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Granular fertilizer caking: A research on the performance evaluation of coating agents

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RESEARCH ARTICLE

ABSTRACT



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Caking formation in fertilizer products during handling and storage is a frequently encountered problem. Caked fertilizers become together as lumps, lose their free-flowing properties, and create inefficient applications. Several parameters such as relative humidity and temperature of the environment, pressure by pile height, and physicochemical properties of fertilizer product such as moisture content, presence of fines and chemical composition, etc. cause or accelerate caking mechanism. Internal or external anticaking products have been proved to be effective in preventing the caking of fertilizer particles. Liquid or wax anticaking agents are applied externally as coatings on the fertilizer surface. This study focuses on the performance evaluation of commercially available liquid-wax anticaking agents coated on NP 20-20-0 fertilizer, classified as three main groups: petroleum-derived, alkyl amine-fatty acid containing mixtures and bio-based composition. Caking performance of coating products are evaluated via small-bag storage test in 4 sets, according to the results, petroleum derived and alkyl amine-fatty acid containing anticaking products show similar performance, whereas bio-based products' performance is moderate. This study focuses on a practical approach that will help fertilizer industry about which type of coating product will be effective on the anticaking properties of nitrogenous fertilizers and gives brief results of the effect of commercially available products with various ranges of chemical composition on the caking process.

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1. Introduction

Fertilizers, which are one of the most important agricultural inputs, provide higher yield in crops and mediate soil fertility, which improves human livelihood and offers a better food security and qualified nutrition. Naturally occurring soil nutrients are the major yield-limiting factors and the agronomic importance of synthetic fertilizers is that they procure an efficient and profitable crop production by serving macro- and micro-nutrients to the plant during their growth. Thus, in order to meet the increasing food demand and to give a balanced and qualified diet to the global population, fertigation has great importance [1,2]. Providing the fertilizer product to the end-user at a reasonable quality and affordable cost is as important as the application of the right grade of fertilizer to the plant for meeting its nutritional needs. Fertilizers must be developed according to the plant needs as well as global food demands, and they must have good physical resistance against degradation during transport and handling. The problems related with the quality of fertilizer frequently encountered and studied can be stated as caking, degradation, dust formation, nonhomogeneous distribution of micro-nutrients etc. [3].

Caking can be defined as the formation of contact points between solid particles, also known as liquid and solid bridges,

or the formation of undesired agglomerates during storage period. Caking can also be explained as the transformation of free-flowing particles into lumps and then into agglomerated solids. The caking mechanism in fertilizers generally occurs due to the formation of salt bridges or capillary adhesion, and the severity of caking is affected by various parameters such as chemical composition, moisture content, particle structure, critical relative humidity (CRH), storage conditions (relative humidity, temperature & pressure), and storage period [4-6]. Salt bridges and phase contacts, characterized by the sum of ionic bonding and hydrogen bonding, play an important role in supramolecular chemistry [7]. They are formed at the contact points between fertilizer particles during long-term storage periods after the production of fertilizer products, ambient conditions in the storage area, ongoing internal chemical reactions, dissolution, and recrystallization processes as a function of the hygroscopicity of the fertilizer material. Capillary adhesion or adhesive contacts, also known as Van der Waals forces, are relatively weak interactions and they occur due to the molecular interaction between contacting surfaces. Plasticity of the fertilizer material and the pressure exerting on the fertilizer particle during storage whether in bags or piles as bulk, are the most important parameters affecting capillary adhesion [6]. Caking tendency is investigated and controlled by

storage simulation tests such as small or big-bag storage tests [8]. According to European Fertilizer Manufacturers Association (EFMA) standards, the physical and chemical structure of fertilizer products should maintain and do not degrade in a two-year storage period [18]. However, these long-term observations are mostly not preferred due to their high cost, location occupation, and labor requirements. Thus, lab-scale small-bag storage tests are commonly conducted. Small-bag storage test exerts a coherent mass under an applied force on the fertilizer granules during predetermined storage periods and helps to observe the degree of caking in lab-scale. As a result of exerting pressure, fertilizer particles at the interfaces begin to bond and agglomerate as a function of storage time [8].

Caking is a complex phenomenon, and its mechanism is dependent both on material structure and ambient conditions [9,10]. To decrease the negative impacts of caking phenomena on the physical quality of fertilizers, some optimizations are conducted by the fertilizer industry. Effective drying and cooling processes have importance in order to obtain a product with low moisture content and temperature, respectively [4]. Additionally, granulation, prilling and pelletizing processes provide products with a larger and uniform particle size distribution, thus the low surface area of these particles disables the formation of contact points between particles [8]. Although these precautions significantly reduce the caking tendency of fertilizers, they do not entirely solve the caking problem [11]. Application of quality improvement additives internally or externally is another precaution taken to decrease the caking tendency. These additives are also called as anticaking agents or conditioners, allow fertilizer particles to maintain their free-flowing and dust-free properties. Utilization of anticaking agents allows long-term storage without cake or dust formation (or keep caking and dust formation at minima) and ease in application of fertilizers onto soil [3]. Most of the anticaking agents aim to give prevention by using inorganic salts and stabilizers during the production of the fertilizer as an internal filler or by coating the finished product with an agent including mostly based on petroleum raw materials and surfactants. There have been various kinds of suggestions cited in the literature explaining how anticaking agents prevent the formation of caking phenomena such as disabling the contact of saturated solutions on the surfaces of different adjacent particles, creating a hydrophobic film layer on the surface of the particles, inhibiting dissolution or recrystallization processes, etc. since friction between two interacting surfaces has maxima, enabling an increase in the contact area as a result of friction [6,12]. Generally, all these additives are classified according to their application methods; externally applied additives are called as coating agents and the rest is defined as internal conditioners [13-15]. Coating agents generally disable the formation of crystal growth between fertilizer particles and create a hydrophobic film on the surface in order to limit moisture absorption and alter the interfacial tension between solid and liquid phases. Formation of a hydrophobic layer on the particle surface improves water repellency, reduces the capillary adhesion, modifies crystal growth, and reduces the tensile strength of the caking bond. Application of these coatings reduces the interfacial tension and improves the flowability of fertilizers [16]. Additionally, they contain basically a specific component that plays against the cake formation such as sulfonates, alkyl sulfates, amines, silicone, and petroleum-based components, etc. Sulfonates, mainly alkyl aryl sulfonates, are composed of a polar group and a long hydrocarbon chain. Their hydrophobic nature allows efficient wettability of solid surfaces by water, and the ease in application makes them industrially valuable [17]. Alkyl sulfate groups such as sodium alkyl sulfate, potassium alkyl sulfate, mono/di, or triethanol ammonium alkyl sulfates have been reported to be effective in limiting the moisture absorption properties of fertilizers [18]. Amine groups and their

derivatives having varied hydrophobic tails (C12-C22) also have good anti-hygroscopic performance [19]. Lowering the surface free energy with long-chain alkylamine compounds allow the formation of a hydrophobic layer on the substrate surface [20]. Fatty amines, especially those with long carbon chains such as octadecylamine are widely used for creating hydrophobic coatings [21]. Alkylamine groups are also reported to be the key component for creating a functionalized hydrophobic surface [22]. Silicone-based fluids are extensively used as an anticaking agent for powdery materials; however, their application for fertilizers is limited since they are economically not feasible. Petroleum-based products such as fuel-oil and paraffin wax, stearic acid, or mineral oil coatings also forms a hydrophobic layer on the surface. However, these agents are not efficient and environment-friendly, their accumulation affects the aquatic environment and soil in a negative sense [18].

Apart from the scientific point of view, commercial anticaking products are profit-focused and the specific information related with their contents and synthesis routes, etc., cannot be provided because most of these products have patented content under individual confidentiality agreements. This study briefly discusses the commercially available products of three basic classification, i.e., petroleum-based, alkyl amine: fatty acid mixture, and bio-based, in terms of their anticaking performance. Moreover, this study gives a performance comparison of commercial anticaking products with different chemical contents, providing a general approach for the fertilizer industry about which type of anticaking product will decrease the caking percent of nitrogenous fertilizers.

2. Experimental

In this study, NP 20-20-0 compound fertilizer was used to investigate its caking behavior. Fertilizer samples were provided from Toros Agri Industry and Trade Co. Inc., Turkey. Table 1 gives the chemical content of uncoated fertilizer samples.

Table 1. NP 20-20-0 fertilizer content.

Property	w/w (%)
Total nitrogen (N)	20
Ammonium nitrogen (N)	9
Urea nitrogen (N)	11
Water soluble P ₂ O ₅	19
Neutral ammonium citrate and water soluble P ₂ O ₅	20
Total SO ₃	5.5
Total Zn	1

Several types of commercially available liquid coating agents together with some promising bio-based alternative liquid coatings were coated on fertilizer surface for their performance comparison. Table 2 gives the structural details of applied liquid coating agents. Abbreviations given as P, AA and B represent petroleum-derived, alkyl amine- fatty acid based and bio-based samples, respectively. Application rates of commercial products were performed according to the instructions given in their technical datasheets, while bio-based coatings were coated in 800-1200 ppm (0.8-1.2 wt %) range.

2.1. Characterization analyses

The basic characteristics that affect the caking tendency of the prepared fertilizers such as moisture content, particle size distribution, and mechanical strength were conducted according to a drying oven method (TS 2832-1), sieve analysis on Retsch mesh sieve set (EN 1235) and crushing strength of bare fertilizer samples on Lutron FG-5000 A, respectively [23].

2.2. Evaluation of caking by small-bag storage test

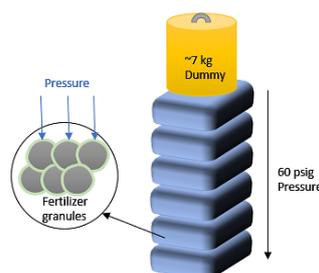
The caking tendency of fertilizer samples was investigated according to small-bag storage test as described by Hignett and

Table 2. Liquid-wax coating agents used in the study

Anti-cake agent	Ingredient base	Physical structure
P1	Petroleum-derived	Viscous liquid
AA1	10% Alkylamine + 20% Fatty acids	Wax
AA2	50% Alkylamine + 50% Fatty acids	Wax
AA3	3-10% Alkylamine + 2.5-10% Organic P-compounds	Wax, creamy
P2	Petroleum-derived	Wax
B1	Vegetable oil based	Liquid
B2	Vegetable oil based	Viscous, sticky
AA4	25-100% Petroleum-derived + 10-20% Alkyl amine	Wax

Table 3. Physical characteristics of uncoated NP 20-20-0 fertilizer.

Physical properties	Results
Moisture (%)	0.72
Crushing strength (kg/granule)	1.98
Granule size (mm)	2-4
Grain class (the content of 2-4 mm granules, %)	95

**Figure 1.** Small-bag storage caking test scheme [8].

Walker *et al.* [8,24]. Schematic illustration of test setup is given in Figure 1. Although big-bag storage tests represent approximately actual caking tendency, utilization of large quantities of materials, occupation of a large test area, and a considerable amount of labor are the disadvantages in the application. Small-bag storage test allows the elimination of these disadvantages [8]. In this test, a small number of samples were stored in six moisture-resistant bags and exposed to a predetermined pressure during a specified time to simulate the actual storage conditions. In order to investigate the performance of different anticaking agents, various types of products (i.e., petroleum-based, bio-based, alkylamine derivatives), were coated on NP 20-20-0 compound fertilizer. Uncoated fertilizer sample as reference was treated together with coated fertilizer samples in the same conditions. These small-bags were stacked up with a stable weight in order to apply predetermined pressure onto each bag sets. The test was conducted in a climate-controlled room, simulating warehouse conditions for 10 days. Temperature and relative humidity values were measured by Vaisala Humicap HM 41 hygrometer on a daily basis. After 10 days, the bags were opened and caking % was measured by weighing the caked parts.

3. Results and discussion

3.1. Basic characteristics of uncoated compound fertilizer NP 20-20-0

NP and NPK type inorganic fertilizers are basically a combination of powdered raw materials and multicomponent salt systems granulated upon heating and humidification, which are highly hygroscopic and have a high tendency for agglomeration [25].

The basic properties of uncoated NP 20-20-0 fertilizer such as moisture content, crushing strength, grain class, and granule size were determined to understand the source of caking tendency of the fertilizer used in the study. The results of basic analyses were listed in Table 3.

The moisture content of the bare fertilizer was determined as 0.72 %, which is commonly between the limits, <1% and it can be stated as one of the most important parameters affecting

caking tendency [5,6,10]. High moisture content allows a high concentration of dissolved fertilizer ions, triggering the severity of caking. In many caking mechanisms, the main reason of agglomeration is the remained excess water in fertilizer structure which increase the cake formation reactions. Therefore, keeping the moisture content of fertilizers under control during production and during the storage period in the warehouse is essential to prevent cake formation [6,9]. Moisture absorption rate of a fertilizer depends on the fertilizer substrate's hygroscopicity and it can be compared via measuring the critical relative humidity (CRH) values [26]. NP 20-20-0 fertilizer used in this study has 20% nitrogen content, in which 9% of nitrogen content is ammonium and 11% is urea based. Ammonium sulfate fed to the granulation bed is the resource of 5.5% SO₃ and partially ammonium nitrogen which comes from ammonia fed to the pipe reactor during the production process. Blending of these ammonium nitrogen resources (ammonia and ammonium sulfate) within the fertilizer formulation allows adjusting the moisture absorption rate and critical relative humidity of resulting product. The mechanical strength of NP fertilizer was analyzed as 1.98 kg/granule by using a crushing strength test device. The hardness of fertilizer granules is highly effective on the caking tendency. Weak granules always tend to break down under pressure during storage or by handling while transportation and application. Therefore, these deformed granules may have more contact areas to interact with each other and cake [27]. Moreover, increased dustiness enables the interactions between fertilizer particles, accelerating the caking mechanism. Particle size distribution of the fertilizer sample was determined by sieve test, in which 95% of the particles were in 2-4 mm range. According to the regulation on chemical fertilizer products used in agriculture issued by Ministry of Agriculture and Forestry, Turkey; particles smaller than 1 mm should not exceed 5 wt. % in a compound fertilizer, and the optimum particle size should be between 2 and 4 mm. According to the sieve test results, 95% of particles are >2-4 mm range, thus it can be presumed that dust formation will be low. Uniform particle size distribution is of great importance in order to minimize cake formation since dimension equity will reduce the contact area and minimize cake formation.

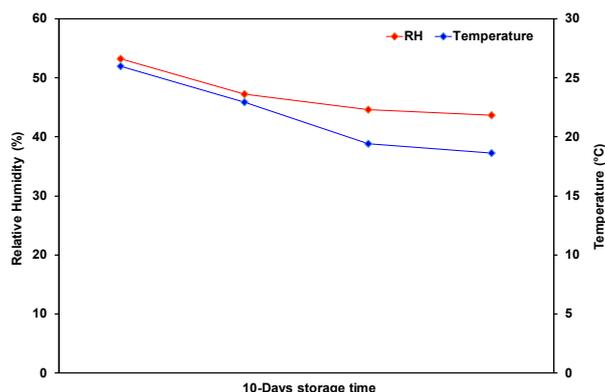


Figure 2. Temperature and humidity changes in the test environment.



Figure 3. Caked parts of uncoated fertilizer sample.

After the small-bag test, the weak granules break and come together to form a cake during the test. Because the weak and brittle particles are agglomerated, they are not in the proper form to control their strength in the crush test. For this reason, the agglomerated part is evaluated as fragile and weak particle amount and calculated in percentage.

3.2. Anticaking performance of coatings

Temperature and relative humidity changes of the environment during the small-bag storage test are shown in Figure 2. The ambient physical conditions of all experiment sets are calculated as 21.74 °C temperature and 47.2% relative humidity. It is important to follow the environmental conditions for interpreting the mechanisms that may occur in the fertilizer. The fertilizer products of multi-component salt systems usually have lower critical relative humidity than those of which are composed of a single salt type. Especially when certain fertilizer salts such as ammonium nitrate and urea come together, dramatic decreases can occur in critical relative humidity. The nitrogen source of the compound fertilizer used in this study is both ammonium sulfate and urea based. Ammonium sulfate has a CRH of 75% at 30 °C, and urea has a CRH of 70-75% at 30 °C, whereas the CRH of a mixture of these two fertilizers is approximately 55%. In addition, the CRH of most fertilizers decreases with an increase in temperature [8]. During small-bag storage, it was observed that the relative humidity rose to around 53% and approached the critical relative humidity of NP 20-20-0 (55%). However, this situation is not considered critical. Because the temperature was below 30 °C during the experiment and in this case, the CRH of the fertilizer increases above 55%. Therefore, it was interpreted that environmental conditions have a dominant effect on fertilizers throughout the experiment.

The caked parts after small-bag test in the uncoated fertilizer sample are shown in Figure 3. In all of the experimental sets, approximately 14 g of fertilizer caked in small bags of 80 grams.

Particle size distribution of fertilizer granules were analyzed by sieve test before small-bag storage test. 95% of the granules were resulted to be in the >2-4 mm range, in accordance with the requirements issued by the regulation on chemical fertilizer products used in agriculture by Ministry of Agriculture and Forestry, Turkey. Although samples were not further analyzed in order to determine whether the granules have cracked under pressure during small-bag storage test, of samples after the bags were opened showed a non-uniform particle size distribution, explaining that the fertilizer samples had degraded (i.e. cracked or partially dusted) upon exerting pressure.

The performance of alternative coating products in NP 20-20-0 fertilizer, which has a very high caking tendency of 30 % wt., is shown in Figure 4. It is seen that fertilizer without any coating (reference) is highly caked after a 10-day storage simulation.

In order for a fertilizer to be considered suitable in terms of caking tendency, it must be 15% or less caked by weight in a two-year storage period [18]. In this case, it can be said that AA3 and AA4 showed poor performance although they meet this requirement. According to small-bag storage test results, petroleum-based anticaking agent samples have shown better anticaking performance, and P2 reduces the amount of caking to 6.26%. Despite their better anticaking performance, P1 and P2 are petroleum-derived samples, and these products can be stuck to some laws and regulations and their use might be eliminated in the future. Therefore, while evaluating the performance of the products, these potentially biohazardous properties and possible regulatory amendments should also be considered. Moreover, continuous application of petroleum-based anticaking agent coated fertilizers for landfarming applications might cause phytotoxic effects in the long term, and the possible transfer of these hydrocarbon-based materials from fertilizer to soil and finally to plant tissue needs detailed studies because these plants enter the food chain [28]. Although many of the soil microorganisms can perform the transformation of petroleum-based hydrocarbons into nontoxic compounds, this process is

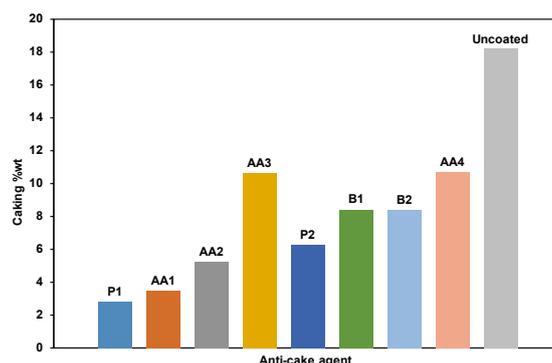


Figure 4. Anticaking performances of applied coatings.

also called as bioremediation, long-term effect of these materials due to accumulation via landfarming applications on microbiological, biochemical, and biological properties might be adverse [29].

P1 is followed by AA1 and AA2, with 3.5% and 5.21% caking, respectively. AA1 and AA2 are chemically alkylamine-fatty acid mixture samples having different alkylamine/fatty acid ratios by weight. Coating a nitrogenous fertilizer with materials including primary, secondary, and tertiary amines might improve its physical properties, and according to these results, it can be said that the decrease in alkylamine: fatty acid ratio in the product formulation increases the anticaking performance. However, these two products have shown similar anticaking performances. AA3 is an alkylamine-organic P compound mixture that has shown 10.64 % caking, and it can be said that introduction of an organic P compound in the formulation has an adverse effect on the caking performance. This effect might be due to the affinity of phosphorus react with oxygen. Moreover, phosphorus and nitrogen are located close to each other in the periodic table, and the moisture absorption potential of these two elements is similar to each other. Finally, AA4 is a mixture of petroleum-derived distillates and a small amount of alkyl amine, has shown an anticaking performance of 10.68%. It is also shown that alkylamine ratio is very effective to provide caking. The reason this product performs lower than other alkylamine included anticaking products is because it has a lower alkylamine content. Similarly, it can be said that the increase in alkylamine ratio increases the anticaking performance.

B1 and B2 are bio-based coating agents, which have a promising future as being an alternative to petroleum-derived products. Bio-based products are based on renewable sources, and they have gained increasing attention due to global warming and stricter amendments in the regulations related to the environment [30]. B1, with an anticaking performance of 8.42%, is a mixture of mostly glycerin, in addition to trace amounts of alcohol, salt, heavy metals, free fatty acids, unreacted mono-di-tri glycerides and methyl esters derived from waste bio-oil [31]. B2 has shown 8.40% caking, performing a very similar anticaking effect to B1 is derived from biomass, enabling the consumption of large amounts of organic wastes [32]. Thus, B1 and B2 are cleaner and environment-friendly products, the utilization of these kinds of products derived from renewable resources will be of great interest since they do not harm the environment and are promising alternatives to petroleum-derived products. Although the performance of these products is close to the regulatory limits, a caking tendency of around 8% can still be considered as a necessarily good performance in the environmental point of view. According to the sustainable development goals, it is major importance to minimize waste generation through

recycling and re-use [33]. Therefore, the utilization of these bio-based products should be encouraged.

After the anticaking application, the amount of cake in the fertilizer was sieved and weighed to determine the amount of caking in the fertilizer. Fertilizer particles, which are crushed by pressure and turned into powder during small-bag storage tests, form a cake. In other words, the measured pellets actually contain crushed manure particles. Therefore, the analysis of agglomerates also refers to crushed, dusted, and unsuitable fertilizer particles. The crushing test can be done by selecting coated fertilizer granules one by one; However, this simple test showing the crushing resistance of a single granule may not be as useful as the result of the small-bag test.

It is highly important to underline that the coating materials do not change the physicochemical properties of fertilizer particles in order to inhibit the phase contact, they are coated externally to improve the physical properties of the fertilizer particles by creating an interface between the fertilizer particles in order to keep their contact in a minimum. Application of these external conditioners helps to control the degradation rate in cost-effective manner.

The coating materials were coated on the fertilizer sample after physically melting, heated in a water bath and also in a rotary chamber kept at room temperature. Sudden temperature changes during coating application can cause partial solidification of the samples, affecting the efficiency of the coating. The inhomogeneous distribution of coatings on fertilizer granules can also be an important parameter in determining the caking performance of the coating. The coating distribution can be observed visually during coating. The coating is prevented from having an inhomogeneous structure. However, technically, surface coating distribution and coating efficiency cannot be measured in fertilizers. The lack of this test causes difficulties in our work. Whether the coating is complete or not can only be examined visually or can be calculated on the basis of weight. However, there is no test that can see the coating distribution on the fertilizer granule surface or measure the coating thickness.

From the past to present, the addition of petroleum derivatives to improve the quality performance of nitrogen fertilizers is a type of chemical agent that researchers have focused on. The good penetration of petroleum derivatives on nitrogenous fertilizer surfaces with porous surface and their high surface tension in terms of oxygen transfer have caused the frequent use of petroleum derivatives in the surface coating industry until today. However, due to the destruction caused by petroleum derivatives such as fuel oil on the soil, today the trend towards petroleum derivative chemicals with alternative chemical content, amine based. In this study, instead of petroleum-based coating materials having high performance in inhibiting caking but with dissolution problems, a comparison of alternative antiquarian chemicals has been made, and thus

an important alternative chemical evaluation has been provided to solve the problem of caking. However, as suggested by organizations that play an active role in setting the standards, such as EFMA (European Fertilizer Manufacturers Association), determining concentrations such that the application rate of any coating chemical used does not exceed 1% is important in terms of the results obtained.

4. Conclusion

Fertilizer performance on crop yield is highly important to meet global food demands, thus it is a great importance to maintain the fertilizer products' physical quality during storage and transportation. Caking is an important problem in the fertilizer industry, and the application of anticaking agents mainly inhibits the phase transition and lowers the friction effect, thus preventing the formation of agglomerates.

This study gives a brief summary of the anticaking performance of commercially available coating products on NP 20-20-0 type fertilizer, basically classified as three different groups: petroleum-based, bio-based and alkylamine-fatty acid containing mixtures. Experiments were conducted in a 10-day test period. Temperature and RH % of the experiment environment were recorded daily, and calculated as 26 °C and 47.2 %, approximately. According to test results, petroleum-based coating agents and alkylamine-fatty acid mixtures have similar performances. Oil products derived from fatty acid alkyl esters and biodiesel products seem to decrease the caking rate of fertilizer more effective, and bio-based coating agents showed a moderate performance; however, these products are advantageous in an environmental manner, since they are derived from vegetable-based wastes.

Fertilizer sample used in the study contains both ammonium sulfate and urea as nitrogen sources, and the mixture of these salts approximately has a critical relative humidity of 55% at 30 °C. Below this temperature, CRH value tends to increase. Since the average temperature is 21.74 °C (below 30 °C) during the test period, it can be concluded as the ambient physical conditions were not as effective as the coating samples on caking.

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Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.

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Ethical approval: All ethical guidelines have been adhered.

Sample availability: Samples of the compounds are available from the author.

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