METHOD OF MANUFACTURING A HEAT INSULATING/FIRE RETARDANT MATERIAL

Abstract: A method of manufacturing a heat insulating/fire retardant material includes forming a reaction mixture by mixing together colloidal silica, a sodium-containing clay and water and allowing the reaction mixture to react for a sufficient time to form the heat insulating/fire retardant material in the form of a gel or emulsion. The reaction mixture may further include a silicon-containing polymer and a suitable dye. The heat insulating/fire retardant material may be used as a heat barrier or heat shield. An artefact (10) includes a body defining a panel encapsulating the heat insulating/fire retardant material, which artefact can be used as a heat barrier or heat shield.
METHOD OF MANUFACTURING A HEAT INSULATING/FIRE RETARDANT MATERIAL

This invention relates to heat insulation. In particular, it relates to a method of manufacturing a heat insulating/fire retardant material, to a heat insulating/fire retardant material and to an artefact including the heat insulating/fire retardant material.

According to one aspect of the invention, there is provided a method of manufacturing a heat insulating/fire retardant material, which method includes the steps of forming a reaction mixture by mixing together colloidal silica, a sodium-containing clay and water and allowing the reaction mixture to react for a sufficient time to form the heat insulating/fire retardant material.

In this specification, the term "heat insulating material" should be interpreted broadly to include heat dissipating material.

The colloidal silica may be silica dispersed in a silica sol. The colloidal silica may have a specific surface area to concentration ratio of 300/30%. A colloidal silica having a specific surface area to concentration ratio of 200/30% may also be used but with much lower heat resistance. The colloidal silica may be, for example, the colloidal silica sold under the trade name "LEVASIL" by BAYER A.G. (Germany).

The sodium-containing clay may be a sodium-rich clay such as attapulgite, montmorillonite, or the like. The montmorillonite may be
a powder having a pH which is equal to or greater than 10.2. Preferably, the pH is about 10.5. The montmorillonite powder may be the montmorillonite powder sold by GW BASE AND INDUSTRIAL MINERALS (South Africa) under the trade name "ENVIROBENT". The attapulgite may be that sold by G W BASE AND INDUSTRIAL MINERALS (SOUTH AFRICA) under the trade name "ATTAPULGITE".

The water is preferably distilled water or otherwise purified water.

The reaction mixture may be formed using 3-8% m/m colloidal silica, 8-15% m/m sodium-containing clay and 77%-89% m/m water.

Preferably, the reaction mixture is formed using 3-5% m/m colloidal silica, 8-12% m/m sodium-containing clay and 83%-89% m/m water. Even more preferably, the mixture is formed by mixing together 3% m/m colloidal silica, 12% m/m sodium-containing clay and 85% m/m water.

The reaction mixture may have a pH in the range 8-9. Preferably the pH of the reaction mixture is 8.6.

The reaction mixture may be formed by optionally mixing together a silicon-containing polymer, and a suitable dye, in addition to the colloidal silica, the sodium-containing clay and water.

The silicon-containing polymer may be a silicone polymer. Typically, the silicone polymer is in the form of a curable aqueous
emulsion. It may be a 60% non-ionic aqueous silicone emulsion of a reactive dimethyl silicone polymer, for example, the product sold under the trade name "SM 2138(E)" or a 35% anionic aqueous silicone emulsion of a high viscosity silanol fluid made by emulsion polymerisation, for example, the product sold under the trade name "SM 2068(A)(2068E)", both of which are sold by GE BAYER SILICONES GMBH & CO. KG. (Germany). When the silicone emulsion is "SM 2138(E)", the reaction mixture is mixed until a jelly-like slurry is obtained and the heat insulating/fire retardant material obtained is in the form of a gel. When the silicone emulsion is "SM 2068(A)(2068E)", the reaction mixture is mixed until it starts to froth and the heat insulating/fire retardant material obtained is in the form of an emulsion. The reaction mixture used to form the emulsion may contain an emulsifier, for example, 1% m/m of the emulsifier sold as "CRILLET 1" by Croda Chemicals (United Kingdom). By the term "gel" is meant a jelly-like apparently solid colloidal solution. By the term "emulsion" is meant a colloidal suspension of one liquid in another.

Typically, the heat insulating/fire retardant emulsion is lighter than the heat insulating/fire retardant gel.

An alternative curable silicon-containing polymer which can be used is a silicone emulsion containing 35% phenyl fluid such as PH300 or PN200, 3.5% emulsifier such as "RENEX 36" (ICI) and balance water. This silicone emulsion has a higher temperature range than the silicone emulsions mentioned above, however, it is significantly more expensive.
The method may include pre-mixing the aqueous silicone emulsion and the water.

The method may include first heating the water. Typically, the water is pre-heated to a temperature of between 40°C and 60°C. Preferably, the water is heated to a temperature of about 60°C.

The reaction mixture may be formed using 3-8% m/m silicon-containing polymer and 0.1-0.2% m/m dye. Preferably, the mixture is formed using 3-5% m/m silicon-containing polymer and 0.1-0.2% m/m dye. Even more preferably, the mixture is formed using 3% m/m silicon-containing polymer and 0.1% m/m dye.

The dye may be methylene blue.

The invention extends to a heat insulating/fire retardant material whenever manufactured in accordance with the method as hereinbefore described.

The invention extends to a heat insulating/fire retardant material which includes an admixture of colloidal silica, a sodium-containing clay and water. The heat insulating/fire retardant material optionally further includes a silicon-containing polymer and a suitable dye.

The colloidal silica, the sodium-containing clay, the silicon-containing polymer and the dye, and their relative proportions may be as hereinbefore described.
The heat insulating/fire retardant and heat dissipating material of the invention may be used in a range of heat insulating/fire retardant and heat dissipating applications. It may for example be used as a heat barrier or heat shield to protect a surface, typically by smearing the gel to a thickness of 6-10mm over the area to be protected, the quantity of gel required being dependent on the heat being applied and the thickness of the area being protected.

Accordingly, the invention extends to a method of protecting a surface by applying the heat insulating/fire retardant material as hereinbefore described to at least part of the surface.

According to a still further aspect of the invention, there is provided an artefact which includes a body defining a panel encapsulating a heat insulating/fire retardant material as hereinbefore described.

The panel may include a front wall and a rear wall and a number of longitudinal passages or flutes extending end-to-end therebetween, within which the heat insulating/fire retardant material is encapsulated. The panel may be an extrusion of polypropylene or polyethylene.

Typically, the artefact is used as a heat barrier between engine and passenger compartments in vehicles and as a heat barrier in building and construction applications.

The invention will now be described, by way of example, with reference to the following non-limiting Examples and with reference
to the drawing which shows a three-dimensional view of an artefact in accordance with the invention.

EXAMPLE 1

A heat insulating/fire retardant gel according to the invention was manufactured by the method described below. Water (81.6% m/m) at approximately 60°C, montmorillonite powder (12% m/m) having a pH of 10.5, a curable silicone polymer in the form of a curable 60% non-ionic aqueous silicone emulsion (3% m/m), colloidal silica (3% m/m) and methylene blue (0.1% m/m) were combined in the following manner.

The water was first added to a twin-speed mixer and the mixer was operated at a low speed (700rpm). The mixer used was a twin propeller stainless steel shear type mixer. The methylene blue dye was slowly added to the water while the mixer was still running. The silicone emulsion and the colloidal silica were then slowly added to the methylene blue and water mixture. The mixer was operated at the low speed (700rpm) for approximately 15-20 minutes so as to homogenise the mixture. Thereafter, montmorillonite powder was screened through a 60 US mesh screen in order to make the powder fluffy. The montmorillonite powder was then slowly added to the mixture while the mixer was still operating at a low speed (700rpm). The montmorillonite powder was spread over a wide area. Care was taken to ensure that no lumps formed in the slurry. Thereafter, the mixture was switched to a high speed (1400rpm) for approximately 15 seconds and then returned to the low speed (700rpm). A visual check was made for the presence of lumps in the slurry. If lumps were present then the mixer was operated at a high speed (1400rpm) for approximately 15 seconds in order to disintegrate the lumps. The process was repeated viz. a visual check for
lumps was conducted and the mixer was operated at a high speed (1400rpm) for approximately 15 seconds until all lumps present in the slurry disintegrated. Thereafter, the mixer was operated at a high speed (1400rpm) until a smooth jelly-like substance formed. The mixture was then allowed to stand for approximately 1 to 2 days during which a gas evolved. Thereafter the jelly-like slurry was decanted into 50kg buckets and left open to enable any further gas to evolve. The buckets were sealed after approximately 2 days. It was observed that the jelly-like slurry changed into a gel after approximately 3-6 hours.

The montmorillonite was that sold by GW BASE AND INDUSTRIAL MINERALS (South Africa) under the trade name "ENVIROBENT". The curable non-ionic aqueous silicone emulsion was that sold by GE BAYER SILICONES GMBH & CO. KG. (Germany) under the trade name "SM 2138(E)". The colloidal silica was that sold by BAYER A.G. (Germany) under the trade name "300/30% LEVASIL". The water was distilled water or otherwise purified water.

**EXAMPLE 2**

Example 1 was repeated using a different curable silicone polymer viz. the curable silicone polymer sold in the form of a 35% anionic aqueous silicone emulsion under the trade name "SM2068(A)(2068E)" by GE BAYER SILICONES GMBH & CO. KG. (Germany). An emulsifier (1% m/m), sold under the trade name "CRILLET 1" by Croda Chemicals (United Kingdom), was also added to the reaction mixture together with the silicone emulsion. The method used was the same as that used in Example 1 except that once all lumps disintegrated, the mixer was
operated at a high speed (1400rpm) until the mixture started to froth. Instead of being a gel, the final product was an emulsion.

EXAMPLE 3

Referring to the drawing, reference numeral 10 generally indicates an artefact in accordance with the invention. The artefact 10 includes a body which defines a panel 12. The panel 12 has a front wall 14 and a rear wall 16 and a number of passages or flutes 18 extending end-to-end between the walls 16, 18. The panel 12 encapsulates the heat insulating/fire retardant material manufactured in accordance with Example 1 within the passages 18. The ends of the passages 18 are sealed by a sealing method whereby plugs made of a compatible material are ultrasonically welded into each end of the passages. Alternatively, the ends of the passages 18 are sealed by means of mechanically bonding the panel material under pressure and heat. The panel 12 is a polypropylene extrusion and is sold in South Africa under the trade name "CORUPLUS" by AMPAGLAS S.A. (PTY) LTD.

A sample of the panel 12 having a thickness of 8mm, an area of 1m², and a mass of 2400g/m² was subjected to the following test. The panel 12 was exposed to an oxy-acetylene torch flame having a temperature of approximately 3000°C. Photographs of the panel were taken every 30 seconds for evaluation purposes. The flame took 7 minutes to burn through the panel 12 without ignition of the panel 12 occurring. As a comparative test, the panel 12 encapsulating sand instead of the gel of the invention was exposed to the oxy-acetylene torch flame. The result was that the panel 12 ignited after about 10 seconds and the flame burnt through after about 60 seconds. Because
of the rapid ignition that took place, photographs of the panel were taken at 20 second intervals.

In another test, a South African two rand coin was placed on the panel 12 encapsulating the gel of the invention and exposed to the oxy-acetylene torch flame for about 7 minutes. The coin did not melt as the panel 12 functioned as a heat sink effectively dissipating the heat of the coin.

In another test, the panel 12 was exposed to a controlled oven temperature of 200°C for a period of one hour. No deformation, melt-down or ignition took place.

In a further test, the panel 12 was exposed to a naked flame having a temperature of 3000°C for a period of 25 seconds before isolated ignition and flame commenced. The flame was limited to the area of flame impingement without spread.

The panel 12 passed the REI 100, REI 200 and REI 300 tests.

The Applicant believes that it is an advantage of the invention that it provides a heat insulating/fire retardant or fire resistant material in the form of either a gel or an emulsion which can be used in a range of heat resistant applications. For example, the heat insulating/fire retardant material can be

(a) applied to the hands, arms and other parts of a person's body to protect the body from heat, e.g., when working on hot engines or hot exhaust systems;
(b) applied to an accident victim to protect the victim from the heat of a cutting torch used to free the victim from a motor vehicle. In this application the material is liberally smeared to a depth of at least 5mm onto any area of the victim in close proximity to the cutting torch;

(c) sprayed as a fire-extinguisher (by spraying the emulsion under pressure, for example, by using CO₂ gas) directly into a fire;

(d) used as a cable lubricant and protection for cables installed in conduits over long distance, for example, electrical cables or television cables. In this application the material is used as a lubricant when slipping the cable within the conduit. After the slip process has been completed, the ends of the cable are sealed to ensure air tightness;

(e) used to prevent heat damage to paint work, electrical insulation, wood, plastic and glass;

(f) used for protection of metallic or other surfaces in order to prevent warping and distortion during heating; the material should be smeared to a depth of at least 5mm over the whole area to be protected prior to a heating, welding or cutting process. Routine experimentation should be carried out to establish the optimum parameters for the process being used;

(g) used for the very high temperature lubrication of metallic parts or components. Applications include high duty lubrication of very hot, highly stressed bearing surfaces such as encountered within an artillery breach mechanism;

(h) used in jewellery manufacture and repairs where precious stones need protection from welding heat; and

(i) used in oil well drilling mud for the protection and cooling off of drilling bits.
Generally speaking, the heat insulating/fire retardant material can be used for the protection of operatives when working in a hot environment; thus the material can be used in conjunction with specially designed protection gloves when handling hot or very hot materials or for the protection of hands, arms, upper body, etc. when working in a hot environment. Examples of such hot environments occur in the glass, ceramic, metallurgical, chemical, foundry and automotive (exhaust repair) industries; the material can also be used to enhance the performance of existing protective clothing for operating personnel where the risk of exposure to a high temperature environment is considered particularly high, for example, firemen, rescue workers, electricity supplies workers etc.

When working in a hot environment the objective is to protect the hands from the effect of touching hot surfaces and protect the body (including the hands) from the general effects of radiant heat. Operatives working in hot environments typically wear protective clothing consisting of overalls, aprons, leggings, heavy-duty boots, gloves, hoods, balaclava’s etc. This clothing is manufactured using various heat-resisting materials such as Du Pont’s "KEVLAR" (registered trade mark).

By the use of the heat insulating/fire retardant material of the invention, it is possible to significantly enhance the performance of industrial clothing and, thus, improve working conditions and safety. The heat insulating/fire retardant material also allows operatives to work more efficiently in demanding situations with high operating temperatures.
For hand and forearm protection, "KEVLAR" (registered trade mark) is a suitable glove material because of its good mechanical strength and relatively high decomposition temperature (>400°C).

A knitted glove construction with a fairly tight cuff around the wrist and forearm may be used. This facilitates impregnation with the material. A cuff length above the wrist of about 200mm may be used.

For hand and forearm protection, enough material should be placed inside each glove to fully impregnate the palm and finger area of the glove. This can be done by rolling the cuff back and dropping the material into the glove. The operative should then insert the hand into the glove and make sure the palm and finger areas are fully impregnated and protected by massaging the product into place.

The amount of material required to provide adequate protection depends on the specific application and routine experimentation should be used to establish the correct or optimum parameters.

As a rough guide, for palm and finger protection, about 0.5kg of material is required for each glove and the material must be clearly visible on the outside of the palm area of the glove prior to use.

Further, the invention provides an artefact in the form of a panel which panel encapsulates the heat insulating/fire retardant material of the invention which can be used, for example, as a heat barrier between the engine and the passenger compartments in motor vehicles,
to encapsulate a motor vehicle battery within its battery compartment, to provide a heat barrier within the walls of safes in order to neutralise the effect of cutting torches, and as a heat barrier in building and construction applications.
CLAIMS:

1. A method of manufacturing a heat insulating/fire retardant material, characterized in that the method includes the steps of forming a reaction mixture by mixing together colloidal silica, a sodium-containing clay and water and allowing the reaction mixture to react for a sufficient time to form the heat insulating/fire retardant material.

2. A method as claimed in Claim 1, characterized in that the colloidal silica has a specific surface area to concentration ratio of 300/30%.

3. A method as claimed in Claim 1, characterized in that the sodium-containing clay is attapulgite or montmorillonite.

4. A method as claimed in Claim 1, characterized in that the reaction mixture is formed using 3-8% m/m colloidal silica, 8-15% m/m sodium-containing clay and 77%-89% m/m water.

5. A method as claimed in Claim 1, characterized in that the reaction mixture has a pH in the range 8-9.

6. A method as claimed in Claim 1, characterized in that the reaction mixture is formed by mixing together a silicon-containing polymer in addition to the colloidal silica, the sodium-containing clay and water.

7. A method as claimed in Claim 6, characterized in that the reaction mixture is formed using 3-8% m/m silicon-containing polymer.
8. A method as claimed in Claim 1, characterized in that the water is pre-heated to a temperature of between 40°C and 60°C.

9. A method as claimed in Claim 1, characterized in that the heat insulating/fire retardant material is in the form of a gel.

10. A method as claimed in Claim 1, characterized in that the reaction mixture further includes an emulsifier.

11. A method as claimed in Claim 10, characterized in that the heat insulating/fire retardant material is in the form of an emulsion.

12. A heat insulating/fire retardant material whenever manufactured in accordance with the method as claimed in Claim 1.

13. Use of the heat insulating/fire retardant material as claimed in Claim 12 as a heat barrier or heat shield.

14. A method of protecting a surface, the method being characterized in that the heat insulating/fire retardant material as claimed in Claim 12 is applied to at least a part of the surface.

15. An artefact characterized in that it includes a body defining a panel encapsulating a heat insulating/fire retardant material as claimed in Claim 12.

16. An artefact as claimed in Claim 15, characterized in that the panel includes a front wall and a rear wall and a number of longitudinal
passages or flutes extending end-to-end therebetween, within which the heat insulating/fire retardant material is encapsulated.

17. Use of the artefact as claimed in Claim 15 or Claim 16 as a heat barrier or heat shield.
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C04B30/00 F16L59/00 //C04B30/00,14:06,14:10

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)
IPC 7 C04B

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 practical, search terms used)
WPI Data, PAJ, EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Further documents are listed in the continuation of box C.

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Patent family members are listed in annex.

+ Special categories of cited documents:

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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