

Release of selected nutrients from polymer-coated fertilisers in the soil environment

Małgorzata Włodarczyk^{*1)} , Hanna Siwek¹⁾ ,
Krzysztof Lubkowski²⁾ , Anna Buchwał¹⁾ 

¹⁾ West Pomeranian University of Technology in Szczecin, Faculty of Environmental Management and Agriculture, al. Piastów 17, 70-310 Szczecin, Poland

²⁾ West Pomeranian University of Technology in Szczecin, Faculty of Chemical Technology and Engineering, al. Piastów 17, 70-310 Szczecin, Poland

* Corresponding author

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Abstract: The aim of the study was to evaluate the release of $\text{NH}_4\text{-N}$ and $\text{PO}_4\text{-P}$ from polymer-coated fertilisers in the soil environment, and to analyse their impact on pH and conductivity of the soil leachates. In this investigation mineral NPK(S) 6-20-30(7) fertiliser (as a starting material), commercial, controlled-release Osmocote™ fertiliser (as a reference material) and four polymer-coated fertilisers have been used. Biodegradable polybutylene(succinate-co-dilinoleate), polyethylene(succinate-co-terephtalate) and chitosan have been used as coating materials. The experiments were conducted in the laboratory conditions, in PVC columns filled with air-dry soil. The nutrients release from the investigated materials was explained based on the diffusion mechanism and it was interpreted with the use of the Korsmeyer–Peppas kinetic model. Two mechanisms dominate in the release process of nutrients: the mechanism based on quasi-Fickian diffusion and non-Fickian (anomalous case) mechanism. The largest changes of pH and electrical conductivity (EC) of soil leachates occurred in the initial period of research for all tested fertilisers (pH: 9.5–20.3% – loamy sand (S1) 7.9–20.6% – sandy loam (S2); EC: 438–1667% – S1, 771–1509% – S2). The polymer coating significantly reduces the nutrient release from the fertiliser core. The size of these changes depends on the type and thickness of the polymer layer and the physicochemical properties of the soils.

Keywords: biodegradable polymer-coated fertiliser, diffusion coefficient, nutrients, release kinetics, soil leachate

INTRODUCTION

The United Nations estimates that the world population will increase to approximately 9.7 bln people in 2050 and may reach 10.4 bln by the end of the 21st century (UN, 2024). In order to ensure food security, resulting from the rapid population growth, global food production is estimated to increase nearly 70% from current levels (Dijk *et al.*, 2021; Noulas, Torabian and Qin, 2023). Presently, the increase in food production is achieved mainly by increasing crop areas as a result of deforestation and destroying animal habitats. This has a negative impact on the natural environment due to climate and biodiversity changes. It is

estimated that changes in the land use contribute 12% to the total greenhouse gas emissions leading to global warming (Chataut *et al.*, 2023). The challenge is to increase the efficiency of agriculture, mainly through the use of various fertilisation techniques, while reducing the negative impact of agriculture on the environment. Effective fertilisation of crop plants is a key element in maximising production and increasing yields (Noulas, Torabian and Qin, 2023).

The political priorities of the European Union, including proposals for the European Green Deal becomes the main challenge for the entire fertiliser sector. In view of the EU's ambitious pro-environmental goals (Communication, 2019),

actions will focus on ensuring food security and supporting a sustainable agricultural sector by encouraging producers and final recipients to make efforts to effectively use the mineral components of fertilisers.

Conventional mineral fertilisers contain at least one of the three essential nutrients (nitrogen, phosphorus and potassium), provided in a form that is easily absorbed by plants. Correctly targeting the application of fertilisers maximises the uptake of nutrients by plants, ensuring their productive growth. Unfortunately, after the application of conventional mineral fertilisers, large losses of nutrients are observed as a result of various physical and chemical processes. It is estimated that losses may reach 30–50% for nitrogen, 10–25% for phosphorus, and 50–60% for potassium (Shaviv and Mikkelsen, 1993; Trenkel, 2010). The use of conventional fertilisers also results in increased gas emissions into the atmosphere, soil acidification, water eutrophication, and soil erosion (Sapek *et al.*, 2021; Skorupka and Nosalewicz, 2021). The prevention of such phenomena is now becoming a priority of the successively implemented European Union policy.

Optimisation of the nutrient use efficiency (*NUE*) by plants, and limitation of the negative impact of fertilisers on the natural environment, can be achieved by using the intelligent fertilisers, including slow- or controlled-release fertilisers (Trenkel, 2010; Lawrencja *et al.*, 2021; Kassem *et al.*, 2024). Slow-release fertilisers are chemically or biologically decomposed materials with a high molecular weight, complex structure and small solubility, whereas controlled-release fertilisers are materials in case of which the release of mineral components takes place through a polymer layer or a membrane (Trenkel, 2010). Controlled-release fertilisers have been produced commercially since the early 1960s, and since then methods for their preparation and characterisation have also been known (Newman *et al.*, 2006; Trenkel, 2010; Lawrencja *et al.*, 2021). However, it should be pointed out that these fertilisers were and still are produced using polymers that do not degrade in the natural environment. A conducive solution to environmental protection may be the production of controlled-release fertilisers using biodegradable materials, both of natural origin (Jamnongkan and Kaewpirom, 2010; Lubkowski, 2014; Pawelec *et al.*, 2021; Jayanudin *et al.*, 2022; Vermoesen *et al.*, 2023), and organic synthesis products (Lubkowski *et al.*, 2015; Lubkowski *et al.*, 2016; Treinyte *et al.*, 2018; Lawrencja *et al.*, 2021; Kassem *et al.*, 2024).

The aim of the presented research was to evaluate the release of selected nutrients ($\text{NH}_4\text{-N}$, $\text{PO}_4\text{-P}$) from the polymer-coated fertilisers with the controlled-release properties, prepared with the use of biodegradable polymer materials. Nutrient release from such fertilisers was previously determined, however those experiments were performed only in water (Lubkowski, 2014; Lubkowski *et al.*, 2015; Lubkowski *et al.*, 2016). The currently presented study was obtained as a result of experiments carried out in the soil environment, under laboratory conditions. In order to explain the release process, a diffusion model (Lambie, 1987; Shaviv and Mikkelsen, 1993; Shaviv, 2001; Shaviv *et al.*, 2003; Du, Zhou and Shaviv, 2006; Malekjani *et al.*, 2020; Lawrencja *et al.*, 2021) was used whereas the kinetics of the process were interpreted using the Korsmeyer–Peppas model. Another objective of the research was also to analyse the impact of nutrients on the pH and conductivity of the soil leachates. As far as we know, research on this type of fertilisers (biodegradable polymer-coated fertilisers) has not yet been conducted in soil conditions.

MATERIALS AND METHODS

FERTILISERS

Multi-component granulated mineral fertiliser NPK(S) 6-20-30(7) Polifoska®6 (GA ZCh Police SA) was used as the starting material (P). According to the data declared by the fertiliser manufacturer, the content of three main components of the fertiliser is as follows: 5.7 wt% $\text{NH}_4\text{-N}$, 19.8 wt% P_2O_5 , and 29.5 wt% K_2O .

As coating materials biodegradable chitosan (Lubkowski, 2014; Jayanudin *et al.*, 2022), two organic synthesis products: polybutylene(succinate-co-dilinoleate) – PBS/DLA (Kozłowska *et al.*, 2008; Lubkowski *et al.*, 2015), and polyethylene(succinate-co-terephthalate) – PES/PET (Lubkowski *et al.*, 2016) were used.

For comparative purposes, the commercial controlled-release Osmocote™ fertiliser (OS) was also tested. Osmocote™ is the first commercially manufactured resin-coated fertiliser, where the alkyd resins (copolymers of diclopentadiene with a glycerol ester) were used (Lambie, 1987; Goertz, 1991). According to the manufacturer's declaration (The Scotts Company) it contains 14% of total nitrogen, 9% of P_2O_5 , 11% of K_2O .

METHODS OF COATING WITH POLYMER SOLUTIONS

Granules of multicomponent NPK fertiliser with a grain size of 3–4 mm were covered with the solutions of chitosan (in acetic acid), PBS/DLA and PES/PET (in chloroform), according to the previously described procedures (Lubkowski, 2014; Lubkowski *et al.*, 2015; Lubkowski *et al.*, 2016). As a result, the materials with mass ratios of polymer to fertiliser (P_1/F) 0.24 (CH), 0.19 (PES/PET_1) and 0.26 (PES/PET_2, PBS/DLA) were obtained.

RELEASE EXPERIMENT

The release of $\text{NH}_4\text{-N}$ and $\text{PO}_4\text{-P}$ from fertilisers was tested in the laboratory conditions, in PVC columns with the following dimensions: length 40 cm and diameter 4.0 cm, filled with air-dry soil. Two soils were used in the research: loamy sand (S1) and sandy loam (S2). Soil characteristics are presented in Table 1. The soil level in the column was equal to the depth of the arable layer and was 20 cm, the irrigation area was 0.00126 m². Fertilisers in the amount of 10 granules with a precisely defined weight were placed in columns at a height of 15 cm, and then to minimise losses related with NH_4^+ volatility covered with a 5 cm layer of soil. Three replicates were prepared for all combinations. The kinetics of $\text{NH}_4\text{-N}$ and $\text{PO}_4\text{-P}$ release into the soil solution for fertilisers were conducted for 15 days (at intervals of one or two days). Additionally, for fertilisers PES/PET_1, PES/PET_2, PBS/DLA and OS, kinetic tests have been extended to 32 days (at intervals of two or three days). Soil leachates were obtained by irrigation the soil in columns with 100 cm³ of distilled water. Water was dosed using a peristaltic pump. The leachates were collected for 24 h. Conductivity of distilled water was 0.01 mS·cm⁻¹.

The spectrophotometric methods were used to determine the content of $\text{NH}_4\text{-N}$ and $\text{PO}_4\text{-P}$ in soil leachates (UV-VIS Spectrophotometer Evolution 201, ThermoScientific). The phosphorus was determined in the form of PO_4^{3-} using the blue molybdate method at a wavelength of 890 nm, nitrates were

Table 1. Selected physicochemical parameters of soils

Parameter	Measurement unit	Soil	Value
Water capacity	%	S1	34.63
		S2	35.77
Organic carbon		S1	0.83
		S2	1.20
Nitrogen		S1	0.075
		S2	0.099
Phosphorus		S1	0.033
		S2	0.046
Sulphur		S1	0.0076
		S2	0.0001
pH	H ₂ O	S1	5.56
		S2	6.09
	KCl	S1	4.28
		S2	5.13
Hh	cmol·kg ⁻¹	S1	3.33
		S2	2.36
CEC		S1	16.10
		S2	0.853
TEB		S1	19.43
		S2	3.215
BS		S1	82.88
		S2	26.53

Explanations: S1 granulometric group – loamy sand, S2 granulometric group – sandy loam, Hh = hydrolytic acidity, CEC = cation exchange capacity, TEB = total exchangeable bases; BS = base saturation; definitions of Hh, CEC, TEB and BS in Osman (2013).

Source: own study.

determined in the form of NH₄⁺ with Nessler's reagent at a wavelength of 400 nm.

In order to control soil leachates, their pH (pH meter Elmetron CX-505) and conductivity (conductometer WTW Cond 730) were measured.

The content of the released nutrients into the soil leachates were corrected for the values of control samples.

RELEASE KINETICS MODEL

The mechanisms of NH₄-N and PO₄-P release from fertilisers were investigated using a semi-empirical kinetic model, known as the power law or the Korsmeyer–Peppas model (Siepmann and Peppas, 2012; Lubkowski, 2014; Malekjani and Jafari, 2020; Lawrencja *et al.*, 2021):

$$\frac{M_t}{M_0} = Kt^n \quad (1)$$

where: M_t/M_0 = the fraction of nutrient released, M_t = nutrient release (mg), M_0 = the total amount of nutrient (mg), t = time (day), K = the release rate constant (day⁻¹), n = the exponent of release.

Differences between fertilisers were analysed statistically using the Tuckey's test with a significance level of $p \leq 0.05$ (STATISTICA 13.3).

RESULTS AND DISCUSSION

POTENTIAL OF HYDROGEN (pH)

The impact of fertilisers on the pH of soil leachates is varied and depends on the type of soil (Fig. 1a, 1b). In general, the tested fertilisers reduce the pH of soil leachate, which is confirmed by research (Newman *et al.*, 2006; Radulov *et al.*, 2011; Ozlu and Kumar, 2018).

Compared to control samples (soil without fertiliser) for soil S1 decrease of the soil leachate pH was recorded in the initial period of the experiment for all coated fertilisers and the multi-component fertiliser P and ranges from 4.2 to 20.3%. Significant pH changes lasted the longest in the case of coated fertilisers (PES/PET_2, PBS/DLA) – 12 days and OS – 17 days (Fig. 1a). A similar relationship was obtained for soil S2 (Fig. 1b) with the exception of the slow-release fertiliser OS. In the case of OS, there were no significant changes in the pH of the collected soil leachates. Additional factor determining changes of the soil leachate pH is the thickness of the polymer layer. Comparing PES/PET_1 and PES/PET_2 with a mass ratios of polymer to fertiliser (P₁/F) 0.19 and 0.26, respectively, it was found that increasing the thickness of the polymer layer extends the acidification time of the soil solution (S1), or this process is postponed in time (soil S2).

Research shows that the chemical composition, especially N-fertiliser form, have a significant impact on the soil acidification process. Generally NH₄⁺ has the potential to contribute to intensifying acidification of soil. The NH₄⁺ as the main source of N in the multi-component fertiliser P and coated fertilisers (CH, PES/PET_1, PES/PET_2, PBS/DLA) can displace base cations (Ca²⁺, Mg²⁺, K⁺, Na⁺) from soil sorption complex and make them easy to leach out of soils reducing their buffering against acidification. Additional processes which cause soil acidification are connected with the nitrification of NH₄-N, ammonia volatilisation, and the absorption by microorganisms and/or plants of the NH₄⁺ and the secretion of H⁺ (physiological acidity) (Tian and Niu, 2015; Tkaczyk *et al.*, 2020).

The leachate pH is influenced by ratios of NH₄⁺ to NO₃⁻. The pH of leachate decreasing when the amount of NH₄⁺ relative to NO₃⁻ increases. In the present study for the OS fertiliser the amount of N as NH₄⁺ was at the level 73% – this confirms the ability of the OS fertiliser to acidify the soil solution and the results for soil S1. The results obtained for soil S2 (no changes in the pH of soil leachate) indicate a significant impact of the physicochemical properties of soils on changes in their pH under the impact of OS. Similar relationships for controlled release fertilisers were obtained in studies of Newman *et al.* (2006). However, the increase in the pH of soil leachates is related to the leaching of alkaline compounds from the soil, which are components of the soil sorption complex.

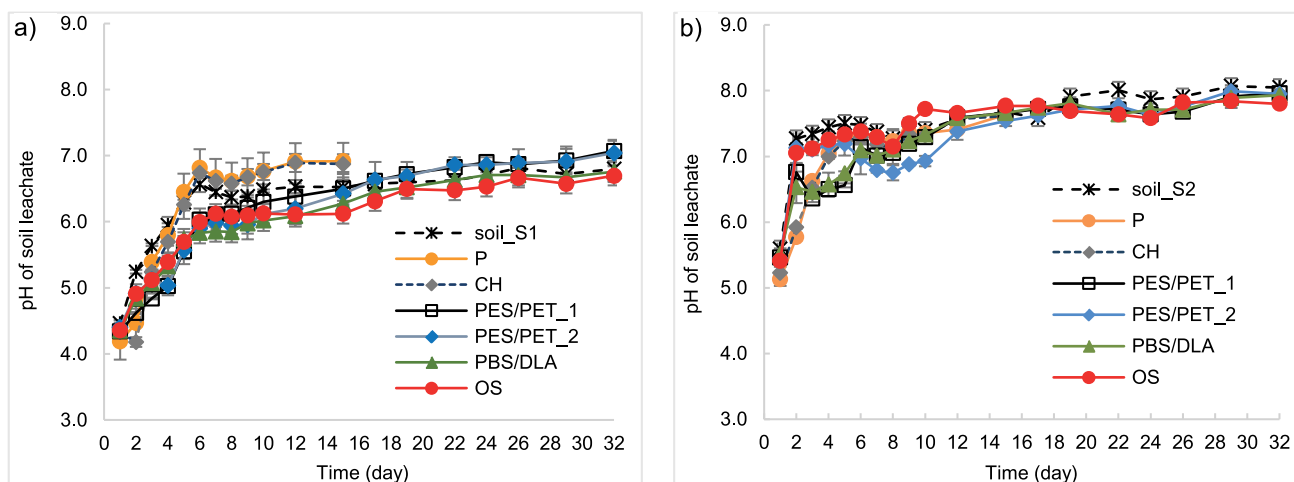


Fig. 1. The influence of fertilisers on the pH of soil leachates: a) soil S1, b) soil S2; P = commercial fertiliser Polifoska[®]6, CH = Polifoska[®]6 coated by chitosan, PES/PET_1 = Polifoska[®]6 coated by PES/PET – mass ratios of polymer to fertiliser = 0.19, PES/PET_2 = Polifoska[®]6 coated by PES/PET – mass ratios of polymer to fertiliser = 0.26, PBS/DLA = Polifoska[®]6 coated by PBS/DLA, OS = control-release fertiliser Osmocote[™]; source: own study

ELECTRICAL CONDUCTIVITY (EC)

It was found that the *EC* of leachates depends on the soil type. The tested fertilisers influence the conductivity of soil leachates in different ways, but for both soils the general trend of changes is similar and in accordance with research of Newman *et al.* (2006) and Adams, Frantz and Bugbee (2013). The leachates obtained from soil samples with fertiliser are characterised by statistically significantly higher *EC* values (according to Tuckey's test at $p \leq 0.05$) compared to control samples (Tabs. 2–3). The biggest changes of soil leachates *EC* were recorded in the first two days of the research for the multi-component fertiliser P. The difference between fertiliser P and control at the 1st day was at the level of 4.16–4.98 $\text{mS}\cdot\text{cm}^{-1}$ for S1 and S2, respectively and at the 2nd day

at the average level of $2.33 \pm 0.004 \text{ mS}\cdot\text{cm}^{-1}$ for both soil. Large changes of soil leachates *EC* were also found for fertiliser coated with biodegradable chitosan (CH). For S1, on the first day the changes were at the level of $0.53 \text{ mS}\cdot\text{cm}^{-1}$, while on the 2nd day the CH caused an increase in the salinity of the soil solution by $3.87 \text{ mS}\cdot\text{cm}^{-1}$. In the case of S2, in the first two days, the changes of leachates *EC* were at a more similar level of $2.47\text{--}2.96 \text{ mS}\cdot\text{cm}^{-1}$ for S1 and S2, respectively. From the 3rd day of the experiment, the leachate conductivity for P and CH decreased significantly, additionally from 3rd to 15th day the differences in *EC* between these fertiliser formulations are insignificant.

Significantly smaller changes in the conductivity of soil leachates (compared to the control combinations) were noted for coated fertilisers PES/PET_1, PES/PET_2, PBS/DLA and the

Table 2. Influence of fertilisers on electrical conductivity of soil leachate – data for soil S1

Day	Control soil 1	P	CH	PES/PET_1	PES/PET_2	PBS/DLA	OS
	$\text{mS}\cdot\text{cm}^{-1}$						
1 st	2.72 ± 0.25 ^a	6.88 ± 0.31 ^b	3.25 ± 0.16 ^c	3.16 ± 0.15 ^c	2.94 ± 0.20 ^c	3.09 ± 0.02 ^c	3.26 ± 0.12 ^c
2 nd	0.23 ± 0.08 ^a	2.56 ± 0.20 ^b	4.10 ± 0.46 ^c	1.76 ± 0.13 ^d	0.64 ± 0.17 ^e	1.06 ± 0.02 ^f	0.68 ± 0.08 ^e
3 rd	0.08 ± 0.01 ^a	0.25 ± 0.01 ^b	0.32 ± 0.07 ^{bc}	0.81 ± 0.03 ^c	0.37 ± 0.14 ^{bde}	0.60 ± 0.11 ^d	0.44 ± 0.04 ^e
4 th	0.06 ± 0.01 ^a	0.16 ± 0.00 ^b	0.19 ± 0.04 ^b	0.63 ± 0.08 ^c	0.63 ± 0.15 ^c	0.30 ± 0.01 ^d	0.30 ± 0.04 ^d
5 th	0.05 ± 0.00 ^a	0.11 ± 0.00 ^b	0.13 ± 0.02 ^b	0.29 ± 0.03 ^c	0.41 ± 0.03 ^d	0.21 ± 0.03 ^e	0.19 ± 0.00 ^e
6 th	0.05 ± 0.01 ^a	0.09 ± 0.00 ^b	0.10 ± 0.03 ^b	0.18 ± 0.00 ^c	0.36 ± 0.10 ^d	0.29 ± 0.08 ^d	0.15 ± 0.02 ^e
7 th	0.04 ± 0.00 ^a	0.08 ± 0.00 ^b	0.09 ± 0.02 ^b	0.14 ± 0.01 ^c	0.23 ± 0.00 ^d	0.24 ± 0.09 ^d	0.13 ± 0.02 ^c
8 th	0.04 ± 0.00 ^a	0.08 ± 0.00 ^b	0.09 ± 0.02 ^b	0.11 ± 0.00 ^c	0.21 ± 0.07 ^d	0.20 ± 0.05 ^d	0.11 ± 0.00 ^c
9 th	0.03 ± 0.00 ^a	0.08 ± 0.00 ^b	0.09 ± 0.02 ^{bc}	0.10 ± 0.01 ^c	0.24 ± 0.05 ^d	0.16 ± 0.04 ^d	0.11 ± 0.01 ^c
10 th	0.04 ± 0.00 ^a	0.07 ± 0.00 ^b	0.08 ± 0.02 ^{bc}	0.09 ± 0.00 ^c	0.15 ± 0.02 ^d	0.15 ± 0.05 ^{cd}	0.10 ± 0.01 ^c
12 th	0.04 ± 0.00 ^a	0.07 ± 0.00 ^b	0.08 ± 0.02 ^b	0.11 ± 0.00 ^c	0.18 ± 0.04 ^d	0.14 ± 0.04 ^{cd}	0.13 ± 0.02 ^{cd}
15 th	0.05 ± 0.01^a	0.08 ± 0.01^b	0.09 ± 0.02^{bc}	0.11 ± 0.01^c	0.17 ± 0.02^d	0.12 ± 0.01^{bc}	0.14 ± 0.01^d

Explanations: P, CH, PES/PET_1, PES/PET_2, PBS/DLA, OS as in Fig. 1; values marked with the same letter for a given time do not differ significantly, according to the Tuckey's test at the significance level of $p \leq 0.05$.

Source: own study.

Table 3. Influence of fertilisers on electrical conductivity of soil leachate – data for soil S2

Day	Control soil 2	P	CH	PES/PET_1	PES/PET_2	PBS/DLA	OS
	mS·cm ⁻¹						
1 st	3.13 ±0.24 ^a	8.11 ±0.85 ^b	5.60 ±0.70 ^c	3.35 ±0.14 ^a	3.12 ±0.18 ^a	2.99 ±0.18 ^a	3.27 ±0.33 ^a
2 nd	0.21 ±0.05 ^a	2.53 ±0.10 ^b	3.17 ±0.1 ^c	1.82 ±0.15 ^d	0.52 ±0.06 ^e	1.72 ±0.16 ^d	0.48 ±0.11 ^e
3 rd	0.11 ±0.00 ^a	0.34 ±0.02 ^b	0.40 ±0.03 ^c	0.68 ±0.05 ^d	0.33 ±0.02 ^b	0.78 ±0.21 ^d	0.39 ±0.08 ^c
4 th	0.10 ±0.00 ^a	0.23 ±0.01 ^b	0.25 ±0.02 ^{bc}	0.42 ±0.05 ^c	0.54 ±0.07 ^d	0.55 ±0.03 ^d	0.30 ±0.04 ^e
5 th	0.09 ±0.00 ^a	0.19 ±0.01 ^b	0.20 ±0.01 ^b	0.28 ±0.02 ^c	0.56 ±0.06 ^d	0.45 ±0.07 ^d	0.25 ±0.04 ^c
6 th	0.09 ±0.00 ^a	0.17 ±0.01 ^b	0.18 ±0.02 ^b	0.22 ±0.01 ^c	0.72 ±0.04 ^d	0.24 ±0.02 ^c	0.23 ±0.01 ^c
7 th	0.09 ±0.00 ^a	0.16 ±0.00 ^b	0.16 ±0.01 ^b	0.19 ±0.01 ^c	0.41 ±0.06 ^d	0.19 ±0.01 ^c	0.20 ±0.01 ^c
8 th	0.08 ±0.00 ^a	0.15 ±0.01 ^b	0.17 ±0.03 ^b	0.18 ±0.01 ^b	0.25 ±0.01 ^c	0.17 ±0.01 ^b	0.17 ±0.01 ^b
9 th	0.09 ±0.00 ^a	0.15 ±0.01 ^b	0.18 ±0.05 ^b	0.16 ±0.01 ^b	0.31 ±0.08 ^c	0.17 ±0.00 ^b	0.21 ±0.03 ^d
10 th	0.09 ±0.00 ^a	0.14 ±0.01 ^b	0.18 ±0.07 ^{bc}	0.16 ±0.01 ^b	0.25 ±0.03 ^c	0.16 ±0.00 ^b	0.22 ±0.01 ^c
12 th	0.11 ±0.00 ^a	0.14 ±0.01 ^b	0.19 ±0.08 ^{bc}	0.19 ±0.01 ^c	0.26 ±0.03 ^d	0.19 ±0.01 ^c	0.21 ±0.01 ^d
15 th	0.13 ±0.01^a	0.18 ±0.02^b	0.17 ±0.03^b	0.21 ±0.01^c	0.26 ±0.02^d	0.21 ±0.02^c	0.23 ±0.02^c

Explanations as in Tab. 2.

Source: own study.

reference OS, which is also confirmed by research of Newman *et al.* (2006) and Adamus, Frantz and Bugbee (2013). The main factors determined the level of *EC* changes were the type of polymer layer and its thickness. The PES/PET_1 caused higher salinity of soil leachates only in the first days of the experiment (up to day 3rd) at the maximum on the 2nd day at the level of 1.53–1.61 mS·cm⁻¹ for S1 and S2, respectively. For the PES/PET_2, the maximum changes of soil leachates *EC* were noted on the 4th (S1) and 6th (S2) day at a significantly lower level, 0.57 and 0.62 mS·cm⁻¹, respectively. The fertiliser coated with PBS/DLA shows some similarity to the PES/PET_1 in terms of the analysed parameter. The reference fertiliser OS was characterised by the lowest variability of soil leachate salinity throughout the entire experiment and similar to PES/PET_2. Tables 2 and 3 present the *EC* values of soil leachates only for 15 days of the experiment, because during this time the greatest changes in this parameter were recorded for all fertilisers. Extending the experiment to 32 days did not significantly affect on the soil leachates *EC*. For the soil S1, the salinity level of the soil solution for individual fertilisers remained practically constant, while for the soil S2, the *EC* of soil leachates decreased over time by an average of about 0.01 mS·cm⁻¹.

The use of excessive doses of mineral fertilisers is one of the most important causes of soil salinity in areas intensively used for agriculture. Increased concentration of minerals has a negative impact on the soil and plants. It brings about changes in the composition of the soil sorption complex, increases the leaching of alkaline cations and the dispersion of soil colloids. As a result of chemical sorption, the absorption of phosphorus (the main plant nutrient) and microelements that stimulate proper growth and development of plants decreases. At the same time, the ionic balance in cells is disturbed, which contributes to a reduction in enzyme activity. The resulting oxidative stress, in long-term exposure, leads to the death of organs and entire plants. Soil with excessive salt concentration imparts a detrimental impact on soil

microbial population as well as their activities (Lemanowicz, 2013; Safdar *et al.*, 2019; Zhao *et al.*, 2020).

The proposed technological solution, namely the use of controlled release fertilisers (CRF), allows to reduce the level of soil salinity. This is confirmed by measured values of *EC* which are a function of the concentration of ions released from CRF into the analysed medium (water, sand, soil). The *EC* measurements showed that a physical barrier created by the polymer coating is able to control the release of nutrients, however this process depends on the quality of the coating, its source and concentration of polymer used. The results of the presented experiments confirmed that both the fertilising factors (qualitative and quantitative composition of the core, solubility, polymer layer thickness, biodegradability of the coating material) and environmental factors (physicochemical parameters of soil, granulometric composition, organic matter, microorganisms, temperature) play an important role in the release of nutrients (Lubkowski *et al.*, 2016; Treinyte *et al.*, 2018; Vermoesen *et al.*, 2023; Kassem *et al.*, 2024).

AMMONIA NITROGEN (NH₄-N)

The amount of NH₄-N leachate depends on the tested fertilisers formulations and physicochemical properties of the soil (Fig. 2a, 2b). Generally, concentrations of N-NH₄ in the soil leachate were highest and fluctuated often in the initial period of the research (Newman *et al.*, 2006; Adams, Frantz and Bugbee, 2013; Jayanudin *et al.*, 2022). For both soils, the highest percentage of the applied NH₄-N dose was determined in soil leachates for the fertiliser P on the 2nd day of the experiment, at the level of about 50% for S1 and 28% for S2. In the following days, the amount of NH₄-N in soil leachates is significantly lower, and on the last day it reached the level of 1.4–1.7% for S1 and S2, respectively, of the applied dose. In total, within 15 days, from 87% (S1) to 71% (S2) of NH₄-N used in the form of the P was released into the soil

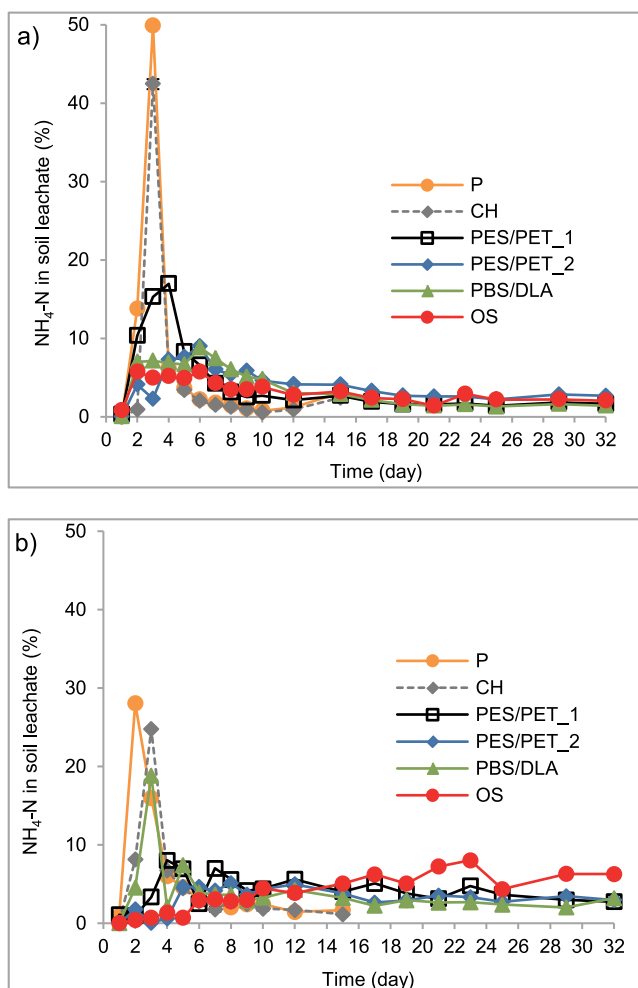


Fig. 2. Concentration changes of $\text{NH}_4\text{-N}$ in soil leachates: a) soil S1; b) soil S2; explanations as in Fig. 1; source: own study

solution. The polymer coatings had a varied impact on the release process of $\text{NH}_4\text{-N}$ from fertiliser core (Newman *et al.*, 2006; Lubkowski, 2014). The fertiliser coated a biodegradable layer of chitosan (CH) is very similar to the Polifoska®6. The highest percentage of the $\text{NH}_4\text{-N}$ dose was determined in the initial days of the experiment for soil S1 on 2nd day at a quite high level of 44%, for soil S2 on 3rd day at the level of 25%. In the case of fertiliser CH, the total percentage of available $\text{NH}_4\text{-N}$ in the leachate is slightly lower in relation to P and on the 15th day of the experiment, for both soils reached a similar level of $62 \pm 1.5\%$ of the initial dose. Soil leachates obtained for coated fertilisers PES/PET_1, PES/PET_2, PBS/DLA and reference fertiliser OS were characterised by significantly lower $\text{NH}_4\text{-N}$ contents, and the amplitude of their changes was significantly lower. Compared to P, the total release of the analysed nutrient was significantly lower, for S1 from 11.2% (PES/PET_1) to 37.9% (OS), for S2 from 13.9% (PBS/DLA) to 43.3% (OS). The results obtained for PES/PET_1 and PES/PET_2 indicate that increasing the polymer coating thickness significantly slows down the process of $\text{NH}_4\text{-N}$ release into the soil solution by about 15% on the 15th day of the experiment for both soils (Fig. 3a, 3b). On the last day of the experiment, the influence of the polymer layer thickness is more varied and depends on the soil conditions (S1 = 7.5%, S2 = 20.4%).

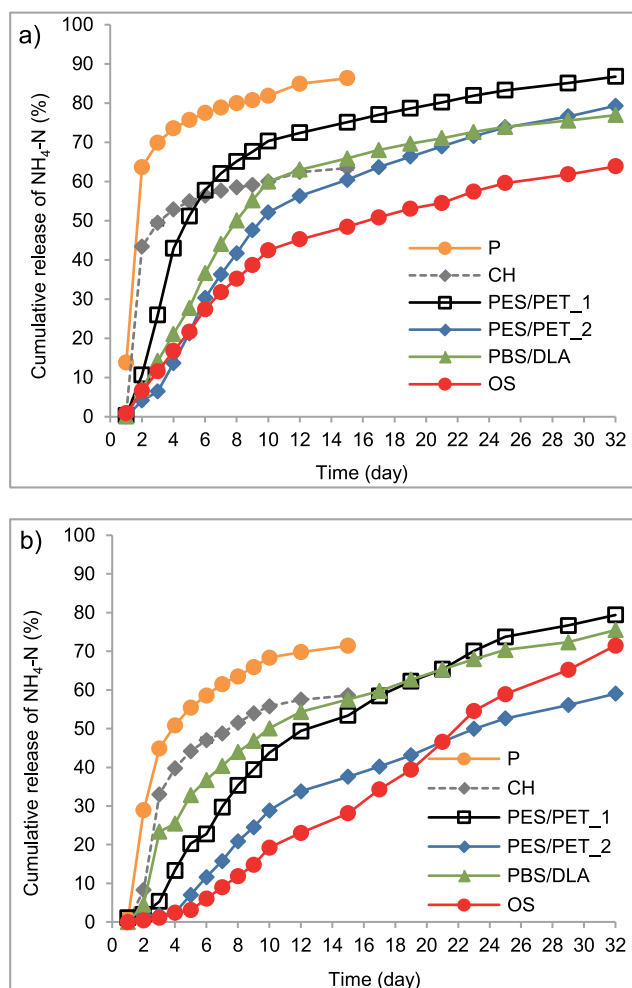


Fig. 3. Kinetics of $\text{NH}_4\text{-N}$ release from fertilisers in the soil environment under laboratory conditions from soil: a) soil S1; b) soil S2; explanations as in Fig. 1; source: own study

According to literature data the nutrient release process is complex and non-linear. Generally the release process from control release fertilisers (CRF) takes place in three different stages: lag period (I), constant release (II) and decay period (III), what can be described by a sigmoidal (S-shaped) release profile (Shaviv, 2001; Shaviv *et al.*, 2003; Du, Zhou and Shaviv, 2006; Lawrenciana *et al.*, 2021).

Based on kinetic data for soil S1, the $\text{NH}_4\text{-N}$ release process of all tested fertiliser forms included only two stages: constant release (II) and decay period (III). For soil S2, the release process was varied. For P and CH, the process included only stages II and III, for coated fertilisers PES/PET_1, PES/PET_2 and PBS/DLA a lag period (I) lasting a maximum of 4 days for PES/PET_2 can be distinguished. For the reference OS fertiliser, the lag period lasted the longest, the second stage – constant release – was clearly distinguished, while stage III – decay period was not achieved during the 32 days of the experiment (Fig. 3a, 3b).

The release data were fitted following the Korsmeyer–Peppas model (Eq. 1) in order to determine release mechanism by calculating the n value. All the parameters calculated were summarised in Table 4. Based on the n values, two dominant variants of the release mechanism can be distinguished. The first one, where the constant n has low values < 0.5 , indicating a mechanism based on quasi-Fickian diffusion (S1: P, CH and

Table 4. The release parameters of $\text{NH}_4\text{-N}$ release from fertilisers in soil

Fertiliser	Soil 1				Soil 2			
	K	<i>n</i>	R	time 75% release	K	<i>n</i>	R	time 75% release
P	0.439	0.284	0.922	7	0.235	0.457	0.943	13
CH	0.305	0.306	0.929	19	0.155	0.547	0.933	18
PES/PET_1	0.240	0.398	0.935	18	0.070	0.726	0.983	26
PES/PET_2	0.098	0.630	0.965	25	0.031	0.871	0.980	39
PBS/DLA	0.144	0.517	0.950	24	0.131	0.533	0.971	27
OS	0.097	0.564	0.976	38	0.008	1.312	0.997	32

Explanations: K = the release rate constant (day^{-1}), *n* = the exponent of release, R = correlation coefficient, P, CH, PES/PET_1, PES/PET_2, PBS/DLA, OS as in Fig. 1.

Source: own study.

PES/PET_1, S2: P). To the low *n* value in the release kinetic model, may have contributed high porosity of the fertiliser surface (P) and polymer surface to allowed an instantaneous water penetration into the matrix, dissolving the soluble content and release them, which was similar to the reports from previous researchers (Du, Zhou and Shaviv, 2006; Malekjani and Jafari, 2020; Jayanudin *et al.*, 2022). The higher diffusion exponent *n* value ($0.5 < n < 1.0$) obtained for S1: PES/PET_2, PBS/DLA, OS, S2: CH, PES/PET_1, PES/PET_2, PBS/DLA confirmed that addition to the diffusion, other mechanisms like swelling, sample dissolution or polymer decaying contribute to the release of $\text{NH}_4\text{-N}$. Only in one case, for the reference fertiliser OS (soil S2) obtained $n > 1$ which indicate an extreme case of transport mechanism represents relaxation behaviour (Malekjani and Jafari, 2020).

The release time of 75% $\text{NH}_4\text{-N}$ was determined for all tested fertilisers and ranges from 7 to 38 days for S1 and from 13 to 39 days for S2. It can be concluded that, in accordance with the recommendation of the European Committee for Standardization (CEN), the release rate of $\text{NH}_4\text{-N}$ from the tested coated fertilisers into the soil solution was slower compared to the Polifoska®6 fertiliser, but it depended on the type and thickness of the polymer layer.

ORTHOPHOSPHATE AS PHOSPHORUS ($\text{PO}_4\text{-P}$)

Phosphorus is one of the most important plant nutrients. The major part of soil phosphorus is not available to plants and is therefore often a limiting nutrient in crop production (Mengel *et al.* (eds.), 2001; Khan *et al.*, 2023). Plants take up phosphorus from the soil solution mainly in the form of orthophosphate ion therefore, investigating the process of phosphorus release from fertilisers, its concentration in the form of $\text{PO}_4\text{-P}$ was analysed.

The concentrations of $\text{PO}_4\text{-P}$ in the soil leachate were the highest in the initial period of research but the amplitude of changes was significantly lower (Fig. 4a, 4b), which is consistent with the research of Du, Zhou and Shaviv (2006), Newman *et al.* (2006), and Adams, Frantz and Bugbee (2013). This relationship can be observed the most in the case of fertiliser P. The highest concentration of $\text{PO}_4\text{-P}$ was determined in the first days of the experiment with a maximum peak on 2nd day at the level of 14–33.5% for S1 and S2, respectively. In total, within 15 days, from

50% (S1) to 76% (S2) of $\text{PO}_4\text{-P}$ used in the form of the multi-ingredient fertiliser P was released into the soil solution. In the case of CH, PES/PET_1, PES/PET_2 and PBS/DLA significant influence of the type and thickness of the polymer coating on the $\text{PO}_4\text{-P}$ release process was found. For CH, the release trend of $\text{PO}_4\text{-P}$ was similar to fertiliser P. The maximum $\text{PO}_4\text{-P}$ concentration was determined on 3rd day at level 7% (S1) and 22% (S2). In total, for CH from 32.4% (S1) to 51.3% (S2) of phosphorus in the form of $\text{PO}_4\text{-P}$ were released into the soil environment. For PES/PET 1, the maximum $\text{PO}_4\text{-P}$ release was determined on the 5th day at the level of 6.2% for S1 and on the 4th day at the level of 15% for S2. In the case of PES/PET_2, the $\text{PO}_4\text{-P}$ release process was more stable with the maximum of 2.5% for S1 and 4.3% for S2 on the 10th day. The release profile of $\text{PO}_4\text{-P}$ from the fertiliser coated with PBS/DLA was determined by soil conditions. For soil S1 was close to PES/PET_2 with a maximum of 3.4% on 10th day, while for soil S2 was close to PES/PET_1 with the maximum concentration of $\text{PO}_4\text{-P}$ on 5th day at a level of 13% of the applied dose.

The reference OS is characterised by a different release profile of $\text{PO}_4\text{-P}$ into the soil solution. For both soils, the amplitude of changes its concentration in leachates was very low and remained at the level of 0–0.5% for S1 and from 0 to 2.5% for S2 during the entire experiment, which indicates a very small tendency of $\text{PO}_4\text{-P}$ to release. Compared to fertiliser P, for all coated fertilisers on the 15th day of the experiment a significantly lower total degree of $\text{PO}_4\text{-P}$ release into the soil solution was obtained and are: S1 from 2.6% (OS) to 35.8% (PES/PET_1); S2 from 8.9% (OS) to 57.6% (PES/PET_1). Extending the research time to 32 days a higher degree of phosphorus release was obtained, but its general tendency to release is the same: PBS/DLA > PES/PET_1 > PES/PET_2 > OS (Fig. 5a, 5b).

Kinetic data (Fig. 5a, 5b) indicate that the $\text{PO}_4\text{-P}$ release profile from P and CH fertilisers include only two stages: constant release (II) and decay period (III). For coated fertilisers PES/PET_1, PES/PET_2 and PBS/DLA release profile of $\text{PO}_4\text{-P}$ corresponding to a sigmoidal release profile with a selected stage I – lag period, lasting several days, which is particularly visible for soil S1. For the reference OS, release $\text{PO}_4\text{-P}$ profile included only a clearly marked stage I (lag period) and the beginning of stage II (constant release), stage III – decay period was not reached within

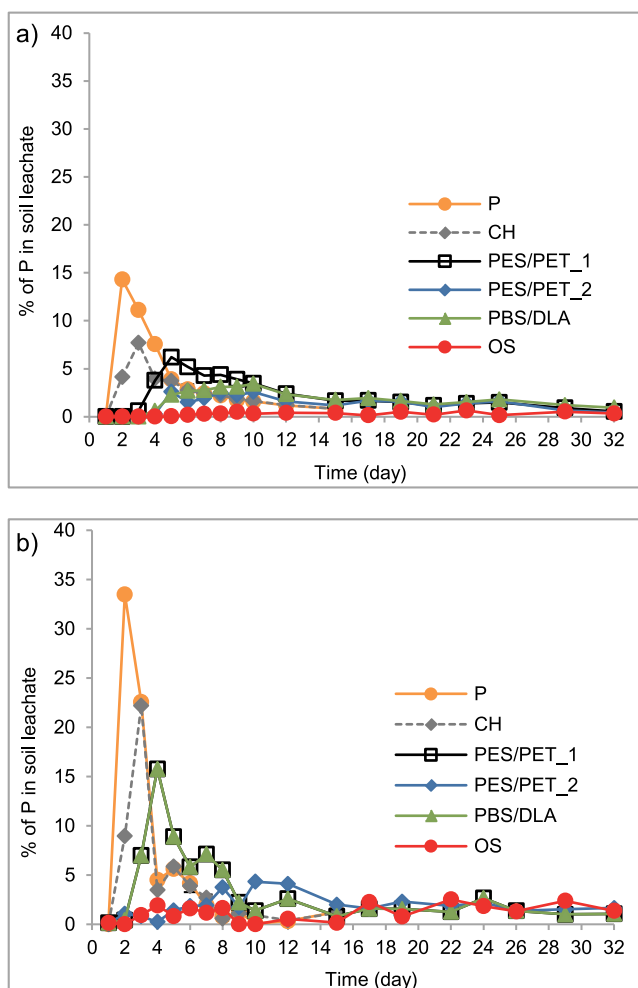


Fig. 4. Concentration changes of $\text{PO}_4\text{-P}$ in soil leachates: a) soil S1; b) soil S2; explanations as in Fig. 1; source: own study

32 days. The diffusion constants n determined based on the semi-empirical model (Eq. 1) confirms the complexity of the mechanism of $\text{PO}_4\text{-P}$ release from fertilisers, which is also influenced by the physicochemical properties of the soils. On loamy sand soil (S1) for P fertiliser and coated fertilisers CH, PES/PET_1, PES/PET_2, PBS/DLA obtained n values from 0.528 to 0.867 indicates that in addition to diffusion the phosphorus release process into the soil solution depends on additional mechanisms such as dissolution, swelling and polymer decomposition. Only for OS n value is close to 1.0 which corresponds to non-Fickian transport (case II), and $\text{PO}_4\text{-P}$ release is typical of zeroth order kinetics mechanism governed by phenomena of polymer degradation or degradation/dissolution of fertiliser core. For sandy loam (S2), two $\text{PO}_4\text{-P}$ release mechanisms dominate. The first one where the constant n has low values (<0.5), which indicates a mechanism based on quasi-Fickian diffusion (P, PES/PET_1, PBS/DLA) in which $\text{PO}_4\text{-P}$ diffuses partially through the a swollen matrix and water filled pores in the fertiliser granules, and the second where n takes values $0.517 < n < 0.905$ (CH, PES/PET_2, OS) which indicates anomalous transport or non-Fickian transport (Tab. 5).

The polymer coatings significantly extends the time of 75% release of $\text{PO}_4\text{-P}$ into the soil solution, which is in accordance with the recommendation of the European Standardization

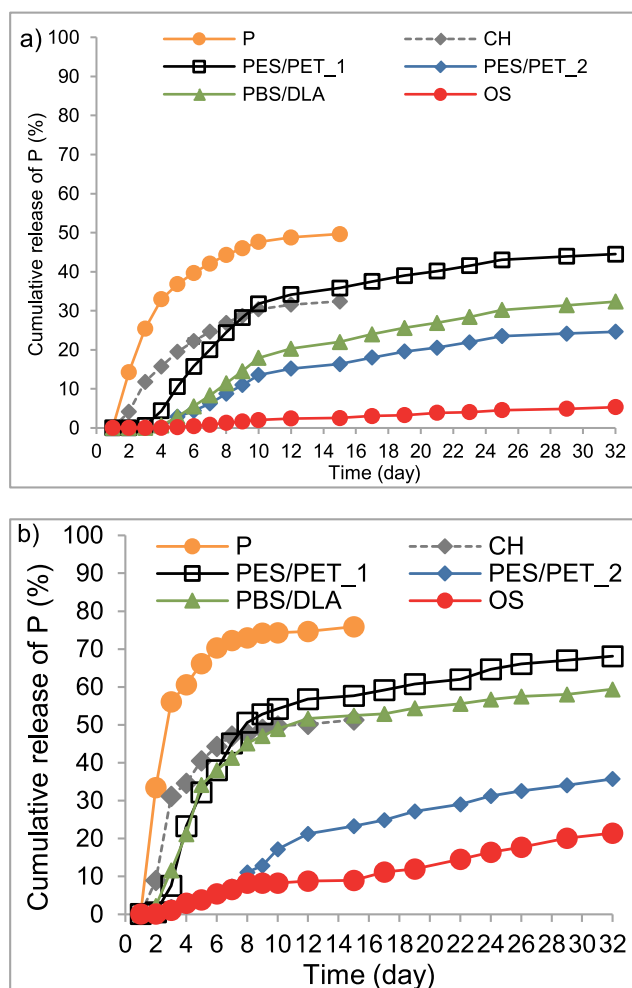


Fig. 5. Kinetics of $\text{PO}_4\text{-P}$ release from fertilisers in the soil environment under laboratory conditions from soil: a) soil S1; b) soil S2; explanations as in Fig. 1; source: own study

Committee. The calculated times of 75% $\text{PO}_4\text{-P}$ release can be arranged in the order $\text{P} < \text{CH} < \text{PES/PET}_1 < \text{PBS/DLA} < \text{PES/PET}_2 < \text{OS}$ (Tab. 5). Research shows that an important factor influencing the determined parameter are the physicochemical properties of the soil, including its permeability. On a soil of the sandy nature (S1), characterised by higher permeability, the obtained values of 75% $\text{PO}_4\text{-P}$ release are significantly longer, which may be due to the shorter contact time of the fertiliser with water/soil solution. This is important in the processes of dissolution of the fertiliser material, swelling and degradation of the polymer coating.

Generally, the release rate of phosphates is lower than that of NH_4^+ , which is confirmed by a series of studies, in which the differences in release rates vary and range from 10% to 75% (Shaviv, 2001; Du, Zhou and Shaviv, 2006; Lubkowski, 2014; Lawrencja *et al.*, 2021). This is due to presence of strong interactions among nutrients in the fertiliser granule and differences in nutrient solubility. Additionally these processes are greatly affected by the type of the medium used. Numerous studies show that fastest nutrient release rate is observed in water and in the case of CRF depends on the polymer structure and its content in the investigated materials (Du, Zhou and Shaviv, 2006, Adams, Frantz and Bugbee, 2013; Lubkowski *et al.*, 2016, Kassem *et al.*, 2024).

Table 5. The release parameters of PO₄-P release from fertilisers in soil

Fertiliser	Soil 1				Soil 2			
	K	n	R	time 75% release	K	n	R	time 75% release
P	0.138	0.528	0.949	25	0.297	0.404	0.912	10
CH	0.059	0.683	0.964	41	0.150	0.517	0.923	22
PES/PET_1	0.052	0.661	0.945	57	0.143	0.487	0.926	30
PES/PET_2	0.014	0.867	0.967	99	0.017	0.908	0.979	65
PBS/DLA	0.019	0.863	0.965	71	0.145	0.443	0.922	41
OS	0.001	1.087	0.984	342	0.009	0.905	0.987	128

Explanations as in Tab. 4.

Source: own study.

CONCLUSIONS

Effective reduction of the negative impact of mineral fertilisers on the environment caused by the nutrient losses may be achieved by the use of controlled-release fertilisers prepared with the use of biodegradable polymer materials. Depending on the used polymers, the obtained fertiliser materials differed from each other with the rate and degree of analysed nutrients release to the soil solution. The largest changes of pH and conductivity of soil leachates occurred in the initial period of release experiments for all tested fertilisers. The amplitude of their changes was the highest for NPK Polifoska®6 fertiliser but it was significantly smaller for the coated fertilisers, especially for the reference Osmocote fertiliser and for the fertiliser coated with polyethylene (succinate-co-terephthalate) with the polymer to fertiliser ratio 0.26. These changes are closely correlated with the amount of nutrients released into the soil solution. The highest release degree was observed for NPK Polifoska®6 fertiliser: the release of NH₄-N was 0.71 for loamy sand soil (S1) and 0.86 for sandy loam soil (S2) and the release of PO₄-P was 0.50 and 0.76 for S1 and S2, respectively. All polymer coatings (CH, PES/PET, PBS/DLA) significantly reduced the nutrients release degree from fertilisers and on the 15th day of release their values were 0.38–0.59 (NH₄-N) and 0.16 – 0.32 (PO₄-P) for the S1 and 0.60–0.75 (NH₄-N), 0.23–0.58 (PO₄-P) for the S2.

The polymer layer thickness is an additional factor modifying the release degree of nutrients. For the fertiliser coated with polyethylene(succinate-co-terephthalate) (PES/PET) with the polymer to fertiliser ratio 0.26 the release degree throughout the entire experiment was lower in comparison with the material with the ratio 0.19 and on 32nd day these differences were 8.6–25.7% for NH₄-N and 44.6–47.6% for PO₄-P. It was proved that using the semi-empirical kinetic model it is possible to specifically describe the release of nutrients from the polymer-coated fertilisers using an exponential relationship between the release and time. The values of the diffusion exponent indicate that two mechanisms dominate in the release process of nutrients: the mechanism based on quasi-Fickian diffusion and non-Fickian (anomalous case) mechanism where the release degree is governed not only by diffusion, but also by swelling, fertiliser dissolution, and polymer decomposition. The investigation showed that an important factor influencing the release degree of nutrients are the physicochemical properties of the soil and

interactions between fertiliser core – polymer coating – soil solution. This is confirmed by differences in degree release and times of 75% release of NH₄-N and PO₄-P. The results of the presented experiments could be used in the preparation of controlled release fertilisers, in the selection of the polymer type and its thickness, as well as in the selection of fertiliser dose that determines the optimal efficiency of nutrient uptake by plants. This approach to the issue could be beneficial both in environmental and economic contexts.

CONFLICT OF INTERESTS

All authors declare that they have no conflict of interests.

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