106-84 M CROSS REFERENCE EXAMINER

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IMPROVED SILICACEOUS INSULATING MATERIAL

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11 Claims. (Cl. 260—2.5) 106-75X

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This invention relates to insulating material, suitable for application either directly to surfaces to be insulated or in the form of blocks, which comprises the reaction products of sodium silicate, and aluminum and more particularly to such a material which comprises the products of adding either an alkyl silicate or certain types of not fully polymerized resinous polymers or both to the reaction mixture of sodium silicate and aluminum.

In preparing foam-like materials from an alkali silicate with a powdered metal reactive therewith, such as aluminum, the foam-like material thereby produced is more rigid, more porous, and has more uniform pores, and has greater overall strength and weather resistance if a small amount of alkyl silicate is added thereto.

Many methods and substances have been proposed or used for insulating walls, ceilings and roofs of buildings and for ovens, refrigerators and like purposes. All of these have disadvantages of one sort or another, such as low strength, low insulating value, high specific gravity, high cost, and others.

It has been found that by utilizing the reaction of aluminum with sodium silicate, an excellent insulating material may be formed. By carrying out the reaction in situ, and applying a mixture of the invention to a wall or other surface before the reaction has been completed, an insulating layer may be formed directly at the place needed. By carrying out the reaction in equipment of suitable shape, blocks may be formed for applying to building walls, for example, or between studs in frame constructions, or to other surfaces.

Many materials and forms thereof are used conventionally for insulating purposes, but in all of these cases the bulk of the material, when shipped, is high because a gas is shipped in the material. In the present invention, the ingredients can be shipped in a gas-free condition and can be formed up into the desired insulating material by mixing or by adding water thereto after they have arrived, following shipment, at or near the site at which they are to be used. This makes it possible to keep the bulk in shipment low and to thereby reduce shipping costs, both as to the shipping space required and the amount of handling necessary. The material has the additional advantage that the ingredients may be formed into the desired insulating material under field conditions with minimum requirements as to bulk in the prior shipment of the material.

An object of this invention is therefore to provide an insulating material.

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Another object is to provide an insulating material having superior strength, insulating, and weight characteristics, and complete fire resistance.

Another object is a method for making such material.

Another object is an insulating material which may be shipped, prior to installation, in a form having a much lower bulk than the final material.

Another object is an insulating material which has superior characteristics and which can be made in situ.

Further objects will become apparent as the following detailed description proceeds.

A principal embodiment of the invention consists of mixing sodium silicate solution with a small quantity of ethyl silicate and of then rapidly mixing in with this a small quantity of aluminum powder and then perhaps mixing in rapidly a quantity of a crushed mineral product such as "Pearlite" (ground expanded volcanic glass), crushed heated cinders, or milled exploded mica. A short period during which there is only a very slight reaction is followed by a very rapid increase in the rate of the reaction until it is completed, coupled with the evolution of heat and hydrogen gas and the resultant expansion of the material to several times its original volume. The reaction comes to completion suddenly, with the material in nearly its final state, but quite warm. During cooling its strength usually becomes greater, and may be increased further by baking in an oven.

During the period following mixing in which the reaction is dormant, the material may be applied to walls or other surfaces of any kind and the completion of the reaction will then form an insulating layer in the desired location.

Blocks or "batts" of insulating material may be formed by pouring the mixture, during the dormant period of the reaction, into a mold or form, and allowing the reaction to come to completion with or without a cover on the mold or form. The molds or forms may either be individual separate structures or a continuous mold or form may be used. In such a continuous process, the unreacted material is poured on to one end of a moving belt which may also have in addition moving sides or a moving cover or both, and the reaction is allowed to take place on this belt and the finished product is removed at the other end, either having been baked by passage through an oven or in the unbaked state.

The invention is illustrated by the following examples, which are given by way of illustration

only and not in any sense by way of limitation. All quantities or parts shown are by weight.

Example 1

The following ingredients were used:

Sodium silicate solution, 49° Baumé	300
Tetraethyl orthosilicate	1
Aluminum powder	17

The tetraethyl orthosilicate was stirred into the sodium silicate, and the aluminum powder was then added with stirring. The mixture was poured on to a flat surface. After a few minutes the rate of reaction had increased until the mass appeared to be boiling violently; it then subsided suddenly, leaving a gray material having several times the thickness of the original layer. The gray material was a porous, hard, solid substance and was found to be a good heat insulator.

Example 2

The following ingredients were used:

Sodium silicate solution, 49° Baumé	300
Urea-formaldehyde resin with acid salt catalyst and 17% inert filler	10
Aluminum powder	15

The urea-formaldehyde resin was stirred into the sodium silicate thoroughly, and the aluminum powder was then added with stirring. The mixture was poured on to a flat surface immediately after stirring in the aluminum powder. After a few minutes, the rate of reaction increased until the mass appeared to be boiling violently; it then subsided very rapidly, leaving a gray material having several times the thickness of the original layer. This gray material hardened over a period of several hours and then constituted a hard tough material having many small pores of relatively uniform size.

Example 3

The following ingredients were used:

Sodium silicate solution, 52° Baumé	300
Tetraethyl orthosilicate	1
Polyester resinous material, consisting of reacting 1 mole glycerol, 1 mole succinic acid, and ½ mole phthalic anhydride	8
Aluminum powder	15

The tetraethyl orthosilicate was stirred into the sodium silicate, and the resin was then stirred into the mixture. The aluminum powder was then added with stirring, and the mixture was immediately poured on to a flat surface. The reaction proceeded at an increasing rate and terminated suddenly leaving a dark gray material having several times the thickness of the original layer. This material was baked in an oven held at 250° F. for 4 hours. The resulting material was found to be an excellent heat insulator and comprised a tough water resistant porous solid.

Example 4

The following ingredients were used:

Sodium silicate solution, 45° Baumé	300
50% hydrolyzed tetraethyl orthosilicate	2
Ground expanded volcanic glass	15
Aluminum powder	15

The expanded volcanic glass and the tetraethyl orthosilicate were added with stirring to the sodium silicate and the aluminum powder was then added with stirring. The mixture was poured on to a flat surface, and after the reaction had come to completion and subsided, a gray material was left having several times the thickness of the original layer. This material was found to be an excellent thermal insulating material and to have excellent water resistance, strength, toughness. Its interior was more porous than were the exposed surfaces.

Example 5

The following ingredients were used:

Sodium silicate solution, 49° Baumé	300
Tetraethyl orthosilicate	1
Urea-formaldehyde resin with acid salt catalyst and 20% inert filler	1
Rock wool	1
Aluminum powder	15

The first four ingredients were mixed together with stirring, and the aluminum powder was then added and stirred in quickly. The material was applied to a vertical surface with a trowel and the reaction then allowed to increase in rate and come to completion. The material was found to adhere very well to the vertical surface and constituted a layer having several times the thickness of the original layer. It was a porous gray material of considerable strength and toughness and water resistance.

Example 6

The following ingredients were used:

Sodium silicate solution, 49° Baumé	300
50% hydrolyzed tetraethyl orthosilicate	4
Rock wool	2
Polyester resinous material consisting of the product of reacting 1 mole succinic acid, and ½ mole phthalic anhydride, 1 castor oil	1
Aluminum powder	3

The first five ingredients were mixed together with stirring, and the aluminum powder was then stirred into the mixture. The resulting mixture was applied to a vertical surface with a roller device before the reaction rate increased noticeably. The reaction rate was then allowed to increase until the reaction terminated rather suddenly. The resulting substance was a very porous material having several times the thickness of the original layer as applied and was hard, tough and water resistant. Its interior was more porous than were the exposed surfaces.

The above examples indicate the breadth of the applicability of the invention, but by no means do they exhaust the possible applications.

The aluminum powder used in the invention may be of any type, of which the following are given only as examples and are not to be considered as in any way limiting. The aluminum powder may be a plain uncoated powder; it may be an aluminum powder coated with an oil or grease such as for example the stearate-coated aluminum powder sold as "Aluminum Bronze" and used extensively in paints; it may be a plain or coated aluminum powder heated in an oven for some period prior to use; or it may be any of these types of powder suspended in water, alcohol, mineral oil, or any other liquid, as a paste.

The examples have disclosed the use of two types of resins, but this disclosure is not limited since other resins such as any polyester resin (any product of the reaction between a poly carboxy acid and a poly hydric alcohol), any urea formaldehyde resin, or any phenolic resin may

be used successfully. Any thermosetting resin is suitable for the invention which can be incorporated in the mixture in a monomeric or only partially polymerized form prior to carrying out the reaction with the aluminum powder.

The examples have disclosed the use of two extenders or fillers: ground expanded volcanic glass and rock wool. This is not limiting as any other filler suitable for use as a thermal insulating material may also be used, such as, for example: expanded mica, cork particles, shreds, or granules, ground baked cinder material, asbestos, vegetable, wood, or other cellulose blocks, fibers, or mats.

The examples have disclosed the use of one heat treating or baking step, but this is not limiting since it has been found that with various compositions, heat treatment or baking may be carried out with useful results for from 5 minutes to 24 hours at temperatures of from 115° F. to 500° F. Within this range it has been found preferable to conduct heat treatment, when this is desirable, at 150° F. to 350° F. for periods of from 15 minutes to 5 hours.

The examples have shown the use of tetraethyl orthosilicate and partially hydrolized tetraethyl orthosilicate, but these are not to be taken as limiting, since, for example, other silicates such as the condensed silicates, such as methyl and ethyl disilicates and methyl trisilicate and also tetra methyl silicate, common secondary and tertiary glycerol silicates, primary and secondary ethylene glycol silicates, and condensed poly hydric alcohol silicates are also suitable.

The examples have disclosed the use of sodium silicate in the form of a 45° to 52° Baumé solution in water, but this is in no way limiting. Either the meta or the ortho silicate may be used and the concentration may be that of a 40° Baumé solution to a 69° Baumé solution, but 45° to 65° Baumé is preferable.

The useful and preferred proportions of the several ingredients are not limited to the proportions shown in the examples but are as follows:

	Useful	Preferred
	Percent	*Percent*
Sodium silicate solution	80 -98	88 -95
Aluminum powder	2 -10	3 - 7
Alkyl silicate	0.1- 4	0.1- 2
Synthetic resin	0.1- 5	0.2- 1.5
Extender or filler	0.0-10	0.0- 5

The reaction between the free alkali in the sodium silicate solution and the aluminum is thought to occur as follows:

$$6NaOH+2Al \rightarrow 2Na_3AlO_3+3H_2$$

and other reactions are possible which would deposit SiO_2 (silica) (by hydrolysis of the silicate), Al_2SiO_5 and $Al_2O_3 \cdot Na_2O \cdot 6SiO_2$. Thus, although the possible reactions and reaction products are believed to be known, we do not think it wise to restrict ourselves to any one theory as to what these are or how they take place.

It is thus apparent that the invention is broad in scope and is not to be restricted except by the claims in which it is our intention to cover all novelties inherent in the invention as broadly as possible in view of prior art.

Having thus disclosed our invention, we claim:

1. The method of making a porous insulating material which comprises the steps of: mixing 80% to 98% of sodium silicate solution with 0.1% to 4% of an alkyl silicate, rapidly mixing 2% to 10% of aluminum powder thereinto, spreading the resultant mixture on a surface before the reaction reaches the apparent boiling stage, and allowing the reaction to become completed.

2. The method of producing an insulating layer on a surface which comprises the steps of: mixing 80% to 98% of a sodium silicate solution and 0.1% to 4% of an alkyl silicate and 0.1% to 5% of a not fully polymerized thermosetting synthetic resinous material and 2% to 10% of aluminum powder, spreading the resultant mixture on a surface before the reaction reaches the apparent boiling stage, and allowing the reaction to become completed.

3. The compositions of matter comprising the reaction products of aqueous sodium silicate, aluminum powder and alkyl silicate in the following proportions:

	Percent
Sodium silicate solution	80 to 98
Aluminum powder	2 to 10
Alkyl silicate	0.1 to 4

4. The compositions of matter comprising the reaction products of aqueous sodium silicate, aluminum powder and alkyl silicate in the following proportions:

	Percent
Sodium silicate solution	88 to 95
Aluminum powder	3 to 7
Alkyl silicate	0.1 to 4

5. The compositions of matter comprising the reaction products of aqueous sodium silicate, aluminum powder and alkyl silicate in the following proportions:

	Percent
Sodium silicate solution	88 to 95
Aluminum powder	3 to 7
Alkyl silicate	0.1 to 2

6. The compositions of matter comprising the reaction products of aqueous sodium silicate, aluminum powder, alkyl silicate and not fully polymerized thermosetting synthetic resinous material in the following proportions:

	Percent
Sodium silicate solution	80 to 98
Aluminum powder	2 to 10
Alkyl silicate	0.1 to 4
Said resinous material	0.1 to 5

7. The compositions of matter of claim 6 further characterized by said not fully polymerized synthetic resinous material being selected from the group consisting of polyester resins, urea-formaldehyde resins and phenolic resins.

8. The compositions of matter comprising the reaction products of aqueous sodium silicate, aluminum powder, alkyl silicate and not fully polymerized thermosetting synthetic resinous material in the following proportions:

	Percent
Sodium silicate solution	88 to 95
Aluminum powder	3 to 7
Alkyl silicate	0.1 to 4
Said resinous material	0.1 to 5

9. The compositions of matter of claim 8 further characterized by said not fully polymerized synthetic resinous material being selected from the group consisting of polyester resins, urea-formaldehyde resins and phenolic resins.

10. The compositions of matter comprising the reaction products of aqueous sodium silicate, aluminum powder, alkyl silicate and not fully

polymerized thermosetting synthetic resinous material in the following proportions:

	Percent
Sodium silicate solution	88 to 95
Aluminum powder	3 to 7
Alkyl silicate	0.1 to 2
Said resinous material	0.2 to 1.5

11. The compositions of matter of claim 10 further characterized by said not fully polymerized synthetic resinous material being selected from

the group consisting of polyester resins, urea-formaldehyde resins and phenolic resins.

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References Cited in the file of this patent

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Number	Country	Date
571,284	Great Britain	Aug. 17, 1945