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(71) Applicant: **3M INNOVATIVE PROPERTIES COM-
 PANY** [US/US]; 3M Center, Post Office Box 33427, Saint
 Paul, Minnesota 55133-3427 (US).

(72) Inventors: **HU, Weili**; 8 Xing Yi Road, Maxdo Center 38/
 F, Shanghai 200336 (CN). **LIN, Weigang**; 8 Xing Yi Road,
 Maxdo Center 38/F, Shanghai 200336 (CN). **SHI, Zhiyu**; 8
 Xing Yi Road, Maxdo Center 38/F, Shanghai 200336 (CN).
LI, Yu; 8 Xing Yi Road, Maxdo Center 38/F, Shanghai
 200336 (CN).

(74) Agent: **GALLAGHER, Ann K.**, et al.; 3M Center, Office
 of Intellectual Property Counsel, Post Office Box 33427,
 Saint Paul, Minnesota 55133-3427 (US).

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 PRODUCT PREPARED FROM THERMAL INSULATION MATERIAL

(57) Abstract: Provided in embodiments of the present disclosure are a thermal insulation material, a method of preparing the thermal
 insulation material, and a product prepared from the thermal insulation material. The thermal insulation material of the embodiments
 of the present disclosure comprises: a non-woven substrate having a thickness retention ratio of at least 70% under a pressure of 1
 kPa; a xerogel; and a cationic surfactant comprising a quaternary ammonium salt and uniformly distributed in the xerogel; wherein
 the content of the non-woven substrate in the thermal insulation material in percentage by weight is 20%-96.7%, the content of the
 xerogel in the thermal insulation material in percentage by weight is 3%-60%, and the content of the cationic surfactant in the thermal
 insulation material in percentage by weight is 0.3%-20%.



**THERMAL INSULATION MATERIAL, METHOD OF PREPARING THERMAL
INSULATION MATERIAL, AND PRODUCT PREPARED FROM THERMAL
INSULATION MATERIAL**

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Technical Field

The present application relates to a thermal insulation material, a method of preparing the thermal insulation material, and a product prepared from the thermal insulation material. Specifically, the present application relates to a thermal insulation material including a xerogel, a method of preparing the thermal insulation material, and a product thereof.

Background

Thermal insulation materials are widely used in clothing, shoes, gloves, etc., or used in bedding such as quilts. In order to achieve a thermal insulation effect, most of the structures of thermal insulation materials can retain air therein, thereby reducing heat energy dissipation.

In some applications where space is limited and a thermal insulation effect is required, for example, in footwear applications, a thermal insulation material would be compressed and consequently the thermal insulation effect is affected. Therefore, currently there are technologies that utilize the characteristics of low density and porosity of aerogels or xerogels to maintain the thermal insulation effect.

In some documents, the terms “xerogel” and “aerogel” are used to describe a porous solid formed by drying a gel. Generally, the difference between the xerogel and the aerogel is based on the porosity and density of the structure. The porosity of the xerogel is usually 20%-40% and the density thereof is 0.5-0.8 g/cc; while the density of the aerogel is usually 0.1-0.2 g/cc and the porosity thereof is at least 75%.

However, the aerogels or xerogels are fragile due to the presence of many pores, and are therefore only applied to limited fields. How to maintain structural integrity in a

compressed state is also an important issue. Among existing aerogel or xerogel products, there are those that use various technologies to increase the physical structure strength of sols thereof. For example, in WO 2013053951, a fiber reinforced material is used in a process of preparing a xerogel; disclosed in WO 1997023675 A2 is a composite material
5 formed by thermoplastic fibers and aerogel particles; disclosed in WO 2017087511 is a synthetic fiber including an aerogel particle and a polymer material; and disclosed in US 20160060808 A1 is a thermal isolation sheet containing a silica xerogel and a non-woven fabric. However, these existing technologies still cannot overcome the problem of fragility of aerogels or xerogels; in addition, many processes of preparing aerogels use subcritical or
10 supercritical conditions, or require solvent exchange for preparation, such as CN 102964111 A, and therefore the processes involve complicated steps and high expenditure.

In addition, there are also some technologies in which polymers are covered on the surface of aerogel or xerogel to overcome the problem of fragility, but these methods would sacrifice the air permeability of thermal insulation materials and increase the weight thereof.

15 Summary

In view of this, provided in embodiments of the present disclosure are a thermal insulation material, a method of preparing the thermal insulation material, and a product prepared from the thermal insulation material. The thermal insulation material provided in the embodiments of the present disclosure does not need to be prepared under harsh
20 conditions such as supercritical and subcritical conditions or by means of solvent exchange methods, and are therefore helpful to simplify preparation processes and reduce preparation costs, and the resultant thermal insulation material still maintains certain structural strength, thermal insulation level, and air permeability, and can therefore be widely applied to various products. In addition, because the thermal insulation material of the embodiments of the
25 present disclosure further has the advantage of antibacterial property, and also maintains a thermal insulation effect in a compressed state by means of a stable structure, the thermal

insulation material is also applicable to environments prone to bacteria breeding, such as but not limited to footwear.

Generally, the thermal insulation material described in the embodiments of the present disclosure comprises a non-woven substrate, a xerogel, and a cationic surfactant.

- 5 The non-woven substrate has a thickness retention ratio of at least 70% under a pressure of 1 kPa. The cationic surfactant includes a quaternary ammonium salt and is uniformly distributed in the xerogel, wherein the content of the non-woven substrate in the thermal insulation material in percentage by weight is 20%-96.7%, the content of the xerogel in the thermal insulation material in percentage by weight is 3%-60%, and the content of the
- 10 cationic surfactant in the thermal insulation material in percentage by weight is 0.3%-20%.

In some embodiments, the material of the non-woven substrate is selected from at least one of a polyester fiber, a nylon fiber, an acrylic fiber, a polypropylene fiber, a polylactic acid fiber, and a cellulose fiber.

- In some embodiments, the initial thickness of the non-woven substrate under a
- 15 pressure of 0.02 kPa is 2-50 mm.

In some embodiments, the xerogel is formed from a gel comprising an organosilicon oxide precursor, and the organosilicon comprises an alkoxysilane.

- In some embodiments, the alkoxysilane comprises an alkyltrialkoxysilane, and optionally, the alkyltrialkoxysilane is methyltrimethoxysilane, vinyltrimethoxysilane, and a
- 20 combination thereof; or, the alkoxysilane comprises a dialkoxysilane, and optionally, the dialkoxysilane is selected from diethoxysilane, dimethoxysilane, and a combination thereof.

- In some embodiments, the gel further comprises a solvent and a catalyst. The solvent comprises an alcohol, for example, methanol, ethanol, or a combination thereof; the catalyst comprises an acidic catalyst (such as oxalic acid, hydrochloric acid, or a combination
- 25 thereof) and a basic catalyst (such as ammonium hydroxide, urea, or a combination thereof).

In some embodiments, the quaternary ammonium salt of the cationic surfactant is selected from a quaternary ammonium halogen salt, for example, hexadecyl trimethyl

ammonium bromide, hexadecyl trimethyl ammonium chloride, benzalkonium bromide, benzalkonium chloride, or a combination thereof.

Further provided in the embodiments of the present disclosure is a method of preparing the thermal insulation material, comprising: (a) providing a sol and a cationic surfactant, the cationic surfactant comprising a quaternary ammonium salt; (b) co-condensing the sol and the cationic surfactant to form a gel; (c) providing a non-woven substrate having a thickness retention ratio of at least 70% under a pressure of 1 kPa; (d) impregnating the non-woven substrate in the gel; and (e) heating and drying the non-woven substrate impregnated with the gel under atmospheric pressure to form the thermal insulation material having a xerogel bonded onto the non-woven substrate, wherein the content of the non-woven substrate in the thermal insulation material in percentage by weight is 20%-96.7%, the content of the xerogel in the thermal insulation material in percentage by weight is 3%-60%, and the content of the cationic surfactant in the thermal insulation material in percentage by weight is 0.3%-20%.

Further provided in the embodiments of the present disclosure is a product prepared from the foregoing thermal insulation material.

Brief Description of the Drawings

The accompanying drawings are used to provide a further understanding of embodiments of the present disclosure, and constitute a part of the description, and serve to explain the present disclosure together with the embodiments of the present disclosure, but do not impose a limitation to the present disclosure. The above features, other features and advantages will become more apparent to those skilled in the art by describing detailed example embodiments with reference to the accompanying drawings, in which:

FIG. 1 is a photograph of a thermal insulation material prepared in an embodiment of the present disclosure.

Detailed Description

In order to enable those skilled in the art to better understand the technical solutions of embodiments of the present disclosure, a thermal insulation material, a method of preparing the thermal insulation material, and a product prepared from the thermal insulation material provided in embodiments of the present disclosure would be described in detail below with reference to the accompanying drawings.

The embodiments of the present disclosure would be adequately described hereinafter with reference to the accompanying drawings, but the illustrated embodiments may be embodied in different forms, and the embodiments of the present disclosure should not be construed as being limited to the embodiments set forth in the present disclosure. Rather, the purpose of providing these embodiments is to make the present disclosure thorough and complete, and to enable those skilled in the art to fully understand the scope of the present disclosure.

Without contradiction, each embodiment of the present disclosure and each feature in the embodiments can be combined with each other.

The terms used in the present disclosure are only used to describe specific embodiments, and are not intended to limit the present disclosure. The term “and/or” as used in the present disclosure includes any and all combinations of one or a plurality of related listed items. The singular forms “a” and “the” as used in the present disclosure are also intended to include plural forms, unless clearly specified in the context otherwise. The terms “comprise” and “prepared from...” as used in the present disclosure designate the presence of the described feature, entirety, step, operation, component, and/or assembly, but do not exclude the presence or addition of one or a plurality of other features, entireties, steps, operations, components, assemblies, and/or groups thereof.

Unless otherwise defined, the meanings of all terms (including technical and scientific terms) used in the present disclosure are the same as those commonly understood by those of ordinary skill in the art. It would also be understood that those terms such as

those defined in commonly used dictionaries should be interpreted as having meanings consistent with the meanings in the background of related art and the present disclosure, and would not be interpreted as having idealized or excessive formal meanings, unless explicitly defined as such in the present disclosure.

5 Unless otherwise defined, the following terms or descriptions in the present application have the following meanings:

 “Xerogel” is used to describe a final product of a porous solid formed by drying a gel.

 “Sol” refers to a raw material used to form a gel.

10 “Gel” refers to a colloidal substance formed by gluing and co-condensing a sol; in the present application, both sol and gel are used to describe a gel before the formation of a xerogel.

 “Organosilicon oxide precursor” refers to a material that forms an organosilicon oxide when hydrolyzed and co-condensed.

15 “Initial thickness” refers to an average thickness of a non-woven substrate measured under a pressure of 0.02 kPa.

 “Gsm” means weight in grams, which means grams per square meter, i.e., the weight in grams of a material per square meter.

 “Clo value” is a parameter to evaluate the thermal insulation capability of a material,
20 which is substantially a thermal resistance value. The larger the value is, the better the thermal insulation capability is. When a person who is quiet or engaged in mental work (calorific value at 209.2 kJ/m²•h) feels comfortable in an environment where the temperature is 21°C, the relative humidity is less than 50%, and the wind speed does not exceed 0.1 m/s, the Clo value of clothes worn thereby is defined as 1.

25 The description of “A to B” or “A-B” includes the value of A, the value of B, and any value greater than A and less than B.

“Percentage by weight of A in B” means that A is a part of B, and refers to the percentage of the weight of A when the weight of B is taken as 100%.

Provide in the embodiments of the present disclosure is a thermal insulation material, including a non-woven substrate, a xerogel, and a cationic surfactant, wherein the cationic
5 surfactant is dispersed in the xerogel, and the xerogel is uniformly distributed on the surface and in pores of the non-woven substrate and bonded to the non-woven substrate, so that the non-woven substrate can capture air by numerous pores of the xerogel, so as to achieve a thermal insulation effect.

In some embodiments, the material of the non-woven substrate may be selected
10 from polyester fibers, such as a vertically lapped 100% polyester fiber (vertically lapped non-woven polyester), the weight in grams thereof may be 50-1240 gsm, which is not limited thereto though.

In addition, the initial thickness of the non-woven substrate is preferably 2-50 mm, and the thickness retention ratio under a pressure of 1 kPa is at least 70%, that is, the
15 thickness of the non-woven substrate under the pressure of 1 kPa should not be less than 70% of the initial thickness, so that the final thermal insulation material prepared has a certain thermal insulation effect.

Certainly, other artificial fibers or natural fibers, such as nylon fibers, acrylic fibers, polypropylene fibers, polylactic acid fibers, cellulose fibers, and other fiber materials may
20 also be used as the non-woven substrate in the embodiments of the present disclosure, without limitation of using only the polyester fibers.

The xerogel in the embodiments of the present disclosure is formed by drying a gel, and the sol used to prepare the gel may include ingredients of an organic silicon oxide precursor, a solvent, a catalyst, and the like. In the embodiments of the present disclosure,
25 an organosilicon of the organosilicon oxide precursor includes an alkoxysilane, for example, an alkyltrialkoxysilane or a dialkoxysilane, and the alkyltrialkoxysilane may be selected

from methyltrimethoxysilane (MTMS), vinyltrimethoxysilane, and a combination thereof, and the dialkoxysilane may be diethoxysilane, dimethoxysilane, or a combination thereof.

The solvent in the sol used to prepare the gel is selectively added, and may be selected from alcohols, which, illustratively, may be methanol, ethanol, or a combination thereof, but is not limited thereto.

The catalyst includes an acidic catalyst and a basic catalyst. The acidic catalyst may be added to the solvent and the organosilicon oxide precursor, so as to use the acidic catalyst to facilitate an early stage of hydrolysis reaction of a co-condensation process, while the basic catalyst is added after the acidic catalyst, so as to accelerate the condensation reaction to form the gel. In some embodiments, the acidic catalyst may be optionally selected from oxalic acid, hydrochloric acid, or a combination thereof; and the basic catalyst may be selected from ammonium hydroxide, urea, and a combination thereof, which are not limited thereto though.

A sol formulation usable in the embodiments of the present disclosure is described in, for example, Chem. Mater. 2005, Vol. 17, 2807-2816 (Dong et al.), Chem. Mater. 2004, Vol. 16, No. 11, 2041 (Loy et al.), Chem. Mater. 2006, Vol. 18, 541-546 (Dong et al.), J. Colloid Interface Sci. 2006, 300, 179-285 (Rao et al.), WO 2010080239 A2, etc., which are incorporated herein by reference.

The xerogel in the embodiments of the present disclosure further includes a cationic surfactant, which may be a quaternary ammonium salt surfactant, and may be preferably a quaternary ammonium salt surfactant comprising a halogen salt, for example, hexadecyl trimethyl ammonium bromide, hexadecyl trimethyl ammonium chloride, benzalkonium bromide, benzalkonium chloride, or a combination thereof. The addition of the surfactant can facilitate the dispersion of the ingredients in the sol. In the embodiments of the present disclosure, the use of the quaternary ammonium salt surfactant can further help to facilitate a polymerization reaction, secure the xerogel structure after drying, and enables an antibacterial effect of the xerogel.

The method of preparing the thermal insulation material in the embodiments of the present disclosure can generally include providing a surfactant and a sol including a solvent, an organosilicon oxide precursor, and an acidic catalyst (wherein the solvent is a selectively added ingredient). In the embodiments of the present disclosure, the surfactant is preferably a cationic surfactant including a quaternary ammonium halogen salt, and the cationic surfactant is uniformly mixed with the ingredients of the sol, and the mixture is then left to stand for a period of time, for example, 24 hours, so that the mixture can undergo hydrolysis and an early stage of condensation reactions, and then a basic catalyst is added to initiate gelling and co-condensation reactions, so as to co-condense the sol and the cationic surfactant to form a gel, and impregnate the non-woven substrate in the gel, so that the gel is completely distributed on the surface and in pores of the non-woven substrate, and the non-woven substrate impregnated with the gel is subsequently heated and dried under an ambient pressure (for example, 1 atmosphere) to obtain the thermal insulation material of the embodiments of the present disclosure. In some embodiments, the drying conditions of the gel are 1 atmosphere and a temperature of 110-150°C, but are not limited thereto, that is, the embodiments of the present disclosure can prepare a xerogel under ambient pressure, but it is not necessary to use supercritical or subcritical conditions or solvent exchange.

The content of the non-woven substrate in percentage by weight in the thermal insulation material prepared in the embodiments of the present disclosure is 20%-96.7%, the content of the xerogel in percentage by weight is 3%-60%, and the content of the cationic surfactant in percentage by weight is 0.3%-20%, and the thermal insulation material has good thermal insulation properties, air permeability, and antibacterial effects, and has a stable structure. The following examples further illustrate the embodiments of the present disclosure.

25

Table 1. Materials used to prepare the thermal insulation material of the embodiments of the present disclosure

Material	Description	Source
Non-woven substrate	Vertically lapped 100% polyester fiber (CVL, 50-1240 gsm, initial thickness of 2-50 mm)	3M Company
	Meltblown combined mesh (Combo, containing 88% meltblown polyacrylate and 12% polyester staple fiber, 100-400 gsm, initial thickness of 3-9 mm)	3M Company
Cationic surfactant	Benzalkonium bromide, benzalkonium chloride	Jiangxi Tak Shing Pharmaceutical Co. Ltd.
	Hexadecyl trimethyl ammonium bromide, hexadecyl trimethyl ammonium chloride	Sinopharm Chemical Reagent Co. Ltd.
Organosilicon oxide precursor	Methyltrimethoxysilane (MTMS, 95%)	
Catalyst	Oxalic acid (prepared as a 0.01M solution), hydrochloric acid (typically prepared as a 0.01M solution, unless otherwise specified), and ammonium hydroxide	
Solvent	Absolute methanol, absolute ethanol	

5 Test methods of various features of the thermal insulation material in the embodiments of the present disclosure are described as follows:

Thickness measurement

10 The thickness of the non-woven substrate having a size of 30 cm*30 cm is measured under 0.02 kPa by a thickness tester, and the result is an initial thickness (T0). The thickness of the non-woven substrate measured under a pressure of 1 kPa is a compressed thickness (T1).

Thickness retention ratio

Thickness retention ratio = compressed thickness (T1)/initial thickness (T0) * 100%.

Thermal insulation effect

- 5 The effect is represented by a Clo value, and is tested using an ASTM C518 standard test method, and takes an average value of three test results.

Thermal insulation effect in compressed state

The effect is represented by a Clo value obtained when the thermal insulation material to be tested is compressed under a pressure of 1 kPa.

- 10 Air permeability

The air permeability is tested using the method of determination of air permeability as specified in GB/T 24218.15-2018 People's Republic of China national standard test method for non-woven fabrics, in which the test area is 20 cm², and the pressure difference is 200 Pa.

- 15 Antibacterial effect

The antibacterial effect is tested using an AATCC100 standard test method in which Escherichia. coli, Staphylococcus aureus, and Candida albicans are used. If 99.0% or more of the bacteria can be inhibited in 24 hours, it is considered to have an antibacterial effect.

Xerogel stability

- 20 The thermal insulation material is visually observed, after completed and when in a compressed state, whether a xerogel falls off, to evaluate the stability of the xerogel attached to the non-woven substrate.

Table 2. Gel formulations in experimental examples and comparative examples of the embodiments of the present disclosure

Group	Gel ingredient and proportion				Surfactant and weight
	Organosilic on oxide precursor	Acidic catalyst	Basic catalyst	Solvent	
Exp Ex 1	MTMS/ 1L	Oxalic acid/ 0.73L	Ammonium hydroxide/ 1M, 0.73L	Methanol/ 0.28L	Hexadecyl trimethyl ammonium bromide/ 30g
Exp Ex 2	MTMS/ 1L	Hydrochloric acid/ 0.25L	Ammonium hydroxide/ 1M, 0.25L	Ethanol/ 1.63L	Hexadecyl trimethyl ammonium chloride/ 100g
Exp Ex 3	MTMS/ 1L	Oxalic acid/ 0.80L	Ammonium hydroxide/ 1M, 0.80L	Methanol/ 0.35L	Benzalkonium bromide/ 150g
Exp Ex 4	MTMS/ 1L	Hydrochloric acid/ 0.186L	Ammonium hydroxide/ 1M, 0.186L	Methanol/ 0.3L	Benzalkonium chloride/ 300g
Exp Ex 5	MTMS/ 1L	Hydrochloric acid/ 10L, 0.001M	Ammonium hydroxide/ 1M, 0.25L	Not added	Hexadecyl trimethyl ammonium bromide/ 100g
Exp Ex 6	MTMS/ 1L	Hydrochloric acid/ 0.25L	Ammonium hydroxide/ 1M, 0.25L	Not added	Hexadecyl trimethyl ammonium bromide/ 300g
Exp Ex 7	MTMS/ 1L	Hydrochloric acid/ 0.25L	Ammonium hydroxide/ 1M, 0.25L	Not added	Hexadecyl trimethyl ammonium bromide/ 400g
Exp Ex 8	MTMS/ 1L	Hydrochloric acid/ 1.5L	Ammonium hydroxide/ 1M, 0.25L	Not added	Hexadecyl trimethyl ammonium

					bromide/ 30g
Comp Ex 1	MTMS/ 1L	Hydrochloric acid/ 0.25L	Ammonium hydroxide/ 1M, 0.25L	Ethanol/ 1.63L	NA
Comp Ex 2	MTMS/ 1L	Hydrochloric acid/ 0.25L	Ammonium hydroxide/ 1M, 0.25L	Ethanol/ 1.63L	Hexadecyl trimethyl ammonium chloride/ 100g
Comp Ex 6	MTMS/ 1L	Hydrochloric acid/ 5L, 0.001M	Ammonium hydroxide/ 1M, 0.25L	Not added	Hexadecyl trimethyl ammonium chloride/ 10g
Comp Ex 7	MTMS/ 1L	Hydrochloric acid/ 1.5L	Ammonium hydroxide/ 1M, 0.25L	Not added	Hexadecyl trimethyl ammonium bromide/ 200g
Comp Ex 8	MTMS/ 1L	Hydrochloric acid/ 15L, 0.001M	Ammonium hydroxide/ 1M, 0.25L	Not added	Hexadecyl trimethyl ammonium chloride/ 100g
Comp Ex 9	MTMS/ 1L	Hydrochloric acid/ 0.2L	Ammonium hydroxide/ 1M, 0.20L	Not added	Hexadecyl trimethyl ammonium chloride/ 300g

The gels in Experimental Examples 2 and 5-8 and Comparative Examples 1-2 and 5-9 were prepared by uniformly mixing the organosilicon oxide precursor, the solvent, the acidic catalyst, and the cationic surfactant for 30 minutes and then leaving the mixture to stand for 1 hour, and subsequently adding the basic catalyst and uniformly mixing to initiate gelling and co-condensation reactions to form the gels. The gel in Experimental Example 1 was prepared by uniformly mixing the organosilicon oxide precursor, the solvent, the acidic catalyst, and the cationic surfactant for 20 minutes and then leaving the mixture to stand for 24 hours, and subsequently adding the basic catalyst and uniformly mixing to initiate gelling

and co-condensation reactions and leaving the mixture to stand for 24 hours to form the gel. The gel in Experimental Example 3 was prepared by uniformly mixing the organosilicon oxide precursor, the solvent, the acidic catalyst, water, and the cationic surfactant for 30 minutes, and subsequently adding the basic catalyst and stirring for 30 minutes to form the gel. The gel in Experimental Example 4 was prepared by uniformly mixing the organosilicon oxide precursor, the solvent, the acidic catalyst, and the cationic surfactant for 30 minutes, and subsequently adding the basic catalyst and stirring for 30 minutes to form the gel.

Table 3. Formulations of the thermal insulation materials in the experimental examples of the embodiments of the present disclosure

Group	Non-woven substrate	Weight in grams (gsm)	Initial thickness (mm)	Thickness retention ratio (%)	Non-woven substrate proportion	Gel proportion	Quaternary ammonium salt proportion	Quaternary ammonium salt type
Exp Ex 1	CVL	110	4	78.3%	67.3%	31.7%	1.0%	Hexadecyl trimethyl ammonium bromide
Exp Ex 2	CVL	150	6	74.3%	75.3%	22.7%	2.0%	Hexadecyl trimethyl ammonium chloride
Exp Ex 3	CVL	250	8	88.9%	62.8%	32.2%	5.0%	Benzalkonium bromide
Exp Ex 4	CVL	380	12	83.8%	59.5%	30.5%	10.0%	Benzalkonium chloride
Exp Ex 5	CVL	50	2	71.2%	96.7%	3.0%	0.3%	Hexadecyl trimethyl ammonium bromide
Exp Ex 6	CVL	700	20	90.0%	20.0%	60.0%	20.0%	Hexadecyl trimethyl ammonium bromide
Exp Ex 7	CVL	880	30	95.0%	30.5%	54.0%	15.5%	Hexadecyl trimethyl ammonium bromide
Exp Ex 8	CVL	1240	50	98.9%	80.4%	19.0%	0.6%	Hexadecyl trimethyl ammonium bromide
Comp Ex 1	CVL	159	6	74.3%	85.3%	14.7%	0.0%	NA

Comp Ex 2	Combo	140	3	46.4%	70.8%	27.2%	2.0%	Hexadecyl trimethyl ammonium bromide
Comp Ex 3	Combo	100	3	46.4%	100.0%	0.0%	0.0%	NA
Comp Ex 4	Combo	200	5	49.0%	100.0%	0.0%	0.0%	NA
Comp Ex 5	Combo	400	9	52.1%	100.0%	0.0%	0.0%	NA
Comp Ex 6	Combo	52	2	71.2%	92.0%	7.9%	0.1%	Hexadecyl trimethyl ammonium chloride
Comp Ex 7	Combo	490	9	52.1%	82.0%	14.5%	3.5%	Hexadecyl trimethyl ammonium bromide
Comp Ex 8	CVL	117	6	74.3%	97.1%	2.6%	0.3%	Hexadecyl trimethyl ammonium chloride
Comp Ex 9	CVL	638	6	74.3%	17.7%	62.3%	20.0%	Hexadecyl trimethyl ammonium chloride

The thermal insulation material was prepared by immersing each group of non-woven fiber in the aforementioned gel. There were no limitations on the size and weight of the non-woven substrate and the amount of the gel used. The principle was that the non-woven substrate could be completely impregnated with and adsorb the gel. After the

5 impregnation was complete, the non-woven substrate with the gel was fed into a squeeze roller under a squeezing pressure of 5 kg/m² to control the final load of the gel, and then drying was performed under ambient pressure (i.e., approximately 1 atmosphere) at 150°C, to obtain the final thermal insulation material, in which the proportions of the non-woven

substrate, the xerogel, and the surfactant were the percentages by weight thereof in the finally prepared thermal insulation material.

Table 4. Thermal insulation effect, air permeability, and antibacterial effect of the experimental examples and comparative examples

Group	Thermal insulation effect in compressed state (Clo)/ weight in grams of non-woven substrate * 100	Air permeability (L/m ² /s)	Antibacterial effect	Does the xerogel fall off?
Exp Ex 1	0.55	6021.7	Y	N
Exp Ex 2	0.59	4982.3	Y	N
Exp Ex 3	0.52	3983.0	Y	N
Exp Ex 4	0.52	3563.0	Y	N
Exp Ex 5	0.54	*	Y	N
Exp Ex 6	0.56	*	Y	N
Exp Ex 7	0.59	*	Y	N
Exp Ex 8	0.68	*	Y	N
Comp Ex 1	0.45	2435.0	N	Y
Comp Ex 2	0.20	320.1	Y	N
Comp Ex 3	0.29	340.4	N	N
Comp Ex 4	0.34	170.3	N	N
Comp Ex 5	0.29	71.9	N	N
Comp Ex 6	0.40	*	N	Y
Comp Ex 7	0.24	*	Y	N
Comp Ex 8	0.43	*	Y	Y
Comp Ex 9	0.19	*	Y	Y

5 * indicates that testing is not performed.

It can be seen from Table 4 that compared with the comparative examples, the embodiments of the present disclosure have a better thermal insulation effect per unit of the non-woven substrate in a compressed state. If Comparative Example 5 is further compared

with Comparative Example 7, it can be seen that even if the xerogel is added in Comparative Example 7 and the quaternary ammonium salt therein help to produce an antibacterial effect, but they are not helpful to improve the thermal insulation effect of the thermal insulation material in a compressed state. Therefore, it is found in the present application that an appropriate thickness retention ratio of the non-woven substrate can help to improve the thermal insulation effect of the thermal insulation material, wherein the thickness retention ratio of the non-woven substrate is preferably greater than 70%.

If Experimental Example 2 is further compared with Comparative Example 1, or Experimental Example 5 is further compared with Comparative Example 6, it can be seen that when the xerogel lacks a quaternary ammonium salt ingredient, or the proportion of the quaternary ammonium salt is excessively low, the binding strength of the xerogel with the substrate would be affected. Therefore, it can be seen that adding a quaternary ammonium salt to the xerogel can help to strengthen the bonding strength of the xerogel with the substrate. It is found in the present application that the proportion of the quaternary ammonium salt is preferably 0.3%-20%.

From the comparison of Experimental Example 2 with Comparative Examples 8 and 9, it can be seen that even if the non-woven substrate has an appropriate thickness retention ratio and quaternary ammonium salt proportion, if the non-woven substrate proportion and the xerogel proportion exceed a range, the xerogel may fall off. That is, it is found in the present application that the proportion of the non-woven substrate is preferably 20%-96.7%, and the proportion of the xerogel is preferably 3%-60%.

In addition, it can be seen from the comparison of the experimental examples with the comparative examples that the addition of the quaternary ammonium salt not only helps to improve the structural stability of the thermal insulation material, but also enables the thermal insulation material to produce an antibacterial effect. It can also be seen from Experimental Example 2 and Comparative Example 1 that the thermal insulation material provided by the method of the embodiments of the present disclosure has better air

permeability; and if groups with similar weights in grams (Experimental Example 1 and Comparative Example 3, Experimental Example 3 and Comparative Example 4, and Experimental Example 4 and Comparative Example 5) are compared, it can be seen that the thermal insulation material prepared by the method of the embodiments of the present disclosure has excellent air permeability, and the addition of the quaternary ammonium salt in an appropriate proportion also helps to improve the air permeability of the thermal insulation material (Experimental Example 2 compared with Comparative Example 1).

It can thus be seen that provided in the embodiments of the present disclosure is a thermal insulation material having good structural stability, good air permeability and antibacterial effects that can be prepared under ambient pressure, and the thermal insulation material maintains a good thermal insulation capability when compressed, and is therefore suitable for application to various products, such as clothing, bedding, footwear, etc. Because the thermal insulation material of the embodiments of the present disclosure has good air permeability and bacteriostatic capability, and still has a thermal insulation capability in a compressed state, when applied to shoes, hosiery, and other products, the thermal insulation material can thereby avoid generation of unpleasant smell and allow for user comfort and warmth.

The present disclosure has disclosed example embodiments, and although specific terms are used, they are intended to and should only be interpreted as having general descriptive meanings, and are not intended for the purpose of limitation. In some examples, it would be obvious for those skilled in the art that unless clearly indicated otherwise, the features, characteristics, and/or elements described in conjunction with specific embodiments may be used alone, or may be used in combination with features, characteristics, and/or components described in conjunction with other embodiments. Therefore, those skilled in the art would understand that various changes in form and details can be made without departing from the scope of the present disclosure as set forth by the appended claims.

What is claimed is:

1. A thermal insulation material, comprising:
a non-woven substrate having a thickness retention ratio of at least 70% under a
5 pressure of 1 kPa;
an xerogel; and
a cationic surfactant comprising a quaternary ammonium salt and evenly distributed
in the xerogel;
wherein the content of the non-woven substrate in the thermal insulation material in
10 percentage by weight is 20%-96.7%, the content of the xerogel in the thermal insulation
material in percentage by weight is 3%-60%, and the content of the cationic surfactant in the
thermal insulation material in percentage by weight is 0.3%-20%.

2. The thermal insulation material according to claim 1, wherein the material of the
15 non-woven substrate is selected from at least one of a polyester fiber, a nylon fiber, an
acrylic fiber, a polypropylene fiber, a polylactic acid fiber, and a cellulose fiber.

3. The thermal insulation material according to claim 1, wherein an initial thickness of
the non-woven substrate under a pressure of 0.02 kPa is 2-50 mm.
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4. The thermal insulation material according to claim 1, wherein the xerogel is formed
from a gel comprising an organosilicon oxide precursor, and the organosilicon comprises
an alkoxysilane.

25 5. The thermal insulation material according to claim 4, wherein the alkoxysilane
comprises an alkyltrialkoxysilane, and the alkyltrialkoxysilane is methyltrimethoxysilane,
vinyltrimethoxysilane, and a combination thereof.

6. The thermal insulation material according to claim 4, wherein the alkoxysilane comprises a dialkoxysilane, and the dialkoxysilane is selected from diethoxysilane, dimethoxysilane, and a combination thereof.
- 5
7. The thermal insulation material according to claim 4, wherein the gel further comprises a solvent and a catalyst.
8. The thermal insulation material according to claim 7, wherein the solvent comprises
- 10 an alcohol, and the alcohol is selected from methanol, ethanol, and a combination thereof.
9. The thermal insulation material according to claim 7, wherein the catalyst comprises an acidic catalyst and a basic catalyst.
- 15 10. The thermal insulation material according to claim 9, wherein the acidic catalyst is selected from oxalic acid, hydrochloric acid, and a combination thereof.
11. The thermal insulation material according to claim 9, wherein the basic catalyst is selected from ammonium hydroxide, urea, and a combination thereof.
- 20
12. The thermal insulation material according to claim 1, wherein the quaternary ammonium salt of the cationic surfactant is selected from a quaternary ammonium halogen salt.
- 25 13. The thermal insulation material according to claim 12, wherein the quaternary ammonium halogen salt is selected from hexadecyl trimethyl ammonium bromide, hexadecyl trimethyl ammonium chloride, benzalkonium bromide, benzalkonium chloride,

and a combination thereof.

14. A method of preparing the thermal insulation material, comprising:

- 5 (a) providing a sol and a cationic surfactant, the cationic surfactant comprising a quaternary ammonium salt;
- (b) co-condensing the sol and the cationic surfactant to form a gel;
- (c) providing a non-woven substrate having a thickness retention ratio of at least 70% under a pressure of 1 kPa;
- (d) impregnating the non-woven substrate in the gel; and
- 10 (e) heating and drying the non-woven substrate impregnated with the gel under atmospheric pressure to form the thermal insulation material having a xerogel bonded onto the non-woven substrate, wherein the content of the non-woven substrate in the thermal insulation material in percentage by weight is 20%-96.7%, the content of the xerogel in the thermal insulation material in percentage by weight is 3%-60%, and the content of the
- 15 cationic surfactant in the thermal insulation material in percentage by weight is 0.3%-20%.

15. The method of preparing the thermal insulation material according to claim 14, wherein the sol comprises an organosilicon oxide precursor, and the organosilicon comprises an alkoxysilane.

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16. The method of preparing the thermal insulation material according to claim 15, wherein the alkoxysilane comprises an alkyltrialkoxysilane, and the alkyltrialkoxysilane is methyltrimethoxysilane, vinyltrimethoxysilane, and a combination thereof.

25 17. The method of preparing the thermal insulation material according to claim 15, wherein the alkoxysilane comprises a dialkoxysilane, and the dialkoxysilane is selected from diethoxysilane, dimethoxysilane, and a combination thereof.

18. The method of preparing the thermal insulation material according to claim 14, wherein the quaternary ammonium salt is selected from a quaternary ammonium halogen salt.
- 5 19. The method of preparing the thermal insulation material according to claim 18, wherein the quaternary ammonium halogen salt is selected from hexadecyl trimethyl ammonium bromide, hexadecyl trimethyl ammonium chloride, benzalkonium bromide, benzalkonium chloride, and a combination thereof.
- 10 20. The method of preparing the thermal insulation material according to claim 14, wherein the sol further comprises a solvent and a catalyst.
21. The method of preparing the thermal insulation material according to claim 20, wherein the solvent comprises an alcohol, and the alcohol is selected from methanol, 15 ethanol, and a combination thereof.
22. The method of preparing the thermal insulation material according to claim 20, wherein the catalyst comprises an acidic catalyst and a basic catalyst.
- 20 23. The method of preparing the thermal insulation material according to claim 14, wherein the material of the non-woven substrate is selected from at least one of a polyester fiber, a nylon fiber, an acrylic fiber, a polypropylene fiber, a polylactic acid fiber, and a cellulose fiber, and an initial thickness of the non-woven substrate under a pressure of 0.02 kPa is 2-50 mm.
- 25 24. The method of preparing the thermal insulation material according to claim 14, wherein the heating condition in step (e) is 110-150°C.

25. A product prepared from the thermal insulation material according to any one of claims 1 to 13.

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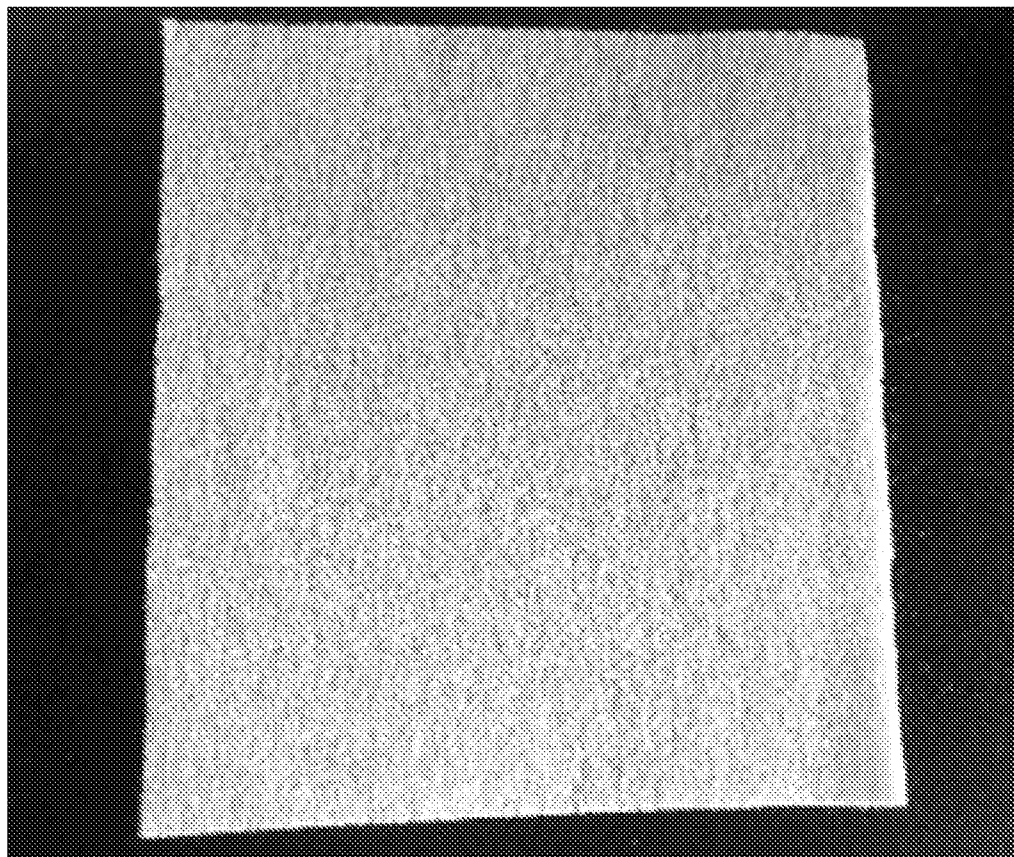


FIG. 1

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2021/056470

A. CLASSIFICATION OF SUBJECT MATTER INV. D06M11/11 D06M11/61 D06M13/192 D06M13/432 D06M13/463 D06M13/507 D06M13/513 D06M16/00 C01B33/16 F16L59/00 D04H1/54 F16L59/02				
According to International Patent Classification (IPC) or to both national classification and IPC				
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) D06M F16L C01B D04H				
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched				
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EP0-Internal, WPI Data				
C. DOCUMENTS CONSIDERED TO BE RELEVANT				
Category*	Citation of document, with indication, where appropriate, of the relevant passages			Relevant to claim No.
X	WO 2016/171558 A1 (SEPALEX S A S [FR]) 27 October 2016 (2016-10-27)			1-23,25
Y	page 1, lines 5-10 page 4, line 5 - page 5, line 7 page 10, lines 1-3; claims 1,9-10; example 1			24
A	----- WO 2019/027046 A1 (SAMSUNG R&D INST JAPAN CO LTD [JP]) 7 February 2019 (2019-02-07) paragraphs [0001], [0006], [0019]; examples 1-6			1-25
T	----- US 2006/223965 A1 (TRIFU ROXANA [US]) 5 October 2006 (2006-10-05) paragraphs [0004], [0040], [0042]; example 1; table 1 ----- -/-			
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.				
* Special categories of cited documents :				
"A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family				
Date of the actual completion of the international search 8 October 2021			Date of mailing of the international search report 03/11/2021	
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016			Authorized officer Menard, Claire	

INTERNATIONAL SEARCH REPORT

International application No

PCT/IB2021/056470

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 2011 136859 A (ASAHI FIBREGLASS CO) 14 July 2011 (2011-07-14) paragraphs [0001], [0009]; claim 14; examples 1-13 -----	24

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Information on patent family members

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