

(19) World Intellectual Property
Organization
International Bureau



(43) International Publication Date
5 February 2004 (05.02.2004)

PCT

(10) International Publication Number
WO 2004/011391 A1

(51) International Patent Classification⁷: **C04B 41/47**,
C09D 191/06, 191/00, D27K 3/50, C09K 3/10

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(21) International Application Number:
PCT/AU2003/000963

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(22) International Filing Date: 30 July 2003 (30.07.2003)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2002950459 30 July 2002 (30.07.2002) AU
2002950460 30 July 2002 (30.07.2002) AU
2003900472 4 February 2003 (04.02.2003) AU
2003902375 16 May 2003 (16.05.2003) AU

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

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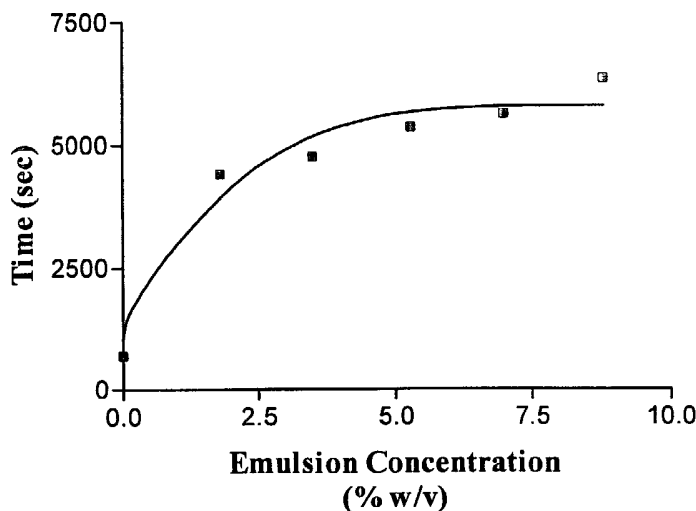
Published:
— with international search report

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: WATERPROOFING CONSTRUCTION MATERIALS WITH HIGH MOLECULAR WEIGHT ESTERS



(57) Abstract: A waterproofing or repelling agent comprising; naturally occurring high molecular weight esters, selected from the group consisting of wool grease, bees wax, carnauba wax, spermaceti wax and the like; a carboxylic acid; and a hydrocarbon solvent. The waterproofing agent may be used on porous building products such as timber and masonry. The waterproofing agent may also be use to repel water from road bases to assist in stabilization of road bases formed from particulate materials. The waterproofing agent may also include preservatives or termite repelling agents to assist in the preservation of timber.

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WATERPROOFING CONSTRUCTION MATERIALS WITH HIGH MOLECULAR WEIGHT ESTERS FIELD OF THE INVENTION

THE INVENTION RELATES TO the use of naturally occurring high
5 molecular weight esters, in particular wool grease, as a waterproofing or
water repelling agent for use on construction materials, including timber,
masonry and road construction materials.

BACKGROUND TO THE INVENTION

Many important building and construction materials are porous, and
10 include cement, concrete, timber, paint, mud bricks and road base
aggregates. Water ingress into these materials can seriously impair
their intended functions. For example, timber will rot, concrete walls will
absorb and transfer massive amounts of water to buildings, and mud
bricks may collapse in heavy rains. Water proofing these types of
15 materials would assist in preserving their inherent properties for much
longer time periods.

The chemistry and technology of wool grease is extensively
covered in the monograph entitled Wool Wax by E. V. Truter (Cleaver-
Hume, London, U.K., 1956). Truter reports the use of wool grease as a
20 rust preventative. It is also known from Truter to use wool grease to
waterproof oilskins to render them impermeable to rainfall, as a vehicle or
plasticizer for paints, in polishing lubricants (e.g. ski wax), as an egg
preservative, a vehicle for insecticide sprays and plant growth hormones,
for printing inks and in carbon paper. However, the water
25 proofing/repelling qualities of wool grease have not been fully utilized in
applications to porous building and construction materials.

Wool grease is relatively stable; it does not go rancid and therefore
is useful for products that must be stored; is tacky and adheres to a range
of surfaces; has no tendency to dry, so it is invaluable where prolonged
30 plasticity is required; and does not crystallize but rather hinders the
crystallization of waxes with which it is blended, making it ideal for a
number of end uses.

Road construction involves the use of road base aggregates and the like to form a foundation for the upper bitumen layer on surfaced roads. Both surfaced and unsurfaced roads are subject to erosion and break down from the effects of water, particularly rain. Rain or water typically ingress into road base layers by seepage through the porous bitumen layer or by capillary action through the shoulders of the road. Over time this ingress of rain leads to a break down in the compacted strength of the road base layers. As the road base foundation starts to break down so does the upper bitumen layer. Providing a water repelling/proofing system for road base materials would assist in preserving their inherent properties for much longer time periods.

Partial stabilization of particulate matter such as road base aggregates and the like has been achieved through the addition of binders, such as calcium lignosulfonate. These lignosulfonates are typically water-soluble and are leached from unsurfaced roads during rain episodes. The leached product is brown in color and very unsightly. Furthermore, upon leaching of the lignosulfonates from road surfaces the binding properties are removed and the road surface becomes subject to the usual problems caused by water.

US patent 4,666,522 describes the use of lignosulfonates as binders for coal, road surfaces, animal feeds and the like. The formulation described in US patent 4,666,522 has 1-50 parts hydrophobe to 100 parts lignosulfonate. A number of hydrophobes are described, such as waxes, plant oils and fats, including animal and plant fatty acids. US patent 4,666,522 only teaches the use of such a composition for binding particulate matter, which is due to the known binding action of lignosulfonates. There is no teaching of waterproofing these materials or the specific use of wool grease.

Prior uses of wool grease for stabilization of soils, road bases or the like have involved emulsions of wool grease requiring the adoption of a highly alkaline medium, using potash, as in US patent 813, 389.

US patent 561,266 uses insoluble calcium or aluminum salts of

fatty acids treated with mineral based oils/waxes; lanolin; naphthalene; stearic acid; palmitic acid or aliphatic alcohols with a flocculent such as calcium hydroxide or aluminum sulfate to provide a water repelling flexible film for use in airfield construction.

5 US patent 575,479 describes the use of ammonium salts of waxes, resins or fatty acids (oleic, stearic, and palmitic) and rendering them insoluble with a multivalent metal salt such as aluminum, iron, copper, and chromium to prevent them from being leached from road bases and the like.

10 US patent 605,057 uses a combination of wool grease, soap of rosin acids and a hydrophobic substance such as bitumen, a heavy metal (litharge) and a filler to create a waterproof plastic compound used in construction. The use of soaps however, does not render reliable
15 stabilization as they break down and leach from the soil, and their effectiveness is short lived. Excess trivalent metal salt is required to react with the soaps applied to the soil, and the trivalent metal salts are usually
soluble and toxic. Any excess is leached to the environment and has detrimental effects on the ecosystems that they enter.

 To date there does not appear to be a simple, long term, cost
20 effective way and environmentally waterproofing or water repelling properties to construction materials and the stabilization of particular materials for use in road construction.

SUMMARY OF THE INVENTION

 In one form, although it need not be the only or indeed the
25 broadest form, the invention resides in a waterproofing or repelling agent comprising;

 naturally-occurring high molecular weight esters, selected from the group consisting of wool grease, bees wax, carnauba wax, spermaceti wax and the like;

30 a carboxylic acid; and
 a hydrocarbon solvent.

 Preferably the hydrocarbon solvent is selected from D-60 (e.g.

Exxsol D-60, Trade Mark), *n*-decane, methoxy propoxy propanol, cineole (eucalyptus oil), d-limonene, turpentine, kerosene, diesel, paraffinic solvents, and the like. More preferably the hydrocarbon solvent is D-60.

5 Preferably the carboxylic acid is selected from straight chain or branched fatty acids, tertiary carboxylic acids produced by a Koch reaction of olefins and carbon monoxide, or naphthenic carboxylic acids. The straight and branched chain fatty acids may be selected from palmitic acid, stearic acid, isopalmitic acid, isostearic acid, palmitoleic acid, oleic acid or myristic acids. The naphthenic carboxylic acids are preferably
10 obtained from crude oil and may include 2-naphthoic acid. More preferably the carboxylic acid is a straight chain fatty acid such as stearic acid.

Preferably the waterproofing or repelling agent comprises:

30-70% (w/v) naturally occurring high molecular weight
15 esters, selected from the group consisting of wool grease, bees wax, carnauba wax, spermaceti wax and the like; and

1-5% (w/v) carboxylic acid; per volume of hydrocarbon solvent.

Preferably the water proofing or repelling agent comprises:

20 30-70% (w/v) wool grease; and

1-5% (w/v) stearic acid; per volume of hydrocarbon solvent.

More preferably the hydrocarbon solvent is D-60.

The waterproofing or repelling agent may be added to an aqueous solution of alkali hydroxide, comprising 1 part alkali hydroxide in 5,000 to
25 10,000 parts water, to form a waterproofing or repelling emulsion.

The alkali hydroxide is selected from sodium or potassium hydroxide. More preferably the alkali hydroxide is sodium hydroxide.

The waterproofing or repelling emulsion may be diluted to comprise
30 5 to 12 % (w/v) naturally occurring high molecular weight esters, selected from the group consisting of wool grease, bees wax, carnauba wax, spermaceti wax and the like. Preferably the naturally occurring high molecular weight ester is wool grease.

The water proofing or repelling agent may further comprise binders, such as lignosulfonate or bitumen; preservatives and/or termite repellents, such as quaternary ammonium compounds, benzalkonium lignosulfonates and benzalkonium lauryl sulfates. Preferably the binder is calcium or sodium lignosulfonate.

In another form the invention provides a method of producing waterproofing or repelling agent comprising naturally occurring high molecular weight esters, selected from the group consisting of wool grease, bees wax, carnauba wax, spermaceti wax and the like; comprising the step of;

forming a solution of naturally occurring high molecular weight esters by mixing said high molecular weight ester and a carboxylic acid with a hydrocarbon solvent and heating to temperatures greater than 50°C.

Preferably the solution of high molecular weight ester is formed by heating between 60°C to 80°C.

The method of producing a waterproofing or repelling agent may further comprise the steps of;

adding a solution of naturally occurring high molecular weight ester to an aqueous solution of alkali hydroxide, comprising 1 part alkali hydroxide in 5,000-25,000 parts of water; and agitating to form a stable W/O/W emulsion, which may also be in equilibrium with an O/W emulsion.

Preferably the waterproofing agent or emulsion further comprises high molecular weight waxy hydrocarbons; binding agents, selected from lignosulfonate and salts thereof, and bitumen, and/or preservatives or termite repellents, selected from quaternary ammonium compounds, quaternary ammonium lignosulfonates or quaternary ammonium lauryl sulfates. More preferably the high molecular weight waxy hydrocarbon is microcrystalline wax.

Preferably the waterproofing agent comprises 4-12% (w/v) of microcrystalline wax per volume of hydrocarbon solvent.

The waterproofing or repelling agent or emulsion may be used to

waterproof porous building materials, such as timber, concrete, and the like; provide water repelling properties to road base to assist in the stabilization; or preserving timber when the emulsion includes one or more preservative and/or termite repellent agents.

5 In yet another form the invention provides a method of waterproofing or water repelling in road construction comprising the steps of;

 applying the said waterproofing agent or emulsion of high molecular weight esters to the surface of particulate material and
10 distributing the waterproofing agent or emulsion throughout an upper layer of the particulate material; and

 compressing the particulate material to form a road base.

 The particulate material may include crusher dust, aggregate, decomposed granite and laterite soils, crushed shale, corona, and the
15 like.

BRIEF DESCRIPTION OF DRAWINGS

To assist in understanding the invention preferred embodiments will now be described with reference to the following figures in which:

FIG 1 is a graph of the time taken for water to ingress into timber
20 treated with different concentrations of wool grease emulsions;

FIG 2 is a graph of the change in area of ingressing water, which is
 inversely proportional to the ingression time, on a wooden
25 surface painted with an inexpensive acrylic water based paint as a function of the concentration of wool grease added to the paint;

FIG 3 is a graph of time taken for water to ingress into treated
 crusher dust as a function of crusher dust particle size;

FIG 4 is a computer generated graph showing the rainfall
30 absorbed by treated crusher dust as a function of waterproofing agent concentration;

FIG 5 is a computer generated graph showing the amount of

rainfall absorbed by treated (1.5% wool grease) and untreated crusher dust as a function of rainfall intensity;

FIG 6 is a graph of water ingress and evaporation of a laterite soil as a function of waterproofing agent concentration; and

5 FIG 7 is a graph of the time taken for a crusher dust mold (60 mm diameter x 5 mm thick) to totally disintegrate under water as a function of waterproofing agent.

DETAILED DESCRIPTION OF THE INVENTION

For ease of description herein after the term waterproofing will be
10 used to include water repelling characteristics of the composition of the invention.

EXAMPLE 1: Preparation of Waterproofing agent

The waterproofing agent is formed using one of the following processes.

15 A. Solution in Hydrocarbon solvent

1. Wool grease and stearic acid are dissolved in a hydrocarbon solvent, preferably D-60, by heating to temperatures greater than 50°C, preferably 60°C, and mixing thoroughly. D-60 is a proprietary paraffinic, dearomatized solvent with a boiling point of 180°C, similar to *n*-decane. It
20 was selected as it is readily available, inexpensive, and environmentally friendly. The stearic acid is required to fortify the wool grease/D-60 mixture. The concentration of wool grease is typically between 30-70% (w/v) of D-60, and the concentration of stearic acid is typically between 1-5% (w/v) of D-60.

25 At higher concentrations of wool grease in D-60 a gel is formed whilst at lower concentrations in D-60 a solution is formed.

2. Wool grease, stearic acid and microcrystalline wax are dissolved in a hydrocarbon solvent, preferably D-60, by heating to temperatures
30 greater than 70°C, preferably 80°C, and mixing thoroughly. The concentration of wool grease is typically between 40-60% (w/v) of D-60, the concentration of stearic acid is typically between 1-5% (w/v) of D-60, and the concentration of microcrystalline wax is typically between 4-12%

(w/v) of D-60.

At higher concentrations of wool grease in D-60 a gel is formed whilst at lower concentrations a solution is formed in D-60. At ambient temperatures microcrystalline wax exists as a gel in the mixture.

5

3. For use as a concrete, cement, mud brick, etc. sealant, the wool grease, stearic acid and microcrystalline wax are dissolved in a hydrocarbon solvent, preferably D-60, by heating to temperatures greater than 70°C, preferably 80°C, and mixing thoroughly. The concentration of wool grease is typically between 8-12% (w/v) of D-60, the concentration of stearic acid is typically between 1-2% (w/v) of D-60, and the concentration of microcrystalline wax is typically between 4-6% (w/v) of D-60.

10

At higher concentrations of wool grease in D-60 a gel is formed whilst at lower concentrations a solution in D-60 is formed. At ambient temperatures microcrystalline wax exists as a gel in the mixture.

15

It will be appreciated by a person skilled in the art that other hydrophobic solvents such as kerosene, diesel, paraffinic solvents, methoxy propoxy propanol, cineole (eucalyptus oil), d-limonene, and turpentine may be used to form a solution of wool grease and stearic acid.

20

B. Emulsion

The solution of A above (except part 3) may be added into water containing sodium hydroxide to form a stable emulsion.

Sodium hydroxide is dissolved in water at a ratio of between 1 part sodium hydroxide to 5,000 – 25,000 parts water. The solution of A above is then added to the water containing sodium hydroxide and agitated, either by pumping, stirring, or air agitation to form an O/W—W/O/W equilibrium mixture containing approximately 5-12% (w/v) of wool grease.

25

The emulsion pH is between 8 and 8.5. Commercial stearic acid (a mixture of stearic and palmitic acids) is added to give a concentration range of 0.15-0.25% (w/v) in the emulsion.

30

The emulsion formed by this process has been found to be stable for several hours without agitation, and stable indefinitely with gentle

agitation.

Several emulsions were formulated. These emulsions appear to have long-term stability and flowed well at 25°C, and they could be readily diluted down to a stable 2% (w/v) wool grease emulsion in water. The optimum formulations and processes have been found to be:

1. 40-60% (w/v) wool grease, 40-60% (v/v) D-60, 50-96% (v/v) water, plus sodium hydroxide to neutralize the free fatty acids naturally present in commercial wool grease. Heat the wool grease and D-60 to 60°C, mix vigorously, and allow to cool. Add 1 part of the mixture to 5-10 parts water containing 1 part sodium hydroxide to 5,000-15,000 parts water. Shake gently to disperse the emulsion.

Preferably 1 part of the mixture to 5 parts water containing sodium hydroxide to 5,000 parts water are used for general water proofing applications, whilst 10 parts water containing 1 part sodium hydroxide to 15,000 parts water is used for road base type applications.

2. 40-60% (w/v) wool grease, 40-60% (v/v) D-60, 1-5% (w/v) stearic acid, 50-96% (v/v) water plus sodium hydroxide to neutralize the free fatty acids added to and present in wool grease. Heat the wool grease, D-60, and stearic acid to 60°C, mix vigorously, and allow to cool. Add 1 part of the mixture to 5-20 parts water containing 1 part sodium hydroxide to 5,000-10,000 parts water. Shake gently to disperse the emulsion.

Preferably 1 part of the mixture to 5 parts water containing sodium hydroxide to 5,000 parts water are used for general water proofing applications, whilst 20 parts water containing 1 part sodium hydroxide to 10,000 parts water is used for roadbase type applications.

3. 40-60% (w/v) wool grease, 40-60% (v/v) D-60, 1-5% (w/v) stearic acid, 4-12% (w/v) microcrystalline wax, 50-92% (v/v) water plus sodium hydroxide to neutralize the free fatty acids added to and present in wool grease. Heat the wool grease, D-60, and stearic acid to 80°C, mix vigorously, and allow to cool. Add 1 part of the mixture to 5 parts water containing 1 part sodium hydroxide to 5,000 parts water. Shake gently to

disperse the emulsion.

The stearic acid facilitates the formation of a stable W/O/W emulsion at low concentrations of wool grease, such as 1-5% (w/v) wool grease. Above this concentration range there are usually enough fatty acids present in commercial wool grease to readily facilitate emulsion formation. However, above about 5% (w/v) wool grease in the emulsion the amount of sodium hydroxide must be 1 part to 5,000 parts water to prevent the W/O/W emulsion from forming a W/O emulsion that is not water miscible. The stearic acid or naturally present fatty acids are converted to an alkali metal salt, preferably a sodium salt, during emulsion formation due to the presence of the sodium hydroxide in the water. It will be appreciated by the person skilled in the art that stearic, palmitic, myristic or similar acids may be used to facilitate the formation of a stable emulsion.

It has been found that only the sodium or potassium salts of the fatty acids have substantial W/O/W and O/W emulsifying power. Sodium bicarbonate was found to be required in high concentration which resulted in a "salting out" effect occurring, converting the W/O/W and O/W emulsions to a W/O emulsion, which is not miscible with water. However, sodium hydroxide was found to be very effective in forming the sodium salt of stearic acid and stabilizing the resulting emulsion.

It has also been found by the inventors that addition of alkali metal salts of the fatty acid, stearic acid, to hydrocarbon solvents such as D-60 are not soluble and therefore these salts are avoided in the dissolution step A, above. Furthermore, the addition of alkali metal salts of stearic acid to water forms a high volume, low weight gel that does not allow for appropriate dispersion of the wool grease through the resultant gel and thus is not effective in waterproofing applications.

The waterproofing agent may further comprise small amounts of microcrystalline wax (or any other similar wax) to form a harder, less greasy surface coating, and as a side benefit it also contributes to waterproofing. This is for both solution and emulsion application. The

microcrystalline waxes are not completely soluble in any known solvent, but ultimately form a gel when heated above their melting point and mixed with any of the solvents listed above and allowed to cool. This gel can then be added to the hot or cool wool grease solution, usually in the range of 1-2 parts microcrystalline wax to 10 parts wool grease. However, in the above examples the microcrystalline wax is added directly to the hot mixture.

Low odor turpentine, mineral turpentine, white spirits and kerosene were found to provide the same stability of W/O/W and O/W emulsions as D-60.

Palmitic acid, stearic acid and commercially available stearic acid (mixture of stearic and palmitic acids) were found to stabilize or fortify the W/O/W and O/W wool grease emulsions to the same level.

Wool grease, D-60, and water were combined in that order at 80°C, allowed to cool, and then the mixture was diluted 10x with water. The proportions of O/W & W/O emulsions were qualitatively assessed, and summarized in Table 1, below.

TABLE 1: Emulsion Formation as a function of Ingredient Concentration

% Wool grease	% D-60	% Water	% O/W Emulsion	% W/O Emulsion
10	80	10	5	95
10	60	30	10	90
10	40	50	50	50
10	20	70	90	10
5	64	31	10	90
2.5	65	32.5	10	90
20	53	27	25	75
5	22	73	75	25
2.5	21	76.5	95	5
18	18	64	40	60
35	35	30	100	0

Whilst the above data indicate that a 100% O/W emulsion can be achieved, in practice this was not achieved during manufacture.

Therefore, 5% W/O emulsion that generally formed in manufacture was transferred from one batch to the next.

Tests over 1-6 months at ambient conditions, 5°C, and 60°C showed no deterioration in the emulsion stability or waterproofing ability of the above emulsions when applied to softwood and hardwood. Emulsion stability of accelerated testing at 54°C for two weeks is generally sufficient to predict a >2 yr stability of the emulsion. Therefore, the emulsion of this invention can be assumed to have a stable lifetime of >2 yr.

Tests over 1-6 months under accelerated UV light conditions when the above emulsions were applied to softwood, showed no deterioration in the waterproofing ability of the coating emulsion.

Tests over 1-6 months of the above wool grease emulsion containing 2% (w/v) benzalkonium chloride, 1.2% (w/v) benzalkonium lignosulfonate, or 2% (w/v) benzalkonium lauryl sulfate was applied to softwood and subjected to outside sun and rain in humid conditions showed no deterioration in waterproofing ability and prevented mould and fungal growth on the timber, while an untreated control showed cracking and splitting and substantial mould growth.

In another test 185 g wool grease supplied from (a) Perth, Western Australia and (b) Warwick, Queensland; 200 ml D-60; and 150 ml water were separately heated to 80°C. The wool grease was dissolved in the D-60, and then the water was added while rapidly mixing the ingredients. After allowing to cool, the mixture was added to water. The Warwick wool grease mixture was miscible with water, indicating that it was a W/O/W emulsion, whereas the Perth wool grease formed a W/O emulsion. When the Perth wool grease was fortified with stearic acid and sodium hydroxide, it readily formed a stable W/O/W emulsion. These results demonstrate the consistency of emulsion formation regardless of the wool grease origin.

Example 2: Applications

A wool grease solution of Example 1 is generally restricted to low volume applications such as hardwood timber, paint, and concrete blocks

due to its gel like consistency and cost restraints resulting from the high volume of D-60 that would be required.

The wool grease emulsion of Example 1 is cost effective for high volume applications such as waterproofing larger areas of timber, particularly softwood timbers.

Computer modeling

Computer-aided modeling eliminates the need for expensive pre-trial field tests and provides an indication as to the waterproofing properties of the emulsion and/or solution of the invention when applied to various materials.

Scientific Principles

Porous building and constructions materials, such as timber, concrete, cement and paint contain porous particles, or form porous layers in the case of paint, through which water can ingress. Water ingresses by capillary action, and the scientific principles behind this are well-known, and are mathematically modeled by the Poiseuille equation:

$$\eta = P \pi r^4 t / 8 V l$$

η = viscosity of a liquid

t = time of flow (i.e. time of water ingress)

(water in this case)

V = volume of liquid

P = pressure applied to the liquid

l = length of capillary

r = radius of the capillary

The equation can be rearranged to the following form:

$$t = 8 \eta V l / P \pi r^4$$

This equation can be modified to test waterproofing ability. The time taken for a certain volume of water to ingress the substance is given by the parameter t . The model is valid for an unsaturated system where the water volume is far less than the total pore volume. All the individual pores can be summed to approximate a single theoretical capillary. As water ingresses this theoretical capillary, its length will shorten until it is zero (full of water), assuming that water does not leave the material. Since the change in theoretical capillary length cannot be measured, the length is kept approximately constant by using a small volume of water

compared to its total capacity. The theoretical capillary volume is measured by immersing a mold of the substance in a container of water and allowing it to absorb all the water that it can. Then the amount absorbed is deduced from the increase in weight. Using a small volume
 5 of water also has the advantage of keeping ingression times reasonably small; therefore 20-500 μL volumes are normally used. The viscosity of water and pressure applied to it remain constant, so all these modifications lead to the following simplified equation:

$$t = a V / r^4 \quad \text{where: } a \text{ is a constant}$$

10 For a given amount of added waterproofing agent, the theoretical capillary radius is a constant, and the Poiseuille equation can be written as:

$$t = bV \quad \text{where: } b \text{ is a constant.}$$

The ingression time against water volume can be readily measured
 15 and the data analyzed by linear regression analysis.

An important measurement is to determine the waterproofing ability of a waterproofing agent that has been added to the material under test. In these measurements the added water volume is kept constant, and the theoretical capillary radius decreases with increasing waterproofing agent
 20 concentration. Therefore the Poiseuille equation gives the following form:

$$t = 1 / (a - b \sqrt{i})^4$$

a & b =constants, but different to those above

i =concentration of water repelling agent

The ingression time of water is measured against various
 25 concentrations of added waterproofing agent and data are analyzed by non-linear regression analysis provided by such computer software as GraphPad PrismTM (GraphPad Software, Inc., USA). Total waterproofing is often required for timber, paint, concrete, etc., and it is interesting to note that it is attained very suddenly. The value of $(a/b)^2$, obtained as part
 30 of the analysis, gives the concentration of waterproofing agent required for total waterproofing. However, evaporation time for a material with high water repelling ability becomes important, and is incorporated in the

equation, thus:

$$t = 1 / [(a - b \sqrt{i})^4 + (1/c)]$$

where a, b, and c are constants;

t = time for the water to evaporate, ingress or both

5 i = concentration of water repelling agent

Again, the term $(a/b)^2$ gives the concentration of waterproofing agent required for total waterproofing, and c is the evaporation time.

Pine wood was painted with patches of emulsions of varying wool grease concentrations applied at 120 mL/ m² and dried for 3 days at 22°C.

10 Water (20 µL) was applied to each patch and allowed to ingress and/or evaporate. This is demonstrated in FIG. 1, and modeling the data gives total waterproofing at a wool grease concentration of 10.0%(w/v), and the time taken for evaporation is 5800 sec.

Since diffusion time is inversely proportional to the area of diffusion, the Poiseuille equation is modified for paint to the following form:

$$A = (a - b \sqrt{i})^4 + c$$

A=area of the diffused water

a, b & c=constants, but different to the above

20 Paint with varying concentrations of wool grease was painted in patches onto plywood with a brush in a thin, even coat. After drying for 24 hrs at 22°C, 500 µl of water was placed onto each patch. The area to which the water diffused was measured after 4 hours, and the initial area subtracted from this. The data are demonstrated in FIG. 2, and modeling gives a wool grease concentration of 10.3% (w/v) for total waterproofing.

25 Since the concrete blocks in this experiment absorbed water like a sponge, it is difficult to compare a treated area with an untreated area (control). Therefore we did not measure actual ingression times for an untreated area because it is so rapid. The minimum criterion that we used was no visible ingression of 2 ml water into a treated area for at least 30 15 minutes, and no total ingression for at least 4 hours. In practice these criteria were easily met when the concrete was first saturated with water and allowed to dry for at least 24 hours (preferably longer) after

application of wool grease/ microcrystalline wax.

For application of the emulsion to road base the above model assumed a constant particle size, which is not necessarily the case with road base aggregate. For example, crusher dust is a mixture of many particle sizes, and the proportion of different sizes can vary. It has been found that the smaller the particles, the greater the surface area that is exposed to the ingressing water, allowing it to ingress more rapidly. Therefore the model is modified to the following form:

$$t = p^2 / (a - b \sqrt{i})^4$$

10

p =average particle diameter

a & b =constants, but different to those above

Variation of water ingression time with crusher dust particle size is demonstrated in FIG. 3, with an r^2 value of 1.

Total waterproofing is attained very suddenly, but it is not always required because most materials used for road base construction require optimum moisture levels for maximum binding strength, and highway bitumen performs better with a degree of porosity. One such material that we have investigated is crusher dust. This is a plentiful and inexpensive material that under normal conditions is useless for road base because the ingression of water rapidly destroys its strength and it falls apart. However, at optimum moisture content, it is quite strong and useful as a road base. By adding wool grease of Example 1 to crusher dust, an optimum amount of moisture is allowed to ingress, but any excess is repelled. The crusher dust maintains its binding and strength, and becomes a plentiful, inexpensive, yet high quality road base.

FIG. 4 demonstrates the amount of rainfall absorbed by crusher dust containing various concentrations of wool grease of Example 1. With just small additions of wool grease, much of the rainfall is repelled, thereby maintaining the moisture content and optimum binding and strength of the crusher dust.

Rain falls at different rates, and this can be incorporated in our model. FIG. 5 demonstrates the difference in rainfall absorbed by

untreated and treated (1.5% w/w wool grease) crusher dust. At low rainfall the water is allowed to enter the crusher dust (maintaining its optimum moisture content), but as the rainfall becomes heavier it is repelled, which protects the crusher dust. This means that an automatic protection process is built into the road base, vital for areas of heavy rainfall such as the tropics.

The Poiseuille equation may be modified to model water ingress into road base materials and also allowing for water evaporation. For some materials that may be used for road bases, water evaporation dominates over water ingress. The equation becomes:

$$t = 1 / [(a - b\sqrt{i})^4 + (1/c)]$$

where a , b , and c are constants;

t = time for the water to evaporate, ingress or both

i = concentration of water repelling agent

The value of $(a/b)^2$ gives the concentration of water repelling agent at which total waterproofing is achieved, and the value of c is the evaporation time at total waterproofing. FIG. 6 demonstrates this for a laterite soil, and total waterproofing is achieved at 0.91% (w/w) of waterproofing agent, and the evaporation time for 20 μ L of water is 4490 sec.

From these computer models, realistic decisions can be made about the concentration of a wool grease emulsion for a specific application. Not only does such a procedure minimize costs, but it also minimizes the amount of extraneous matter added to the original material. Adding too much extraneous matter could significantly compromise the properties of the original material, so this potential hazard is avoided.

Example 2.1: Application to Timber

2.1.1 Hardwood

Painting, spraying, dipping, etc. with waterproofing agent of Example 1 can readily waterproof hardwood and plywood. Microcrystalline wax is added to the solution to give a harder, less greasy surface. Typical concentrations and application rates are:

Wool grease 5% (w/v)

Microcrystalline wax 0.5% (w/v)

Application rate 200 mL/m²

5 The waterproofing agent can also be applied by vacuum or pressure ingression, or as is more common, sequentially by both techniques. However, the product should be diluted about 100x with solvent to obtain more efficient ingression and cover that is not excessive.

2.1.2 Softwood and Hardwood

10 Prepare the wool grease and stearic acid mixture in D-60, add microcrystalline wax in D-60, and add the mixture to water containing 1 in 5,000 parts of sodium hydroxide so that a final concentration of 5-10% wool grease and 0.5-1% microcrystalline wax results. Shake thoroughly and apply at about 10-20 mL/ m² to softwood and 50 mL/ m² to hardwood. Allow to dry at ambient temperature for one or two days. Microcrystalline
15 wax is added to give a harder, less greasy surface.

The emulsion has been found to give superior waterproofing properties compared to the wool grease dissolved in D-60, especially when applied to softwood timbers. It appears initially contradictory that a water-based emulsion is superior to an organic solvent for waterproofing
20 timber. Without wanting to be bound to a theory that the inventors hypothesize, the more favorable results with the water based emulsion may be due to the cellulose fibers being hollow and they and/or their gates collapsing in the presence of an organic solvent, which would result in larger voids within the timber structure.

25 The waterproofing agent can also be applied by vacuum or pressure ingression, or as is more common, sequentially by both techniques. However, the product should be diluted about 20-100x with water to obtain more efficient ingression and cover that is not excessive. However, at such low concentrations a surfactant such as sodium lauryl
30 sulfate needs to be added at about 0.1% to maintain emulsion stability.

There was concern that the use of water-based emulsions in treating timber may lead to water being trapped in the timber that could

lead to rotting. This was evaluated by treating timber with water alone and with 2 to 10% (w/v) wool grease emulsions and allowing the timber to dry at ambient conditions for 2 days followed by heating at 60°C for 5 days. The addition of wool grease emulsions resulted in relative weight gains (absorption) and weight losses (drying) that were identical to the control. These data demonstrate that treating timber with a water-based emulsion does not add water to the timber when it is allowed to dry under normal conditions. Further, it was found that timber at ambient conditions contained about 7-12% water as a normal part of its structure. Drying of treated samples at ambient conditions simply returns the water content to this level.

Example 2.2: Application to Paint

Waterproofing paint and adding an organic UV protector can turn poor quality paint into a paint that can be used for exterior purposes, at little extra cost. Wool grease and stearic acid are dissolved in D-60 at 60°C, the solution is added to water-based paints, and the mixture is shaken thoroughly. The wool grease/ D-60 solution mixes well with the water based paint (that contains its own emulsifiers), and does not separate from the paint on standing. Of course, most paints separate and must be shaken before use, but the addition of wool grease/ D-60 does not hasten this process.

Example 2.3: Application to Concrete

Concrete is so absorbent it must first be saturated with water to prevent a massive ingress of waterproofing agent, which then becomes cost prohibitive. A higher proportion of microcrystalline wax compared to timber is necessary to give a harder surface if the blocks are in constant contact with water, such as in swimming pools or fish ponds. If the concrete is not in constant contact with water, the microcrystalline wax proportion can be lowered to that for timber. Information and data in this section is given for the higher proportion of microcrystalline wax only.

Concrete is saturated with water. The wool grease/ microcrystalline wax mixture is shaken thoroughly, and applied to the

water saturated concrete at a rate of 150 mL/m² by spraying, painting or dipping. A W/O/W emulsion is formed *in situ*, and the concrete is allowed to dry for at least two days.

Example 2.4: Application to Road Base, Crusher Dust or Aggregate

5 Although solution in a hydrocarbon or similar solvent can be used, it is generally preferable to use the emulsion of Example 1 above for high volume applications such as waterproofing road base aggregates, crusher dust, decomposed granite and laterite soils, crushed shale, and corona (crushed coral) for roads, piles of dirt, gravel, coal, ash, etc.

10 Road base materials are often expensive, high quality aggregates. Crusher dust is a plentiful and inexpensive material that under normal conditions is useless for road base construction because the ingress of water rapidly destroys its strength and it falls apart. The application of the emulsions of Example 1 to crusher dust results in water being repelled
15 and optimum moisture content can be maintained within the crusher dust structure. Thus the crusher dust becomes a high-quality, inexpensive, road base material. The invention can use any soil, rock, aggregate, etc. as long as there are some small particles and shrinkage on drying is not a problem.

20 The emulsions of Example 1 are delivered to the road base material by gravity feed or pumping through a spray device normally found on water trucks used for road construction. The road base is then bladed or mixed by other means to obtain an even spread of the product in the road base material.

25 Another means of applying the product to road base material or aggregates is to spray it onto the material on a conveyor belt or similar device, and then pugging or mixing the material by suitable means, after which the final product is delivered to the construction site.

30 In some applications it may be desirable to also add lignosulfonate or other known binders to the emulsion to provide additional binding properties. The lignosulfonate in no way adds to the waterproofing properties of the wool grease. However, the use of the waterproofing

agent has been found to prevent the leaching of any water-soluble binders such as lignosulfonate by rain or ground water when used in road base type applications.

Waterproofing Tests – Crusher Dust and Laterite Soils

5 50 g of crusher dust or laterite soil was mixed with water to give an added moisture concentration of 4% (w/w), complementing the approximately 2% (w/w) moisture already present. A wool grease emulsion was prepared in accordance with Example 1 and added to the moistened crusher dust in varying amounts. The application rate of the
10 wool grease emulsion was around 1 L/m² surface area of crusher dust. However, levels from 0.5-5 L/m² surface area can be used.

 The treated crusher dust was then compacted into molds of 60 mm diameter by 5 mm thick by hitting the mixture with a bolt within the confines of an egg ring. The resulting “biscuits” mimicked a road base
15 and were allowed to dry (22 & 38°C) for appropriate times.

Strength Tests – Crusher Dust

A. Subjected to shock

 50 g of crusher dust was mixed with water or varying amounts of 30% (w/v) calcium lignosulfonate plus water to give an added moisture
20 concentration of 8% (w/w), complementing the approximately 2% (w/w) moisture already in crusher dust. Wool grease emulsions were prepared as in Example 1 and added to the crusher dust, which was compacted into molds 60 mm diameter by 5 mm thick by hitting the mixture with a bolt within the confines of an egg ring. The “biscuits” mimicked a road base
25 and were allowed to dry at room temperature for at least 3 days or at 38°C for at least 24 hrs.

 To test dry strength, a bolt weighing 143 g was dropped onto the sample from a measured height until the sample cracked. Wet strength was tested by allowing 3 ml of water to ingress the sample for exactly 5
30 minutes prior to repeating the above test, with any water not ingressing into the sample being wiped away. Dry strength of crusher dust with and without calcium lignosulfonate was slightly weakened by wool grease. But

more importantly, wet strength of crusher dust was maintained at the dry strength level with the addition of wool grease, whereas without the addition of such the wet strength was almost zero. The wet strength of crusher dust with added calcium lignosulfonate was also maintained at the dry strength, but the ratio of calcium lignosulfonate to wool grease had to be greater than 1.7:1 or the strength decreased.

B. Subjected to water immersion

The molds of section A above were totally submerged in water and the time for complete disintegration recorded. This technique measures how well strength is retained under severe water stress. Fig. 7 demonstrates the enormous difference between treated and untreated crusher dust. Disintegration time without added waterproofing agent was 3 seconds, but with 1% (w/w) added waterproofing agent the time was 582 seconds, a 194-fold increase in resistance to disintegration.

15 Coloring Crusher Dust

When crusher dust is used as a road surface, its pale gray color has a negative aesthetic impact. Red or black iron oxide is readily available and reasonably priced, and is non-toxic and environmentally friendly. To minimize costs, it is pugged (mixed at the mill) with crusher dust to give a 1-2% (w/w) iron oxide in crusher dust mixture, and then placed on top of 95 mm of uncolored crusher dust to a depth of 5 mm. An emulsion of Example 1 is sprayed onto the surface of the crusher dust and allowed to dry in order to seal the surface.

Iron oxide can be purchased in many other colors, such a green, blue and yellow, should other colors be desired.

FIELD TRIALS

The wool grease emulsions of Example 1 above were applied in a number of outside locations to assess its effectiveness in assisting in the stabilization of road bases. In some cases calcium lignosulfonate was added to the emulsion to act as a binder. The binder was used in a 50% (w/v) aqueous solution. In the below example the wool grease emulsion of Example 1 is referred to as "WaterBloc" and the calcium

lignosulfonate is simply referred to as "binder".

Location A

Light traffic access road to a Community Centre. Type 2.5-grade road base was used and treated with WaterBloc. This road has held up very well without any binder, although other trial data suggested that binder may be necessary.

Location	Product Used	Appl'n Rate	Conditions	Results
A	WaterBloc	1 L/ m ²	Light Traffic	Stable and Water Resistant No grading required at this point

Location B

Location B was an all weather sand horse training track. WaterBloc and binder were added to 1800 tonnes of 2.5 grade road base at the quarry, transported to the track, spread over the 1400 metre area, and compacted with a multi-tyre roller. 150mm of sand was put over the top when dry.

Location	Product Used	Appl'n Rate	Conditions	Results
B	WaterBloc & binder	1 L/ m ² of each	Sand Training Track	Stable

Location C

This road was done in two sections using the crusher dust from a quarry.

Section One was a slope, which was treated with WaterBloc only. Minimal erosion after 6 months was observed. This is considered to be a good result due to the slope of the road. This section was subsequently treated with WaterBloc and binder in October 2002. No wear or erosion occurred despite considerable rainfall.

Section Two was treated with WaterBloc and binder and experienced minimal erosion. No maintenance was needed.

Trial Location	Product Used	Appl'n Rate	Conditions	Results
C-Section 1	WaterBloc and binder	1 L/ m ² of each	Light Traffic but steep slope	After 9 months usage this road is still in good condition not requiring grading
C-Section2	WaterBloc-RP and Cooe Binder	1 L/ m ² each	Light Traffic	No wear but some erosion after 6 months Job re-treated with WaterBloc and binder October 2002

Location D

Two Sections of type 2.5 Road Base were treated with WaterBloc and binder. One section was left unsealed, the other has a two coat seal.

- 5 Both sections looked great with no wear, erosion or deviation despite heavy loads from the quarry trucks operating there.

Trial Location	Product Used	Appl'n Rate	Conditions	Results
D - Unsealed	WaterBloc and binder	1 L/ m ² each	Quarry Road Heavy loads	Excellent No visible wear at all
D - Sealed	WaterBloc and binder	1 L/ m ² each	Quarry Road Heavy loads	Excellent No deviation from trucks

Location E

Location E was completed in two unsealed sections.

- 10 The first section was 1 km over a crest in the hill with an S bend and was treated with WaterBloc only. The drought meant very little rain on the road and it dusted up fairly quickly on the S bend. Results showed that WaterBloc alone does not add any shear strength to the roadbase.

- 15 The first section was retreated with WaterBloc and binder mixed into the top 50mm instead of 100mm, thus doubling the concentration of the waterproofing emulsion without extra cost. This section after re-treatment received some heavy rains and has experienced no erosion to

date.

The second section was 0.5 kilometres down the slope of the hill. treated with WaterBloc and binder. This section has performed very well and has not needed to be graded.

Trial Location	Product Used	Appl'n Rate	Conditions	Results
E 1 km section	WaterBloc	1 L/ m ²	Sloped with 'S' bend	Dusted up badly especially on 'S' bend
E 0.5 km section	WaterBloc and binder	1 L/m ² of each	Steep slope	Looks very good; no grading required Small amount of erosion

5

Location F

Laterite material was treated with WaterBloc before applying a 2 coat seal. No erosion or wear was observed after a major wet season.

Location	Product Used	Appl'n Rate	Conditions	Results
F	WaterBloc	1 L/ m ²	Heavy rainfall Sealed Roads	Excellent, with no erosion or potholes

10

Location G

A section of trial was treated with WaterBloc and binder. The trial was located in a position where trucks turn sharply. The treated surface was found to dust up. Additional binder would be required to withstand such shear forces and wear. It is suggested that in a similar location the binder volume be increased 3 times.

15

Trial Location	Product Used	Appl'n Rate	Conditions	Results
G	WaterBloc and binder	1 L/ m ² of each	Heavy trucks turning area	Dusted up on bend

Location H

Heavy transport yard with a sandy laterite type soil. The yard was

treated with WaterBloc and binder in July 2002 but was unsatisfactory. We were not sure why the WaterBloc and binder was not suitable in this application. The area was retreated with WaterBloc-BW, a WaterBloc emulsion containing 3 parts anionic bitumen emulsion to each part of WaterBloc emulsion, in November 2002. It appears that the presence of the bitumen emulsion assisted with binding of the road base. This worked very well and is holding up very well under the weight of the equipment

Trial Location	Product Used	Appln Rate	Conditions	Results
H	WaterBloc	1 L/ m ²	Transport yard heavy loads	Not satisfactory
H	WaterBloc-BW	1 L/ m ²	Transport yard heavy loads	Working very well

10 Location I

Laterite type soil under heavy transport. This trial was done with WaterBloc and binder. Total success, no wear or erosion at this point.

Trial Location	Product Used	Appln Rate	Conditions	Results
I	WaterBloc and binder	1 L/ m ² each	Heavy machinery yard	Excellent

Location J

15 Road in centre of grounds at Location J. Treated with WaterBloc and binder in 2.3 road base material. Looks great, but it has had very little traffic.

Trial Location	Product Used	Appln Rate	Conditions	Results
J	WaterBloc and binder	1 L/ m ² each	Light Traffic Farming Expo road	Excellent

WATER PROOFING AGENTS' PROPERTIES

20 The waterproofing agent of the invention when applied as a surface

coat to timber or similar materials has been found to have maximum surface ingress; provide a non-greasy surface and high waterproofing ability when compared to the current commercially available solvent based wool grease formulations.

5 Surface ingress has been found to be on average 3mm for softwood. The treated surface is non-greasy in nature when the wool grease concentration in the emulsion is at or below 8% (w/v). The waterproofing ability of the wool grease emulsion is high at concentrations above 2% (w/v) wool grease.

10 Ideally, in order to maximize the favorable properties of the waterproofing agent the optimum application rate of a 5-8% (w/v) wool grease emulsion is 1 L/ m² for softwood and 1 L/ 20 m² for hardwood.

ANTI-TERMITE ACTIVITY

15 The use of wool grease emulsions of the invention has been found to protect timber against termite infestation.

 Termites infest timber that is infected with an active microbial growth, such as dry rot. Microbial colonies containing fungal spores and/or bacteria develop, or become active, when timber is moist or wet for an extended period of time. Termites have been known to bore through plastic conduits, lead sheeting, PVC coatings on wiring and healthy or green timber to reach microbe infected timber. Whilst the termites may make their way through PVC tubing, coatings and the like, it is believed that they do not ingest these materials.

25 Attempts to protect timber from moisture with paints and similar coatings have been found to be generally ineffective in deterring termites. The paint chips and flakes over time, allowing moisture to ingress into the timber and in turn encourages or activates microbial infection leading to termite infestation.

30 It has been found that the application of wool grease emulsions to timber is surprisingly effective as termite prevention or deterrent. Termite prevention is preferably attained through the application of a wool grease emulsion containing greater than 7.5% (w/v) wool grease. More suitably

the wool grease emulsion contains greater than 10% (w/v) wool grease.

The wool grease emulsion may be painted or sprayed onto the timber. Alternatively, the timber may be pressure treated with a wool grease emulsion.

5 **Example 3**

The waterproofing or repelling agent of the invention has been found to provide anti-termite activity to timber, either on its own or in combination with other anti-termite agents.

10 Pieces of pine wood (40x20x10 mm) were painted with the O/W emulsion, of Example 1, containing 10%, 5%, 2.5%, and 1.2% (w/v) wool grease at the rate of 1 L/ 6 m² and allowed to dry overnight. Control samples of timber were painted with water and treated in the same manner.

15 Five (5) pieces of timber treated with each concentration of O/W wool grease emulsion and 5 pieces of control timber were placed into plastic containers. Cardboard sheets separated the timber pieces from each other. A 1 m long hole that was about 10mm wide was drilled into termite nests of the *Coptotermes* sp. in a natural environment, and rubber or plastic tubes linked the plastic containers, containing the treated and
20 control timber samples to the inside of the termite nest. All connections were tight to prevent predators entering the nest and moisture escaping.

After two weeks the pieces of wood were removed from the container. The containers had large numbers of termites, mostly workers. The wood samples were very moist and covered in faecal matter. All
25 control timber pieces had been eaten extensively. The timber pieces treated with 1.2%, 2.5%, and 5% (w/v) O/W wool grease emulsion had been eaten about half as much as the control timber. The timber treated with a 10% (w/v) wool grease emulsion had not been eaten at all and had far more faecal cover than the other timber samples. These results were
30 consistent with numerous previous qualitative tests where treated and untreated timber had been placed in close proximity to termite nests.

Whilst the timber sample treated with 10% (w/v) wool grease was

not eaten there were indications that termites had sampled the timber as one corner of the timber sample was covered with mud, an indication to other termites that the timber was unsuitable for digestion.

Laboratory tests demonstrated that total waterproofing of the timber was achieved at about 2% (w/v) wool grease in the emulsion painted onto the timber at a rate of 1 L/ 6 m². The termite data above indicate that an excess of wool grease rather than just enough for waterproofing is desirable to render the timber termite resistant .

Ideally timber is treated with an emulsion comprising 12% (w/v) of wool grease for effective waterproofing and termite prevention.

Additional experiments have been conducted where timber pellets were treated with an aqueous solution of wool grease. The treated pellets were subject to water blasting treatment and remained waterproof, thus indicating that timber once treated with a wool grease emulsion will remain resistant to termite infestations even in harsh conditions.

It is hypothesized that the waterproofing properties of the wool grease emulsion prevent the formation of bacterial and/or fungal infections in treated timber and as a result render the treated timber somewhat, although not totally, unappealing to termites. It is likely that the digestive flora balance of a termite is affected or interfered with by the presence or digestion of the timber treated with a wool grease emulsion. An imbalance in a termite's digestive bacterial flora may lead to death, thus the termite endeavors to maintain the symbiotic relationship with its digestive flora by avoiding timber treated with wool grease.

If resistance to termite infestation is a function of taste or smell the wool grease emulsion may further comprise lignosulfonates, bitumen and the like.

In addition the wool grease emulsion may further comprise antimicrobial treatments such as quaternary ammonium compounds or fungicides, to further control the development of fungal infections in timber, such as illustrated in the following example.

Example 4

Wood blocks of *Pinus radiata* (50 x 40 x 40 mm, with the grain in the 50 mm direction) were treated with quaternary ammonium lignosulfonates with and without wool grease emulsion treatment, and solvent controls of both ethanol and water. The timber samples treated with a range of formulations were placed within PVC tube containers, and inserted into aboveground mound colonies of *Coptotermes* species. Table 2 summarizes the location of treated timber within the termite mound and the treatment used on the respective sample.

TABLE 2. Bait containers installed for 24 days into four *C. lacteus* mound colonies.

ARRANGEMENT OF TUBES IN MOUNDS.	BASE	2	3	4	TOP
Mound # 1 Tube container # 1	A	I	G	H	F
Mound # 1 Tube container # 2	C	G	D	A	E
Mound # 2 Tube container # 1	A	B	D	C	E
Mound # 2 Tube container # 2	D	A	C	J	E
Mound # 3	G	D	A	E	C
Mound # 4 Tube container # 1	B	F	F	B	G & D
150 m underground against dead log					

Key: Base = Wood block nearest cap with wire-vent hole.

Top = Wood block nearest poly pipe into tube container from termite mound.

A = Solvent control (water).

B = Solvent control (ethanol).

C = Wool grease emulsion (1 coat).

D = Wool grease emulsion (2 coats).

E = 50% Wool grease emulsion diluted in water.

F = 0.4% Quaternary ammonium lignosulfonate.

G = 0.8% Quaternary ammonium lignosulfonate

H = 0.2% Quaternary ammonium lignosulfonate + wool grease emulsion.

I = 0.4% Quaternary ammonium lignosulfonate + wool grease emulsion.

J = 0.8% Quaternary ammonium lignosulfonate + wool grease emulsion.

After 24 days located within the termite mounds the tube containers were withdrawn from the active mound colonies, and the extent of termite attack and damage on each wood specimen assessed qualitatively (i.e., visually); the results are summarised in Table 3 below.

5 Table 3 outlines the assessment criteria used in these qualitative assessments. The scale offers an indication of the palatability of treated timber specimens in test, and is not a definitive quantitative assessment scale. Timber treatments refer to any materials added onto or into untreated wood specimens. This includes solvent controls, such as
10 ethanol or water, which have no other additive or toxin. Controls were soaked for 15 min consecutively in ethanol and water. All samples were dried at ambient conditions for 3 days.

TABLE 3. Termite attack and damage rating.

Termite attack & damage scale	Descriptive assessment
Nil	No termites present
TV	Termite visit
TN	Termite nibble
SA	Slight attack - less than 10% attacked
MA	Moderate attack - less than 50% attacked
SV	Severe attack – More than 50% attacked

15

The importance of including 'termite visit' and 'termite nibble' is that when timber specimens are placed in containers and the termites from an active mound colony are allowed to forage freely into the container, termites always 'check out' all the specimens prior to feeding on any
20 particular specimen(s). Another feature of 'termite visit' is that the termites will completely cover the wood specimen that appears unpalatable to them, with a layer of soil and faecal material. Basically, isolating a 'foreign' material or object from their normal foraging and feeding situations.

TABLE 4. Termite damage ratings to the wood specimens from Table 1 above after 24 days in *C. lacteus* mound colonies.

ARRANGEMENT OF TUBES IN MOUNDS	Base	2	3	4	Top
Mound # 1 Tube container # 1	A	I	G	H	F
Termite rating	SV	SA	SA	SA	SA
Mound # 1 Tube container # 2	C	G	D	A	E
Termite rating	SA	TN	SA	SV	SA
Mound # 2 Tube container # 1	A	B	D	C	E
Termite rating	SV	SV	TV	TN	TN
Mound # 2 Tube container # 2	D	A	C	J	E
Termite rating	TV	SV	SA	TV	SA
Mound # 3	G	D	A	E	C
Termite rating	SA	SA	SV	SA	SA
Mound # 4 Tube container # 1 150 m underground against dead log	B	F	F	B	G & D
Termite rating	SV	TV	SA	SV	TN & TV

Key: Base = Wood block nearest cap with wire-vent hole.

Top = Wood block nearest poly pipe into tube container from termite mound.

5

Example 5

In a manner similar to Example 4 above the experiments were conducted with concentrations of benzalkonium lauryl sulfate in ethanol at 0.5, 1.0 and 1.5% respectively. One coat of wool grease emulsion was applied to the wood blocks treated with the anti-microbial solutions, in accordance with the application schedule of Table 5. Controls were soaked for 15 min consecutively in ethanol and water. All samples were dried at ambient conditions for 3 days.

15

TABLE 5. Showing the treatments applied to the wood specimens of *P. radiate* and their identification codes.

Code	Treatment
A	Solvent control (water and ethanol)
B	Wool grease emulsion (single coat applied to wood block)
C	Wool grease emulsion (two coats applied to wood block)
D	0.5% Benzalkonium lauryl sulfate solution
E	1.0% Benzalkonium lauryl sulfate solution
F	1.5% Benzalkonium lauryl sulfate solution
G	0.5% Benzalkonium lauryl sulfate solution + wool grease emulsion (1 coat)
H	1.0% Benzalkonium lauryl sulfate solution + wool grease emulsion (1 coat)
I	1.5% Benzalkonium lauryl sulfate solution + wool grease emulsion (1 coat)

Randomized treated wood blocks were placed within each of five
 5 tube containers (300 x 90 mm), and inserted into aboveground mound
 colonies of *C. lacteus*. Termites were allowed to forage into the tube
 containers for 28 days. After this period, all the tube containers were
 withdrawn from the mound colonies and the feeding response of the
 natural populations of *C. lacteus* towards the various treated wood blocks
 10 was measured using a simple objective rating system of attack and
 damage, as outlined in Table 3 above.

The visual assessment of termites feeding response on the treated
 wood blocks over the 28 day test period is shown in Table 6. While this
 data is qualitative it is important to record termite behavioral responses
 15 when they are presented with a potential active ingredient.

TABLE 6. Termite feeding response to treated wood blocks in the *C. lacteus* aboveground mound colonies in the field experiment as measured by visual ratings after a 28 day test period.

Arrangement of tubes in mounds	Base	2	3	4	5	Top
Mound # 1 Tube # 3	I	H	A	C	G	E
Termite visual rating	TV	TV	SV	MA	TV	MA
Mound # 2 Tube # 4	G	I	D	B	A	H
Termite visual rating	SA	TV	SV	MA	SV	TN
Mound # 3 Tube # 1	A	I	C	G	I	H
Termite visual rating	SC	TV	TV	TV	TV	MA
Mound # 3 Tube # 5	C	G	B	A	-	-
Termite visual rating	TN	TN	MA	SV	-	-
Mound # 4 Tube # 2	H	A	C	G	F	I
Termite visual rating	TN	SV	TV	TV	MA	TV

Key: Base = Wood block nearest cap with wire-vent hole.

5 Top = Wood block nearest poly pipe into tube container from termite mound.

Table 7 summarizes the results. All the solvent control blocks were severely damaged at every mound. Blocks treated with two coats of wool grease emulsion were less palatable to the termites than to those treated with a single coat. Though the specimens were limited, termites were not completely deterred by blocks treated with the anti-microbial treatments alone at the stated concentrations.

Blocks treated with the highest loadings of anti-microbial and one coat of wool grease emulsion showed mostly termite visits and only a single termite nibble. The anomalous responses of the termites to the 0.5 and 1.0% anti-microbial blocks are considered to be due to differences in ingress rates. The wood blocks treated with a 1.5% solution of anti-microbial and wool grease emulsion showed the lowest feeding response after the 28 day test period.

TABLE 7. Summary of results

Code	Treatments	Number	Result
A	Solvent controls (water/ethanol)	5	5 x SV
B	Wool grease emulsion (12%) 1 coat	2	2 x MA
C	Wool grease emulsion (12%) 2 coats	2	2 x TV
D	0.5% Benzalkonium lauryl sulfate solution	1	1 x SV
E	1.0% Benzalkonium lauryl sulfate solution	1	1 x MA
F	1.5% Benzalkonium lauryl sulfate solution	1	1 x MA
G	0.5% Benzalkonium lauryl sulfate + wool grease emulsion (1 coat)	5	3 x TV 1 x TN 1 x SA
H	1.0% Benzalkonium lauryl sulfate + wool grease emulsion (1 coat)	5	1 x TV 2 x TN 2 x MA
I	1.5% Benzalkonium lauryl sulfate + wool grease emulsion (1 coat)	5	5 x TV

Key: TV = Termite visit ; TN = Termite nibble; SA = Slight attack; MA = Moderate attack; SV = Severe attack.

5 The above results indicate that the combination of the wool grease emulsion of the invention and the highest loading of the quaternary ammonium compound (1.5% w/w) completely deterred termite feeding for the period of the test.

10 **EXAMPLE 6**

 The process of Example 5 was repeated using benzalkonium stearate (BSt), benzalkonium dodecyl (lauryl) sulfate (BDS) and benzalkonium lignosulfonate (BLS), but where they were used in combination with wool grease emulsions they were mixed with the emulsion rather than applied separately. Results are summarized below in
15 Table 8.

TABLE 8.

Treatment	Result
Control	4 x SV
6% BDS	TN
1.8% BLS	TN
1.2% BDS + wool grease emulsion	TV
2% BDS + wool grease emulsion	TV
6% BDS + wool grease emulsion	TV
10% BDS + wool grease emulsion	TV
0.6% BLS + wool grease emulsion	4 x TV
5.4% BSt + wool grease emulsion	3 x TV/ 1 x TN
0.4% BSt + wool grease emulsion	MA
0.7% BSt + wool grease emulsion	MA
1.1% BSt + wool grease emulsion	SV

Wool grease emulsion = is a W/O/W emulsion containing wool grease.

5 These data indicate that the quaternary ammonium compounds by themselves are moderate anti-termite agents, and that high molecular weight esters such as wool grease, by themselves, are moderate anti-termite agents. However, benzalkonium lignosulfonate and benzalkonium lauryl sulfate combined with high molecular weight esters, such as wool grease, are powerful anti-termite agents. In contrast, benzalkonium
10 stearate mixed with high molecular weight esters, such as wool grease, is only a moderate anti-termite agent.

15 In yet another variation of the invention it has been found that the O/W, W/O and O/W/O emulsions comprising wool grease and bitumen are also useful for preserving timber against rot or fungi, termites, and against mechanical stress due to contraction and expansion. Mechanical stresses due to contraction and expansion appear to be prevented because water is prevented from ingressing into the treated timber and then evaporating.

An O/W wool grease-bitumen emulsion may comprise 2-10% (w/v)

wool grease and 1-20% (w/v) bitumen. O/W emulsions may also contain carboxylic acid, such as stearic acid, and microcrystalline waxes, such as paraffin. A W/O wool grease-bitumen emulsion may comprise: 1-5% (w/v) wool grease; 5-15% (w/v) bitumen; 0-10% (w/v) microcrystalline wax; 60-90% (v/v) D-60; and 2-20% (v/v) water. Whilst O/W/O wool grease-bitumen emulsion may comprise: 0.2-0.5% (w/v) wool grease; 0.2-0.5% (w/v) bitumen; 0-1% (w/v) microcrystalline wax; 7-40% (v/v) D-60; and 55-92% (v/v) water.

The O/W emulsion is applied by diluting 3-5 times with water, and sprayed or painted onto timber at about 1 L/ 6 m². The W/O emulsion is applied by diluting 2-5 times with D-60, and sprayed or painted onto timber at about 1 L/ 6 m². The O/W/O emulsion is applied undiluted at an application rate of about 1 L/ 2 m². Drying times are 1-2 hrs.

Studies in which timber was painted with the emulsions and left in damp conditions and within termite nests showed no rotting and no termite attack, respectively, within two months. Untreated samples showed extensive rotting and termite attack, respectively, within two weeks.

Timber samples, both softwood and hardwood treated with the wool grease emulsion of the invention, when exposed to ambient conditions, 5°C and 60°C over a period of 1, 2 & 6 months showed no deterioration.

Softwood samples treated with the wool grease emulsion of the invention under accelerated UV light conditions showed no deterioration in the waterproofing properties of the treated surface over a period of 1, 2 & 6 months.

Softwood timber pieces were allowed to soak overnight in solutions of wool grease emulsions described above but containing 2% (w/v) benzalkonium lignosulfonate, 2% (w/v) benzalkonium lauryl sulfate, and 2% (w/v) benzalkonium 5-amino-2-naphthalene sulfonate, and allowed to dry for 3 days. Together with an untreated control timber sample the treated softwood timber samples were placed on concrete blocks covered in mould and exposed to the weather. In this accelerated mould test, after

4 weeks the control was almost totally covered with black mould, but the treated samples were totally free of mould.

Benzalkonium lignosulfonate can be added to the wool grease emulsions up to a concentration of 1.2% (w/v) without compromising emulsion stability. At this concentration, together with 7% (w/v) of the high
5 molecular weight esters, the mixture becomes an effective antitermite/antifungal product, and prevents mould and algal growth.

Benzalkonium lauryl sulfate is an antimicrobial, water resistant, wax-like compound that forms an emulsion and can be added at higher
10 concentrations (up to about 10% (w/v)) to wool grease emulsions used for timber treatment. The benzalkonium lauryl sulfate enhances the water repelling properties of the high molecular weight esters contained in the wool grease emulsions, and at 2% (w/v) provides antimicrobial properties, and the mixture strongly repels termites and prevents rot and mould from
15 attacking timber.

It will be appreciated by the person skilled in the art that the solution and emulsion of naturally occurring high molecular weight esters may also be used to other porous building and construction materials not tested above.

20 Throughout the specification the aim has been to describe the preferred embodiments of the invention without limiting the invention to any one embodiment or specific collection of features.

Throughout this specification, unless the context requires otherwise, the word "comprises", and variations such as "comprise" or
25 "comprising", will be understood to imply the inclusion of a stated integer or group of integers or steps but not to the exclusion of any other integer or group of integers.

CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A waterproofing or repelling agent comprising;
naturally occurring high molecular weight esters, selected
from the group consisting of wool grease, bees wax, carnauba wax,
5 spermaceti wax;
a carboxylic acid; and
a hydrocarbon solvent.
2. The waterproofing or repelling agent of claim 1 wherein, the
hydrocarbon solvent is selected from D-60, *n*-decane, methoxy
10 propoxy propanol, cineole (eucalyptus oil), d-limonene, turpentine,
kerosene, diesel, paraffinic solvents.
3. The waterproofing or repelling agent of claim 1 wherein, the
hydrocarbon solvent is D-60.
4. The waterproofing or repelling agent of claim 1 wherein, the
15 carboxylic acid is selected from straight chain or branched fatty
acids, selected from palmitic acid, stearic acid, isopalmitic acid,
isostearic acid, palmitoleic acid, oleic acid or myristic acids; tertiary
carboxylic acids produced by a Koch reaction of olefins and carbon
monoxide; or naphthenic carboxylic acids.
- 20 5. The waterproofing or repelling agent of claim 4 wherein, the
carboxylic acid is a straight chain fatty acid such as stearic acid.
6. The waterproofing or repelling agent of claim 1 wherein the
waterproofing or repelling agent comprises:
30-70% (w/v) naturally occurring high molecular weight
25 esters, selected from the group consisting of wool grease, bees
wax, carnauba wax, spermaceti wax; and
1-5% (w/v) carboxylic acid; per volume of hydrocarbon
solvent.
7. The waterproofing or repelling agent of claim 1 wherein, the water
30 proofing or repelling agent comprises:
30-70% (w/v) wool grease; and
1-5% (w/v) stearic acid; per volume of hydrocarbon solvent.

8. The waterproofing or repelling agent of claim 1 wherein, the water proofing or repelling agent further comprises binders, preservatives and/or termite repelling agents.
- 5 9. The waterproofing or repelling agent of claim 8 wherein, the binder is sodium or calcium lignosulfonate.
- 10 10. The waterproofing or repelling agent of claim 8 wherein, the preservatives or termite repelling agents are selected from one or more of the following; quaternary ammonium compounds, quaternary ammonium lignosulphonates or quaternary ammonium lauryl sulfate.
- 11 11. A use of the waterproofing or repelling agent of claim 1 in waterproofing porous building materials.
- 12 12. The use of claim 11 wherein, the waterproofing or repelling agent is applied to porous building materials by painting, spraying, dipping,
15 vacuum ingress ion and/or pressure ingress ion of the waterproofing or repelling agent onto the building materials.
- 13 13. The use of the waterproofing or repelling agent of claim 1 in providing water repelling properties to road base to assist in the stabilization thereof.
- 20 14. The use of the waterproofing or repelling agent of claim 1 in the preservation of timber.
- 15 15. The use of claim 13 wherein, the waterproofing or repelling agent further includes one or more preservatives or termite repelling agents.
- 25 16. A method of waterproofing or water repelling in road construction comprising the steps of;
applying the waterproofing or repelling agent of claim 1 of high molecular weight esters to the surface of particulate material and mixing the solution or emulsion throughout the upper layer of the particulate material; and
30 compressing the particulate material to form a road base.
17. The method of claim 16 wherein, the particulate material is

selected from crusher dust, aggregate, decomposed granite and laterite soils, crushed shale, corona.

18. A waterproofing or repelling emulsion formed by adding the waterproofing or repelling agent of claim 1 to an aqueous solution of alkali hydroxide, comprising 1 part alkali hydroxide in 5,000 to 10,000 parts water.
19. The waterproofing or repelling emulsion of claim 18 wherein, the alkali hydroxide is selected from sodium or potassium hydroxide.
20. The waterproofing or repelling emulsion of claim 18 wherein, the waterproofing or repelling emulsion is diluted to comprise 5 to 12 % (w/v) naturally occurring high molecular weight esters.
21. The waterproofing or repelling emulsion of claim 18 wherein, the naturally occurring high molecular weight ester is wool grease.
22. The use of the waterproofing or repelling emulsion of claim 18 in waterproofing porous building materials.
23. The use of claim 22 wherein, the waterproofing or repelling emulsion is applied to porous building materials by painting, spraying, dipping, vacuum ingression and/or pressure ingression of the waterproofing or repelling agent onto the building materials
24. The use of the waterproofing or repelling emulsion of claim 18 in providing water repelling properties to road base to assist in the stabilization thereof.
25. The use of the waterproofing or repelling emulsion of claim 18 in the preservation of timber.
26. The use of claim 25 wherein, the waterproofing or repelling emulsion further includes one or more preservatives or termite repelling agents.
27. A method of waterproofing or water repelling in road construction comprising the steps of;
- applying the waterproofing or repelling emulsion of claim 18 of high molecular weight esters to the surface of particulate material and mixing the solution or emulsion throughout the upper

layer of the particulate material; and

compressing the particulate material to form a road base.

28. A method of producing waterproofing or repelling agent of claim 1, comprising the step of:

5 forming a solution of naturally occurring high molecular weight esters by mixing said high molecular weight ester and a carboxylic acid with a hydrocarbon solvent and heating to temperatures of greater than 50°C.

29. The method of claim 28 wherein, the solution of high molecular weight ester is formed by heating between 60°C to 80°C.

30. The method of claim 28 further comprising the steps of:
 adding a solution of naturally occurring high molecular weight ester to an aqueous solution of alkali hydroxide, comprising 1 part alkali hydroxide in 5,000-25,000 parts of water; and agitating to form a stable W/O/W emulsion, which may also be in equilibrium with an O/W emulsion.

31. The method of claim 28 wherein, the waterproofing agent comprises 4-12% (w/v) of microcrystalline wax per volume of hydrocarbon solvent.

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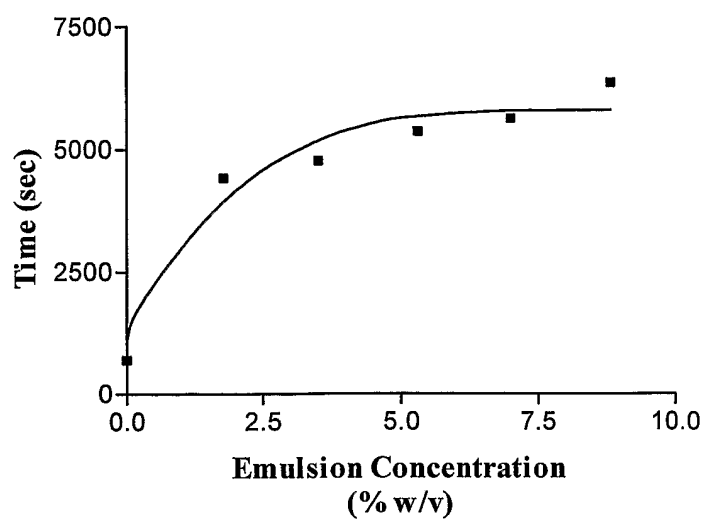


FIG 1

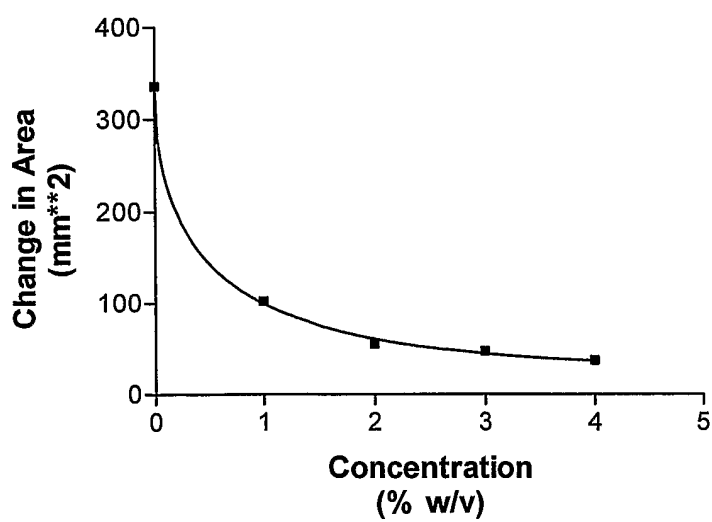


FIG 2

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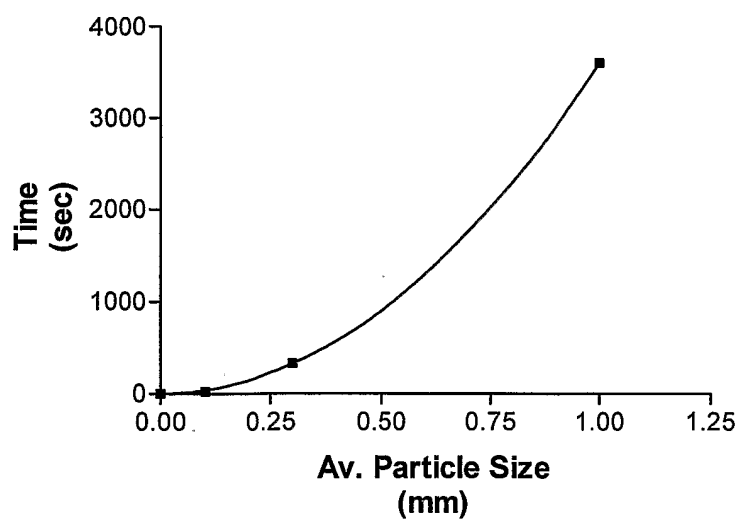


FIG. 3

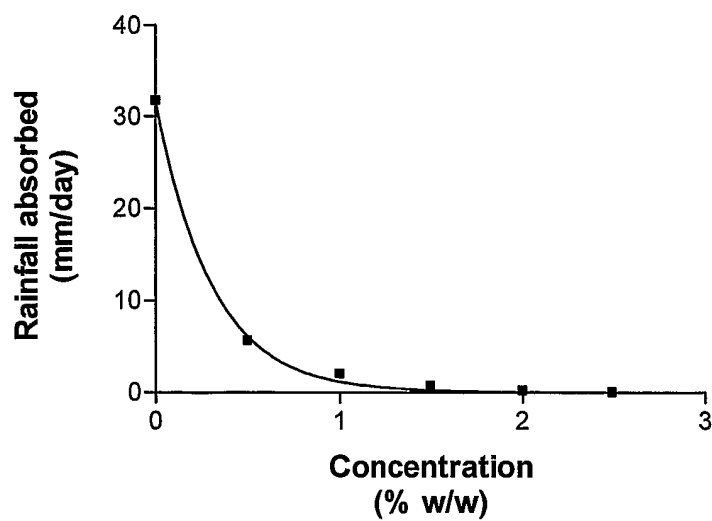


FIG 4

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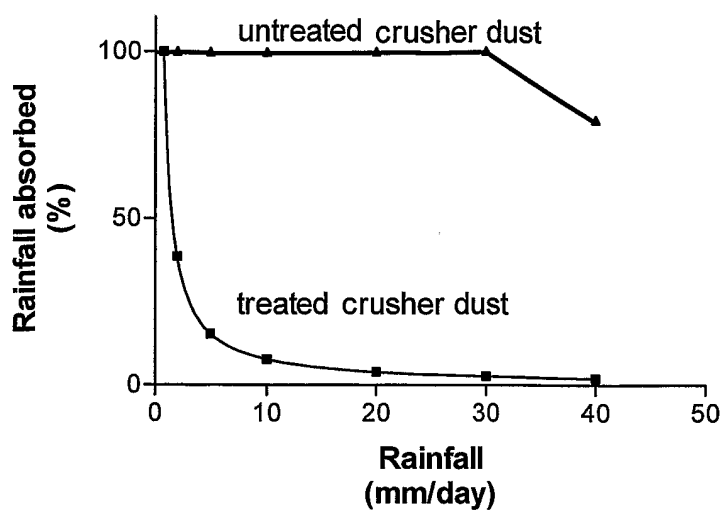


FIG 5

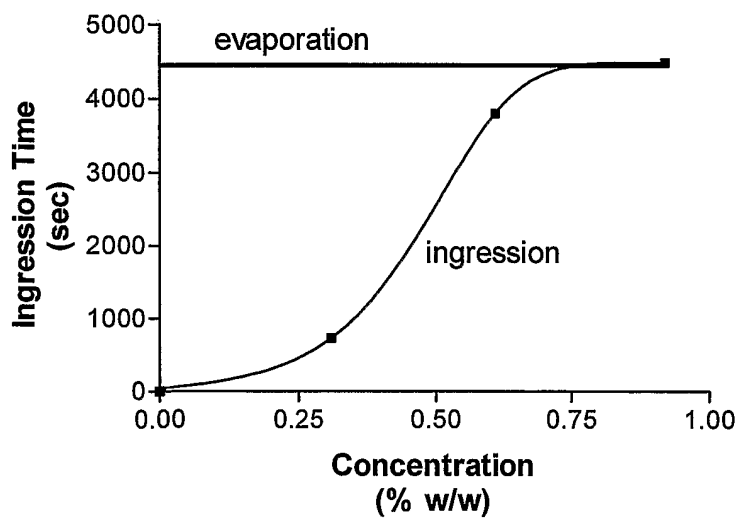


FIG 6

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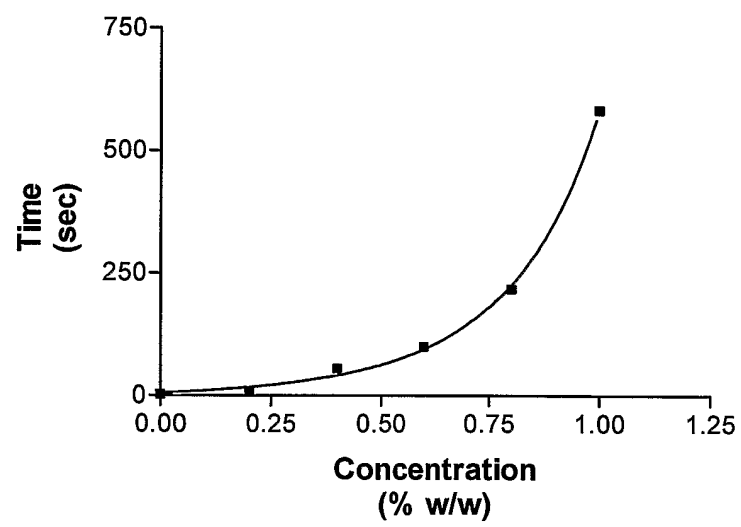


FIG 7

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU03/00963

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl. ⁷: C04B 41/47, C09D 191/06, 191/00, D27K 3/50, C09K 3/10

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)

Refer electronic database consulted below

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)

DWPI IPC C04B 41/47, C09D 191/06, 191/00, D27K 3/50, C09K 3/10 and keywords: stearic, wax, solvent and similar terms, Esp@ce (keywords: stearic, wax, solvent and similar terms) and USPTO

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Patent Abstracts of Japan, JP 10-212445 A (CAR MATE MFG CO LTD) 11 August 1998 See whole abstract	1, 2, 4 - 6, 8, 14, 28 - 31
X	Patent Abstracts of Japan, JP 58-063769 (KAO CORP) 15 April 1983 See whole abstract	1, 2, 4 - 6, 8, 15
X	FURNITUREKNOWLEDGE.COM 1999 Tip #19 (online) (retrieved November 2003) <URL: http://www.furnitureknowledge.com/tiparkiv.htm > See tip # 19: Traditional furniture polishes comprise bees wax as a naturally occurring high molecular weight ester, vinegar (carboxylic acid) and turpentine (hydrocarbon solvent) and are applied as a waterproofing or water repelling agent.	1, 2

☒ 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	"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
"E" earlier application or patent but published on or after the international filing date	"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
"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)	"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
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search
5 November 2003

Date of mailing of the international search report

17 NOV 2003

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU03/00963

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 19952563-A1 (ROMONTA GMBH) 3 May 2001 See whole document	1, 2, 4, 11 - 13, 16 - 19, 22, 27
X	Derwent Abstract Accession No. 14896B/08, Class E19, JP 54006002 (CHUO YUKA KK) 17 January 1979 See whole abstract	1, 2, 4, 5, 6

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU03/00963

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member	
JP	10212445		
JP	58063769		
DE	19952563	WO	0132780
			END OF ANNEX