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## **КАПСУЛЮВАННЯ ДОБРИВ ЯК ОДИН ІЗ МЕТОДІВ ЗМЕНШЕННЯ ЗАБРУДНЕННЯ НАВКОЛИШНЬОГО СЕРЕДОВИЩА**

**Анотація.** Забруднення навколишнього середовища залишками мінеральних добрив з кожним роком зростає та спричиняє погіршення і ґрунтового, і водного та повітряного середовища. Це відбувається внаслідок того, що азотні добрива є добре розчинними та швидко проникають вертикальним ґрунтовим профілем у глибинні шари, недоступні для кореневої системи рослин. Нами проведено аналіз проблеми забруднення навколишнього середовища компонентами мінеральних азотних добрив та запропоновано застосування капсульованих добрив пролонгованої дії, що дасть змогу регулювати швидкість та кількість добрив, які будуть надходити у ґрунт із капсули залежно від ґрунтово-кліматичних умов. А це, своєю чергою, дасть змогу зменшити кількість добрив, які будуть вноситися в ґрунт, та зменшити забруднення навколишнього середовища.

Проведено експериментальні дослідження вивільнення компонентів мінеральних добрив із капсул залежно від водневого показника середовища, та встановлено, що покриття азотних добрив чутливими до зміни рН середовища речовинами дає змогу в широких межах регулювати вивільнення речовин із капсул та створювати добрива залежно від типу ґрунту, де вони будуть застосовуватися.

За рахунок того, що кількість опадів у різних районах є різною, виникає проблема створення капсульованих добрив із різною товщиною покриття. Якщо опадів є багато, то, відповідно, товщина покриття має бути більшою, а в районах, де є недостатня кількість опадів, вона є дуже малою. Тому для регулювання швидкості вивільнення компонентів із капсул проведені експериментальні дослідження впливу внесення легкорозчинної кристалічної речовини до складу полімерного покриття на швидкість вивільнення компонентів із капсул. Встановлено, що такого типу покриття дають можливість застосовувати капсульовані добрива в широких межах, регулюючи швидкість вивільнення вмістом внесеної легкорозчинної кристалічної речовини. В якості якої можна використовувати або ті ж добрива, або інші легкорозчинні поживні речовини, у яких є потреба.

Проведено розрахунок коефіцієнтів масопередачі  $k$  крізь полімерну оболонку та коефіцієнтів дифузії компоненту в полімері.

**Ключові слова:** забруднення, інкапсуляція, викид, мінеральні добрива.

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## FERTALIZERS ENCAPSULATION AS ONE OF THE METHODS OF ENVIRONMENTAL POLLUTION ABATEMENT

**Abstract.** Pollution of the environment with mineral fertilizer residues increases every year and causes deterioration of soil, water and air environment. This is due to the fact that nitrogen fertilizers are well soluble and quickly penetrate the vertical soil profile into the deep layers that are not available for the root system of plants. We have analyzed the problem of environmental pollution with components of mineral nitrogen fertilizers and proposed the use of encapsulated fertilizers prolonged action, which will allow adjusting the speed and amount of fertilizers that will flow into the soil from the capsule depending on the soil and climatic conditions. And this, in turn, will enable reducing the amount of fertilizer that will be introduced into the soil, and reduce environmental pollution.

Experimental researches of release of components of mineral fertilizers from capsules depending on the hydrogen index of environment are conducted. It was found that the coating of nitrogen fertilizers sensitive to changes in the pH of the medium with substances makes it possible to regulate, within wide limits, the release of substances from capsules and to create fertilizers, depending on the type of soil, where they will be applied.

Due to the fact that the amount of precipitation in different areas is different, there is a problem of creating encapsulated fertilizers with different thicknesses of coverage. If there is a lot of precipitation, then the thickness of the coating should be greater, and in areas where there is insufficient number of precipitation, it has very little. Therefore, to control the rate of release of components from capsules, experimental studies have been carried out on the effect of introducing a readily soluble crystalline substance into the polymer coating on the rate of release of components from capsules. It has been established that this type of coating allows using encapsulated fertilizers in wide ranges, regulating the rate of release of the content of the introduced freezing crystalline material. It is possible to use either the same fertilizers or other easily soluble nutrients that need it.

The calculation of mass transfer coefficients through the polymer shell and coefficients of diffusion of the component in the polymer were carried out.

**Key words:** contamination, encapsulation, release, mineral fertilizers.

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### Introduction

Environmental degradation due to the wide and constant use of mineral fertilizers in agriculture is intensifying every year. This concerns both the deterioration of properties and reduction of soil fertility, as well as pollution of underground

and surface water, air chemical elements and compounds, deterioration of agricultural products, etc. And as a result, this leads to environmental degradation, which affects the health of people.

The losses of components of mineral fertilizers are observed, from their production

to the soil environment application. On the one hand, this happens due to the use of outdated equipment for the production of mineral fertilizers, as well as the imperfect technologies of their transportation, storage, preparation and introduction, and on the other hand – as a result of soil processes that occur due to the vital functions of soil microorganisms, migration, atmospheric washings precipitation, etc.

In the soil environment, processes of the transformation of certain substances into others through the activity of soil microorganisms are constantly undergoing. On the one hand, the process of nitrification is a positive process, since the conversion of nitrogen from ammonium and ammonia salts to nitrates (nitric acid salts) is absorbed by plants. This occurs under the influence of nitrifying bacteria, for which oxidation is a source of energy. On the other hand, this process contributes to the formation of nitrates that are not absorbed by the soil absorbing complex, in contrast to ammoniacal nitrogen, which is negative in the absence of plant vegetation. As a result, favorable conditions are created for increasing the migration of nitrates in the deep horizons. In order to limit this process, nitrification inhibitors are used – chemical compounds that inhibit the activity of nitrifying bacteria and ensure the storage of nitrogen in soil and fertilizers in ammonium form. As inhibitors of nitrification use derivatives of pyridines, pyrimidines, triazoles, cyanamides, etc. (Skowrońska & Wiater, 2000), (Mel'nychuk, et al., 2004).

In addition, with the purpose of increasing the coefficient of assimilation of nitrogen fertilizers by plants, it is necessary to take into account the climatic conditions of the area, soil fertility and the availability of phosphorus and potassium, the soil reaction, the need for plants and the duration of the growing season.

An important method of preventing environmental pollution is the fertilization of crops in the period of their greatest need for nitrogen, i.e., the retail supply of nitrogen fertilizers during vegetation and in the phase of the largest absorption of nitrogen.

Since nitrogen fertilizers are predominantly water-soluble salts, one of the methods for preventing soil contamination is the creation of encapsulated fertilizers, that is, prolonged-action fertilizers that can gradually feed their nitrogen during one or several vegetation periods. They are divided into two groups. The first group combines poorly soluble fertilizers in water, whose components become available to plants only as a result of a gradual chemical or microbiological decomposition in the soil. These are urea condensates and various aldehydes,

ammonium humates – nitrogen compounds based on lignensulfonic acid, and the like. The second group – a fertilizer, well soluble in water, but their granules are covered with thin hard-soluble membranes, ie, encapsulated fertilizers. As the encapsulating substances, phenol formaldehyde resins, sulfur, amines, stearin, polystyrene, etc. (Solodovnik, 1980), (Gutcho & Gutcho, 1972), (Devassine et al., 2002) are used.

At present, encapsulated substances are widely used in various industries and microcapsulation products are quite common (Solodovnik, 1980), (Gutcho & Gutcho, 1972), (Devassine et al., 2002). Pharmaceuticals is the main industry where capsulation is applied. Among the other examples of the use of encapsulated products are: the production of microcapsules fibers and their products, food products and cosmetics, perfumery substances, household appliances, as well as some technical materials. Microcapsulation of cellulose, synthetic and inorganic fibers aims to modify the fiber to give it new properties, as well as improve its technical characteristics.

The main advantages of prolonged-action fertilizers are: the full supply of agricultural crops with mineral fertilizer components during the entire period of vegetation and minimization of environmental pollution; the use of well-soluble nitrogen fertilizers in areas where excessive humidification of soils or irrigated lands prevails; reducing the amount of mineral fertilizers that will be introduced during the year, which not only limits the loss of fertilizers to the environment, but also reduces the cost of agricultural production, etc.

Encapsulated substances, and especially mineral fertilizers, are widely used in developed countries, as evidenced by numerous patents and published scientific works. The most prolonged-action fertilizers are produced in the USA, Europe and Japan. The main scholars involved in this topic in Ukraine and abroad are A. Kondratov, V. Solodovnik, I. Kovshinnikov, Wahid R. Ali, V. Babtsov, M. Rodson, H. Scher, Y. Shapiro, E. Polyansky, D. Waldman, P. Markusch, J. Rosthauser, M. Buiser, E. Aksnes, L. Kilaas, S. Baldwin et al. (Taran et al., 2008), (Kuvshinnikov, 1987), (Babtsov et al., 2005).

The widespread use of encapsulated fertilizers in Ukraine is limited by the fact that they are expensive. Therefore, there is a problem of the development of quality prolonged fertilizers depending on the soil-climatic conditions of the region of their introduction and needs of agricultural crops at affordable prices.

The objective of the paper is to create and research of prolonged action of encapsulated fertilizers depending on the soil-climatic conditions.

### 1. Theoretical part

In the application of capsular mineral fertilizers in agriculture one of the main tasks is to use materials that are not harmful to the environment, as well as the creation of fertilizers that have certain predetermined properties. The main effect of encapsulated fertilizers is the gradual release of components from the capsule, therefore, it is necessary to regulate the permeability of the polymer shell, depending on the physico-chemical and soil-climatic conditions of the environment, in order to minimize the losses of components of nitrogen fertilizers to the environment. The release time in this case depends on the properties of the polymer coating itself, its thickness, and so on. Creating a polymeric coating of a certain thickness allows to regulate the process of release of the target component within a fairly wide range, but sometimes it is necessary to create films of a significant thickness to prevent the rapid release of fertilizer components in conditions of excessive moisture and vice versa, to create very thin polymeric capsules for the use of fertilizers under conditions of insufficient precipitation. In this case, it is advisable to regulate the permeability of the polymer coating by introducing into the polymer a readily soluble crystalline substance, which will significantly expand the limits of application of these fertilizers.

The main parameters determining the release time of a substance from a capsule are the mass transfer coefficient across the polymer shell and the diffusion coefficient of the component in the polymer. The diffusion of the mineral fertilizer component through the polymeric shell can be described by the mass transfer equation, which takes into account three diffusion supports (inside the capsule, through the shell and external diffusion) (Fig. 1)

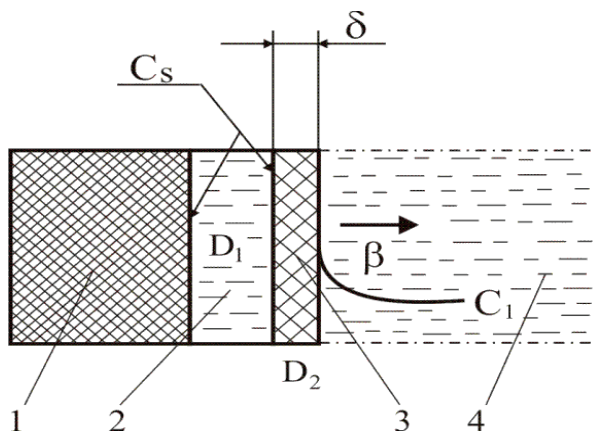


Fig. 1. Scheme of the release process of mineral fertilizers through a polymeric membrane:  
1 – mineral fertilizers in solid state;  
2 – saturated liquid fertilizer solution; 3 – polymeric membrane; 4 – surrounding water environment

Given that the diffusion resistance inside the capsule is insignificant, the equation has the following form:

$$M = k \cdot F \cdot (C_s - C_1), \quad (1)$$

where  $k = \frac{1}{\frac{\delta}{D_2} + \frac{1}{\beta}}$  – coefficient of mass transfer of

mineral fertilizers through a polymeric membrane,  $M/c$ ;  $C_1$  – concentration of the component in the external environment,  $\kappa\text{g}/\text{m}^3$ ;  $C_s$  – concentration of saturation, which is equal to the concentration on the inner surface of the shell,  $\kappa\text{g}/\text{m}^3$ ;  $D_2$  – coefficient of diffusion of a component in a polymer,  $\text{m}^2/c$ ;  $\beta$  – the coefficient of mass deducing the component from the outer surface of the capsule into the environment,  $M/c$ ;  $\delta$  – shell thickness, m.

To determine the coefficient of mass transfer of mineral fertilizers through a polymeric shell  $k$ , we use a mathematical model for the release of the target component through insoluble shells in the surrounding liquid medium, which is given in (Humnitsky et al., 2006). This mathematical model makes it possible to accurately predict the time and rate of release of matter from mineral fertilizer particles coated with polymeric membranes with sufficient for practical purposes.

According to the solution of this mathematical model, the obtained data can be represented in the form of linear dependence:

$$\frac{1}{3} \cdot \ln \left( \frac{a^3 + R^3}{a^3 + r_0^3} \right) = k \cdot \frac{C_s}{\rho_T} \cdot \frac{R^2}{a^3 + r_0^3} \cdot \tau, \quad (2)$$

where  $a^3 = \frac{3}{4} \cdot \frac{W \cdot C_s}{\pi \cdot (\rho_T - C_s)} - R^3$ ;  $\tau$  – time, c;  $R$  – particle radius

(inner radius of the shell), m;  $r_0$  – particle radius at certain moment  $\tau$ , m;  $W$  – volume of liquid,  $\text{m}^3$ ;  $C_s$  – concentration of saturation,  $\kappa\text{g}/\text{m}^3$ ;  $\rho_T$  – solid particles density,  $\kappa\text{g}/\text{m}^3$ .

To find the coefficient of mass transfer  $\beta$ , it is necessary to use the known criterial dependence for the conditions of natural convection (Zaytsev & Aseyev 1988):

$$Sh = 0.6 \cdot \sqrt{Sc \cdot Gr}, \quad (3)$$

where the number of Sherwood includes the coefficient of mass transfer  $\beta$ .

Calculations are made for ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) with density  $\rho_m = 1725 \text{ kg}/\text{m}^3$ , density of environment  $\rho = 1000 \text{ kg}/\text{m}^3$ , coefficient of kinematic water viscosity  $\nu = 1 \cdot 10^{-6} \text{ m}^2/c$ , diffusion coefficient  $D_1(\text{NH}_4\text{NO}_3) = 1,92 \cdot 10^{-6} \text{ m}^2/c$ . After obtaining the value  $\beta$  and  $k$  the values of the diffusion coefficients to be calculated  $D_2$  according to the equation (1).



The second method that makes it possible to regulate the rate of release of components from capsules is the method of entering certain amount of readily soluble crystalline material into a polymeric film. This enables adjusting the release rate within a wide range. It also makes it possible to create fertilizers that are intended for certain types of soils and areas with varying amounts of precipitation in different periods of cultivating crops.

Fig. 2. schematically depicts a polymer shell (thickness  $\delta$ ) of solid (a) and formed voids (b).

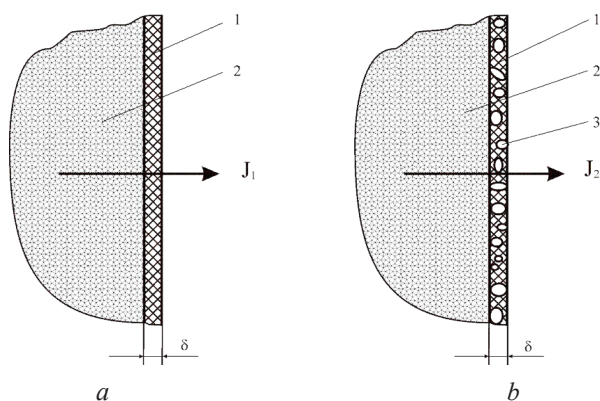


Fig. 2. Scheme of diffusion flows through a continuous polymer shell (a) and a shell with formed voids after dissolution of a readily soluble crystalline substance (b)  
1 – polymer shell; 2 – mineral fertilizers;  
3 – formed emptiness

Water, as a solvent penetrating through a polymeric membrane, in this case, firstly, dissolves a readily soluble crystalline substance (most appropriate to use the same mineral fertilizers or any other, which are needed for plants), which is located in the shell of the capsule. This leads to the emergence of voids in the capsule, due to which the diffusion flow of the main component in the capsule ( $j_2 > j_1$ ) increases. Consequently, with the increase in the amount of readily soluble crystalline material in the shell, the rate of release of the components of mineral fertilizers from the capsules will increase.

## 2. Experimental part

Nitrogen fertilizers, widely used in agriculture, namely ammonium nitrate, have been selected for experimental research.

In order to determine the effect of the hydrogen index of the medium, the encapsulated particles were placed in a liquid medium at pH 1 to 13. The spherical particles were pre-coated with acetyl phthalylcellulose. The medium was chosen to be large enough so that the concentration of the released substance from the particle was significantly less than the saturation concentration of the solution. In our case it was 500 ml. The

acidity of the medium in the range of pH from 1 to 6 was made with hydrochloric acid (HCl), neutral – distilled water, and alkaline medium in the range of pH from 8 to 13 – by means of sodium hydroxide solution (NaOH). Experiments were carried out until the target component was completely released from the encapsulated particle. The analysis of the selected wire was carried out using an ionomer.

To investigate the influence of impurities of a readily soluble crystalline substance, particles of ammonium nitrate (ammonium nitrate) with a particle diameter coated with an insoluble polymeric film, which contained small crystalline ammonium nitrate in quantities of 1, 5, 10, 15, 20, 25, were used to change the coefficient of diffusion of the polymer shell  $d=4\text{ mm}$  and 30% by weight of the polymer. For comparison, particles of mineral fertilizers were coated with pure polymer. Polystyrene was used as a polymer coating.

During experimental studies, the encapsulated particles were placed in a capacity of 500 ml, where distilled water was pouring. After a certain period of time, the encapsulated particle was gently removed from the container, and the test solution was thoroughly mixed to align the concentration. After that, samples were taken from each container. The sample was analyzed on an ion to determine the content of the nitrate ion in the solution under study. All samples taken after analysis were returned to a container where a pre-expanded capsular particle was placed to further release the mineral fertilizer from the capsule. Experiments were conducted for 30 days.

## 3. Results of researches

Taking into account the fact that the hydrogen index of the soil environment in different regions of the country varies depending on the type of soil, location and soil contamination by various harmful substances that affect the pH of the medium, it is important to study the influence of pH on the rate of release of fertilizers from capsules. This will reduce the environmental pollution of mineral fertilizer residues by the use of encapsulated mineral fertilizers, the release of which substances can be regulated by the physical and chemical properties of the coating itself. As a coating of this type, we chose an insoluble polymeric compound made from acetylphthalyl cellulose. The reason for choosing this coating was its non-toxicity, since this substance is used in medicine for the capsulation of drugs, as well as the sensitivity of the polymer coating to the change in the hydrogen index of the medium.

Experimental studies were conducted within the limits  $pH = 1 \div 13$ . The results of experimental studies in the form of the dependence of the

concentration of ammonium nitrate on the pH of the medium at fixed time intervals are shown in Fig. 3.

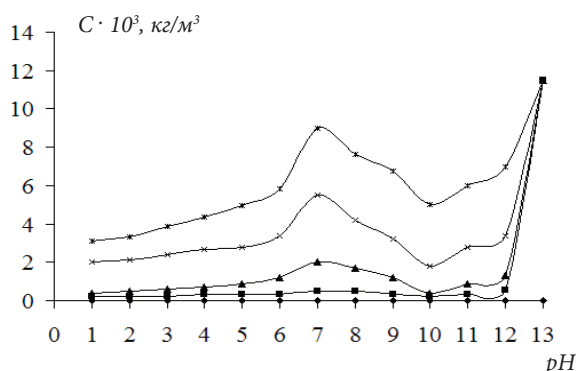


Fig. 3. Dependence of the concentration of the released substance (ammonium nitrate) from the capsule from the pH of the release medium:  
♦ – 0 hours.; ■ – 1,5; ▲ – 3; – 5; – 7

The analysis of experimental data shows a tendency to increase the permeability of the polymer shell in an acid medium with an increase in pH to 7. In an alkaline medium, the rate of release of the target component from the capsule decreases to pH = 10, but thereafter there is an increase in the rate of release of the component, and in the range of pH 12 a sharp increase in the rate of extraction of the component caused by the dissolution of the fertilizer shell occurs.

Insolubility, acid resistance and solubility in aqueous solutions with  $pH > 7$  is an important characteristic of acetylphthalyl cellulose. With the increase of pH from 1 to 7, the degree of swelling of the polymeric membrane increases, resulting in accelerated release rate of the component.

In the alkaline medium, the ionization of the macromolecules is gradually weakened, resulting in the chain of acetyl phthalylcellulose being twisted, which leads to a decrease in the mass transfer coefficient in the polymeric shell and, thus, causes the decomposition of the component release from the capsule particles. Thus, in the case of the achievement of the electroneutrality of the polymer macromolecules, i.e. the isoelectric point, the permeability of the polymer shell is minimal. But the simultaneous substitution of acetyl and phthalic groups by hydroxyl in the alkaline medium still contributes to some increase in the rate of release of the component, as compared to the acidic medium. Then, under pH 12, the chemical interaction of the cellulose ether with the hydroxide and the gradual dissolution of the polymeric membrane occurs.

Presenting research results in the form of a straightforward dependence,  $\frac{1}{3} \cdot \ln \left( \frac{a^3 + R^3}{a^3 + r_0^3} \right) = f(\tau)$

the coefficient of mass transfer to be determined through the polymer shell and values of the diffusion coefficients  $D_2$  are carried out.

As can be seen from the research results, the hydrogen index of the medium has a huge impact on the kinetics of releasing components from encapsulated particles coated with a coating capable of physic-chemical and chemical interaction in a liquid medium. That is why it is possible to regulate the optimal release of the target component from capsules with the thickness of the shell depending on the pH of the soil. In addition, due to the ability of the polymer to gradually dissolve at  $pH \geq 7$ , the problem of soil contamination with the remnants of the shell is eliminated.

The results of experimental studies on the influence of entering readily soluble crystalline substance on the rate of release of components from capsules are presented in Fig. 4.

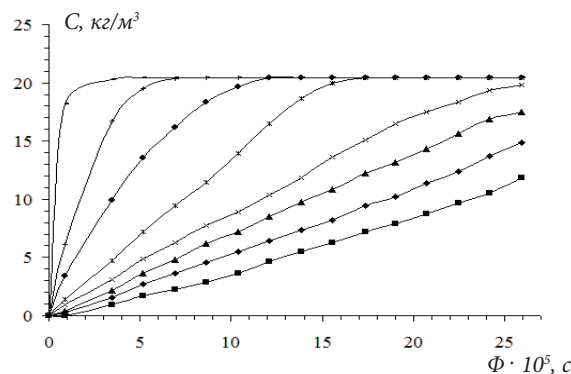


Fig. 4. Kinetics of release of components of capsular mineral fertilizers (ammonium nitrate) provided that different amounts of ammonium nitrate are added to the polymer coating: ■ – 0 mof weight part; ♦ – 0,01; ▲ – 0,05; x – 0,10; \* – 0,15; ● – 0,20; + – 0,25; – – 0,30

Extraction of the target component from the capsule takes place in three stages. The first stage is the descent of water into the polymeric membrane and dissolution of the inclusions of the crystalline material. The second stage is the penetration of the solvent into the capsule, the dissolution of the mineral fertilizer and diffusion of the dissolved substance into the environment. This process is the longest and therefore limits the overall release of the component. The third stage begins after the complete dissolution of the mineral fertilizer inside the capsule and the release of the component to the equilibrium, which is determined by the equality of concentrations inside and outside the capsule.

The rate of release of the components from the capsules depends on the amount of light-soluble substance that was entered into the polymer shell. According to the results of experimental studies, the application of encapsulated fertilizers, which

will contain a part of a readily soluble crystalline substance in the polymer coating, will allow the use of nitrogen fertilizers on all types of soils in any region of Ukraine, regardless of the amount of precipitation.

The experimental data obtained for determining the mass transfer coefficient were presented in the form of linear dependence  $\frac{1}{3} \ln \left( \frac{a^3 + R^3}{a^3 + r_0^3} \right) = f(\tau)$ , to determine the coefficient

of mass transfer through the polymer sheath and values of the diffusion coefficients  $D_2$ .

The values of the coefficients of mass transfer  $k$  obtained are presented as a graphical dependence  $k = f(x)$  fig. 5, which can be approximated by the dependence of the coefficient  $k$  on concentration:

$$K = 10^{-9} \cdot (1,159 + 5,5104 \cdot x + 44,127 \cdot x^2) \quad (4)$$

where  $x$  – mass part of readily soluble crystalline material in a polymeric shell. Determination level is  $R^2 = 0,9989$ .

## Conclusions

Consequently, the use of encapsulated mineral fertilizers allows slowly, depending on the permeability of the polymer coating, releasing the components from the capsules over a long period of time, ensuring uniform fertilization of the crops. In this way, soil fertilization is reduced

in underground water levels and, as a result, contamination of drinking water basins and lower layers of soil, where there is no absorption of fertilizer components by root plant systems.

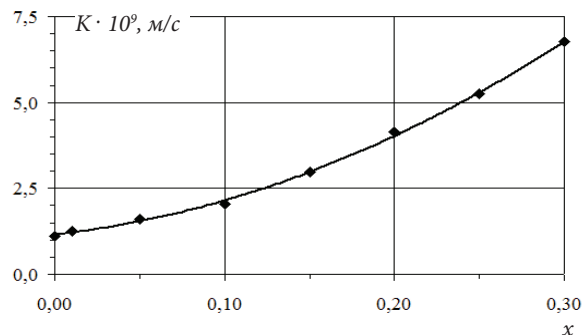


Fig. 5. The kinetics of the change in the mass transfer coefficient  $k$  depending on the content of readily soluble crystalline substance in the polymer coating: ---- calculated value by the formula (4), points – experimental values.

The regulation of the release of components from capsules can be carried out within wide limits by the thickness of the shell itself, or by making the polymer a readily soluble crystalline substance. Since the reaction of soil soils in most cases ranges from slightly acid to very alkaline in Ukraine, application of encapsulated fertilizers, based on the physical and chemical properties of the environment, is very important.

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