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(54) Title: A MOULDABLE ALUM COMPOSITION

(57) Abstract

A moulded alum composition is formed by mixing crushed solid alum with a polyol plasticiser followed by heating to form a slurry, and then pouring the mixture into a mould. No mechanical cutting and grinding is required and weakening cracks and fissures present in the raw alum are removed.

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TITLE

A MOULDABLE ALUM COMPOSITION

THIS INVENTION relates to a mouldable composition and particularly to a mouldable alum composition.

BACKGROUND ART

Alum is an inorganic compound and generally contains two metals, two sulphate groups and water. A compound of this type is called a hydrated double salt. Alums are hydrated double salts that have similar compositions and similar crystalline structures.

Alums have a variety of uses. For instance, alums have been used in the dyeing industry, for water purification, for paper sizing, for fire-proofing fabrics, in fire extinguishers, and in medicinal and cosmetic fields.

It is the medicinal and cosmetic field where alums have particular interest. For instance, potassium alum has astringent properties, and is used in medicine to treat certain skin conditions, to reduce excessive perspiration, and to stop bleeding from small cuts. Dilute solutions (1 - 4%) have been used as mouth washes and gargles. Solutions of 5 - 10% are used to harden the skin, especially of the feet. Powdered forms of alum, or strong aqueous solutions of alum, are used as styptic for minor cuts and abrasions. Compositions containing talc and alum have been used as foot powders. Recent applications of alum containing solutions include bladder irrigations, and for the control of bladder haemorrhaging.

Commercially, the two most important alums are potassium alum and ammonium alum. Ammonium alum is manufactured by crystallisation from an aqueous solution of ammonium sulphate and aluminium sulphate. Ammonium alum crystals are also produced by treating a mixture of aluminium sulphate and sulphuric acid with ammonia.

Potassium alum occurs naturally in the minerals

alunite and kalinite, but can also be artificially manufactured by treating aluminium oxide with sulphuric acid and potassium sulphate. Another method of production involves heating of alunite followed by treatment with sulphuric acid to obtain crystals of the alum.

It is known that alums, and especially potassium alum and ammonium alum, can be used as solid crystalline deodorants, especially for under-arm use.

For solid deodorant use, it is necessary to manufacture the alum into a stick-like shape having rounded corners and no sharp or abrasive edges or portions.

Currently, the only method available to make such an alum deodorant stick is by cutting and grinding raw alum lumps, which is labour-intensive and also places limitations on the shape and size of the products.

Currently, crystalline lumps of the alum (which can be naturally-occurring alunite, or kalinite, or artificially manufactured crystals), are cut into sections by mechanical cutting devices and then core-drilled to produce a solid cylinder of alum. The cylinder has flat top and bottom ends and a cylindrical side wall and is used as a deodorant stick.

The method of cutting and core drilling results in a large amount of waste product which can not be used as deodorant sticks. Also, the cylinder has a flat top wall which results in a fairly sharp edge between the top wall and the side wall. Some manufacturers further shape the stick by mechanically grinding the cylinder of alum to provide a rounded top portion which is then smoother to use.

Another disadvantage with the naturally-occurring or artificially made alums is that they have numerous cracks and fissures throughout them. This makes mechanical cutting and core drilling difficult and results in many accidental breakages with subsequent discarding of the product. Also, consumers generally do

not like the cracks and fissures which detracts from the appearance of the deodorant stick. The cracks and fissures also reduce the strength of the deodorant stick making it susceptible to breakage if dropped or handled roughly, or during the packaging and transportation of the product to the retail outlets.

Attempts have made to shape alums into deodorant sicks or other shapes without requiring mechanical cutting, grinding or other shaping techniques. For instance, alum such as the dodecahydrate form of potassium alum has a melting point of 92.5°C. However, it is found that melting the alum followed by re-solidification transforms the alum into a weak and crumbly structure making it useless as a commercial deodorant stick. While not wishing to be bound by theory, it appears that when the alum is melted, the hygroscopic nature results in the weakening of the alum structure upon re-solidification and exposure to air.

OBJECT OF THE INVENTION

The present invention is directed to a method by which an alum can be moulded thereby doing away with, or reducing, the need for mechanical cutting or grinding, and where the moulded alum has sufficient strength to make it useful as a deodorant stick, or useful for other applications.

In one form, the invention results in a method of producing a shaped alum containing composition, the method comprising -

1) granulating an alum composition of Formula 1,

$$M1\ M2\ (XO4)_2.YH_2O$$

wherein

M1 is a positive univalent ion;

M2 is a positive trivalent ion;

X is sulphur or selenium; and

Y is a number between 0 - 30.

2) Mixing the product of Step 1 with a plasticiser to form a mouldable product,

3) moulding the mixture of Step 2 into a desired

shape.

Optionally, the mixture of Step 2 can be heated to promote conversion of the mixture to the mouldable product. It is preferred that the mixture is in the form of a slurry.

Preferably M1 is selected from the group consisting of sodium, potassium, rubidium, caesium, ammonium, thallium, silver, hydrazine, hydroxylamine, organic amines and lithium.

Preferably M2 is selected from the group consisting of aluminium, iron, chromium, manganese, indium, gallium, iridium, titanium, vanadium, cobalt and rhodium.

Preferably Y is 12.

Preferred components are potassium alum $KA1(SO4)_2.12H_20$ and ammonium alum NH_4 $A1(SO4)_2.12H_20$.

Other alums may include caesium alum, iron alum, chrome alum, and chromoselenic alum.

The plasticiser may be a liquid plasticiser or a plasticiser which is in a liquid or substantially liquid form during the mixing step. Preferably a liquid plasticiser is used.

The liquid plasticiser may comprise an organic polyol. The organic polyol may be a linear polyol having between C_2-C_{10} carbons. Suitable such polyols can include glycerol, and/or sorbitol ($C_6H_{14}0_6$). Other plasticisers and solvents may also be used.

Preferably, the plasticiser is one which is stable during the mixing and moulding steps, and therefore glycerol is a preferred plasticiser. If other plasticisers are used may discolour during the mixing and any heating step, such a plasticiser may still be acceptable depending on the end use of the moulded product. If the discolouration is to be masked or converted, it is possible to add dyes, colorants and the like to the mixture.

The amount of plasticiser used can vary depending on the type of plasticiser, the type of alum,

and the end use of the moulded product. The end use may require consideration of hardness, durability, and lasting properties. A plasticiser ratio of between 0.1 to 50% by weight of the alum can be used. If the plasticiser is glycerol, and the alum is potassium alum or ammonium alum, approximately 0.5 to 15% by weight, or by volume of glycerol, can be used.

During the method of preparing the product, various additives can be used. These additives may include fragrances, perfumes, borax, conditioners, natural oils, and medicinal compounds such as antiseptic agents. It may be necessary to adjust the process parameters such as mixing times and heating and select the additives depending on their properties such as boiling point, stability, and the like. Such a choice would be apparent to a person skilled in the art.

The mixing of the plasticiser with the granulated alum composition may be achieved manually, mechanically, automatically, semi-automatically, or by other means.

Similarly, the moulding of the mixture to a desired shape can be done by pouring, pumping or pressing the mixture into a mould or by other means.

BEST MODE

EXAMPLE 1: Potassium alum is crushed into a particle size approximating that of sugar granules, that is, typically between 0.1 to 3 millimetre diameter granules. The crushed potassium alum is added to a mixing device (many types of mixing devices can be used) and 0.5% to 15% by weight or volume of glycerin is added. The mixture is mixed and heated to between 100 to 110°C during or after which it forms a mouldable slurry. The slurry is poured into moulds which are cylindrical and have a domed bottom wall. When cooled, the mould is removed to provide a deodorant stick which can be about 16 centimetres long, 5 centimetres in diameter, with a small taper from a wider base to a narrower domed top. The base of the stick can be wrapped to allow it to be

touched and can be used as a solid deodorant stick.

EXAMPLE 2: Potassium alum is crushed in a manner similar to Example 1. Between 0.5 to 15% by weight or volume of glycerin is added to the mixture. The mixture is mixed and heated until water displaced from the alum begins to boil. At this boiling, or substantially at the boiling point, additives such as borax or fragrances can be added. The mixture is then cooled and rehydrated by addition of water, and the cooled rehydrated mixture is poured into moulds to form desired shapes, such as deodorant sticks, and the like.

EXAMPLE 3: 1000g of potassium alum is crushed as per example 1 and is added to a plastic container. 20g of glycerin is added to the container. The container containing the mixture is subjected to microwave heating (700 watt output) for 9 minutes. At this stage sufficient heating and water displacement has ocurred. The heated mixture is removed from the microwave source and is stirred for 10-15 seconds to ensure mixing of the contents. the mixture is then poured into a mould and allowed to cool before being removed from the mould.

Example 2 illustrates that borax can be mixed in to the slurry to form a useful product. As glycerol is unstable at higher temperatures, the heating to boiling point is kept to a minimum time before cooling occurs. Also, any additives that are added at the elevated temperature must be such that do not immediately boil away or decompose.

It can be seen that the above method will allow alum, such as potassium alum and ammonium alum, to be moulded to any desired shape and does not require extensive cutting or grinding. There is little or no wastage as any off-cuts can be simply ground and added to the mixture.

An advantage of the moulding step is that any fissures and cracks are closed to provide a stronger and more visually appealing product. The plasticiser overcomes the previous disadvantage of crumbling which

occurs when the alums are heated and cooled by themselves. While not wishing to be bound by theory, it appears that polyol plasticisers may hydrogen bond with, or become included with, the alum structure.

The method creates an uniformly perfect product every time without the problems of cracking or crumbling. The moulding method facilitates mass production with little or no wastage, and does not have any significant limitation to size or shape.

If glycerine is used as the plasticiser, a further advantage is that glycerine is an accepted skin conditioner. Thus, the moulded alum is less harsh on the skin than cut and shaped alum deodorants which have a high acidity factor and can result in skin rashes occurring on some people.

The addition of borax in Example 2 reduces the acidity of the alum to allow the deodorant stick to be used on the most sensitive skin types.

The deodorant need not contain currently used ingredients such as aluminium chlorohydrate, which appears to have detrimental qualities.

By being able to add plasticisers and also other additives, fragrances, skin conditioning agents, and the like can be moulded entirely through and into the alum, whereas currently cut and shaped alums can only coat the outside of the stick with such additives.

It should be appreciated that various other changes and modifications can be made to the embodiments described. For instance, the moulded alum can be used other than for deodorants. It can be used to provide relief of tinea, dermatitis, cold sores, acne, shaving rash, cuts and skin irritations.

CLAIMS

1. A method of producing a shaped alum containing composition, the method comprising -

1) granulating an alum composition of Formula 1,

$$M1\ M2\ (XO4)_2.YH_2O$$

wherein

M1 is a positive univalent ion;

M2 is a positive trivalent ion;

X is sulphur or selenium; and

Y is a number between 0 - 30.

2) Mixing the product of Step 1 with a plasticiser to form a mouldable product,

3) moulding the mixture of Step 2 into a desired shape.

2. The method of claim 1, wherein M1 is selected from the group consisting of sodium, potassium, rubidium, caesium, ammonium, thallium, silver, hydrazine, hydroxylamine, organic amines and lithium.

3. The method of claim 2, wherein M2 is selected from the group consisting of aluminium, iron, chromium, manganese, indium, gallium, iridium, titanium, vanadium, cobalt and rhodium.

4. The method of claim 3, wherein the alum is selected from the group consisting of potassium alum and ammonium alum.

5. The method of claim 4, wherein the plasticiser is an organic polyol.

6. The method of claim 5, wherein the polyol is a linear polyol having between 2 -6 carbon atoms.

7. The method of claim 6, wherein about 0.5-15% by weight or volume of plasticiser is added.

8. The method of claim 7, wherein the plasticiser is selected from the group consisting of glycerol and sorbitol.

9. A method of producing a shaped alum containing composition comprising granulating potassium alum or ammonium alum to a particle size of about 0.1mm to 5mm,

adding between 0.1-15% by weight or by volume of glycerol, mixing the alum and glycerol together, heating the mixture to about 100-110C to form a slurry, pouring the slurry into a mould, cooling the mixture and removing the mixture from the mould to form the shaped alum composition.

10. An alum composition formed by the method of any one of the preceding claims.

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/AU 95/00384

A. CLASSIFICATION OF SUBJECT MATTER

Int Cl[6]: A61K 7/38, 33/06, 9/06

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 A61K 7/38, 33/06, 9/06

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
AU: IPC as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
DERWENT: alum: potass: ammoni: plastic: antipersp: deord:
WPAT: alum: potass: ammoni: plastic: antipersp: deord:

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 400 546 A1 (HELENE CURTIS, INC.) 5 December 1990 See abstract and claim 1	1-10
X	EP 0 448 278 A1 (COLGATE-PALMOLIVE COMPANY) 25 September 1991 See examples II, IV	1-10
X	EP 0454 127 A1 (TRADITIONAL CHINESE MEDICINE RESEARCH LABORATORIES INC.) 30 October 1991 See page 15	1-10

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

[X] See patent family annex

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Date of the actual completion of the international search	Date of mailing of the international search report
20 September 1995	13 OCTOBER 1995
Name and mailing address of the ISA/AU AUSTRALIAN INDUSTRIAL PROPERTY ORGANISATION PO BOX 200 WODEN ACT 2606 AUSTRALIA Facsimile No.: (06) 285 3929	Authorized officer **Sudath Kumarasinghe** Telephone No.: (06) 283 2269

INTERNATIONAL SEARCH REPORT

International Application No.
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C (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 94/13255 (CHURCH & DWIGHT CO.) 23 June 1994 See pages 8-11	1-10
P,X	WO 94/28866 (THE GILLETE COMPANY) 22 December 1994 See page 6 lines 29-33 & page 9 lines 6-17	1-10

INTERNATIONAL SEARCH REPORT
Information on patent family members

International Application No.
PCT/AU 95/00384

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					
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WO	94/28866	AU-A1	7202294	US-A	A 5417964		

END OF ANNEX