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(19)

(54) IMPROVED METHOD OF TREATING FERTILIZERS USING ANTICAKING COMPOSITIONS

(71) We, EXXON RESEARCH AND ENGINEERING COMPANY, a Corporation duly organised and existing under the laws of the State of Delaware, United States of America, of Linden, New Jersey, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the application of anticaking compositions to fertilizers to reduce the tendency of the fertilizer particles to agglomerate and form large bulky lumps during storage.

It is well known that fertilizer particles tend to agglomerate during storage to form lumps that can become extremely big in bulk storage. It has been suggested in for example United States Patent 2,772,833 and British Patent 755,516 that this problem may be overcome by spraying the fertilizer granules with an aqueous solution of an alkylaryl sulphonate. However, although this reduces the tendency of the fertilizer to cake since the presence of water is the prime cause of caking the use of an aqueous solution is not entirely satisfactory. It has also been proposed to coat the granules with a hydrophobic liquid such as a hydrocarbon oil and since it is important that the hydrocarbon oil be compatible with the normally aqueous surface of the granules it has been proposed in for example Belgian Patent 568,338 to include fatty acids or fatty amines in the hydrophobic liquid to improve the compatibility of the liquid with the granules. This technique suffers from the disadvantage that the fatty acids have low anticaking activity whilst the fatty amines have a limited solubility in hydrophobic liquids at room temperature so it is generally necessary to store the solutions at elevated temperatures which is expensive. Furthermore the solutions of the fatty amines must be applied at temperatures higher than that at which the fertilizer is stored thus requiring further expense.

We have now found that a hydrophobic liquid containing an alkylaryl sulphonate derived from an alkylaryl sulphonic acid of molecular weight greater than 400 and a fatty amine is a particularly suitable anticaking agent especially at the normal temperatures used for storing fertilizers.

The present invention therefore provides a method for reducing the tendency of fertilizers to cake comprising coating fertilizer granules with a hydrophobic liquid having dissolved therein at least 0.01% and preferably from 0.01% to 20% by weight of an alkylaryl sulphonate derived from an alkylaryl sulphonic acid of molecular weight greater than 400 and from 0.1 to 10 wt % of the composition of a fatty amine.

The invention also provides fertilizer granules coated with an anticaking composition comprising a hydrophobic liquid having dissolved therein at least 0.01% and preferably from 0.01% to 20% by weight of an alkylaryl sulphonate derived from an alkylaryl sulphonic acid of molecular weight greater than 400 and from 0.1 to 10 wt % of the composition of a fatty amine.

In order to reduce caking tendencies fertilizer granules are sprayed with an anticaking composition after drying and the anticaking composition is most conveniently sprayed onto the dried granules at the end of the manufacturing process particularly when the granules are in a rotating drum which helps towards uniform coating. The techniques of the present invention are particularly useful in reducing the caking tendency of nitrogenous fertilizers, especially the complex Calcium Ammonium Nitrate, N.P.K. and ammonium nitrate fertilizers. These materials are highly hydrophilic and tend to cake when they are damp and this caking tendency is thought to be reduced by the anti-caking composition acting as a waterproofing layer for the granules. It is therefore preferred that the anticaking composition be applied shortly after the drying step in the process of fertilizer manufacture.

The hydrophobic liquid used in our invention therefore acts as a waterproofing layer and the presence of the sulphonate enhances compatibility of the liquid with the surface

of the fertilizer granules both on application of the composition and under the conditions at which the fertilizer is stored.

The choice of the hydrophobic liquid that is used in the present invention will depend upon the character of the surface of the fertilizer granules and the method by which the composition is applied to the fertilizer. Any suitable liquid that has a sufficiently high flash point to ensure that there are no fire hazards may be used. Examples of hydrophobic liquids include both paraffinic and naphthenic mineral oils and also synthetic fluids such as synthetic isoparaffins, esters of synthetic or natural acids or polyacids with synthetic or natural alcohols or polyols. We prefer to use a mineral oil and where the composition is sprayed onto the fertilizer we prefer to use an oil having a suitable viscosity at the spraying temperature. Many conventional sprayers operate at around 70°C and in these circumstances we prefer to use a mineral oil of viscosity from 5 to 20, preferably from 10 to 15 centistokes at the spraying temperature.

The alkylaryl sulphonate used in the present invention must be soluble in the hydrophobic liquid and we find that alkylaryl sulphonates derived from alkylaryl sulphonic acids of molecular weight greater than 400 should be used. In particular we prefer to use sulphonates derived from alkylaryl sulphonic acids of a molecular weight greater than 460 especially those acids having a molecular weight in the range 480 to 530. The sulphonates used may be a mixture of sulphonates derived from sulphonic acids of different molecular weights and in this situation the reference to the molecular weight is to the average molecular weight of the sulphonic acids from which the sulphonates are derived. The sulphonate may be an alkali or alkaline earth metal sulphonate or may be the sulphonate of a nitrogen containing compound such as ammonia, ethanolamine, ethoxy amines or fatty acid amines.

The amount of sulphonate or sulphonate mixture that should be used is not critical providing it is sufficent to ensure that the hydrophobic liquid is compatible with the surface of the fertilizer granules and remains so during storage of the fertilizer. We find that the composition should contain at least 0.01% by weight of the sulphonate and from an economic point of view one uses as little as possible and we find that compositions containing from 0.01 to 20% preferably from 0.5 to 15% especially from 1% to 15% are particularly suitable. The amount of the composition that should be applied to the fertilizer depends upon the chemical nature and the morphology of the fertilizer. We find however that good anticaking may be achieved using from 0.005 wt.% to 0.4 wt.% of the composition based on the weight of the fertilizer.

We have found that the presence of the fatty amine is particularly effective especially with Calcium Ammonium Nitrate fertilizers. We have found a solution of an ethanolamine sulphonate and a fatty amine to be particularly useful. We also find that we can use the fatty amines that have hitherto required special heating when used as anticaking agents without the need for this special heating since the presence of the sulphonate seems to aid the solubility of the amine. Any suitable fatty amine may be used and may be saturated or unsaturated and we prefer that it contains at least 12 carbon atoms. Most commercially available fatty amines are mixtures of amines and we have found that those containing predominantly amines with 16 and 18 carbon atoms are especially useful. We have found that compositions containing 1% to 4% by weight of the composition of the amine are particularly useful.

It is known to treat fertilizers with powders such as chalk or diatomaceous earth to reduce their tendency to cake. The techniques of the present invention may be used in combination with powder treatment and this is our preferred method with NPK fertilizers. The powder may be applied to the fertilizer simultaneously with our techniques or sequentially therewith.

Our invention may be used with most types of fertilizer and it tends to work better the dryer the fertilizer and we prefer that the fertilizer contain less than 0.4 wt.% more preferably less than 0.35 wt.% of water and thus our invention is most useful shortly after the drying stage of the manufacturing process.

The present invention is illustrated by reference to the following examples in which the degree of caking of the fertilizer is measured according to the "caking number" which is a number characterising the effort needed to disintegrate the fertilizer. In the test the fertilizer is stored under a standard pressure of 40 kilogrammes per square centimeter in a split cylinder for 24 hours and the force required to separate the two parts of the cylinder is measured and converted according to the formula:

$$n = \frac{\text{separating force (in grams)}}{38}$$

to give n the caking number of the fertilizer. A caking number less than 20 indicates satisfactory anticaking but less than 10 is preferred.

Example 1

17% by weight of the sodium salt of an alkylaryl sulphonic acid of molecular weight 520 was dissolved in a paraffinic mineral oil having a viscosity of 3.55 centistokes at 100°C and 11.3 centistokes at 50°C and blended with an equal amount of a 20 wt.% fatty amine solution (commercially available as

Noram (Registered Trade Mark) SH). This solution was then sprayed onto 200 tons of dried Calcium Ammonium Nitrate fertilizer treated with a filler comprising 75 wt.% Belgian chalk and 25 wt.% Welsh chalk. This fertilizer contained about 26% Nitrogen, 13% Ammonia and 0.34 wt.% water. 0.23 to 0.25 wt.% of the composition was applied to the fertilizer as it passed through a coating drum for storage to give a caking number between 7 and 12 and the fertilizer then stored. After 6 months storage in bulk or in bags the fertilizer easily handled in normal use.

Example 2

A composition comprising the sulphonate solution used in Example 1 containing in addition 1.4 wt.% of a commercially available fatty amine containing 30% C_{16} amines and 60% C_{18} amines was applied to the same type of fertilizer as was used in Example 1 immediately upon completion of fertilizer manufacture to give treat rates of 0.10 wt.%; 0.15 wt.% and 0.20 wt.% based on the fertilizer.

The caking index of these fertilizers was as follows:

Anti-Caking Composition	Treat Rate	Caking Number
None	0.10%	4
Sodium Sulphonate of Example 1+1% Fatty Amine Sodium Sulphonate of Example 1+2% Fatty Amine	0.15%	about 4
1% Fatty Amine alone	0.20%	4
2% Fatty Amine alone		
Ethanolamine Sulphonate of Example 3+1% Fatty Amine		
Ethanolamine Sulphonate of Example 3+2% Fatty Amine		

The caking number of the untreated fertilizer was from 10 to 12.

Example 3

An ammonium nitrate fertilizer containing 36 wt.% nitrogen which untreated had a caking number of 32 was treated with a 9 wt.% solution of the diethanolamine salt of the sulphonic acid from which the sodium salt of Example 1 is derived containing 1% of a fatty amine to a treat rate of 0.1 wt.% based on the fertilizer.

The caking number of the treated material was found to be 4.

Example 4

Two commercially available Calcium Ammonium Nitrate Fertilizers containing 26 wt.% Nitrogen were coated with various anti-caking compositions some comparative and the caking number measured with the following results:

Treat Rate Wt. % Fertilizer	Fertilizer/Caking Number A	B
0	132	41
0.1	9	7
0.1	7	4
0.1	75	
0.1	20	
0.1	11	8
0.1	7	5

It should be appreciated that there is considerable variation of properties within a bulk fertilizer and from one batch to another of fertilizers produced in the same manner particularly in the morphology and water content of the fertilizer. Thus, the values given for caking number are average values and the purpose of including the values is to show the trend obtained when using the various anticaking compositions.

WHAT WE CLAIM IS:—

1. A method of reducing the tendency of fertilizers to cake comprising coating fertilizer granules with an anticaking composition comprising a hydrophobic liquid having dissolved therein at least 0.01% and preferably from 0.01% to 20% by weight of an alkylaryl sulphonate derived from an alkylaryl sulphonic acid of molecular weight greater than 400 and from 0.1% to 10% by weight of the composition of a fatty amine.

2. A method according to claim 1 in which the molecular weight of the alkylaryl sulphonic acid from which the sulphonate is derived is at least 460.

3. A method according to claim 2 in which the molecular weight of the alkylaryl sulphonic acid is in the range 480 to 530.

4. A method according to any of the preceding claims in which the sulphonate is an alkali or alkaline earth metal sulphonate.

5. A method according to any of claims 1 to 3 in which the sulphonate is the sulphonate of a nitrogen containing compound selected from ammonia, ethanolamine, ethoxy amines and fatty acid amines.

6. A method according to any of the preceding claims in which the fatty amine contains at least 12 carbon atoms.

7. A method according to any of the preceding claims in which the composition contains from 1% to 4% by weight of the fatty amine.

8. A method according to any of the preceding claims substantially as hereinbefore

described with particular reference to the accompanying examples.

9. A method according to any of the preceding claims in which the fertilizer is coated with from 0.005 wt.% to 0.4 wt.% of the anticaking composition.

10. A method according to any of the preceding claims in which the fertilizer is also treated with an anticaking powder.

11. A method according to claim 10 in which the powder is chalk or diatomaceous earth.

12. A method according to claim 10 or claim 11 in which the powder is applied to the fertilizer before application of the anticaking composition.

13. Fertilizer granules whenever produced by a method according to any one of the preceding claims.

14. Fertilizer granules according to claim 13 in which the fertilizer is Calcium Ammonium Nitrate.

15. Fertilizer granules according to claim 13 substantially as hereinbefore described with particular reference to the accompanying examples.

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