CI&CEQ
Chemical Industry & Chemical Engineering Quarterly

Available online at
Association of the Chemical Engineers of Serbia *AChE*
www.ache.org.rs/CICEQ

Chem. Ind. Chem. Eng. Q. 30 (2) 143–149 (2024) CI&CEQ

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SCIENTIFIC PAPER

UDC 631.84:66:502/504

ECO-FRIENDLY COATINGS COMPOSED OF LIGNOSULFONATE-MODIFIED BIOPOLYMER AND VEGETABLE WAXES FOR NITROGENOUS FERTILIZERS

Article Highlights

- Biopolymer and vegetable oil coatings are strong alternatives to paraffinic fertilizer coatings
- Biopolymers perform better anti-caking effects than petroleum derivative coatings
- Vegetable coatings showed a significant moisture-repellent behavior
- The polymeric coatings showed high efficiency in both mechanical strength and hydrophobicity

Abstract

Fertilizer coatings are considered mandatory to protect the physical quality of fertilizer granules. They continue to be developed due to compliance with novel fertilizer types and, most importantly, future environmental and animal-plant health regulations. As known, bio-based contents are sustainable and eco-friendly compared to petroleum-based materials. However, many types of coatings are commonly composed of unsustainable, costly, and can be ecologically toxic, such as paraffin or mineral oil. This article presents a comparative research study to provide eco-friendly anticaking coatings composed of lignosulfonate-modified biopolymer and vegetable waxes instead of conventional coatings. This research mainly aims to find alternative ingredients instead of a petroleum-derivatives in conventional coatings. According to the results, an anticaking coating containing lignosulfonate-modified biopolymer improved the granule structure of calcium ammonium nitrate fertilizer. It showed the best anticaking performance compared to other coating types. Vegetable-based coatings, on the other hand, gave results in appropriate intervals, especially at low concentrations, and showed a valuable way to develop better versions in future studies. As a result, it is seen that biopolymers can replace paraffin-based products.

Keywords: anticaking coatings, caking, bio-based coatings, crushing strength, moisture absorption.

The fertilizer industry depends on agriculture and has seasonal sales; therefore, the products are shipped in large quantities and stored for long storage times. It is quite possible to encounter quality problems such as degradation, dusting, water uptake, and, accordingly, caking in fertilizers due to the exposed harsh climatic conditions and intense handling. The caking tendency of fertilizer is mainly caused by its characteristics such as moisture absorption capacity, crushing strength, and abrasion resistance and is related to its chemical structure. In addition, it is strongly triggered by environmental conditions where it is stored, such as temperature, humidity, and pressure [1]. Caking proceeds with the phase change and the adhesion mechanisms between contact points of fertilizer granules that show substantial caking with increasing temperature, humidity, pressure, and handling. The application of a suitable anti-caking agent controls these parameters, and the tendency to cake remains

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Paper received: 14 February, 2023
Paper revised: 1 June, 2023
Paper accepted: 25 July, 2023

https://doi.org/10.2298/CICEQ230214019K

significantly low. Consequently, chemical agents are used to minimize that caking phenomenon. External chemical agents are applied as a coating which is important to improve a hydrophobic surface against high humidity conditions, and a film layer to provide mechanical strength on fertilizer granules [2].

The most common coating agents are paraffin oil-based. Mineral oils of high paraffin content can be considered particularly more effective. Purified paraffin-based oils are distinguished from aromatic oils because they biodegrade, albeit slowly, and are not directly toxic [3]. Traditional paraffin-based coatings usually comprise various ratios of alkylamines, petroleum-based oils, fatty acids, paraffins, or waxes combined with an inert powder. All of these ingredients are strongly hydrophobic molecules. However, the use of heavy oils such as fuel oil is carcinogenic because it contains PAHs above the restrictions, and it is prohibited by regulations in many countries [4–5]. For this reason, paraffinic oils, which are a controversial issue and banned in and around the EU, are substances that may be prohibited around the whole world by future environmental regulations. Therefore, there is a need for research on sustainable alternative products [6–7]. In addition, many anti-caking materials contain fatty amines coated on the fertilizer granule within paraffin or mineral oil solvents and act as surfactants [4]. Instead of amine derivatives, lignin derivatives and vegetable fatty acid (e.g., coconut fatty acid, lauric acid) ingredients that work as surfactants to increase the hydrophobic effect are also being investigated. Moreover, lignin, the second largest biomass resource on earth, can be a sustainable and non-toxic feedstock for fertilizer coating [8].

Today, the fertilizer agents' biodegradability has become an important parameter. The use of products with the potential to create toxic effects in fertilizers is dangerous for human health and creates soil contamination. These products can be expected to be removed from the shelf in the next few years [9]. Eco-friendly coatings that are alternative to traditional coatings should have an ecologically non-toxic oil base instead of mineral oil or paraffin base, application concentrations should have a reasonable and economical price, and the granular healing performance should be at a level that can compete with commercial size paraffin-based coatings [10]. In recent years, studies have been carried out on vegetable oils as oil derivatives, biodegradable polymers, and natural by-products of vegetable oils [8, 11–12]. Vegetable oils do not contain branched and cyclic alkanes, i.e., PAHs, like mineral oils. For this reason, it draws attention to its non-toxicity and eco-friendly nature. Although it has hydrophobic solid structures, it may lag in terms of performance in a moisture absorption test compared to paraffinic oils [13–15].

Developing inhibitory mechanisms to comply with the green consensus process is essential to contribute to sustainable agricultural management due to both greenhouse gas production and ecological transformation for the fertilizer production industry and the use of produced fertilizers in agricultural areas. In this respect, there are initiatives by developed countries to develop models for sustainable development goals (SDG) within the scope of the green agreement on a global scale. Thus, models supporting the inhibitory mechanism, such as biopolymer, can be considered an important alternative to the manufacturer in the green accord compliance process.

The presented research study explains conventional amine-containing paraffin, amine-free paraffinic, biodegradable polymer, and vegetable oil-based coatings. It provides information on the possibility of replacing amine-derived coatings with plant-natural products and biodegradable polymers, which are defined as sustainable and eco-friendly in terms of their resources and following limitations from the environmental point of view. Three concentrations of each coating were tried on CAN/26 fertilizer to determine the optimum dosage for the desired coating. The coatings' performance on the fertilizer's physical properties could be compared. For CAN/26, the best coating type and ratio will be seen for different features.

MATERIALS AND METHODS

Experimental

In this study, five different commercially available coatings were applied to CAN/26 fertilizer. The coating process was applied in three different concentrations between commercially recommended minimum and maximum dosages of coating agents. The main ingredients and application rates of coating agents are given in Table 1.

All fertilizer samples were produced in Toros Agri Ind., and the research was conducted in Toros R&D Center.

The unique prill form and the diameter between 2 and 4 mm were selected for the experiments. The fertilizer granules were heated to 60 °C, and anti-caking agents were injected at around 80 °C on fertilizer. Finally, fertilizer samples were coated in a manually rotated plastic chamber, and final products were evaluated in terms of crushing strength, moisture uptake capacity, critical relative humidity value, and caking tendency by comparing with uncoated CAN/26 and CAN/26 coated with conventional coatings which

Table 1. The main ingredients and dosages of coating agents.

Fertilizer coatings	Type	Dosage	Surfactant
A	Biopolymer	500-1000-2000 ppm	Sodium Lignosulfonate in biopolymer
B	Paraffinic	500-1000-3000 ppm	Amine-free fatty acids in paraffinic oil.
C	Vegetable	500-2000-3000 ppm	Plant-based fatty acids in vegetable oil.
D	Vegetable	500-2000-3000 ppm	Plant-based natural polymers in water.
R (Reference)	Paraffinic	500 ppm	Amines in paraffinic oil.
E	Petroleum-based	<1000 ppm	3-10% Alkylamine + 2.5-10% Organic P-compounds

have paraffines and alkyl amines. With this aim, crushing strength, moisture absorption, and accelerating caking tests were done. All tests based on granules were run in at least ten parallels, while the other tests were run in 3.

Crushing strength analysis

Crushing strength (CS, kg/granule) is the maximum load at which 2 mm fertilizer granule can sustain. It was measured with universal test equipment by applying a compression pressure on one granule until it collapsed [16]. In this study, a crushing strength testing machine, Lutron FG-5000A, was used. Average crushing strength values were determined by testing ten granules for each fertilizer sample.

Moisture absorption test

Moisture absorption test (MA, wt.%) was performed in a JSR JSPC-200C model climate-controlled chamber by exposing fertilizer samples to a specific humidity range at a constant temperature. In this study, around 30 g of fertilizer samples were loaded in the cylindrical volumetric vessels, and the samples were kept at 25 °C and 40, 50, 60, and 70% humidity values each for 3 hours [17].

It was calculated as the moisture uptake capacity from the highest amount of moisture the fertilizers can hold. In addition, the relative atmospheric humidity value at which the fertilizers tested at a constant temperature increasing humidity value first started to retain moisture was determined as the critical relative humidity value of that fertilizer.

Accelerating caking test

An accelerated caking test is a procedure to evaluate the caking tendency of fertilizer over time by simulating storage conditions. To simulate the conditions of storage, transportation, and handling of fertilizers, the samples were exposed to a climatic cycle in the climatic chamber. In this study, fertilizer samples, which were prepared in polyethylene sample bags for the accelerated caking test, were kept in a JSR JSPC-200C model climate-controlled chamber for 32 h at 32 °C and 80% relative humidity conditions and then three days under the conditions, 40 °C and 20% relative humidity [18]. After climatic conditioning, the fertilizers were stored under a pressure of 0.28 kg/cm^2 for three days in a conventional small bag storage test, as shown in Figure 1. In the small bag storage test, dummy weights were used to apply a pressure of 0.28 kg/cm^2 on the surface of the lowest fertilizer bag. This test represented a situation where fertilizer was stored in bags at a standard suitable pressure which is a standard suitable pressure to keep fertilizer in good quality. After the test, caking tendencies were calculated by measuring the caking parts in the fertilizers.

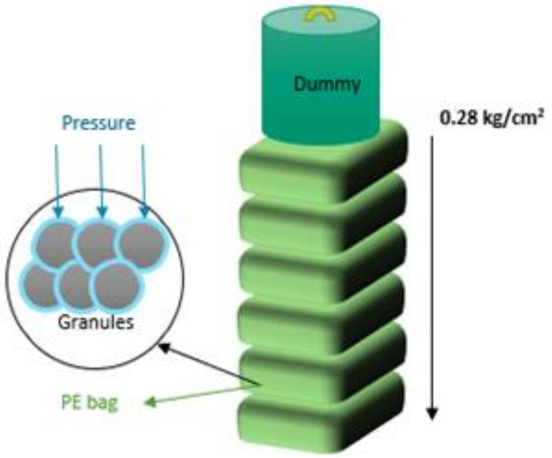

Figure 1. Small-bag storage test scheme.

RESULTS AND DISCUSSION

The crushing strength results of prepared fertilizer samples with various types of coatings at different dosages are given in Table 2. A crushing strength test was employed to determine the effect of coating type, coating ratio, and climatic cycle on the crushing strength of CAN/26.

According to the results, the uncoated fertilizer sample has the weakest granules, probably due to any coating providing a thin film cover on the granules against mechanical interactions and handling. In general, the crushing strength of fertilizer granules is

Table 2. Crushing strength test results for fertilizer samples.

Coating	Dosage (ppm)	CS (kg/granule)	CS after climatic cycle (kg/granule)
Uncoated	-	1.72	1.55
	500	2.24	1.77
A	1000	2.39	2.35
	2000	2.50	2.03
	500	2.46	1.96
B	1000	2.29	1.84
	3000	2.19	2.15
	500	2.46	2.16
C	2000	2.30	1.98
	3000	2.48	1.79
	500	2.43	1.93
D	2000	2.38	2.06
	3000	2.59	2.39
R	500	2.60	1.95
E	<1000	1.41<CS<2.70	<2.40

expected to be at least 2 kg/granule [3]. However, after the climatic cycle test, some fertilizer samples showed less than 2 kg/granule crushing strength, which is probably due to the low moisture resistance of their coating. Thus, although all coatings provided mechanical film protection, the protection of most of the coating decreased by about 10-20% when exposed to moisture and temperature.

After the climatic cycle, it can be said that the coatings are compatible with good performance for the fertilizer samples that still have a crushing strength of more than 2 kg/granule. The fertilizer sample coated with 1000 ppm of biopolymer coating-A shows 2.35 kg/granule crushing strength after the climatic cycle, probably due to the intermolecular forces such as van der Waals among the polymeric chains [12]. The presence of long-chain polymers in the coating enhances the adhesion of the coating on the fertilizer surface by increasing a polymer-salt interaction by forming a hydrogen bonding network [19–20]. The fertilizer coated with 3000 ppm of amine-free paraffinic coating-B shows a competitive crushing strength of 2.15 kg/granule, while the fertilizer coated with 500 ppm reference coating, which contains amine and paraffines, has 1.95 kg/granule after the climatic cycle. Since it does not contain amines which are moisture-repellent supportive compounds, its performance was good at a higher coating rate. Both coatings (B & R) have paraffins, which are hydrophobic mixtures of linear and branched alkanes with a carbon number of 20–40 that show moisture-repellent properties. Petroleum waxes contain mostly branched alkanes with a carbon number of 30–80, and most anticaking agents are paraffin-based, requiring highly branched alkanes [21]. However, although it was effective, B-coating may not be economically interpreted as a 3000 ppm dosage.

According to the complete vegetable coating materials, 500 ppm of coating-C and 2000–3000 ppm of coating-D have good results for crushing strength, such as 2.16, 2.06, and 2.39 kg/granule, respectively. Compared with paraffinic and biopolymeric coatings, the durability of vegetal-based coatings is competitive, probably due to vegetable coatings having branched chains that enhance coating-fertilizer surface adhesion. Vegetable oils contain glycerol trailers from C12–C24 fatty acids, and several double bonds are susceptible to crosslinking or polymerization reactions [22]. Basically, the function of a coating is to cover the granule with a protective film layer in order to avoid interactions between granules [23]. Therefore, it can be pointed out that the compatibility between coating and fertilizer surface enhances granules' mechanical resistance and gives relatively higher crushing strength.

Moisture absorption and CRH results of prepared fertilizer samples with various types of coatings at different dosages are given in Table 3. Moisture absorption tests of CAN/26 were carried out to understand the interaction of fertilizers with moisture and to evaluate the effect of coating type and coating ratio on this situation.

Table 3 shows that biopolymer-coated fertilizers kept above the critical humidity value (at 70% RH) retained a maximum moisture content of 1.39-1.41%. A 1.58% moisture uptake was observed in the fertilizer to

Table 3. Moisture uptake and CRH values for fertilizer samples.

Coating	Dosage (ppm)	Moisture uptake% @70 RH	CRH @25 °C
Uncoated	-	2.40%	60%
A	500	1.39%	58%
	1000	1.40%	58%
	2000	1.41%	58%
B	500	6.29%	58%
	1000	1.38%	58%
	3000	1.31%	58%
C	500	1.46%	60%
	2000	1.37%	58%
	3000	2.95%	52%
D	500	1.42%	58%
	2000	1.59%	52%
	3000	2.67%	52%
R	500	1.58%	60%
E	<1000	0,7%	60%

which the reference coating was applied. All concentrations of the biopolymer coating-A showed better moisture repellency than the reference paraffin coating. It can be due to the good adhesion of polymeric coatings on the fertilizer surface with crosslinking reactions and the lignin-based moisture-repellent components, which have the potential for use in anticaking agents for fertilizer due to hydrophobicity, bonding ability, and film formation capability [24]. On the other hand, it has been observed that the amine-free paraffin coating performs better than the amine-containing coating. It shows that some fatty acid derivatives, such as coconut and lauric acids, which have a very high moisture-repellent function, can replace amine contents. Vegetable coatings' moisture-repellency performance was found to be good at most dosages. Due to the hydrophobic nature of the high content of vegetable fatty acids, vegetable oils showed a significant hydrophobic effect [25–26]. Table 4 shows accelerating test results of prepared fertilizer samples to determine the effect of the coating type and coating concentration on free-flowing fertilizer.

Biopolymer coatings cover the surface in a way that does not allow the passage of air molecules on the particle surface due to its surface wetting or surface tension-increasing properties. In addition, the film layer formed on the fertilizer surface is more advantageous than other organic coating chemicals due to the cross-linking feature of biopolymers, that moisture molecules reach the fertilizer surface. Because the effect of the cross-link structure to slow down moisture diffusion has also been reported in the literature [27]. Considering the results of the accelerated caking test, fertilizer samples were coated with 500 ppm of amine-free paraffinic coating-B, 500 ppm of vegetable coating-C, and all concentrations of biopolymer-containing coating-A

Table 4. Accelerating test results of prepared fertilizer samples.

Coating	Dosage (ppm)	Caking(%)
Uncoated	-	34.70
A	500	2.52
	1000	2.40
	2000	3.00
B	500	3.63
	1000	15.00
	3000	13.72
C	500	7.71
	2000	21.17
	3000	31.58
D	500	23.10
	2000	49.75
	3000	44.58
R	500	3.14
E	<1000	<5

were found to be in proper free-flowing. It has been determined that the biopolymer coating performs better than other coatings in reducing the tendency to caking. Even after the climatic cycle, the granule quality is within appropriate intervals, and the tendency to cake is below 3%. It is because polymer coatings and lignin-based surfactants enhance the resistance to crushing and moisture absorption [11,26]. When amine-containing and non-amine-containing paraffin coatings were compared, the caking tendencies of the 500 ppm applied fertilizers were around 3% and close to each other. Many anti-caking materials contain fatty amines that cause the displacement of ammonia by reacting with ammonium salts in the nitrogenous fertilizer, which ensures a good coating adhesion. It has been observed that crystals form on the surface of fertilizer coated with fatty amine when subjected to high humidity cycles. These crystals may spill from the surface and cause dust formation in the future. Although similar anticaking performance has been obtained, it has been reported that dust formation can occur on the surface, especially in ammonium nitrate fertilizers, in amine-containing coatings, but this dusting risk does not exist in coatings containing fatty acids that do not contain amines [28]. When the anticaking performance of the coatings produced entirely with vegetable ingredients was evaluated, it was seen that the low-concentration coatings of 500 ppm vegetable-based products performed slightly better. Vegetable coating-C was more effective on caking tendency than coating-D. Since the vegetable D-coating is water-based, it has been determined that it penetrates and easily dissolves the fertilizer structure at high concentrations and causes caking.

CONCLUSION

In this study, three coating amounts were tried for each coating material in CAN/26 fertilizer, and optimum concentrations were determined. For different properties of CAN/26, the best coating type and ratio was determined as 1000 ppm biopolymer coating ratio. When the physical strength properties of CAN/26 fertilizers prepared with different coatings were compared, the highest tensile strength was obtained in vegetal-containing coatings; however, the agglomeration tendency was observed to increase at high concentrations of these coatings. It was determined that the critical humidity value of CAN/26 for coating types decreased. However, uncoated CAN/26 fertilizer can hold up to 2.4% of its weight when moisture retention is taken into account; In coated fertilizers, this ratio has generally decreased below 1.5%. The coating type and ratio with the best moisture repellency can be listed as 1000 and 1200 ppm amine-free paraffinic coating and 1000 ppm biopolymer coating. It has been observed that the vegetal-containing coatings show higher performance in terms of agglomeration and moisture repellency, especially in low-concentration applications such as 500 ppm for CAN/26 fertilizer. The 500 ppm concentration of the oil-based vegetable coating showed an anti-caking performance of about 7%. Finally, a comparison with petroleum-based formulas would be an excellent addition.

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NAUČNI RAD

EKOLOŠKI PREMAZI ZA AZOTNA ĐUBRIVA OD LIGNOSULFONATOM MODIFIKOVANOG BIOPOLIMERA I BILJNIH VOSKOVA

Premazi za đubriva se smatraju obaveznim za zaštitu fizičkog kvaliteta granula đubriva. Ona nastavljaju da se razvijaju zbog usklađenosti sa novim tipovima đubriva i, što je najvažnije, budućim propisima o zaštiti životne sredine i zdravlja životinja i biljaka. Kao što je poznato, sadržaji na bazi bioloških materijala su održivi i ekološki prihvatljivi u poređenju sa materijalima na bazi nafte. Međutim, mnoge vrste premaza se obično sastoje od neodrživih, skupih i često ekološki toksičnih, kao što su parafin ili mineralno ulje. Ovaj članak predstavlja rezultate istraživanja ekološki prihvatljivih premaza protiv zgrušavanja koji se sastoje od biopolimera modifikovanog lignosulfonatom i biljnih voskova umesto konvencionalnih premaza. Ovo istraživanje uglavnom ima za cilj pronalaženje alternativnih sastojaka umesto derivata nafte u konvencionalnim premazima. Prema rezultatima, premaz protiv zgrušavanja koji sadrži biopolimer modifikovan lignosulfonatom poboljšao je strukturu granula đubriva kalcijum-amonijum-nitrata. Pokazao je najbolje performanse protiv zgrušavanja u poređenju sa drugim tipovima premaza. Premazi na bazi povrća su, s druge strane, dali rezultate u odgovarajućim intervalima, posebno pri niskim koncentracijama, i pokazali vredan način za razvoj boljih verzija u budućim istraživanjima. Jasno je da biopolimeri mogu zameniti proizvode na bazi parafina.

Ključne reči: premazi protiv zgrušavanja, zgrušavanje, premazi na biološkoj bazi, čvrstoća na drobljenje, apsorpcija vlage.