#### **ORIGINAL ARTICLE**



# Encapsulated organic-mineral fertilizers with nanoporous structure

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#### Abstract

Research to obtain a long-acting encapsulated fertilizer has been carried out. The performed physicochemical studies confirmed the production of a high-quality phosphate-containing shell on the carbamide prill surface. Solubility of proposed samples were analyzed. The effectiveness of the developed compositions of the encapsulated fertilizer shell was determined. Studies in soil columns were carried out to assess nitrogen leaching. The method of scanning microscopy with elemental microanalysis was used to study the quantitative and qualitative characteristics of the shell and the interface between the outer phosphate layer of the coating and the surface of the urea prill. It is shown that the presence of deep tortuous nanopores in the structure of the phosphate-containing coating allows the penetration of soil moisture into the granule and the successive dissolution of the organic part of the shell and the nitrogen core of the encapsulated granule.

Keywords Organo-mineral fertilizers · Surface structure · Superphosphate shell · Carbamide core · Pan granulator

# Introduction

The concept of green innovation advancement, aimed at ensuring sustainable and inclusive growth, causes the need to transform the approaches to rural development, encompassing sustainable farming, management of natural resources, soil protection (Plyatsuk et al. 2019), and bioagriculture (Kurbatova and Hyrchenko 2018; Kurbatova and Perederii 2020; Prokopenko and Shkola 2014). Moreover, the new growth strategy European Green Deal (European Green Deal 2021) is targeted at protecting, conserving, and enhancing the EU's natural capital, and protecting the health and well-being of citizens from environment-related risks and impacts (Sotnyk et al. 2020). Therefore, the issues of soil protection, reduction of anthropogenic pressure on the environment caused by using mineral fertilizers, and improvement in food security are urgent and need to be addressed in terms of forward-looking perspectives (Shkola et al. 2016).

Mineral, organic, and organo-mineral fertilizers (OMF) are widely used in agriculture. Effect of applying mineral fertilizers has studied by Mattiello et al. (2021). Negative impact of nitrogen fertilizers on the environment and human health owing to the gases emitted has scrutinized by Bai et al. (2021). The specifics of using organic fertilizer (nanovermicompost) under conditions of drought, causing reduced mineral uptake, has examined by Ahanger et al. (2021). Simultaneously, nano-vermicompost application compensates for the decline of mineral elements, notably nitrogen, potassium and phosphorous.

Given far-looking the ambition of the European Union as a global leader on climate and environmental measures, as well as consumer protection, to achieve at least 25% of the European Union's agricultural land under organic farming and a significant increase in organic aquaculture by 2030, organo-mineral fertilizers are the most promising for the agricultural application and offer several advantages over organic or mineral fertilizers, which are used separately (Klyosov and Orekhovskaya 2021; Vakal et al. 2021). An attempt to use NPK elements, released from wet blue leather having been the waste of leather industry, and potato peel, while transforming into an eco-friendly bio-organic NPK fertilizer showed that content of the released free ammonia in soil with bio-organic NPK has been six times more than with chemical fertilizer (Majee et al. 2021).



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Organo-mineral fertilizers could be obtained by mixing of carbamide and minerals with dried manure, but a disadvantage of this organo-mineral fertilizer is that the release of nitrogen from urea and minerals is very fast (Patent US 2007/0095118A1 2007). In modern fertilizers, a slowrelease of carbamide is desired (Crusciol et al. 2020).

The most common method of modifying the fertilizer product to provide controlled nutrient release is to control the solubility of the fertilizer. In the case of carbamide, such products can be made by its reaction with aldehydes. Factors such as soil moisture, pH, temperature, oxygen, and nutrient content influence the rate of nitrogen release from N-containing fertilizer (Crusciol et al. 2020). Fertilizer granules must have sufficient mechanical strength to withstand normal handling and storage without significant fracturing and creation of excessive dust. The abrasion resistance and high static strength are required to prevent formation of fine particles and caking problems (Crusciol et al. 2020). Water sorption from the soil by the fertilizer granules is also important as the granule water content determines the rate of dissolution and chemical diffusion (Thapa et al. 2017; Artyukhov et al. 2020; Jafari 2017). Soluble fertilizers could be coated in different ways to adjust the nitrogen release: with impermeable membranes, with Sulphur, lignosulphonate, acrylonitrile, or polymer coatings (Patent US 2007/0095118A1 2007). At present, methods of encapsulating nitrogen fertilizers have received significant development not only with coatings, which are, as a rule, ballast, but coatings that are fertilizers themselves. Methods of encapsulation of urea and ammonium nitrate, superphosphate, and ammophos are known.

Release of the nutrients can be varied by changing the thickness and composition of the coating. The dissolved fertilizer diffuses through the enlarged pores in the coating.

The OMF are long-release fertilizers with slow dissolution rate of the shell and rapid dissolution rate of the granule core. The rapid dissolution of the nitrogen core of the granule is slowed down, mainly due to the mineral composition of the coating, and lasts from several days (first release of the nutrient) to several months, during which carbamide is likely to be released.

The release times depend on the product composition, texture, size, pH of the soil, the soil moisture, the soil temperature and the presence of microorganisms, etc. (Klyosov and Orekhovskaya 2021). One of the first and most significant steps in the practice of encapsulation technology is to find suitable material for encapsulation. This choice is depending on the active component and the acceptable properties of the final product. These characteristics can be reconciled concerning the physicochemical structure of the material in the capsules, the encapsulated product's storage conditions, the scale and density of the materials to be coated in the process, and the economic cost of production



(Artyukhov et al. 2020; Jafari 2017; Kotenko et al. 2019; Vakal et al. 2020a, b). For the production of organo-mineral fertilizers with the shell composition, which is discussed in this article, the most effective is the method of pelletizing in a pan granulator (Artyukhov et al. 2020). In this article, we propose multicomponent organo-mineral fertilizers based on carbamide granules with phosphate shell with potassium and calcium humate as a plasticizer, and biochar as the addition to the shell. The composition and structure of obtained fertilizers were investigated. Due to this unique combination of slow dissolution of the phosphate shell and rapid dissolution of the urea core, a controlled release of organic fertilizer was obtained.

The choice of a pan granulator as the main equipment for encapsulation is justified on the basis of a comparative analysis of granulation equipment of various types (Shanmugam 2015; Muralidhar et al. 2016; Sahoo et al. 2016; Suresh et al. 2017; Obodiak et al. 2020) with different degrees of turbulence of flows (Artyukhov and Artyukhova 2019).

# Experimental

# Materials

As the core of the granule, we used a commercial fraction of prilled urea containing 46.2% nitrogen with preliminary separation of granules with a size of 2–3 mm. To form a phosphate-containing shell, a phosphate–glauconite concentrate of the Novo-Amvrosiivsky deposit with a  $P_2O_5$  content of 15% and a  $K_2O$  content of 1.5% was used. The concentrate was preliminarily grinded before passing through a 0.2 mm sieve.

Currently, the only phosphorite deposit in Ukraine is the Novo-Amvrosiivsky deposit, where industrial extraction and enrichment of raw materials is carried out (Jafari 2017) [7].

To assess the phosphate raw material quality of this deposit and the possibility of its application in the production of phosphorus-containing mineral fertilizers, appropriate physicochemical studies were conducted.

The data of radiographic studies of Novo-Amvrosiivsky phosphorite sample (Fig. 1) allow us to conclude that the main crystalline phase of this phosphorite is fluorapatite  $Ca_5F(PO_4)_3$  in crystal-amorphous form; the mineral also contains impurities of dolomite (CaMg(CO<sub>3</sub>)<sub>2</sub>) and quartz.

The results of chemical analysis of Novo-Amvrosiivsky phosphorite are given in Table. 1.

Ballast potassium and calcium humates obtained on a model homogenizer were used as a plasticizers. The humate solutions were filtered through a 0.125 mm sieve. The chemical composition of potassium and calcium humates is shown in Table 2.



**Fig. 1** XRD analysis of Novo-Amvrosiivsky phosphorite (initial angle=10; final angle=70; step=0.2; exposure=2.0; speed=1; intensity=6584). Main phases: **1** SiO<sub>2</sub>–Silicon Oxide; **2** CaCO<sub>3</sub>–Calcite; **3** Ca<sub>5</sub>F(PO<sub>4</sub>)<sub>3</sub>–Fluorapatite

Table 1 Physico-chemical   parameters of Novo-	Indicator name and measurement unit		Test results
Amvrosiivsky phosphorite	1	Mass fraction of total phosphates (sum of fast- and slow-acting forms of $P_2O_5)~(\%)$	
	2	Mass fraction of digestible phosphates in terms of $P_2O_5$ (%)	16.48
	3	Mass fraction of total potassium in terms of K <sub>2</sub> O (%)	2.2
	4	Mass fraction of water (%)	0.62
	5	Mass fraction of Cd (mg/kg)	4
	6	Mass fraction of Pb (mg/kg)	2
	7	Mass fraction of As (mg/kg)	1
	8	Balance on the sieve	
		With a grid № 2 K (%)	0
		With a grid № 02 K (%)	37
	9	The effective specific activity of natural radionuclides (Bq/kg)	370

Indicator name and measurement unit		Test result		
		Potassium humate	Calcium humate	
1	Mass fraction of water (%)	83.8		88.2
2	Mass fraction of humates in terms of dry matter (%)	18.18		19.81
3	Suspension pH	9.63		9.8

According to IUPAC classification, in the nanometer size range, micro- (less than 2 nm), meso- (2–50 nm), and macropores (more than 50 nm) can be distinguished. This work is devoted to the study of mesopores and macropores in the granule structure.

Within the framework of the research, the task was set to study the structure and size of nanopores, which should freely pass water (molecule size 0.28 nm) into the organic shell, and then (after the shell dissolves at a given rate) to the core (mineral fertilizer) of the granule. Obtaining data on the structure of nanopores determines the further applied problem of research in nanoscale materials to predict the rate of dissolution of the organic shell.

### Methods

granulator

**Fig. 2** Scheme of a model installation based on a disk

Samples of encapsulated organo-mineral fertilizers were obtained on a laboratory installation based on a disk granulator (Fig. 2).

The installation consists of containers of phosphate powder 1, potassium or calcium humate 2 and urea 3, dispenser 4, pan granulator 5, and pallet 6. Minor amounts of biochar can be added to container 1. The consumption of urea, phosphate–glauconite concentrate, potassium or calcium humate, and biochar was determined by the weight method on a VLKT-500 balance. Screening of the encapsulated product was carried out on a RKF-2U device with the release of a commercial fraction (2–5) mm. The resulting product samples were dried in an SNOL-3.5.3.5.3.5 / 3.5-I1 drying oven. The strength of the obtained samples of the encapsulated product was determined using an IPG-1 M device according to standard methods. Samples of the resulting product were sent for physical and chemical research.

Investigation of the surface morphology and internal structure of the granule (linear size and shape of pores, surface microrelief of various objects): scanning electron microscope SEO-SEM Inspect S50-B.

Features of SEO-SEM Inspect S50-B:



Table 3	Chemical	analysis	of enca	psulated	carbamide
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Indicator name and measurement unit		Test result			
		Carbamide with phosphate shell and potassium humate	Carbamide with phosphate shell and calcium humate	Carbamide with phosphate and biochar shell and potassium humate	
1	Mass fraction of nitrogen (%)	21.36	23.40	25.62	
2	Mass fraction of phosphorus in terms of $P_2O_5$ (%)	8.1	8.2	8.9	
3	Mass fraction of potassium in terms of $K_2O$ (%)	0.39	0.13	2.2	
4	Mass fraction of water (%)	0.79	0.45	0.15	
5	Static strength, MPa	1.54	1.36	1.06	





Fig. 3 SEM images of surface: a degree of coating uniformity; b non-porous surface elements and "mechanical" pores; c the presence of nutrient inclusions; d tortuous pores

• AZtecOne energy dispersion spectrometer with X-MaxN20 detector (manufacturer Oxford Instruments plc);

• source of electrons in the gun:tungsten cathode with thermo-electron emission.

# Characterization

The process of encapsulating urea prill was carried out as follows. Powder of phosphate-glauconite concentrate was supplied to the prills of carbamide, pre-moistened with potassium or calcium humate, in the tray of the granulator, during parallel moistening with a plasticizer. To study the diffusion properties of the shell, a metered amount of biochar was introduced into the phosphorite powder. The ratio of the coating weight to the carbamide weight was kept equal to 1. The granulation process on the plates was visually controlled. The moisture content of the encapsulated carbamide at the outlet from the plate of the granulator was 6.42%. The wet granules were dried for 3 h at 65 °C. The product was dispersed to obtain a commercial fraction





Fig.4 SEM image of granule cross-section: 1 phosphate shell, 2 interface between shell and core, and 3 carbamide core

of 2–5 mm. The chemical composition of the sample is presented in Table 3.

Due to the additional preparation of humate and more uniform wetting of the prill surface during its encapsulation, the surface of the phosphate-containing coating had a denser structure and visually differed from the samples of previous batches.

To determine the effectiveness of the developed compositions of the encapsulated fertilizer shell, studies in soil columns were carried out to assess nitrogen leaching. Five washes were performed according to the standard procedure (Vakal et al. 2020b). During the first wash, the amount of nitrogen washed from the shell with the biochar and calcium humate as a plasticizer was 2.3 times less than standard urea and 1.5 times less than the phosphate-containing shell, with the potassium humate as a plasticizer. The fourth and fifth wash showed that the amount of nitrogen from the shell with a potassium humate plasticizer and pure urea are at the same level, and from the granule with a shell with a plasticizer humate calcium and biochar exceeded 1.25 times that confirms the effectiveness of the developed composition of the shell.

# **Results and discussion**

### SEM investigation of organo-mineral fertilizer

Evaluation of the efficiency of the obtained phosphorus-containing shell required studying the quantitative and qualitative characteristics of the shell and the interface between the outer phosphate and inner (urea prill surface) layers by scanning microscopy with elemental microanalysis.



The quality assessment of the structure of the shell and interface was carried out according to the following indicators:

- uniformity of coating on the core surface and uniformity of coating thickness;
- uniformity of the nanoporous surface of the shell;
- the presence of winding curved pores for the potential penetration of soil moisture into the shell;
- absence or small proportion of "mechanical" pores resulting from mechanical action on the granule and/or temperature stresses in the core of the granule;
- reliability and uniformity of the attachment of the shell to the granule core;
- the presence of nutrients from the shell in the granule core.

This type of study of surface morphology and internal structure has proven to be effective when the authors of the article studied other coatings, for example, on porous ammonium nitrate after the stage of moistening and heat treatment (Artyukhov et al. 2017).

The surface of an encapsulated granule at a magnification of  $\times 200$  and  $\times 1600$  is presented in Fig. 3.

The study of the surface morphology of the encapsulated sample shows that the phosphate coating is uniform over the surface without large differences in thickness ( $\pm 10$  from the average value). On the surface, there are practically no coarse inclusions of the phosphate-glauconite concentrate, but there are inclusions of organic fertilizer, which are reliably fixed in the shell and do not have a tendency to chipping at external mechanical influence. This fact is important to ensure the transport of pellets from the place of manufacture to the place of soil fertilization. At the same time, sinuous nanopores are present on the granule surface, and the area of this surface is 45–55% of the total surface of the granules. This is sufficient for the successful penetration of moisture into the shell and its dissolution. There are no "mechanical" pores on the surface, which may appear due to thermal stresses during heat treatment of the granule.

The interface between the phosphate-containing coating boundary and the prill surface is shown in Fig. 4. Position 1 denotes the area of the phosphate-containing coating, position 2 denotes the interface of the coating and the core of the granule, and position 3 denotes the core of the granule.

As follows from Fig. 4, in the encapsulated granule, the transition zone (2) has a certain thickness due to the interpenetration of the dissolved part of the prill surface and the phosphate-containing shell. It is the uniform thickness of this zone that ensures the coating adhesion and the strength of the encapsulated granule itself, as well as the diffusion properties of the coating.



Fig. 5 SEM images of phosphate granule coating:  $\mathbf{a}$  cross-section of the coating;  $\mathbf{b}$  phosphate shell,  $\times 1600$ ;  $\mathbf{c}$  interface,  $\times 800$ 

The main barrier that characterizes the prolonging action of the encapsulated granule is the phosphate-containing coating. Thus, the study of its structure and the quality of adhesion to the prill surface is especially urgent. The shell structure at  $\times 200$ ,  $\times 1600$  magnification and the interface of the shell contact with the prill surface at  $\times 800$  magnification are shown in Fig. 5. The study of Fig. 5 allows us to conclude that there is a uniform nanoporous structure throughout the entire shell thickness. A network of sinuous nanopores can provide moisture penetration throughout the entire thickness of the shell. The presence of phosphate particles in the shell creates the necessary frame of the shell and ensures its strength, which is illustrated by the higher static strength of the encapsulated granule in comparison with the urea prill. Figure 5c illustrates a fairly homogeneous structure of the transition zone from the shell to the core of the granule with an almost complete absence of pores due to the interpenetration of the dissolved part of the prill surface and particles of phosphate–glauconite concentrate and potassium humate. This structure ensures gradual penetration of soil moisture into the granule core and allows for a sequential mechanism of dissolution, first of the shell





Fig. 6 SEM images of nanoparticles, with distribution of elements: a cross-section of granule; b granule interface with coating elements on the core

pores, and then the granule core. The absence of defects in this part of the granule ensures reliable adhesion of the shell to the prill surface.

The study of the distribution of chemical elements over the volume of the encapsulated granule, both in the coating layer and in the transition zone and the structure of the granule core (Fig. 6), shows that the greater dissolution of the granule surface allows the nitrogen component to migrate into the depth of the coating. Outside the transition zone, the formation of the coating occurs mainly by the mechanism of agglomeration of the phosphate–glauconite concentrate using a plasticizer.

The conducted studies on the modernization of the technology of carbamide granule encapsulation with phosphate–glauconite concentrate using a plasticizer—potassium and calcium humates, and biochar made it possible to obtain samples of organo-mineral fertilizers with prolonged action with improved physicochemical characteristics.

# Conclusions

1. The proposed technology of capsulated fertilizer formation allows to get uniform nanoporous structure on the surface and in depth of the shell.

- 2. Improving the physicochemical properties of the encapsulated granule is reached by achieving a homogeneous transition layer in the contact zone of the shell and the granule core.
- 3. The network of deep sinuous nanopores allows the penetration of soil moisture into the granule and sequentially dissolves first the organic part of the shell, and then the nitrogen core of the encapsulated granule.
- 4. The granules are without mechanical defects; the shell is tightly attached to the core. Such granules can be transported to the soil fertilizer site without loss of strength properties.
- 5. Due to the presence in the batch of a small number of granules with a nonuniform shell, the further improvement of the proposed in this article technology for the production of organo-mineral fertilizers is required.
- 6. To determine the effectiveness of the developed compositions of the encapsulated fertilizer shell, studies in soil columns were carried out to assess nitrogen leaching. After fourth and fifth wash, the amount of nitrogen from the shell with a potassium humate plasticizer and pure urea were at the same level, and from the granule with a shell with a plasticizer humate calcium and biochar exceeded 1.25 times that confirms the effectiveness of the developed composition of the shell.



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#### Declarations

**Conflict of interest** On behalf of all authors, the corresponding author states that there is no conflict of interest.

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