(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
14 January 2021 (14.01.2021)

WIPO | PCT

(10) International Publication Number
WO 2021/004555 A1

(51) International Patent Classification:
C04B 16/08 (2006.01) *C08J 9/224* (2006.01)
C04B 28/26 (2006.01) *C08J 9/236* (2006.01)

(21) International Application Number:
PCT/CZ2020/000018

(22) International Filing Date:
26 May 2020 (26.05.2020)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
PV 2019-445 05 July 2019 (05.07.2019) CZ

(71) Applicant: **FIRST POINT A.S.** [CZ/CZ]; Brněnská 4404/65a, 69501 Hodonín (CZ).

(72) Inventors: **CHLANDOVÁ, Gabriela**; Jana Žižky 500, 51724 Borohrádek (CZ). **ŠPANIEL, Petr**; Bož. Němcové 61, 51771 České Meziříčí (CZ).

(74) Agent: **ŠKODA, Milan**; Nahořanská 308, 549 01 Nové Město nad Metují (CZ).

(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JO, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:
— *of inventorship (Rule 4.17(iv))*

Published:
— *with international search report (Art. 21(3))*

(54) **Title:** INSULATING MATERIAL AND METHOD FOR ITS PRODUCTION

(57) **Abstract:** An insulating material, in particular a permeable fire-proof insulating material comprising water glass and polystyrene, consisting of a hardening mixture which contains 1 to 32.4 wt% of expanded polystyrene, 57.5 to 96.0 wt% of aqueous sodium silicate solution, 2 to 6 wt% of aluminium hydroxide, 0.8 to 2.6 wt% water glass hardener and 0.1 to 0.5 wt% of water glass stabiliser, while the surface of the expanded polystyrene is provided with carbon black, the carbon black making up 0.1 to 1 wt% of total weight. A method for the production of insulating material, in particular a method for the production of permeable fire-proof insulating material comprising water glass and polystyrene, according to which firstly the polystyrene beads are mixed with an aqueous solution of carbon black so as to coat their entire surface, then is added to the aqueous sodium silicate solution aluminium hydroxide and the whole is mixed so as to form an insulating mixture, and then a water glass stabiliser is added to the aqueous sodium silicate solution, and then to this solution is mixed water glass hardener, with this solution being further stirred for 1 to 10 minutes to form a binder solution, and the insulating mixture is added to the binder solution with constant stirring, and the whole is mixed, and the resulting mixture is then poured into the application site.

WO 2021/004555 A1

Insulating material and method for its production

Technical Field

The invention relates to an insulating material, in particular a permeable fire-proof insulating material containing water glass and polystyrene, and to a method for its production.

State of the Art

From current technology the use of expanded polystyrene as an insulating material for various types of buildings is known. Its disadvantage is lower fire resistance.

For the insulation of horizontal surfaces, polystyrene panels as well as modern sprayed insulation made of PUR foam are used. The disadvantage of this foam is its lower fire resistance and rapid ageing.

Another known method of insulating horizontal and vertical surfaces is mineral wool insulation. Mineral wool has a higher fire resistance, but it is absorbent, so it loses its insulating properties and mould forms in it.

From the patent application CZ PV2017-127 an acoustic and thermal insulator for use in construction is known, which consists of a slurry in an air-hardening mixture containing 5 to 76 wt% of bulk thermal insulation material with a specific volumetric mass of less than 300 kg/m^3, 9 to 36 wt% of brick dust fractions 0.001 to 1 mm, 6 to 30 wt% of water glass, 7 to 30 wt% of water and up to 5 wt% detergent. The disadvantage of this material is that it has lower thermal insulation properties, higher flammability and less cohesion.

From the utility model CZ 31095 a mixture for a permeable fire-proof lightweight polystyrene thermal insulation system is known, which contains 10 wt% of expanded polystyrene beads with a diameter of 3 to 6 mm, 88 wt% of sodium silicate water glass, 1 wt% carbon black, and 1 wt% water glass stabiliser - hydrophilic alkoxy alkyl-ammonium salts. The disadvantage of this mixture is that the carbon black is not a protection on the surface of the balls, but is freely dispersed in the insulating material, which causes higher thermal conductivity of the insulating

material and low thermal stability and thus limited fire-proofing properties, lower resistance to UV radiation and therefore it degrades very quickly.

From the aforementioned current technology it is clear that main disadvantages of current technology are the lower insulating properties of the known materials and the higher rate of their degradation.

The object of the invention is the construction of a light insulating material which will have high fire resistance, while at the same time being flexible and pliable and, be resistant to degradation.

Principle of the Invention

The above-mentioned drawbacks are largely eliminated and the objects of the invention are fulfilled by an insulating material, in particular a permeable fire-proof insulating material containing water glass and polystyrene, which according to the invention is characterised by that it consists of a hardening mixture containing 1 to 32.4 wt% of expanded polystyrene, 57.5 to 96.0 wt% of aqueous sodium silicate solution, 2 to 6 wt% aluminium hydroxide, 0.8 to 2.6 wt% water glass hardener and 0.1 to 0.5 wt% water glass stabiliser, while the surface of the expanded polystyrene is provided with carbon black, which constitute 0.1 to 1% wt% of the total weight. The advantage of this insulating material is significantly higher thermal stability as well as significantly improved fire-proof properties, the resistance to UV radiation is higher and the degree of degradation is significantly lower. An advantage is also very good permeability. To improve flame retardancy, the mixture contains aluminium hydroxide. The advantage of providing the surface of the expanded polystyrene with carbon black is that the carbon black thus provided reduces thermal conductivity, with the carbon black to advantage being absorbed to a certain extent into the polystyrene beads, thereby stabilising their bond with the polystyrene beads in the resulting mixture. A further advantage is that the carbon black acts as a flame co-retardant. The insulating material to further advantage comprises a hardener, which may be glycerol mono to triacetate or a mixture of these.

To advantage, the expanded polystyrene beads are with a diameter of 3 to 6 mm. The advantage is the possibility of optimising the structure of the material with regard to optimal arrangement.

It is also to advantage that the water glass stabilisers are hydrophilic alkoxy alkyl-ammonium salts.

A great advantage is then that the aqueous sodium silicate solution has a density in the range of 1370 to 1400 kg/m^3 and the molar ratio of SiO_2 and Na_2O is in the range of 3.2 to 3.4. The molar mass ratio of silica to sodium oxide and the associated solution density and solution concentration have a significant effect on the rheological properties of water glass as a polymer mixture, on the electrical properties, compressibility and adhesive strength as in an electrolyte, further to hardness, strength, etc. The advantage of the above stated parameters is that the resulting insulating material is partially flexible and pliable after solidification.

The mentioned disadvantages are largely removed and the objectives of the invention are fulfilled by a method for producing insulating material, specifically a method for producing permeable fire-proof insulating material containing water glass and polystyrene, which according to the invention is characterised by that firstly polystyrene beads are mixed with an aqueous carbon black solution in such a way that their entire surface is coated, then aluminium hydroxide is added and the whole is mixed to form an insulating mixture, and then a water glass stabiliser is added to the aqueous sodium silicate solution, and then a water glass hardener is mixed into the solution. this solution is stirred for 1 to 10 minutes to form a binder solution, and then the insulating mixture is poured into the binder solution while constantly stirring, and the whole is mixed, and then the resulting mixture is poured onto the application site. The advantage is that it is possible to produce both solid products, such as insulation panels and fittings, and the insulation material can even be applied in its liquid state.

It is advantageous if the resulting mixture is poured into the application site, which is a mould, and further the amount of binder solution is extruded from the resulting mixture by means of a press so as to produce the desired ratio of insulating mixture and binder solution. The advantage is that it is possible to easily produce a product with precise parameters.

It is also advantageous if the resulting mixture is finally left to stand until cured. The advantage is that the resulting insulation can be created exactly with respect to the shape parameters of the insulated space, with the fact that due to the fact that the curing length can be regulated, the insulation material can be precisely shaped into the desired shape.

The main advantage of the insulating material and the method of its production according to the invention is that it has comparable insulating properties as the polystyrene products used so far, while unlike existing materials it is non-flammable, vapour permeable, resistant to rainwater and moisture, anti-fungal, strong, flexible, resistant to external influences such as UV radiation. Another advantage is the simple methods of application. It is possible to create both cladding boards and fittings from the insulating material, and it can be easily applied as a liquid mixture by stretching, casting and spraying. The insulating material is thus suitable for floors and ceilings, horizontal and slightly sloping roofs, where it replaces mineral wool, polystyrene concrete or polyurethane foam. Unlike insulation with mineral wool or polystyrene boards, it is well applied to hard-to-reach places and to rugged edges of the surface. It has good adhesion to various substrates, including trapezoidal and folded sheets, Eternit or asphalt, which are usually on roofs. At the same time, the insulating material is strong enough, it can also be walkable. A great advantage of the insulating material according to the invention over the existing materials is also the possibility of combining boards and a liquid mixture. One of the problems associated with the anchoring of conventional polystyrene boards is the filling of joints between the boards and the holes around the dowels. Thanks to the possibility of filling these gaps and openings with a liquid form of insulating material, a uniform surface without thermal bridges is created very easily and quickly. A great advantage is also that the semi-finished insulating material in the form of a liquid mixture can be applied as an insulating lining in industry, e.g., appliances, electrical technology, automotive, etc.

Examples of the Performance of the Invention

Example 1

The permeable fire-proof insulating material is composed an air-hardening mixture which contains 10 wt% of expanded polystyrene, which are spheres with a diameter of 3 to 6 mm, 83.0 wt% of aqueous sodium silicate solution, 4 wt% of aluminium hydroxide, 0.3 wt% water glass stabiliser and 2.3 wt% hardeners.

The surface of the expanded polystyrene is provided with carbon black, the carbon black making up 0.4 wt% of total weight.

The water glass stabilisers are hydrophilic alkoxy alkyl-ammonium salts, in the form of a 98% aqueous solution of N,N,N',N'-Tetrakis (2-hydroxypropyl) ethylenediamine.

The aqueous sodium silicate solution has a density in the range of 1390 kg/m^3 and a molar ratio of SiO_2 to Na_2O of 3.3.

The water glass hardener is a mixture of pure glycerol diacetate / triacetate in a ratio of 7: 3 by volume parts, with a concentration of 2.8 wt% to pure water glass.

According to the method for producing the insulating material, firstly the polystyrene beads are mixed with an aqueous solution with a concentration of 25 wt% of carbon black so that their entire surface is coated with carbon black, then aluminium hydroxide is added and the whole is mixed to form an insulating mixture, then added to an aqueous solution of sodium silicate is a water glass stabiliser, followed by a water glass hardener being added to the solution, this solution being mixed for 5 minutes to form a binder solution, and then the insulating mixture is added to the binder solution with constant stirring, and the whole is mixed, and the resulting mixture is poured into an application site which is a silicone mould, and further, from the resulting mixture such an amount of binder solution is extruded by means of a press so that the desired ratio of insulating mixture and binder solution is obtained.

Finally, the resulting mixture is left undisturbed until hardened. The resulting product is an insulating board, or an insulating layer arranged on an OSB board, more precisely between two OSB boards

Example 2

The permeable fire-proof insulating material is composed of an air-hardening mixture which contains 1 wt% of expanded polystyrene, which are spheres with a diameter of 3 to 6 mm, 96.0 wt% of aqueous sodium silicate solution, 2 wt% of aluminium hydroxide, 0.1 wt% water glass stabiliser and 0.8 wt% hardeners.

The surface of the expanded polystyrene is provided with carbon black, the carbon black making up 0.1 wt% of total weight.

The water glass stabilisers are hydrophilic alkoxy alkyl-ammonium salts, in the form of a 98% aqueous solution of N,N,N',N'-Tetrakis (2-hydroxypropyl) ethylenediamine.

An aqueous solution of sodium silicate having a density in the range of 1370 kg/m^3 and a molar ratio of SiO_2 to Na_2O in the range of 3.2.

The water glass hardener is a mixture of pure glycerol diacetate / triacetate in a ratio of 7:3 by volume parts, with a concentration of 0.8 wt% to pure water glass.

According to the method for producing the insulating material, firstly the polystyrene beads are mixed with an aqueous solution with a concentration of 25 wt% of carbon black so that their entire surface is coated with carbon black, then aluminium hydroxide is added and the whole is mixed to form an insulating mixture, then added to an aqueous solution of sodium silicate is a water glass stabiliser, followed by a water glass hardener being added to the solution, this solution being mixed for 1 minute to form a binder solution, and then the insulating mixture is added to the binder solution with constant stirring, and the whole is mixed, and the resulting mixture is poured into a flat, divided attic space, spread, surface-treated and left undisturbed to harden.

Example 3

The permeable fire-proof insulating material is composed of an air-hardening mixture which contains 32.4 wt% of expanded polystyrene, which are spheres with a diameter of 3 to 6 mm, 57.5 wt% of aqueous sodium silicate solution, 6 wt% of aluminium hydroxide, 0.5 wt% water glass stabiliser and 2.6 wt% hardener.

The surface of the expanded polystyrene is provided with carbon black, the carbon black making up 1 wt% of total weight.

The water glass stabilisers are hydrophilic alkoxy alkyl-ammonium salts, in the form of a 98% aqueous solution of N,N,N',N'-Tetrakis (2-hydroxypropyl) ethylenediamine.

An aqueous solution of sodium silicate having a density in the range of 1400 kg/m^3 and a molar ratio of SiO_2 to Na_2O in the range of 3.4.

The water glass hardener is a mixture of pure glycerol diacetate / triacetate in a ratio of 7:3 by volume parts, with a concentration of 4.5 wt% to pure water glass.

According to the method for producing the insulating material, firstly the polystyrene beads are mixed with an aqueous solution with a concentration of 25 wt% of carbon black so that their entire surface is coated with carbon black, then aluminium hydroxide is added and the whole is mixed to form an insulating mixture, then added to an aqueous solution of sodium silicate is a water glass stabiliser,

followed by a water glass hardener being added to the solution, this solution being mixed for 10 minutes to form a binder solution, and then the insulating mixture is added to the binder solution with constant stirring, and the whole is mixed, and the resulting mixture is then poured onto the outer wall of a building provided with formwork with a silicone surface and finally, the resulting mixture is left undisturbed to harden, after which the formwork is removed.

Industrial Application

The insulating material according to the invention can in particular be used to create a permeable fire-proof insulating system in the building industry.

Patent Claims

1. An insulating material, in particular a permeable fire-proof insulating material comprising water glass and polystyrene, **characterized in that** it consists of a hardening mixture which contains 1 to 32.4 wt% of expanded polystyrene, 57.5 to 96.0 wt% of aqueous sodium silicate solution, 2 to 6 wt% of aluminium hydroxide, 0.8 to 2.6 wt% water glass hardener and 0.1 to 0.5 wt% of water glass stabiliser, and the surface of the expanded polystyrene is provided with carbon black, the carbon black making up 0.1 to 1 wt% of total weight.
2. The insulating material according to Claim 1, **characterized in that** the expanded polystyrene is spheres with a diameter of 3 to 6 mm.
3. The insulating material according to any one of the preceding claims, **characterized in that** the water glass stabilisers are hydrophilic alkoxy alkyl-ammonium salts.
4. The insulating material according to any one of the preceding claims, **characterized in that** the aqueous sodium silicate solution has a density in the range from 1370 to 1400 kg/m^3.
5. The insulating material according to one of the preceding claims, **characterized in that** the aqueous sodium silicate solution has a molar ratio of SiO_2 to Na_2O in the range of 3.2 to 3.4.
6. A method for producing insulating material, in particular a method for the production of a permeable fire-proof insulating material comprising water glass and polystyrene, according to any one of claims 1 to 5, **characterized in that** firstly the polystyrene beads are mixed with an aqueous solution of carbon black so as to coat their entire surface, then is added to the aqueous sodium silicate solution aluminium hydroxide and the whole is mixed so as to form an insulating mixture, and then a water glass stabiliser is added to the aqueous sodium silicate solution, and then to this solution is mixed water glass hardener, with this solution being further stirred for 1 to 10 minutes to form a binder solution, and the insulating mixture is added to the binder solution with constant stirring, and the whole is mixed, and the resulting mixture is then poured into the application site.

7. The method for producing insulating material according to claim 6, **characterized in that** the resulting mixture is poured into the application site, which is a mould, and further, from the resulting mixture such an amount of binder solution is extruded by means of a press so that the desired ratio of insulating mixture and binder solution is obtained.
8. The method for producing insulating material, according to any one of Claims 6 and 7, **characterized in that** finally, the resulting mixture is left undisturbed to harden.

INTERNATIONAL SEARCH REPORT

International application No
PCT/CZ2020/000018

A. CLASSIFICATION OF SUBJECT MATTER
INV. C04B16/08 C04B28/26 C08J9/224 C08J9/236
ADD.

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)
C04B C08J

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)
EPO-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	CZ 31 095 U1 (SPANIEL PETR) 25 October 2017 (2017-10-25) cited in the application	1-5
Y	paragraphs [0023], [0025]; claims -----	6-8
A	US 5 242 494 A (CALLAGHAN IAN C [GB] ET AL) 7 September 1993 (1993-09-07) column 2, lines 1-16 column 4, line 65 - column 5, line 11 -----	1-8
A	RU 2 545 287 C1 (ESAULOV SERGEJ KONSTANTINOVICH [RU]) 27 March 2015 (2015-03-27) paragraphs [0024], [0121] -----	1-8
	-/--	

[X] Further documents are listed in the continuation of Box C.

[X] 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	Date of mailing of the international search report
11 August 2020	19/08/2020

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Theodoridou, K

1

Form PCT/ISA/210 (second sheet) (April 2005)

INTERNATIONAL SEARCH REPORT

International application No
PCT/CZ2020/000018

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2012/019988 A1 (BASF SE [DE]; ULANOVA TATIANA [DE] ET AL.) 16 February 2012 (2012-02-16) page 10, lines 15-30	1-5
A	WO 2007/023091 A1 (BASF AG [DE]; ALLMENDINGER MARKUS [DE] ET AL.) 1 March 2007 (2007-03-01) page 2, lines 15-39 page 3, line 33 - page 4, line 21 page 4, line 33 - page 5, line 17 page 5, line 36 - page 6, line 7 page 6, line 34 - page 7, line 4 page 8, lines 32-39; examples	1-5
Y	EP 0 620 246 A1 (ALGOSTAT GMBH & CO KG [DE]) 19 October 1994 (1994-10-19) column 2, lines 44-54 column 3, lines 26-39	6-8
A	EP 0 004 846 A1 (CROSBY & CO LTD [GB]) 31 October 1979 (1979-10-31) page 2, lines 13-31; claims 1,3,8 page 4, line 31 - page 5, line 5	1-8

1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/CZ2020/000018

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
CZ 31095 U1	25-10-2017	NONE	
US 5242494 A	07-09-1993	CA 2048881 A1	11-02-1992
		EP 0472273 A1	26-02-1992
		JP H04270110 A	25-09-1992
		KR 920004482 A	27-03-1992
		US 5242494 A	07-09-1993
RU 2545287 C1	27-03-2015	NONE	
WO 2012019988 A1	16-02-2012	EP 2603550 A1	19-06-2013
		WO 2012019988 A1	16-02-2012
WO 2007023091 A1	01-03-2007	AT 427335 T	15-04-2009
		AU 2006283921 A1	01-03-2007
		CA 2621731 A1	01-03-2007
		CN 101248120 A	20-08-2008
		CN 101253231 A	27-08-2008
		DE 102005039976 A1	08-03-2007
		DK 1919990 T3	29-06-2009
		EP 1919990 A1	14-05-2008
		ES 2322506 T3	22-06-2009
		JP 2009506150 A	12-02-2009
		KR 20080049753 A	04-06-2008
		PL 1919990 T3	31-08-2009
		SI 1919990 T1	30-06-2009
		US 2008234400 A1	25-09-2008
		WO 2007023091 A1	01-03-2007
		ZA 200802533 B	28-10-2009
		ZA 200802580 B	28-10-2009
EP 0620246 A1	19-10-1994	AT 176261 T	15-02-1999
		DE 9305431 U1	11-08-1994
		DK 0620246 T3	13-09-1999
		EP 0620246 A1	19-10-1994
EP 0004846 A1	31-10-1979	EP 0004846 A1	31-10-1979
		GB 1583308 A	21-01-1981