United States Patent Office 3,305,491 Patented Feb. 21, 1967

3,305,491

ANTI-CAKING AGENT

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No Drawing. Filed May 23, 1963, Ser. No. 282,565
6 Claims. (Cl. 252—384)

It is a well-known inconvenience of salts, such as fertilizers, of becoming a self-compacted mass, this phenomenon being ascribable to the action of pressure and variations in atmospheric parameters such as air moisture and temperature, which develop alternate dissolution and crystallization on the surface of salt crystals which finally become solidly caked to one another.

This caking and therefore the compaction of salts is usually counteracted by incorporating to the salt mass small amounts of compounds adapted to inhibit the aforesaid compacting or adhesive influence by interposing themselves between the crystals constituting the salt mass. The action consists in either separating the crystals mechanically (as by the addition of inert substances) or coating them with a thin water-repellent and isolating film (by using organic reagents having a special affinity for the salts to be treated).

However, certain amino compounds used in this last instance are objected to because they are considered, to a certain extent, as physiologically allergenic.

The present invention constitutes the outcome of long research works aiming at eliminating the drawbacks likely to arise from the use of composite anti-compaction agents which taken together are nevertheless at least as efficient as the best known agents used separately, while having a considerably lower final cost.

This result is obtained according to this invention by mixing and/or combining, among the anti-compaction agents having a substantial affinity for a salt to be treated, anionic agents on the one hand and cationic agents on the other hand, the various groups of component agents being water-repellent and yielding by their mixtures and/or their interreactions anio-catio-active anti-compaction composite agents having a higher efficiency.

According to this invention and with a view to further ensure and increase the anti-compaction efficiency of the product, it is advantageous to add to at least one basic anti-compaction cationic substance such as an amine having the property of reducing the surface tension existing on the surface of the crystals to be coated of the salt to be protected, at least one anionic anti-compaction agent adapted on the one hand to combine with the cationic substance and on the other hand to create an acid medium promoting the action of said cationic anti-compaction substance and, therefore, the combined anti-compaction of said cationic and anionic agents.

The two effects set forth hereinabove may be obtained by adding at least two different anionic agents, such as two fatty acids. If desired, a single anionic agent such as a fatty acid adapted to combine partially with the cationic substance or substances while leaving a free portion for creating the desired acid medium may be added to excess to the cationic substance or substances.

Thus, for example in the case of potassium chloride or other salts responsive on the one hand to the anti-compaction action of certain organic acids such as the stearin constituents, and on the other hand to certain amines (or their salts), with substantially the conventional proportion in the mixture of said acids and amines and at a lower price, an anti-compaction efficiency at least as considerable is obtained, the above-mentioned physiological inconvenience being removed completely.

It appears that the respectively anionic and cationic

component elements stimulate each other and produce a synergetic effect accounting for the surprising results obtained.

Considering more particularly the practical embodiment of the invention in the specific case of the inhibition of compacting action in potassium chloride or similar salts, the substances acting by forming a water-repellent film on the salt crystals are in most cases either salts of fatty acids such as aluminium stearates, zinc stearates and other similar substances, or compounds having a characteristic affinity for the salts to be treated, such as fatty amines, as well as their acetates or chlorides, and also certain fatty acids.

According to this invention and by way of example, in this last case the composite anio-catio-active agents consist advantageously of mixtures of fatty acids and fatty amines, which may be considered as salts of fatty acids of fatty amines, these salts being characterized mainly in that they are associated with a certain amount of uncombined fatty acid and correspond to the formula:

$$R_nCOOH,\ R_pNH^2+xR_nCOOH$$

wherein x is any positive number and R_n and R_p are radicals having from 8 to 22 carbon atoms, preferably 12 to 18.

Natural fatty acids of animal or vegetable origin, such as fatty acids from tallow, palm oil, soy beans, copra, etc., as well as any suitable synthetic organic acids may be used for preparing these mixtures.

Also by way of example, the amines obtained from the aforesaid fatty acids are also used to advantage.

In a practical application of this invention for counteracting the compaction of thermal KCl, tests have been conducted with two composite anio-catio-active products wherein the amino component is a free raw amine according to the general formula R—NH^2 wherein R is a chain mixture with C_{18} predominance and comprising about 30% of palmitylamine, 30% of stearylamine and 40% of oleylamine.

In a first case (case 1) one fraction of this amino compound is mixed in the molten condition with three parts by weight of saponification stearin also in the molten state and comprising:

	Percent
Lauric acid	2.4
Myristic acid	1.4
Palmitic acid	51.6
Palmito-oleic acid	2.7
Stearic acid	41.8

In another case (case 2) the same amino compound is also mixed in the molten state and in the same proportion with a fatty acid or a mixture of recovery, reclaimed or recycled fatty acids comprising for instance:

	Percent
Neutral substances (unsaponified fats)	10
Stearin+palmitin	30
Olein	59
Impurities (including water)	1

Samples were prepared on the one hand for the two aforesaid exemplary cases of anti-compaction products according to this invention (Case 1 and Case 2), and on the other hand:

Case 3, for KCl treated a stearin as set forth hereinabove, taken separately;

Case 4, for KCl treated with the aforesaid recycled fatty acid, taken separately;

Case 5, for KCl treated with the aforesaid amino product, taken separately;

Case 6, for untreated KCl (reference sample).

In each case, the proportion of anti-compaction agent was 0.036%.

The various salts thus obtained are subjected to the sinterizing conditions or factors. Then the samples are inspected and the different degrees of anti-compaction effect obtained are measured.

All the data collected from case to case on the different appearances of the salt are also recorded.

In the following table the average results obtained during several successive tests are summarized;

	Degree of anti-compaction obtained in each case (percent)	Flowability of salt
Case 1. KCl plus 0.036% of the mixture of 3 parts stearin plus 1 part amine.	100	Free-flow.
Case 2. KCl plus 0.036% of the mixture of 3 parts recycling acid plus 1 part amine.	90	Salt will flow under thin surface film.
Case 3. KCl plus 0.036% of stearin alone	92.6	Salt will flow under firm surface film
Case 4. KCl plus 0.036% of recycling acid alone	85.2	Salt mass slightly sintered.
Case 5. KCl plus 0.036% of amine alone	100	Free-flow.
Case 6. KCl without any addition (reference sample).	0	Salt set into a single block.

From the above comparative table it will be seen that the examples of composite products according to the invention display either the same efficiency (Case 1) or an efficiency (Case 2) approaching that of the best hitherto known anti-compaction agents, that is, amines (Case 5) while permitting substantial savings in cost.

Thus, it will be seen that the thermal-prepared and therefore non-floated KCl (and having a strong propensity for compacting into a block) is efficiently protected against sintering by a mixture comprising three parts of saponification stearin and one part of raw-tallow non-hydrogenated amine, this mixture being used in the proportion of .036%.

In another test with KCl of different structure the following results have been obtained comparatively:

(a) KCl+.036% of fatty acid from tallow alone, the efficiency of the anti-compaction effect is 54.5%.

(b) KCl+amine alone, the efficiency of the anti-compaction effect is 65.4%, and

(c) KCl+a mixture of four parts of fatty acid with one part of amine; the efficiency of the anti-compaction effect is 71.1%.

The above examples are concerned with the simplest case of the addition of a single fatty acid in excess to the cationic agent.

Similarly, two fatty acids, for example (1) saponification stearin and (2) recycling fatty acid (as mentioned in the preceding examples) may also be added to the cationic agent.

The last named fatty acid (recycling acid) combines with and neutralizes the amine, and the former (stearin) constitutes the free acid providing the acid medium promoting the exaltation of the anio-catio-active agents.

In a typical form of embodiment of the method of this invention for treating thermal KCl, one part of the aminated component used in the aforesaid examples (Case 1 and Case 2) and 1.203 parts of recycling acid are mixed in the molten state. 1.58 parts of saponification stearin, also in the molten state, are added to this mixture. The M.P. of the product thus obtained is 50° C. This anti-compaction agent, as far as the acidity:alkalinity ratio is concerned, corresponds to the products cited in Cases 1 and 2 hereinabove, wherein there are 3 parts of acid (recycling acid or stearin) per part of amine.

The anti-compaction effect obtained was 92.3%. It is nearly as high as that (92.5%) obtained with a reference sample incorporating the amine alone. A reference sample comprising the two acids but without any amine had an anti-compaction efficiency of only 55.4%.

Therefore, in the present example there is likewise a synergetic effect and a mutual exaltation of the anti-compaction actions of the associated acids and amine.

Of course, the specific examples of practical application of this method should not be construed as limiting the present invention. They illustrate the mutual exaltation of the anionic and cationic component elements constituting the anio-catio-active anti-compaction agent.

As a rule, it may be said that by mixing or combining natural or synthetic fatty acids (or other anionic substances) and amines (or other cationic substances), provided that these products have an inherent anti-compaction power for a given salt to be treated, very efficient and economical anio-catio-active (or more exactly catio-anio-active) anti-compaction products can be obtained. Any number of said products may be mixed (as a rule by fusion) through a wide range of proportions, but a substantial excess of acids (or, more exactly, of acidity) should be available in relation to the amine (or to the amines or other cationic agents). This excess is of primary importance notably from the point of view of cost, since acids are much cheaper than amines.

Among other advantageous features, the properties displayed by these anti-compaction products permitting of substituting high-acid and low-amine mixtures of fatty acids and fatty amines for the conventional amines and amine salts, such as chlorides or acetates, result inter alia, notably for treating KCl, from the possibility of:

Achieving an anti-compaction effect as efficient as that of known products, but at a considerably lower cost;

Obtaining this protection at a lower cost, without resorting to additions exceeding by weight the conventional additions;

Attaining under practical conditions a better result than those obtained in current practice due to the mutual stimulaion of the component elements;

Reducing considerably the physiological effects caused by allergies, as a consequence of the preponderance of acid with respect to amine in the mixture according to this invention.

Of course, the examples concerning the embodiment of the method of this invention which are described hereinabove should not be construed as limiting the invention since the application of many other anti-compaction agents may be contemplated by adding one or several compounds to the anio-catio-active substances of this invention which are taken as basic reagents having or not a mixture fluidizing property and promoting the synergetic effect of said basic reagents as well as the mutual stimulation concerning the anti-compaction effect in addition to all the separate activities thereof.

This invention also concerns, by way of novel industrial products, any anio-catio-active mixtures such as those set forth in the above examples for carrying out the method of this invention, as well as all similar methods.

I claim:

1. A catio-anio surface active composition for addition to salts to prevent caking of said salts, said composition being constituted by an amino component comprising a fatty amine salt of fatty acid, and another component comprising at least one free fatty acid which is in addition to the fatty acid employed in forming said amino

component, said composition being a mixture of the formula:

$$R_nCOOH, R_pNH_2+xR_nCOOH$$

wherein x is a positive number and R_n and R_p are hydrocarbon radicals having from 8 to 22 carbon atoms.

2. A catio-anio surface active composition according to claim 1, wherein the fatty acid of said amino component is the same as said fatty acid of said another component.

3. A composition according to claim 1, wherein said amino component is prepared from a free raw amine mixture having a predominance of C_{18} and comprising about 30% of palmitylamine, 30% of stearylamine and 40% of oleylamine.

4. A composition according to claim 3, wherein said amine mixture is mixed in the molten state with three parts of saponification stearin also in the molten state, which comprises:

	Percent
Lauric acid	2.4
Myristic acid	1.4
Palmitic acid	51.6
Palmito-oleic acid	2.7
Stearic acid	41.8

5. A composition according to claim 3, wherein said amine mixture is mixed also in the molten state, in the same proportion, with a mixture of reclaimed fatty acids such as recycling acids, comprising:

	Percent
Neutral substances (unsaponified fats)	10
Stearin-palmitin	30
Olein	59
Impurities (including water)	1

6. A composition according to claim 1, wherein said amino component is prepared from a free raw amine mixture having a predominance of C_{18} and comprising about 30% of palmitylamine, 30% of stearylamine and 40% of oleylamine, one part of said amino component being mixed in the molten state with about 1.58 parts of saponification stearin also in the molten state, which comprises:

	Percent
Lauric acid	2.4
Myristic acid	1.4
Palmitic acid	51.6
Palmito-oleic acid	2.7
Stearic acid	41.8

and with 1.203 parts of a mixture of reclaimed fatty acids such as recycling acids, comprising:

	Percent
Neutral substances (unsaponified fats)	10
Stearin+palmitin	30
Olein	59
Impurities (including water)	1

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