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Valorisation of Deinking Paper Sludge for Fertiliser Purposes: New Perspective in Sustainable Agriculture

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Abstract: The growth of the global population, coupled with concomitant economic development, has resulted in the generation of a substantial quantity of waste. The transition of the European Union's economy towards a closed-loop model is prompting a comprehensive search for waste management concepts across a range of industrial sectors. The objective of this study is to valorise deinking paper sludge, which has a high potential for soil formation due to its high organic matter content. To produce organic–mineral fertiliser, the deinking sludge was subjected to acid hydrolysis, then neutralised with KOH solution and enriched with poultry litter ash. The final products were characterised in terms of their nutrient and heavy metal content. The bioavailability of phosphorus, along with the forms in which it occurs in fertilisers, was determined through the implementation of a five-step fractionation procedure. Furthermore, an eight-week incubation period was conducted to assess the fertilisers' performance in soil. Soil samples were tested on a weekly basis for pH, water-soluble and bioavailable phosphorus content using the spectroscopic method after previous extraction in water and Bray's solution, and catalase activity using the titrimetric method. The resulting fertilisers were found to meet the requirements for organo-mineral fertilisers and were categorised as PK-type fertilisers with a total nutrient content of 24.6–39.3%. Fractionation studies demonstrated that the fertilisers contained 20–30% of the total potentially bioavailable phosphorus. Furthermore, the long-term release of phosphorus from the fertilisers was confirmed through incubation studies. Additionally, the fertilisers were observed to contribute to an increase in catalase activity in the soil.

Keywords: fertilisers; organic matter recycling; deinking paper sludge; phosphorus fractionation; waste management

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1. Introduction

The global agricultural sector is confronted with a multitude of challenges that impinge upon its capacity to ensure food security for an expanding population [1]. On the one hand, there is the adaptation of crops to changing vegetation zones and extreme weather conditions [2], disrupted supply chains due to armed conflicts or depleted raw material resources [3]. On the other hand, the degradation of productive functions and ecosystems due to the loss of biodiversity [4], the impact of fertilised crops on environmental degradation and the adaptation of agriculture to the requirements of the European Union (EU) strategy [5]. For example, the EU agricultural strategy has the objective of reducing nutrient losses by a minimum of 50% by 2030. This is of particular significance in the context of soil degradation and the eutrophication of water bodies. The strategy encourages the utilisation of organic fertilisers, precise fertilisation techniques and crop rotation. Moreover, the EU strategy advocates the recovery and reuse of nutrients from organic waste streams, thereby facilitating the closure of nutrient cycles. A significant focus is placed on the enhancement of organic matter content within the soil, which serves as a reservoir for nutrients [6].

In addition to crop rotation, mulching, conservation tillage [7], and maintaining plant cover, the use of organic and organo-mineral fertilisers represents a further key practice [8].

These fertilisers enrich the soil with organic matter, thereby improving its structure, water retention capacity and aeration. They also support the development of soil microorganisms and provide the necessary nutrients in a timely manner [5].

The implementation of the principles of the circular economy has led to an increase in the use of biomass as a source of organic matter in fertilisers [9,10]. This biomass is produced from municipal and industrial waste materials, including plant residues, manure, lignocellulosic biomass, paper waste as well as compost and digestate [11]. The mineral component of these fertilisers is ash produced after incineration of biomass like sewage sludge, chicken manure, or wood [12,13]. In light of the EU strategies and the considerable quantity of lignocellulosic biomass produced (exceeding 18 billion tons per year) as well as waste generated from the paper industry and recycling (11 million tons in the EU, of which 7.7 million are from recycling), it can be seen that its intrinsic properties, including the capacity to enhance soil structure, mitigate erosion, and stimulate microbial activity, render it a promising renewable organic raw material with significant fertilising potential following appropriate processing [14–17]. These processes include composting [18,19], fermentation [15], and enzymatic or chemical hydrolysis [20–22].

The utilisation of waste materials derived from the paper industry as components of substrates or soil improvers has been the subject of extensive analysis [23,24]. The waste in question is characterised by a high cellulose content and a high carbon-to-nitrogen ratio, in addition to a high biological oxygen demand BOD, which causes short-term nitrogen sequestration in the soil [24]. Following the processes of composting or hydrolysis, this waste has the potential to serve as an organic component of fertilisers [25]. The findings of recent research [14,19,26] indicate its use as a means of supplementing compost with organic matter and minerals, thereby representing a potential component of gardening substrates. Zawadzińska et al. [14] corroborated the assertion that a compost of superior quality, formulated from pulp and paper mill sludge, fruit and vegetable waste, processed mushroom substrate and rye straw, has a beneficial impact on the yield and quality of cherry tomato fruit (*Lycopersicon esculentum* Mill). The impact of paper waste and peat mixtures on the yield of marigold (*Calendula officinalis* L.), petunia (*Petunia* × *hybrid* L.) and Matthiola (*Matthiola incana* L.) plants was investigated by [26]. It was demonstrated that up to 30% of paper waste can be substituted for peat in the cultivation of marigold and petunia plants, resulting in an increased number of buds but a concomitant reduction in chlorophyll content and a decrease in both height and diameter of the plants.

A further problematic by-product of the recycling of paper is the residue resulting from the deinking process. The deinking stage, which occurs subsequent to repulping, serves to augment the strength and visual appeal of the final product. Flotation is the most common method employed for this process [23]. These wastes are distinguished by a high concentration of carbon and nutrients, which renders them suitable for utilisation in organic plant fertilisation and mulching [27–29].

A review of the literature reveals a dearth of information concerning the utilisation of this specific type of waste in the production of organo-mineral fertilisers. The objective of the presented research is to investigate the potential use of deinking paper sludge, following hydrolysis, as a source of organic matter in the composition of organo-mineral fertilisers based on other waste materials, specifically ashes from the incineration of poultry litter. The resulting fertilisers were evaluated in accordance with the relevant regulatory framework and their efficacy was assessed through fractionation tests and a six-week incubation period in soil. The potential for utilising valuable components from waste materials to support both organic and mineral fertilisation represents a significant advancement in the implementation of circular economy principles.

2. Materials and Methods

2.1. Materials

2.1.1. Waste Paper Pulp

Deinking paper sludge was obtained from a local company (Małopolska region, Poland) specialising in the recycling of paper products. The material is generated as a by-product of the flotation stage. The characterisation of the material is presented in Table 1.

Table 1. Characterisation of deinking paper sludge (values expressed on dry weight basis, except moisture content).

	pH	8.75 ± 0.05
	Moisture content (%)	65.1 ± 0.5
	Organic matter content (%)	68.2 ± 0.005
Main nutrients	K_2O (%)	0.15 ± 0.01
	P_2O_5 (%)	0.01 ± 0.002
Secondary nutrients	Ca (%)	1.22 ± 0.02
	Mg (%)	0.25 ± 0.01
Micronutrients	Zn (mg/kg)	226 ± 7
	Fe (mg/kg)	3724 ± 26
	Cu (mg/kg)	178 ± 10
	Mn (mg/kg)	338 ± 4
	Co (mg/kg)	22 ± 1
Heavy metals	Cd (mg/kg)	5.0 ± 0.4
	Pb (mg/kg)	<18
	Cr (mg/kg)	42 ± 1
	Ni (mg/kg)	56 ± 8

The high moisture content of deinking paper sludge makes impossible its thermal utilisation. We analysed waste rich in organic matter (68.2%), which is beneficial for soil. In addition, it contains secondary nutrients and micronutrients. The concentration of heavy metals in deinking paper sludge was found to be higher than that reported in the literature [27,30], which is primarily attributable to the source of the paper. The agricultural utilisation of such sludge can pose some environmental hazards. Heavy metals are characterised by their persistence in the environment and their capacity to be absorbed by soil particles and taken up by plants, which can result in a reduction in crop yield and the contamination of food items. Furthermore, heavy metals have the ability to cause a decline in microbial diversity, penetrate into aquatic systems and subsequently lead to adverse effects on ecosystems and human health [31,32].

2.1.2. Poultry Litter Ash

The poultry litter ash used in the study was derived from an industrial incinerator. The ash was found to contain 24.7% K_2O and 18.7% P_2O_5 of potassium and phosphorus, respectively, which makes it a promising alternative raw material for the production of fertilisers. Additionally, poultry litter ash is a rich source of secondary nutrients and micronutrients, which are essential for the optimal growth and development of plants. Given the alkaline pH and relatively high content of Cr and Ni, the direct use of ash as a fertiliser is not recommended (Table 2). The availability of heavy metals from poultry litter ash is contingent upon the prevailing soil conditions, particularly the pH value. In acidic conditions, the heaviest metals become more soluble, thereby increasing their potential for leaching. An elevated concentration of H^+ ions reduces the absorption of metal ions in the soil, thereby releasing them into the soil solution. The potential leaching of heavy metals from the soil is also significantly influenced by organic matter and water content, as well as soil structure [33,34].

Table 2. Characterisation of poultry litter ash (values are expressed on dry mass).

pH		12.0 ± 0.2
Main nutrietns	K_2O (%)	24.7 ± 0.8
	P_2O_5 total (%)	18.7 ± 0.3
	P_2O_5 water soluble (%)	0.07 ± 0.01
Secondary nutrients	Ca (%)	9.68 ± 0.47
	Mg (%)	6.27 ± 0.03
Micronutrients	Zn (mg/kg)	2288 ± 29
	Fe (mg/kg)	782 ± 1
	Cu (mg/kg)	694 ± 19
	Mn (mg/kg)	28 ± 1
	Co (mg/kg)	76 ± 5
Heavy metals	Cd (mg/kg)	5.3 ± 0.1
	Pb (mg/kg)	19.1 ± 0.4
	Cr (mg/kg)	175 ± 3
	Ni (mg/kg)	110 ± 8

2.1.3. Soil

The fertilisers were incubated using soil that had been previously subjected to a drying process at 105 °C and subsequently ground. The detailed characteristics of the soil are presented in Table 3.

Table 3. Characterisation of soil.

pH	pH_{H_2O}	5.26 ± 0.02
	pH_{KCl}	4.40 ± 0.05
Organic matter content (%)		4.94 ± 0.01
Main nutrients	K_2O (%)	0.014 ± 0.002
	P_2O_5 total (%)	0.118 ± 0.005
	P_2O_5 water soluble (%)	0.010 ± 0.001
Secondary nutrients	Ca (mg/kg)	405 ± 20
	Mg (mg/kg)	392 ± 14
Microelements	Zn (mg/kg)	77 ± 3
	Fe (mg/kg)	9203 ± 41
	Cu (mg/kg)	10 ± 1
Heavy metals	Cd (mg/kg)	2.1 ± 0.3
	Pb (mg/kg)	<12
	Cr (mg/kg)	39 ± 3
	Ni (mg/kg)	13 ± 1
Bulk density (g/cm^3)		0.943 ± 0.032

The soil was distinguished by a relatively low concentration of essential nutrients. It exhibited notable quantities of iron (9203 mg/kg). Based on the determined pH, it can be concluded that the analysed soil belongs to the category of acidic soils. The soil pH value has a considerable influence on numerous factors that determine its physicochemical properties, including the availability of nutrients. The concentration of heavy metals in the soil was found to be at a relatively low level.

2.2. Methods

2.2.1. Fertiliser Manufacture Process

Acid hydrolysis was carried out to partially decompose deinking paper sludge and obtain a consistency that would allow the production of a homogeneous fertiliser product. Table 4 shows the conditions for acid hydrolysis of waste. Concentrated sulphuric acid, 75% sulphuric acid solution, and a mixture of concentrated sulphuric acid and phosphoric acid in a volume ratio of 1:1 were tested as hydrolysis agents. In addition, different ratios of liquid (LF) to solid phase (SF) were tested.

Table 4. Deinking paper sludge hydrolysis conditions.

Lp	LF:SF	Hydrolysis Agent	Neutralisation Agent		pH Value	Organic Matter Content (%)
			KOH Solution	Poultry Litter Ash		
1	1:1	H_2SO_4 (98%)	−	+	1–2	6.64
2	1:1		+	+	6–7	5.80
3	2:1		−	+	1–2	12.1
4	2:1		+	+	5–6	9.80
5	3:1		−	+	1–2	16.5
6	3:1		+	+	6–7	16.7
7	1:1	H_2SO_4 (75%)	+	+	6–7	7.43
8	2:1		+	+	6–7	14.0
9	3:1		+	+	6–7	21.1
10	1:1	H_2SO_4 (98%) + H_3PO_4 (75%)	+	+	6–7	7.30
11	2:1		+	+	6–7	14.7
12	3:1		+	+	6–7	21.7

When deinking paper sludge was hydrolysed with concentrated sulphuric acid, an attempt was made to neutralise the resulting pulp using only alkaline poultry litter ash. However, the amount of ash added was too low to neutralise an acidity of the pulp to the required level due to its thick consistency, so a 40% potassium hydroxide solution had to be used for this purpose. The addition of the KOH solution increased the content of the main component of the fertiliser, potassium, and also made it possible to obtain a pH of the mixes at the level of 6–7, which is beneficial for the soil.

The selection of fertiliser mixtures to the next stages of research was based on their organic matter content and pH. According to the requirements [35], the minimum organic matter content in organo-mineral fertilisers should be 20%. In the case of mixtures 9 and 12, the organic matter content was higher than the required value, allowing them to be included in this group. These mixtures also met the pH requirements, which allowed them to be selected as potential fertiliser products.

In the next stage, fertiliser mixtures 9 and 12 were produced on a larger scale (500 g). The diagram of the production process is shown in Figure 1. Deinking paper sludge was hydrolysed with 75% sulphuric acid or a mixture of concentrated sulphuric and phosphoric acid (1:1) in the ratio SF:LF 3:1, neutralised with 40% potassium hydroxide solution and enriched with poultry litter ash until the mixture reached pH 6–7. The resulting fertiliser mixture was then pelletised and dried at 105 °C.

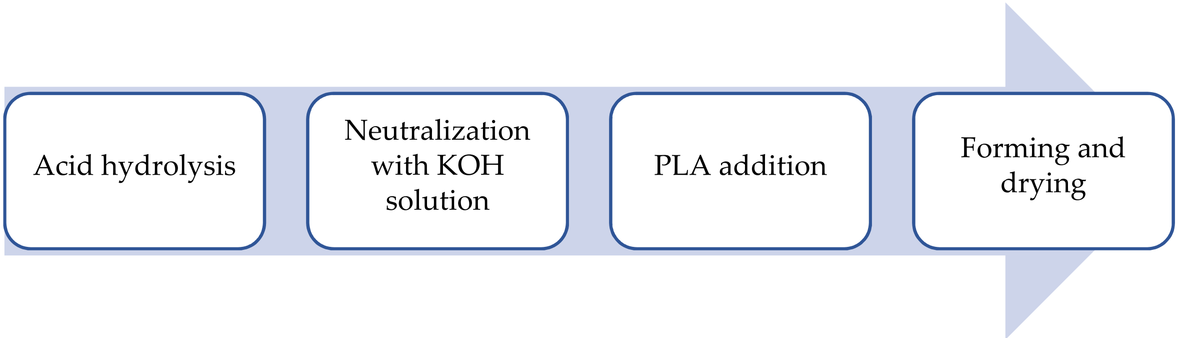

Figure 1. Diagram of fertiliser production process.

Figure 2 shows the composition of the fertilisers produced. In the case of both fertilisers, the mass of deinking paper sludge was about 30% of the total mass of the fertiliser. The addition of PLA, which constituted 11–13% of the total mass of the fertiliser, made the content of alternative raw materials in the fertilisers 41–42%, depending on the fertiliser.

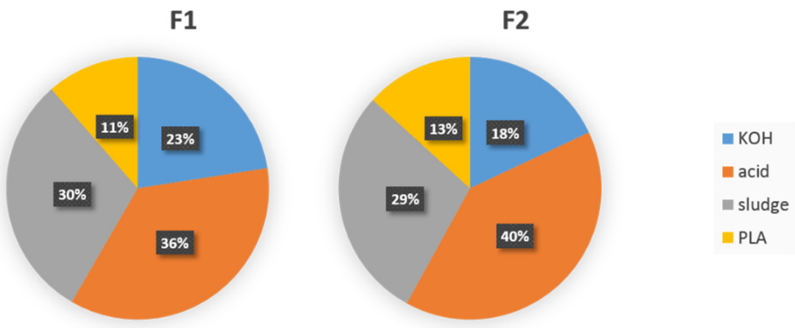

Figure 2. Composition of fertilisers.

2.2.2. Elemental Analysis

The raw materials (deinking paper sludge, poultry litter ash), fertilisers and soil were subjected to analysis with the objective of determining the concentration of selected elements. The atomic absorption spectroscopy (AAS) (AAnalyst 300, PerkinElmer, Waltham, MA, USA) was employed to determine the content of calcium (Ca), magnesium (Mg), potassium (K), iron (Fe), zinc (Zn), copper (Cu), manganese (Mn), cobalt (Co), nickel (Ni), chromium (Cr), lead (Pb), and cadmium (Cd) after digestion in aqua regia [36].

2.2.3. Phosphorus Determination

Two colorimetric methods were used to determine the phosphorus content in the tested samples. The molybdenum blue method was employed for the analysis of water-soluble and available phosphorus content in soil samples after previous extraction in water and Bray's solution the answers [37]. The vanadate-molybdate method according to [38] was used for the determination of phosphorus content in poultry litter ash, soil, and fertilisers.

2.2.4. Organic Matter Determination

The organic matter content of deinking paper sludge and fertilisers was determined by subjecting the samples to a three-hour roast at 550 °C in a muffle furnace, after which the mass loss on ignition was calculated based on the mass difference [39].

2.2.5. pH Determination

In order to determine the pH of the soil, both a solution in water and a 1 M KCl solution were employed [40]. To determine the pH of the fertilisers and soil during incubation, 10% aqueous solutions were prepared. Following a period of two hours, the pH value of the supernatant solution was determined using a CX-701 metre (Elmetron, Zabrze, Poland).

2.2.6. Phosphorus Fractionation

Phosphorus fractionation in fertilisers and poultry litter ash was conducted by adding 50 mL of the extraction solution to the analysed material (weighing approximately 2 g) and agitating for a specified time period, as outlined in Table 5. Subsequently, the samples were subjected to centrifugation for 20 min at 6000 rpm. After separation, the liquid phase was transferred to a separate container and allowed to stand for spectrophotometric molybdenum blue analysis. The sediment was then rinsed with distilled water and covered with a further extraction solution. The sediments remaining after fraction IV were subjected to mineralisation in aqua regia in order to determine the residual phosphorus content [41].

Table 5. Scheme of phosphorus fractionation.

Fractions	Extraction Solution	Time	Phosphorus Occurence Forms
I	1 M NH_4Cl	1 h	Potentially available p
II	0.11 M $Na_2S_2O_4/NaHCO_3$	1 h	Fe-bounded
III	1 M NaOH	18 h	Al-bounded
IV	0.5 M HCl	18 h	Ca-bounded
V	Aqua regia		Residual

2.3. Soil Incubation

In order to ascertain the impact of fertilisers on soil properties, as well as on the content of nutrients and the forms in which they occur, an eight-week incubation period was conducted, during which fertilisers were introduced into the soil. For this purpose, 50 g of previously dried and ground soil (with a particle size of less than 0.2 mm) was weighed into 100 mL of plastic containers. Subsequently, 1 g of fertiliser was weighed and an amount of water equivalent to the maximum sorption capacity of the soil at 70% was added. Additionally, control samples of soil devoid of fertiliser were prepared. The containers were hermetically sealed and placed in an incubator maintained at 25 °C. Subsequently, after each of the eight weeks, the following parameters were subjected to analysis:

The following parameters were analysed at regular intervals:

- pH
- water-soluble phosphorus content
- bioavailable phosphorus content (Bray test)
- organic carbon content
- catalase activity

2.4. Catalase Activity

In order to ascertain the activity of catalase in soil, the manganometric method was employed [42]. The method entails titrating the residual hydrogen peroxide present in the sample, which has not been decomposed by the catalase present in the sample. The titrant employed is potassium permanganate in an acidic environment. In order to perform the determination, the soil sample was incubated for 20 min with 5 mL of a 0.3% hydrogen peroxide solution. Subsequently, 5 mL of 1.5 M sulfuric acid was added to the sample and filtered into volumetric flasks. The determination of catalase activity entails titrating the filtrates with 0.01 M potassium permanganate until a pale pink colour is observed.

2.5. Organic Carbon Content

The organic carbon content was determined by employing the modified Tiurin method [43]. The process entails the oxidation of organic carbon to carbon dioxide in a strongly acidic environment, utilising $K_2Cr_2O_7$ as the oxidant. A solution of 0.1 M Mohr's salt is employed as the reducing agent for the excess oxidant. To perform the determination, approximately 2 g of previously dried and ground soil sample was weighed into an Erlenmeyer flask. Subsequently, 20 mL of a 0.067 M potassium dichromate solution and 15 mL of concentrated sulfuric acid were added. The contents of the flask were heated until boiling point and maintained at this temperature for a period of five minutes. Subsequently, the solution was cooled and transferred to a 200 mL flask, where it was filtered. The organic carbon content was determined spectrophotometrically at a wavelength of 590 nm.

3. Results

3.1. Characterisation of Fertilisers

Table 6 shows the characterisation of the fertilisers obtained. Based on the content of the main nutrients, the fertilisers can be classified as PK-type fertilisers. Phosphorus fertilisation of plants is extremely important, especially at the beginning of their vegetation. It is responsible for proper rooting, plant tillering, increases resistance to disease and also limits the accumulation of harmful nitrates. Phosphorus deficiency results in poor root development, making it difficult for plants to absorb water and other nutrients [44]. The F2 fertiliser was characterised by a phosphorus content five times higher than the F1 fertiliser, due to the use of phosphoric acid in the F2 fertiliser production process. Potassium is a nutrient that influences most of the biochemical and physiological processes that affect the proper growth and metabolism of plants [45]. The potassium content in both fertilisers was similar. The source of potassium in the fertilisers was poultry litter ash and potassium hydroxide solution. For the produced organo-mineral fertilisers F1 and F2, the sum of the main nutrient contents (P and K) was 24.6% and 39.3%, respectively, which is characteristic for mineral fertilisers. The fertilisers met the requirements for organic matter content in accordance with Polish regulations.

Table 6. Characterisation of fertilisers (values are expressed on dry mass).

		F1	**F2**	**Requirements [35]**
	Organic matter content (%)	21.0 ± 0.1	23.1 ± 0.1	20
Main nutrients	N (%)	0.22 ± 0.05	0.27 ± 0.1	1
	P_2O_5 (%)	3.12 ± 0.05	15.3 ± 0.02	0.5
	K_2O (%)	21.5 ± 0.6	23.9 ± 0.3	1
Secondary nutrients	Mg (%)	0.69 ± 0.02	0.68 ± 0.02	
	Ca (%)	8.4 ± 0.4	6.1 ± 0.2	
Microelements	Zn (mg/kg)	157 ± 7	164 ± 2	
	Fe (mg/kg)	945 ± 11	1128 ± 10	
	Co (mg/kg)	7 ± 0.2	8 ± 0.3	
	Cu (mg/kg)	252 ± 8	275 ± 6	
	Mn (mg/kg)	627 ± 8	687 ± 12	
Heavy metals	Pb (mg/kg)	<31	<31	140
	Cr (mg/kg)	16 ± 0.2	17 ± 0.4	100
	Ni (mg/kg)	17 ± 1	16 ± 1	60
	Cd (mg/kg)	4 ± 0.4	3 ± 0.2	5

Fertiliser products were characterised by a high content of secondary nutrients such as calcium and magnesium. The presence of magnesium affects the photosynthetic process, nitrogen management in plants and also activates enzymes. Calcium, in turn, supports the functioning of the root system and is also a building block of cell walls [46]. The F1 and F2 fertilisers produced are enriched with microelements (Fe, Zn, Cu, Mn). Based on the characteristics of fertilisers, it can be expected that their addition to soils poor in nutrients has a beneficial effect on the development and proper functioning of plants. The content of heavy metals in fertilisers did not exceed the maximum levels allowed by Polish legislation.

3.2. Phosphorus Fractionation Results

One of the most crucial elements in the context of plant utilisation of nutrients is the form in which they are presented in fertilisers. The fractionation analysis, results of which is presented in Figure 3, enabled the isolation of five fractions of phosphorus and an evaluation of its potential bioavailability for plants.

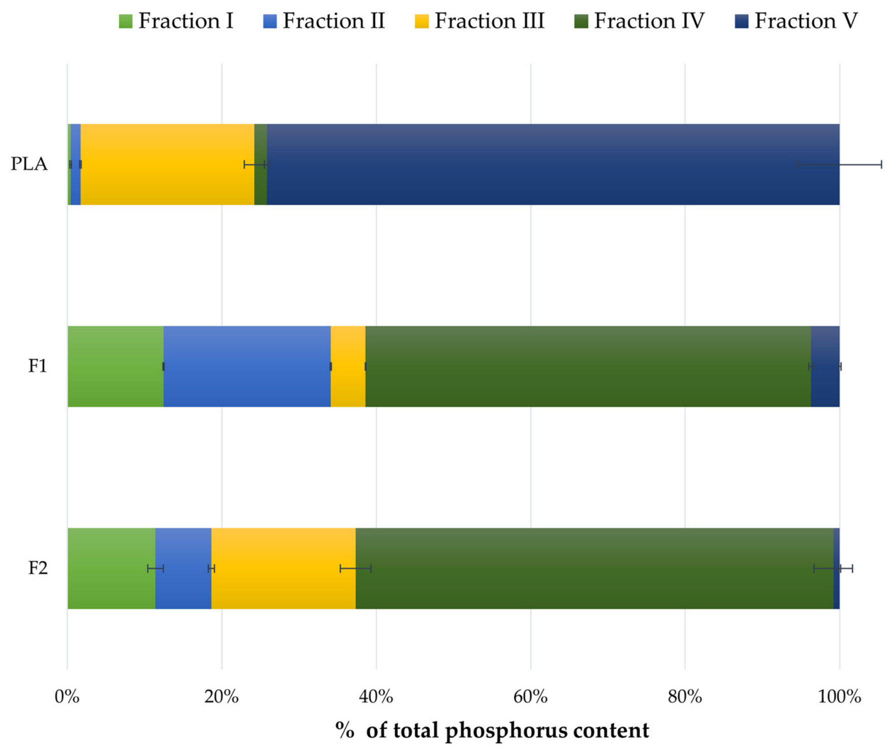

Figure 3. Phosphorus fractionation results.

In the case of poultry litter ash, the phosphorus content of fraction V was found to be 74%. This represents permanently immobilised phosphorus, which is unavailable to plants. It is phosphorus in the form of amorphous calcium phosphate and aluminosilicates associated with potassium, calcium, manganese and iron [47,48]. The ash was found to contain only 0.4% readily available phosphorus, with phosphorus bound to aluminium, iron or calcium representing 26% of the total phosphorus content. The results of the analysis demonstrate that chemical reactions occurring during the production of fertilisers result in a change in the forms of occurrence of the phosphorus present in the ash. The proportion of readily available phosphorus in fertilisers F1 and F2 was found to be 12% and 11% of the total phosphorus content, respectively. The proportion of phosphorus bound to aluminium, iron, and calcium is approximately 85% of the total phosphorus content. It is hypothesised that this form of phosphorus may become available to plants over time, contingent on soil conditions such as moisture content, pH or temperature [41]. The phosphorus fraction designated as V, or residual fraction, constituted 4% of the total phosphorus content in fertiliser F1 and 1% in fertiliser F2.

The outcomes of phosphorus fractionation permit an initial evaluation of the accessibility of phosphorus from fertilisers for plants. However, the behaviour of phosphorus in soil is a highly complex issue, shaped by a number of factors including chemical reactions, interactions with soil components, and the environmental conditions of the soil. The soluble form of phosphorus can be absorbed onto soil particles over time, which consequently reduces its immediate availability to plants. Moreover, phosphorus can undergo transformation into stable mineral forms, including aluminium and iron phosphates in acidic conditions and calcium phosphates in alkaline conditions. The unused phosphorus may leach out, which could have implications for the environment [49].

3.3. Soil Incubation Results

3.3.1. pH Value

The optimal soil pH has a considerable influence on the absorption of phosphorus by plants. Phosphorus is predominantly absorbed in the form of dihydrogen phosphate ions ($H_2PO_4^-$), which are present in the soil solution within a pH range of 5.5 to 7.0 [50]. In both cases, when the pH level is below or above the optimum range for phosphorus uptake, phosphorus can be absorbed on soil particles or precipitate as iron or aluminium phosphates (pH < 5.5) or calcium phosphates (pH > 7.5). As a result, it becomes unavailable to plants. Figure 4 illustrates the fluctuations in soil pH throughout the eight-week incubation study. A similar trend was observed for all samples, with an increase in pH value up to six weeks of incubation and then a slight decrease. In the case of control soil pH value increased from 6.31 to 6.92 and after eight weeks it was 6.49. Due to its slightly acidic pH, the addition of F1 fertiliser lowered the soil pH to 6.18. The maximum pH value of the soil (6.71) was reached in the sixth week of incubation studies.

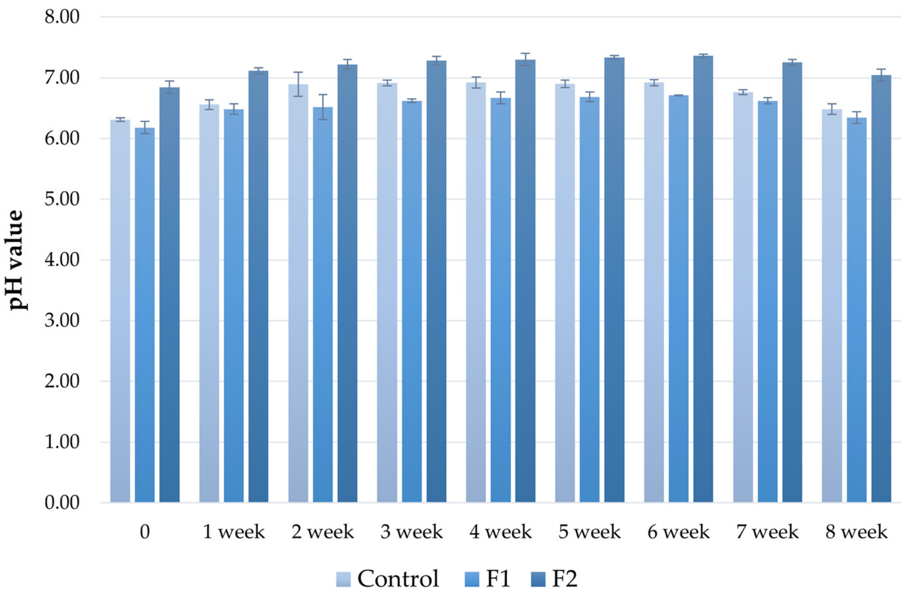

Figure 4. Changes in soil pH values during incubation studies.

In soils with an acidic pH, this fertiliser may contribute to an increase in soil pH, which in turn may lead to an increase in the solubility of phosphates associated with iron or aluminium. The effect of F1 fertiliser on the soil is diametrically opposed. The addition of the fertiliser to the soil resulted in a slight decrease in the pH value throughout the course of the experiment, which increased the solubility of calcium phosphates.

3.3.2. Organic Carbon Content

The presence of organic matter in soil can markedly enhance the accessibility of nutrients for plants, thereby influencing their storage within the soil. It serves as the primary reservoir for nitrogen and sulphur, in addition to iron and copper. Furthermore, it has the capacity to strongly bind toxic heavy metals, rendering them unavailable to plant roots [51]. Figure 5 presents a comparison of the organic carbon content in soil treated with F1 and F2 fertiliser and in the control sample.

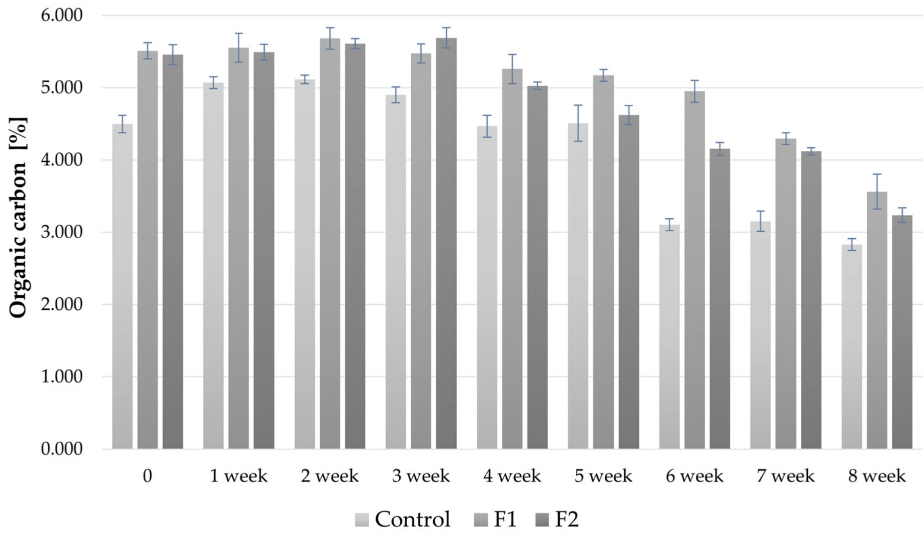

Figure 5. Changes in organic carbon content during incubation studies.

The addition of fertilisers has been demonstrated to result in a notable increase in the organic carbon content of soil, with a mean increase of approximately 20%. The organic carbon content remained at a comparable level for the samples treated with fertilisers throughout the initial three-week period of the experiment, exhibiting a slight increase in the case of the control sample. A decline in organic matter content was observed from week three onwards, which can be attributed to the mineralisation processes occurring in the soil, leading to the decomposition of organic matter. Soil microorganisms play a pivotal role in these processes. They are involved in the decomposition of a wide range of organic compounds, including cellulose, starch, and fats [52]. Their activity is closely correlated with the soil pH. The greatest microbial activity in soil is observed at a pH of 6 to 7. From week four to week eight, a distinct decline in organic carbon content is evident across all soil samples. In the case of the control sample, there was a 37% reduction in organic carbon content. In comparison, the fertilised soil samples exhibited a decrease of 32% and 36% for fertilisers F1 and F2, respectively.

3.3.3. Phosphorus Availability

Water-soluble phosphorus is characterised by immediate availability to plants. Figure 6 illustrates the alterations in water-soluble phosphorus concentration within the soil over the eight-week incubation period. The graph illustrates that the addition of fertilisers to the soil significantly affects the content of water-soluble phosphorus. The addition of fertilisers to soil results in a markedly elevated phosphorus content in comparison to the control samples. Furthermore, soil fertilised with F2 fertiliser contains 5–8 times more water-soluble phosphorus, which is a consequence of the higher phosphorus content in the fertiliser. It was observed that for soil fertilised with F1 fertiliser, the phosphorus content exhibited a slight increase up to week three, after which it remained at a similar level. From week seven, however, a decrease in the content of water-soluble phosphorus was noted. In the case of soil fertilised with F2, the content of this parameter remains constant up to week six, after which a slight decrease is observed. A comparable trend is evident in the phosphorus content of unfertilised soil.

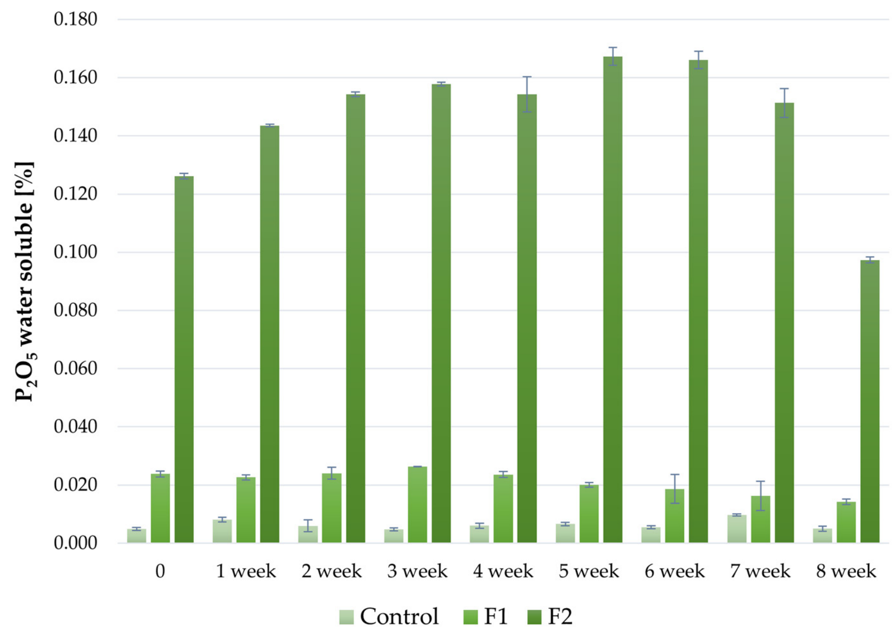

Figure 6. Changes in water-soluble phosphorus content in soil during incubation studies.

Phosphorus is distinguished by its low mobility in soil, which is a consequence of its strong binding to particles in the soil solution. A reduction in moisture content and temperature levels diminishes the rate of phosphorus diffusion towards the roots. The availability of phosphorus is also influenced by soil microbiota, which can compete with plants for phosphorus by producing mycorrhizae in order to enhance the efficiency of phosphorus acquisition [53]. Figure 7 illustrates the temporal dependence of bioavailable phosphorus content. The content of available phosphorus throughout the duration of the experiment remains at a consistent level for both the soil with the addition of fertilisers and the control sample.

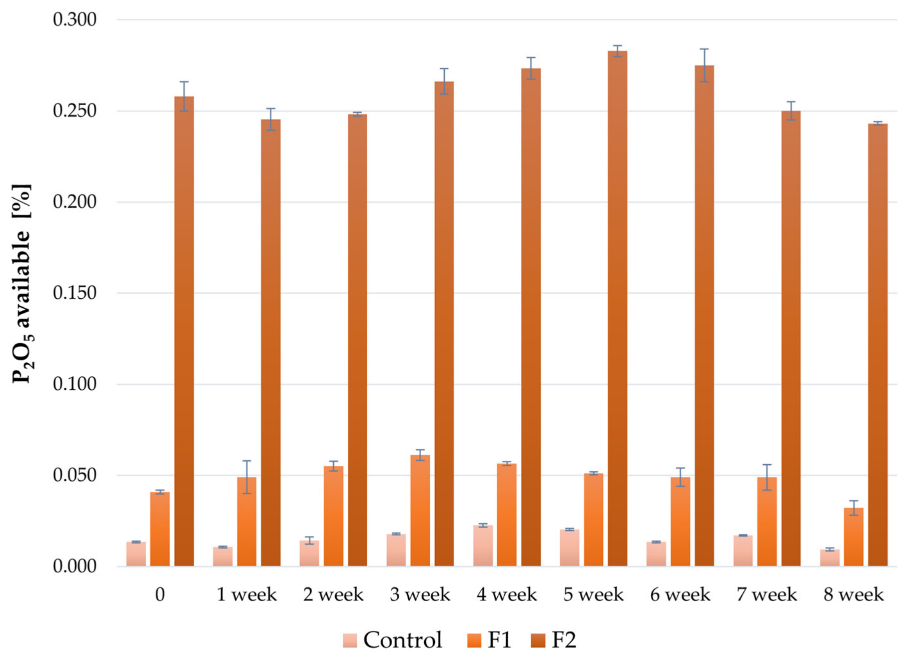

Figure 7. Changes in soil available phosphorus content during incubation studies.

This suggests that the soil underwent gradual, prolonged changes throughout the experiment, resulting in a transformation in the form of phosphorus occurrence. From the fifth week of the experiment, a reduction in the concentration of available phosphorus was observed across all samples. This phenomenon may be attributed to the increasing pH, which has the effect of reversing the chemical equilibrium of phosphorus.

3.3.4. Catalase Activity

Figure 8 illustrates the alterations in catalase activity observed in soil samples treated with fertilisers F1, F2 and the control sample.

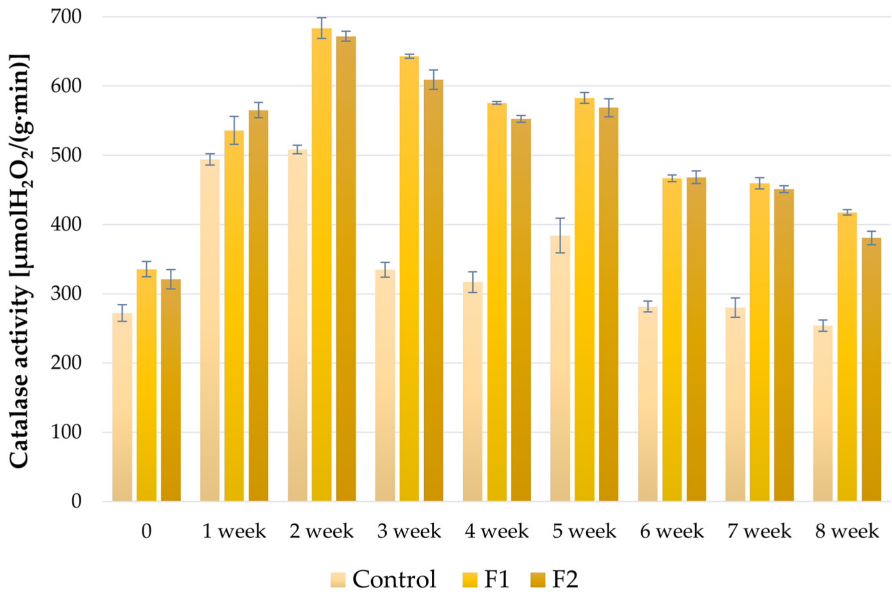

Figure 8. Changes in catalase activity during incubation studies.

During the initial two-week period of the experiment, a notable elevation in catalase activity was discerned, reaching 87% in the control sample, 104% and 109% in the samples fertilised with F1 and F2 fertilisers, respectively. The favourable incubation conditions (in terms of humidity and temperature) and the addition of organic matter in the case of soil with fertilisers stimulated the microbiological population and its activity. A significant correlation was observed between soil catalase activity and the content of organic carbon in the soil, with the former proportional to the latter. As organic carbon is utilised, there is a corresponding decrease in microbial activity, as evidenced by the decline in catalase activity observed in all samples from week two.

4. Discussion

The high organic matter content of deinking paper sludge makes it a potentially valuable resource for agricultural applications. This approach could contribute to the management of this waste and improvement of soil fertility. There are known studies confirming the positive impact of composts containing deinking paper sludge on the physicochemical properties of soil and plant growth (Table 7). Vannucchi et al. [54] demonstrated that a substrate comprising pelletised deinking paper sludge, compost and tephra (pumice 30% + lapillus 60%) is suitable for use in green roofs. The addition of waste reduced the fertility of the substrate, facilitating the coexistence of native stress-tolerant species with sedums and enhancing plant diversity. In additional studies, a substrate composed of mature compost and pellets derived from deinking paper sludge has been demonstrated to be an effective means of promoting the growth of trees in pots within nursery settings, while simultaneously enhancing their resilience to transplanting-related stressors [55].

Table 7. Summary of research results on deinking paper sludge agricultural use.

Type of Waste	Type of Trials, Rate	Results	Country
Compost (deinking paper sludge + manure)	2-years field study, 0, 14, 28 and 42 Mg/ha	• increase in snap bean yield in 17–24% depending on dose of compost • increase in the percentage of P recovered by snap bean in 4.2–15% depending on dose of compost • increase in the percentage of K recovered by snap bean in 39.1–57.5% depending on dose of compost • the concentration of heavy metals in bean tissues was not affected by compost treatment	Canada [56]
Deinking paper sludge	4.5-month greenhouse pot experiment, clay substitution 0, 20, 40, 60, 80% with deinking paper sludge, sand substitution 0, 20, 30% with deinking paper sludge	• decrease in alder and aspen growth with clay substitution more than 20% • increase in shoot dry weight, height and stem diameter od alder with the increase in sand substitution with deinking sludge	Canada [57]
Deinking paper sludge	2-years field study, 0, 30, 60 Mg/ha	• increase in soil pH with the increase in sludge dose • increase in soil organic matter content in 36–73% in comparison with control • increase in content of available P with the increase in sludge dose • increase in microbial biomass carbon up top 500% at dose of 60 Mg/ha • increase in biomass nitrogen in 16–83% depending on dose	Tunisia [58]

The fertilisers obtained provide not only valuable organic matter but also the main nutrients (P and K) and microelements required by plants, thereby increasing their market value. The results of the incubation study showed that fertiliser application resulted in an increase in soil pH. The highest increase in pH of the fertilised soil was observed in week six. It was 8.6% for the soil treated with F1 fertiliser and 7.4% for the soil treated with two fertilisers. This can be attributed to the fact that the fertilisers were rich in calcium and Ca^{2+} ions could replace the H^{+} ions on the clay–humic complex and consequently decrease the soil acidity [58]. Another reason for the increase in pH, which also occurred in the control soil samples, could be the decarboxylation of organic anions during the decomposition of organic matter [59]. However, during the sixth and seventh weeks, the soil pH slightly decreases, which may be related to the buffering properties of the soil.

The use of organo-mineral fertilisers contributed to an increase in soil organic carbon of around 20%. The source of organic carbon in the fertiliser was deinking sludge, which consists mainly of cellulose, hemicellulose, and lignin. These components serve as a carbon source for many different microorganisms and their degradation is an essential part of the global carbon cycle [60]. Their degradation takes place under the influence of enzymes secreted by specific aerobic–anaerobic bacteria and fungi. Temperature, aeration, moisture content and soil pH have a major influence on the decomposition process [61,62]. In general, the degradation process of biopolymers is rather slow. However, the soil samples were incubated under favourable conditions, which positively influences the degradation of organic components in fertilisers by microorganisms, which was observed in the third week of the study.

Microorganisms are considered the mainly source of enzymes of soils, and enzyme activities are strongly associated with the soil microbiological properties [63]. Catalase is an enzyme that is commonly found in animal cells, fungi, aerobic bacteria, and photosynthetic plants. The function of catalase is to protect organisms from the deleterious effects of oxidative stress [64]. Soil organic matter is the primary source of enzyme substrate [65]. Therefore, the introduction of organo-mineral fertilisers into the soil contributed to a significant increase in catalase activity (92% and 82% for F1 and F2 fertiliser compared with control, respectively), which was most visible in the third week of incubation.

The availability of phosphorus is crucial for its availability to plants. Water-soluble phosphorus is considered to be the fraction of phosphorus that is most susceptible to loss. Studies show a positive correlation between the content of soluble phosphorus in water and the content of phosphorus in leachate during leaching study [66]. Mineral fertilisers are usually characterised by a high content of water-soluble phosphorus, only part of which is used by plants and the remainder can be lost, contributing to eutrophication, loss of biodiversity, and environmental degradation in aquatic systems [67,68]. The content of water-soluble phosphorus in the soil during incubation was very low and remained at a similar level from week one to six for the soil treated with F1 and F2 fertilisers, which is beneficial from an environmental point of view. From the sixth week of incubation, the content of water-soluble phosphorus decreased by 36% and 42% for soils with F1 and F2 fertilisers, respectively, compared to the maximum value. This decrease can be attributed to the with various physicochemical processes occurring in the soil. Water-soluble phosphorus can be absorbed on the surface of Fe, Al oxides, kalionite, clay materials, etc. or precipitated [69]. These processes can occur simultaneously and are dependent on pH, temperature, concentration and ratio of individual ions in the soil solution [49]. Increasing pH promotes the precipitation of calcium phosphates, which probably occurred from week six of the soil incubation studies. The same trend was observed for the potential bioavailable phosphorus content, which was extracted with Bray's solution. This solution is designed to extract adsorbed forms of phosphate and is intended for use in soils with a pH < 7.5. From week five of the study, the phosphorus content in the soil began to decrease, reaching a decrease of 48% for the soil with F1 fertiliser and 14% for the soil with F2 fertiliser, with respect to the maximum content in week eight.

The next step in fertiliser research is to determine the effect of fertilisers on plants through application tests. Phytotoxicity tests will allow the effect of the resulting fertiliser products on the early stages of plant growth to be assessed. Pot tests provide information on the long-term effects of fertilisers on plants, but also on the environment.

5. Conclusions

Deinking sludge is produced in large quantities during the paper recycling process and is often difficult to handle. Since it is characterised by a high content of organic matter, mainly consisting of cellulose, hemicellulose, and lignin, it can be used in agriculture as a soil improver. A promising solution is the use of deinking paper sludge for the production of organic–mineral fertilisers, which has the advantage of providing not only organic matter, but also nutrients and microelements. The fertilisers produced in the study were qualified as PK-type fertilisers, containing 24.6% and 39.3% of the total content of the main nutrients, respectively. The content of readily available phosphorus in fertilisers was in the range of 11–12%, and the content of medium-available phosphorus, which are associated with iron, aluminium and calcium, was approximately 85%. The potassium in the fertilisers was in a readily available form, which indicates that the fertilisers are highly efficacious. However, the release dynamics of nutrients are contingent upon the prevailing soil conditions. Fertilisers were enriched with secondary nutrients (Ca, Mg) and microelements (Fe, Zn, Cu) and met the standard requirements for heavy metal content according to Polish legislation. Incubation studies have shown that the addition of fertilisers to the soil contributes to the increase in pH, organic matter content and the content of soil-soluble and potentially bioavailable phosphorus. In addition, fertilisers have a positive effect on the microbiological activity of the soil. To the authors' knowledge, no previous studies have been conducted on the possibility of producing fertilisers based on deinking paper sludge. Accordingly, further studies are required to ascertain the long-term impact of these materials on soil properties and plant growth and development.

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