

[0021] In the present application, an alcoholic solvent mixture shall be understood as a mixture that essentially consists of one or optionally several lower alcohols (in particular ethanol, methanol, n-propanol, isopropanol, butanols) and an appropriate proportion of a hydrophobicization agent. It will be understood that the mixture can further contain a small proportion of water, unavoidable impurities and optionally—as explained elsewhere—certain additives.

[0022] A hydrophobicization agent shall be understood in generally known manner as a component which provides hydrophobic, i.e. water-repellent, properties. In the present application the hydrophobicization agent and the hydrophobicization process relate primarily to the silicate gel and to the modifications of the properties thereof. The aerogel materials that are produced with the invention's process can be produced in form of granulate material, monolithic bodies or composites. In particular, they can be used for the procedurally simple and hence also economically advantageous production of insulant panels.

[0023] The invention's process comprises gelation of an alkoxide-based silicate sol in an alcoholic solvent mixture that contains at least one catalytically activatable hydrophobicization agent.

[0024] The gelation process is initiated by addition of a diluted base such as ammonia. Optionally, the gel thus

formed, which can also be referred to as “organogel”, is additionally subjected to an aging process. The optionally aged gel now contains all components that are required for the hydrophobicization and for the subcritical drying according to WO2013/053951 A1 or, more specifically, it contains a void fluid with the main components alcohol and activatable hydrophobicization agent, but not the hydrophobicization catalyst.

[0025] Subsequently it is necessary to introduce into the gel the hydrophobicization catalyst in a controlled manner, completely and without additional solvent addition or with just a minimal solvent addition. According to a preferred embodiment, hexamethyldisiloxane (HMDSO) is used as the acid-catalytically activatable hydrophobicization agent.

[0026] It is particularly advantageous if the volume fraction of the hydrophobicization agent in the sol is 20 to 50%, particularly 25% to 40% and more particularly 34% to 38%.

[0027] There are various possibilities for adding or activating the hydrophobicization catalyst for step c).

[0028] According to one embodiment, the hydrophobicization catalyst is formed in situ in the gel by a radical decomposition reaction. Advantageously, the hydrophobicization catalyst is formed by radical decomposition of previously added chlorine-containing organic compounds such as a weakly or non-stabilized PVC, tri-chloromethane, chloroacetone or tetrachloroethylene. This allows releasing of the hydrophobicization catalyst, which is advantageously HCl, at a desired time, which can be accomplished either by electromagnetic radiation (UV, x-ray) or by known radical initiators. For gels with high optical transparency and low thickness, photochemical radical decomposition reactions are preferred.

[0029] According to a further embodiment the hydrophobicization catalyst is released by slow-release agents in the gel, with the release optionally being initiated or accelerated by thermal activation. Also in this case it is preferable to use HCl—or any precursors thereof—as the hydrophobicization catalyst which is released from “slow-release” or “controlled-release” additives such as micro- or nano-capsules or particles which are contained in the sol. Ideally, the activation of these agents occurs via externally controllable process parameters such as pressure, temperature or electromagnetic radiation (light, radio waves, microwaves).

[0030] According to a further embodiment, HCl in gaseous form is used as hydrophobicization catalyst that is brought into contact with the gel. This process is particularly suitable for the production of aerogel materials with a large exposed outer surface such as small objects, granulate material and (micro) structured monoliths or composites.

[0031] According to a further embodiment, HCl or TMCS or a mixture of these two components is used as hydrophobicization catalyst, which is dissolved in a diluted solvent mixture having a similar or identical composition as the void fluid and which is brought into contact with the gel in the liquid phase. According to the present invention, the amount of solvent charged with the catalyst as compared to the gel volume shall be kept as small as possible in order to maintain the benefit of keeping the solvent balance of the invention's process as low as possible. Preferably, the catalyst-containing solution in a batch process or in a continuous process shall represent a volume fraction and volume flow fraction of maximally 30%, particularly of maximally 10%.

[0032] According to a further embodiment, the sol further contains at least one functional silane. Depending on the

selection of the silane, the mechanical properties of the aerogel can be altered in a specific manner, particularly the flexibility or the strength thereof.

[0033] According to a further embodiment, the sol further comprises at least one polymerizable monomer, which is capable of forming a polymeric structure within the aerogel material to be produced. Advantageously, the polymerizable monomer is selected from the group consisting of radical polymerizable substances such as acrylates, vinyl chloride, styrene or divinylbenzene. This makes it possible, in particular, to achieve a strengthening of the gel structure.

[0034] According to a further embodiment, the step c) and/or the step d) is carried out in a pressure vessel at an overpressure of for example, up to 20 bar. At atmospheric pressure, the boiling point of the void fluid is between 80 and 100° C. Operating in the pressure vessel allows carrying out the invention's step c) at significant higher temperatures in the range of 90 to 130° C., thus resulting in a higher reaction rate analogously to the example of a pressure cooker. This allows for a dramatic reduction of the hydrophobicization time (for example from 24 h at 65° C. to only 3 h at 90° C.), thus resulting in a significant increase of the efficacy of the process. If desired, an initial phase of the drying step d) can also be carried out at a higher pressure. In the present context the term "initial phase" shall be understood as a part of the drying process that follows after the hydrophobicization step c). In particular, it will be understood that towards the end of the entire process usually a pressure relief is carried out, after which a further drying can still occur depending on the situation.

[0035] According to a further embodiment the step d) is carried out at reduced pressure. Drying in vacuum has the advantage that it can occur at lower temperature, that is, with a reduced heating energy requirement and at a lower temperature level.

[0036] Particularly at the end of the drying process, operating in vacuum at the same temperature allows ending up with a smaller amount of solvents (residual moisture) in the aerogel material. If the preceding hydrophobicization step c) was carried out at an increased pressure, the thermal energy stored in the hot gel can be used upon initiation of the drying step d) to cause the drying process to run much faster initially by actuating the valve of the pressure vessel to effect controlled release of the void fluid in the form of vapor.

[0037] According to a further embodiment, the sol is added to a fiber-based matrix after initiation of gelation. The term "fiber-based matrix" shall be understood both as filamentous as well as fleece-type structures. In this manner structural reinforcements can be embedded in a specific fashion into the aerogel material. In a similar manner such reinforcements can be formed by adding particles, particularly nano-particles or fibers and the like.

[0038] The preparation of the sol on a laboratory scale is usually carried out in a batch process i.e. by stirring a mixture in a resting vessel. On a technical scale the sol is preferably formed continuously in a flow reactor. In particular, step a) and preferably steps b) and c) of the invention's process are thus optionally carried out in a continuous process. Optionally, also step d) can be integrated into the continuous process. The hydrophobicization agent and other additives (polymerizable monomers, fibers, nano-particles etc.) can be added to the sol at various times, but at the latest during gelation. The sequence of steps and the chemical parameters used for the

addition provide for a possibility of creating new materials with precisely adjustable physical properties.

[0039] The catalytically hydrophobicized gels are subcritically dried. The general process can be applied to a number of aerogel materials, whereby depending on the application one can obtain pure silicate aerogels or organically modified aerogels such as polymer-modified x-aerogels or ormosil-aerogels or hybrids of these compound classes.

[0040] Regarding the shape and type of the aerogel materials described in the invention's process, all the customary embodiments (granulate material, panels, plates, ceiling parts ("blankets"), coatings, films) are possible.

In particular, these can be used in an advantageous manner for producing insulant panels.

[0041] According to a further aspect of the invention, there is provided a precursor product for producing an aerogel material. The latter comprises a silicon oxide sol and/or a silicon oxide gel in an alcoholic solvent mixture and further contains at least one acid-catalytically activatable hydrophobicization agent. In this manner one can provide in an advantageous manner a storable and transportable starting material which will only be hydrophobicized, and if necessary dried, at the desired application site. For the latter step the same processing possibilities as described elsewhere herein can be used in principle.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0042] Exemplary embodiments of the invention will henceforth be described.

[0043] Production of an Aerogel Granulate Material

[0044] A silicon oxide sol concentrate is hydrolyzed by alcoholic hydrolysis of TEOS using a molar ratio of TEOS/water/oxalic acid of 1:3.5:0.004 in ethanol at 50° C. for 8 hours under stirring. The silicate content expressed in terms of SiO₂ in the sol concentrate is 18%. The sol concentrate is then diluted with HMDSO and ethanol to the final sol concentration of 6% such that the HMDSO content in the sol is 20%. Thereafter, 1% of a diluted ethanolic ammonia solution is added to this sol, thus causing gelation within 7 minutes. The fresh organogel is aged at 70° C. for 2 h in a block.

[0045] The aged gel is then mechanically crushed, and the gel granulate material is transferred into a closed pressure vessel. Subsequently, a mixture of HCl gas and nitrogen with an overpressure of 2.5 bar is added thereto by pressing. The content of the vessel is then heated up to 100° C. within 30 minutes. The mixture is now hydrophobicized for 90 minutes and then cooled to 50° C., and the over-pressure is released. The hydrophobic gel is subsequently dried on a conveyor belt at 150° C. As the final product there is obtained a hydrophobic aerogel granulate material with a raw density between 0.08 and 0.12 g/cm³ and having a typical thermal conductivity in the material of 0.012 to 0.00145 W/(m K). When measured for a loose granulate material with a medium grain size of 3 mm, the thermal conductivity is between 0.019 to 0.022 W/(m K).

[0046] Industrial Production of an Aerogel Granulate Material

[0047] A silicon oxide sol is hydrolyzed in a flow tube reactor by alcoholic hydrolysis of TEOS with a molar ratio of TEOS/water/hydrochloric acid of 1:1.6:0.003 in ethanol at 75° C., whereby the water is added last and the average residence time is about one hour. In the further course of the flow tube reactor this sol is then diluted with HMDSO and

ethanol such that the HMDSO content in the sol is 30% and the silicate content expressed in terms of SiO_2 is 5.2%. Subsequently, an ethanolic ammonia solution is added, and the mixture is homogenized by means of a static mixer. In this manner the sol flows onto a gelation belt maintained at a temperature of 65° C., with a residence time of 25 minutes.

[0048] Thereafter, the gel is crushed in a breaker to form a granulate material while continuously adding an ethanolic HCl solution. The solvent flow of ethanolic hydrophobicization catalyst is 10% of the gel volume flow. The granulate material is then heated to 100° C. in a pressurized conveyor-belt transit reactor, where it is continuously hydrophobicized and simultaneously pre-dried. In a second part of the drying belt there is atmospheric pressure or below atmospheric pressure, and the final drying temperature is 135° C. A hydrophobic aerogel granulate material with a raw density between 0.092 and 0.117 g/cm³ which has a typical thermal conductivity in the material of 0.013 to 0.0015 W/(m K) is formed as final product. When measured for a loose granulate material with a medium grain size of 4 mm, the thermal conductivity is between 0.0195 to 0.023 W/(m K).

[0049] In an alternative embodiment, the gel is broken over the breaker without solvent or hydrophobicization catalyst. The gel is maintained for a time duration of 3 to 5 minutes as hovering granulate particles in an air stream within a drop shaft. The hydrophobicization catalyst is injected sideways as gaseous HCl or as diluted ethanolic HCl solution, whereby all of the granulate particles are uniformly contacted with the hydrophobicization catalyst. As an alternative to the previously described embodiment, the hydrophobicization and the drying steps can also occur in a cyclone-type drop shaft or in a type of turboflow bed dryer.

[0050] Production of a Composite Material

[0051] A sol with a calculated SiO_2 content of 4.8% is obtained by hydrolysis of TMOS in a solvent mixture consisting of methanol (MeOH) and water in the presence of 0.01 M HCl for 2 h at 65° C. Thereafter, the mixture is cooled to room temperature, so that upon the subsequent dilution with HMDSO an acid-catalyzed hydrophobicization of the individual sol particles does not yet start. The dilution with HMDSO is selected in such manner that the volume fractions in the sol mixture are 65% MeOH, 8% water and 27% HMDSO. Subsequently, 2% of the monomeric ethyl-methacrylate and 0.5% of the polymerizable silane linker 3-(trimethoxysilyl)propyl-methacrylate, which were previously activated over aluminium oxide (removal of the inhibitors), are added to the sol. Thereafter, 0.35 volume percent of poly(vinyl chloride-co-vinyl acetate) micro-particles with a medium particle size of 10 μm are dispersed into the mixture. Finally, 0.05% of the radical initiator tert-butyl-hydroperoxide and 1% of a 1M ammonia solution are added at 30° C., the sol is stirred for 1 minute and then poured into a mold. After gelation, the gel is aged at 55° C. for 4 h. Up to this time no hydrophobicization has occurred.

[0052] Thereafter, the gel bodies are transferred into a closed, pressure-resistant vessel, and the whole assembly is heated to 85-90° C. Hereby an overpressure of about 1 atm is registered in the vessel. Upon heating, the radical initiator is activated, which leads to simultaneous activation of two processes: On the one hand, the PVC-containing micro-particles are activated and thus release a precisely dosed amount of the hydrophobicization catalyst HCl to the gel. In this manner the actual hydrophobicization of the gel with HMDSO is initiated. Simultaneously, the radical polymerization of the meth-

ylmethacrylate is initiated. Accordingly, polymeric chains are formed which are directly attached to the silicate framework via the 3-(trimethoxysilyl)propyl-methacrylate linker. This results in a strengthening of the gel network.

[0053] After 3 h incubation, the reactor is allowed to cool, and the modified gel is dried for 2 h at 140° C. At the end, a polymer-reinforced aerogel composite with a thickness of 0.15 g/cm³ is obtained.

[0054] Production of an Insulant Panel

[0055] 200 ml of a polyethoxy disiloxane (PEDS) sol with 22% SiO_2 content are diluted with 150 ml ethanol and 250 ml HMDSO each. Thereafter, 4.0 g of a slow-release agent in the form of micro-particles doped with 10% HCl and 8.2 g of the flexibilization agent dimethyldiethoxysilane (DMDES) are added. After further addition of 2% of a 1M ammonia solution, the thoroughly mixed sol is poured into a mold which was previously covered with a fittingly cut polyester non-woven fiber mat. The mixture undergoes gelling within 10 minutes, and a gel is formed within the fiber nonwoven matrix. After an aging phase of 24 h at 50° C. the gel plate is carefully lifted out of the mold and taken into a closed vessel maintained at a temperature of 65° C. At this higher temperature, HCl is released from the micro encapsulation and can exert its properties as a hydrophobicization catalyst for HMDSO uniformly throughout the entire volume. The hydrophobicization thus initiated is carried out for 24 h. Thereafter, the vessel is opened, and the gel panel is dried at 145° C. in a convection oven. The finished aerogel insulant panel has a density of 0.11 kg/cm³ and a thermal conductivity of 0.0145 mW/(m K). Moreover, the aerogel insulant panel has flexible mechanical properties due to the DMDES addition.

1. A process for the production of an aerogel material, comprising the following steps:

- preparation of a silicon oxide sol in an alcoholic solvent mixture;
- triggering of the gelation of the sol by adding base, whereby a gel is formed, and optionally aging of the gel;
- hydrophobicization of the optionally aged gel;
- removal of the solvent mixture by subcritical drying, whereby the aerogel material is formed;

characterized in that the silicon oxide sol formed in step a) comprises at least one acid-catalytically activatable hydrophobicization agent, where the volume fraction of the hydrophobicization agent in the sol is 5 to 60%, and that the hydrophobicization in step c) is induced as a result of release or addition of at least one hydrophobicization catalyst acting in combination with the hydrophobicization agent.

2. The process according to claim 1, wherein the catalytically activatable hydrophobicization agent is hexamethyldisiloxane (HMDSO).

3. The process according to claim 1, wherein the volume fraction of the hydrophobicization agent in the sol is 20 to 50%, in particular 25% to 40% and more particularly 34% to 38%.

4. The process according to claim 1, wherein the hydrophobicization catalyst is formed by a radical decomposition reaction in situ in the gel.

5. The process according to claim 1, wherein the hydrophobicization catalyst is released by slow-release agents in the gel and optionally by thermal activation.

6. The process according to claim 1, wherein the hydrophobicization catalyst is gaseous HCl.

7. The process according to claim **1**, wherein the hydrophobicization catalyst is trimethylchlorosilane (TMCS) and/or HCl in an alcoholic solution.

8. The process according to claim **1**, wherein the sol further comprises at least one functional silane.

9. The process according to claim **1**, wherein the sol further comprises at least one polymerizable monomer.

10. The process according to claim **1**, wherein the step c) and optionally an initial phase of step d) are performed at overpressure.

11. The process according to claim **1**, wherein the step d) is performed at reduced pressure.

12. The process according to claim **1**, wherein the sol is added to a fiber-based matrix after initiation of gelation.

13. The process according to claim **1**, wherein at least the steps b) and c) are performed in a continuous process.

14. Insulant panel, formed from an aerogel material produced according to claim **1**.

15. A precursor product for producing an aerogel material, comprising a silicon oxide sol and/or a silicon oxide gel in an

alcoholic solvent mixture and at least one acid-catalytically activatable hydrophobicization agent.

16. The process according to claim **2**, wherein the volume fraction of the hydrophobicization agent in the sol is 20 to 50%, in particular 25% to 40% and more particularly 34% to 38%.

17. The process according to claim **2**, wherein the hydrophobicization catalyst is formed by a radical decomposition reaction in situ in the gel.

18. The process according to claim **3**, wherein the hydrophobicization catalyst is formed by a radical decomposition reaction in situ in the gel.

19. The process according to claim **2**, wherein hydrophobicization catalyst is released by slow-release agents in the gel and optionally by thermal activation.

20. The process according to claim **3**, wherein the hydrophobicization catalyst is released by slow-release agents in the gel and optionally by thermal activation.

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