

## Article

# Can Organic Matter from Waste-Derived Amendments Limit Phosphorus Losses from Soil to the Aquatic Environment?

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**Abstract:** When introducing innovative waste-derived amendments, their potential impact on soil retention capacity towards phosphorus (P) should be accounted for to limit its losses and thus water eutrophication. The study was aimed at evaluating P sorption properties of organic waste-derived amendments conifer woodchip biochar (BIO) and brown coal waste (BCW), and assessing their impact on P sorption properties in soil five years after their application in terms of limiting P losses from soils to the water sources. In batch tests samples of amendments and sandy acid soil (Haplic Luvisol) were exposed for 24 h to balancing solutions with a different P content, respectively 0–80 mg P L<sup>−1</sup> for amendments and 0–20 mg P L<sup>−1</sup> for soil. Three treatments were studied: (1) conifer woodchip biochar (BIO), (2) brown coal waste (BCW) and (0) soil without amendment (control). Organic amendments were applied to soil at a rate equivalent of 10.2 t C ha<sup>−1</sup> once at the start of the trial and they amounted to 24.2 t ha<sup>−1</sup> for BCW and 12.8 t ha<sup>−1</sup> for BIO. Based on the results of the batch tests and the Langmuir and Freundlich equations, sorption parameters of P were determined for BIO, BCW and the soil after their application. The maximum sorption capacities ( $S_{max}$ ) of BIO and BCW were 832.3 and 250.4 mg kg<sup>−1</sup>, respectively, and were controlled mainly by the organic surfaces of their structures. Within the range of concentrations used in the balancing solutions (5–20 mg P L<sup>−1</sup>), sorption of P was generally higher in unfertilized soil compared to soil amended with BCW and BIO. The application of BIO significantly ( $p < 0.05$ ) decreased the  $S_{max}$  (17%), while BCW decreased the bonding energy of P ( $k$ ) by 15% compared to the untreated soil. The content of dissolved P increased significantly in the case of both BCW and BIO applications, which may indicate an increase in soil susceptibility to P losses. However, the effectiveness of these amendments in limiting P losses with the use of other doses and in different soil and climatic conditions cannot be excluded. Further research is recommended to better understand the effect of biochar feedstock and temperature of its production on P retention/losses in various types of soils.

**Keywords:** brown coal waste; conifer woodchip biochar; sorption; phosphorus dispersion; Haplic Luvisols



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## 1. Introduction

Phosphorus (P) is a vital soil macronutrient required by plants for optimum growth and development and at the same time it has the most limited bioavailability in soils of all plant nutrients due to fixation [1]. To ensure optimal plant growth, phosphate fertilizers are applied to agricultural soils in excess of plant requirements to overcome soil fixation processes and maintain P in the soil solution at optimal levels for plant growth [2]. Many European soils are excessively fertilized, accumulating soil P pools at levels that are environmentally unacceptable due to the risk of transfer to watercourses and the potential for eutrophication [3]. This is likely to apply to most soils worldwide with a long P fertilizer application history [4]. If fertilizer application is stopped or reduced, dissolved P

is depleted and the equilibrium turns into a slow net solubilization and its desorption from stabilized soil. The desorption rate of P is markedly different between soils of different pH, mineralogy and organic matter content; thus, these factors are likely to be key regulators of plant uptake [5].

The fixation of P in soils is pH-dependent. In acidic soils, the predominance of aluminium (Al) and iron (Fe) oxides in both crystalline and amorphous forms reduces the solubility of inorganic P through fixation on positively charged surfaces and the formation of insoluble Al and Fe precipitates. Fixation of P in soils is often regarded as a serious problem for agriculture globally [6]. In Europe, a 5R strategy has been proposed to improve stewardship, and to act as a blueprint for national and global sustainability of P resources [7].

In order to provide the right amount of P available to plants in acidic soils, farmers tend to apply in excess P fertilizer and lime to saturate soil with Al and Fe ions and enlarge the pH [8]. However, this practice is uneconomical and environmentally unfriendly because of water eutrophication. The challenges of P losses from soils and eutrophication of water resources are addressed in the UN Sustainable Development Goals and under the EU's Green Deal by 2030.

Sorption and desorption properties of P-based fertilizers are prerequisites in predicting the availability of P in different soils, which is of importance in terms of managing soil fertility and environmental impacts. Agricultural soils are the main areal source of P contamination of the aquatic environment [9,10]. Therefore, when introducing innovative fertilizers, such as e.g., waste-derived amendments, their impact on sorption properties, which determine the soil's susceptibility to losses of P, should be taken into account. Currently, the focus of researchers and farmers is shifting towards the use of waste-derived organic amendments, such as manure, composts, biochar, brown coal waste and agro-industrial waste [11,12], that increase the soil organic matter content, enhancing soil resilience to risk of P losses through improvements of physical, chemical and biological properties [13,14]. Biochar and brown coal waste are considered effective, low-cost and eco-friendly soil ameliorants and have ability to immobilize different toxic elements, especially heavy metals [15–18]. Although some evidence on mitigating sorption and fixation of P using organic matter exists [19,20], there is a knowledge gap on the potential of using woodchip biochar and brown coal waste to improve P availability, especially in acidic soils. There are also no reports available on the retention capacity of brown coal waste and its impact on mobility of P in soil.

Brown coal (lignite) is a class of low-rank coal naturally abundant near the surface of the earth [17]. It contains high levels of organic compounds with a substantial content of humic (10–90% d.w.) and fulvic acids, which has attracted great interest in their potential use as a soil additive [21]. These acids are relatively stable large organic complexes with diverse functional groups reported to mediate many different soil processes, e.g., complexation with metals, alleviation of acidity and greenhouse gases abatement [13,22,23]. The high ion-exchange capacity makes brown coal and its waste an excellent adsorbent of both environmental pollutants and plant nutrients (e.g., NPK) [21,24], justifying the current interest in exploring its ability to enrich the soil with micronutrients needed for plant growth and maintaining agronomic objectives. Other studies examining additional soil benefits from brown coal waste use have found increased electrical conductivity in acidic soils [25] and enhanced P uptake from fertilizers [26]. Many studies have traced a wide range of benefits of applying brown coal and its derivatives, provisioning for soil health and fertility but mainly in short-term experiments [14,27,28]. Lack of disturbance and stable environmental factors under controlled conditions can usually lead to results that are not always consistent with those obtained in the field [29].

Biochar has become widely utilized as an amendment in agricultural soils over the past decades [30]; however, the global factors affecting its long-term fate in the environment are so far poorly understood [31–34]. Some research shows the contradictory effects of biochars on P dynamics in different soil types, and emphasizes the importance of considering the

impact of feedstock and pyrolysis temperature, application rates, soil properties and initial P concentration when applying biochar as a soil amendment for agricultural P management. All these conclusions are, however, based mainly on laboratory experiments [35–38].

Due to the significant diversification of physical-chemical characteristics, the P sorption abilities of biochars differ significantly depending on the feedstock and pyrolysis temperature [38–41]. Zhang et al. [40] found that for biochars from rice straw and egg shell the maximum sorption of P was obtained at 700 °C, and it amounted to 5.407 and 6.085 mg g<sup>-1</sup>, respectively. In the case of biochar originated from *Phragmites communis* and sawdust, the maximum P sorption of 7.747 and 3.859 mg g<sup>-1</sup>, respectively, was obtained at 300 °C. The other physical-chemical properties of biochar are also primarily determined by the type of biomass and pyrolysis temperature [33,37,42]. The total content of C and mineral elements depended to the greatest extent on the properties of the raw material, while the specific surface area (SSA) and pH of biochar were the highest when the highest temperature was used [43].

Despite the different origination and method of generation, the properties of biochar and brown coal waste show many similarities, such as porosity, large SSA and presence of chemical functional groups that make them active molecule sorbents. The heterogeneity of these materials means that they show significant differences in sorption capacity towards individual cations, anions or organic pollutants [15,27,44–46].

It was hypothesized that waste-derived amendments (biochar and brown coal waste) applied into the soil may limit losses of P from soil to the aquatic environment. The aim of the research was to evaluate P sorption properties of conifer wood-chip biochar (BIO) and brown coal waste (BCW), and to assess their impact on P sorption properties in the acidic soil five years after their application, as well as the long-term effect of the applied amendments towards healthy soil in terms of pH, carbon and macronutrients contents.

## 2. Materials and Methods

### 2.1. Field Experiment Description

The effect of biochar and brown coal waste on P sorption properties of soil was studied for soil samples taken from a field experiment conducted at the Prof. Marian Górski Experimental Station of the Institute of Agriculture in Skierniewice (Central Poland, 20° 34' E; 51° 58' N). The station is located in a temperate climate region with an average annual total precipitation of 528 mm and a temperature of 7.9 °C [47]. The trial was established in 2017 on Haplic Luvisol according to the IUSS World Reference of Soil Resources [48] with loamy sand texture (7% clay, 6% silt, 87% sand), pH<sub>KCl</sub> = 5.07 and C content of 7.2 g kg<sup>-1</sup> [28]. It was conducted to assess the long-term effect of a single application of organic amendments in two combinations: without or with mineral fertilizers on soil chemical properties and productivity. Each combination comprised a strip of field with an area of 166.5 m<sup>2</sup> separated from each other by a 1 m wide strip, on which 12 plots with an area of 12.5 m<sup>2</sup> (5 m × 2.5 m) were randomly placed. These plots included 4 treatments: unamended control, farmyard manure, BCW and BIO with or without the addition of mineral fertilizers in 3 replications. All organic amendments were added to the soil at a rate of C equivalent to 10.2 t C ha<sup>-1</sup> once at the start of the trial, which corresponds to the doses of: 30 t ha<sup>-1</sup> (farmyard manure), 24.2 t ha<sup>-1</sup> (BCW) and 12.8 t ha<sup>-1</sup> (BIO). Each dose of the amendment was sieved through a sieve with a diameter of 2 mm, hand-spread evenly over the surface of each plot and immediately mixed with the wetted soil. The following crops were grown in subsequent years (2017–2021): maize (*Zea mays*), potato (*Solanum tuberosum*), barley (*Hordeum vulgare*), wheat (*Triticum aestivum*) and oats (*Avena sativa*). Prior to the establishment of the experiment, a productive crop of wheat was carried out in the field. In the first year (2017), maize was sown in late April, and harvested in early August. Potato was planted in late March 2018 and harvested in September. Barley, wheat and oats were sown in early April and harvested in early August, appropriately in 2019, 2020 and 2021. Immediately after each plant was harvested, the crop residues were mixed with the soil using a disc harrow. Before winter, the plots were tilled to a depth of 0.3 m,

while in spring, before sowing the plants, a cultivating and sowing unit was used. For the presented study, 9 plots were selected, including 3 treatments (unamended control, BCW and BIO), each in 3 replications, from the combination (strip) without mineral fertilizers. Yields and content of micro- and macro elements of cultivated plants can be found in the previous paper [28].

The commercially available BIO, obtained from Fluid S.A., Poland, was derived from conifer woodchips by flash pyrolysis at 280 °C. The BCW came from the Bełchatów Coal Mine (Bełchatów, Poland).

## 2.2. Soil Sampling and Analysis

Soil samples were collected at a depth of 0–30 cm after harvested oats in 2021. From each experimental plot 12 incremental samples were taken, from which after mixing, a representative sample was isolated that was used in laboratory tests conducted at room temperature (20 °C). Prior to analyses soil and amendments after air-drying were passed through a 2 mm sieve. Soil pH was determined with the potentiometric method in 1 mol L<sup>−1</sup> KCl [49]. Soil to solution ratio was 1:2.5 (m/v). The total carbon (C) and nitrogen (N) contents in the soil were determined with a CN elemental analyser, and exchangeable aluminium (Al<sub>ex</sub>) after extraction in 1 mol L<sup>−1</sup> KCl (pH 6.0) [50].

The amounts of available P (P<sub>M3</sub>), Ca (Ca<sub>M3</sub>) and Mg (Mg<sub>M3</sub>), whose contents are comparable to exchangeable forms [51] in the soil and amendments, were determined with the Mehlich-3 extraction. The content of Al and Fe extracted with the Mehlich-3 solution is considered to be an indicator of the amount of amorphous (hydro)oxide Al (Al<sub>M3</sub>) and Fe (Fe<sub>M3</sub>), while  $\Sigma(\text{Al}_{\text{M3}} + \text{Fe}_{\text{M3}})$  in mmol kg<sup>−1</sup> is regarded as a good indicator of sorption capacity towards P [52]. The content of metals in extracts was analyzed by means of atomic absorption spectrometry (AAS), while P was determined spectrophotometrically using the ascorbic acid method.

## 2.3. Phosphorus Sorption on Soil and Amendments—Batch Experiment

For the assessment of sorption properties, duplicate 2 g soil, BCW and BIO samples were placed in 50 mL centrifuge tubes and suspended in 20 mL of 0.01 mol L<sup>−1</sup> CaCl<sub>2</sub> solution with graded P concentrations added as KH<sub>2</sub>PO<sub>4</sub>. The concentrations of P in equilibration solutions were of 0, 5, 10, 15 and 20 mg P L<sup>−1</sup> for soils, and 0, 10, 20, 40, 60 and 80 mg P L<sup>−1</sup> for organic amendments. Two drops of chloroform were added to each tube to prevent the microbial growth. The centrifuge tubes were shaken in a reciprocal shaker at a room temperature at 200 rpm. After 24 h, the samples were centrifuged at 3000 rpm for 15 min, filtered through a 0.45 µm membrane filter and analyzed for P content using the ascorbic acid molybdenum blue method [53]. The amount extracted in 0.01 mol L<sup>−1</sup> (P<sub>CaCl2</sub>) after 24 h was taken as the content indicator of dissolved P.

The sorbed P (S, mg kg<sup>−1</sup>) was calculated as the difference between the amount in the solution before and after equilibrium (c, mg L<sup>−1</sup>). All sorption data are presented as arithmetic means. The P sorption data were fit to the Langmuir and Freundlich equations:

The Langmuir equation:  $S = 1/kS_{\text{max}} + c/S_{\text{max}}$

The Freundlich equation:  $S = a_F \times c^{b_F}$

where S<sub>max</sub> is P sorption maximum, mg kg<sup>−1</sup>, k is the constant related to the bonding energy, L mg<sup>−1</sup>, a<sub>F</sub> is the intensity of sorption, mg kg<sup>−1</sup>, and b<sub>F</sub> is the Freundlich linearity constant [54].

The maximum buffering capacity (MBC) is a parameter of practical environmental importance as it informs about the ability of the tested material to maintain a constant concentration of P in the solution both during its depletion and inflow [55]. The MBC derived from the Langmuir sorption model takes into account both the number of free sorption sites and the force of P interaction with the surface as:

$$\text{MBC} = k \times S_{\text{max}}$$

## 2.4. Statistical Analysis

The suitability of the sorption model was evaluated based on the coefficient of determination ( $R^2$ ). Differences in sorption properties between treatments were analyzed using one-way analysis of variance (ANOVA), and significant differences between individual treatments were determined by the Tukey's HSD test at a significance level of  $p < 0.05$  by using IBM SPSS Statistic 28.

## 3. Results and Discussion

### 3.1. Physico-Chemical Properties of Organic Amendments

As a result of high content of lignine, the C content in biochars from wood was relatively higher than that obtained from other feedstocks. In general, the C content is in the range of 62–91% and increases with the temperature of pyrolysis [56]. Despite the low temperature (280 °C) used for the production of conifer BIO, the C content was relatively high (82.6%) (Table 1). Coalification of plant debris preserved in peat mires leads to the formation of humic acids. The net result of coalification is an extension of the humification process to include a continuous enrichment of fixed C with an increasing rank. In younger coals, e.g., brown coal, the C content is in the range of 30–40% for lignites (low ranks) and 40–65% for subbitumines (high ranks) [18]. Thus, the BCW used in the research had a C content typical for lignite (43.6%), with organic carbon (OC) as the dominant fraction (60–80%) present mainly in the form of humic acids [13] and which, compared to BIO, is less stable in the environment due to lower molar ratio H/C and higher O/C [57].

**Table 1.** Properties of tested brown coal waste (BCW) and conifer woodchip biochar (BIO).

| pH * |     | SSA *<br>m <sup>2</sup> g <sup>-1</sup> | C *<br>% | Total * |      |      |     |      | Mehlich-3 |                 |                  |                  |                  |                  |
|------|-----|-----------------------------------------|----------|---------|------|------|-----|------|-----------|-----------------|------------------|------------------|------------------|------------------|
|      |     |                                         |          | N       | P    | Ca   | Mg  | Al   | Fe        | P <sub>M3</sub> | Ca <sub>M3</sub> | Mg <sub>M3</sub> | Al <sub>M3</sub> | Fe <sub>M3</sub> |
|      |     |                                         |          |         |      |      |     |      |           |                 |                  |                  |                  |                  |
| BCW  | 5.4 | 2.15                                    | 43.8     | 0.53    | 0.17 | 11.0 | 0.6 | 2.64 | 0.74      | 0.070           | 3.5              | 0.54             | 0.272            | 0.07             |
| BIO  | 9.5 | 4.80                                    | 82.6     | 0.87    | 0.92 | 9.4  | 1.0 | 1.07 | 2.48      | 0.005           | 2.3              | 0.23             | 0.005            | 0.65             |

SSA—specific surface area; \* data from Amoah-Antwi et al. [28].

The specific surface area (SSA) is the parameter that largely determines the physico-chemical properties of sorption processes. The SSA of BCW was more than twice smaller than BIO. The low temperature used for the production of the analyzed BIO could be the main reason for the relatively small SSA (4.80 m<sup>2</sup> g<sup>−1</sup>). For biochars from wood obtained at higher temperatures, the SSA may be even of 100–200 m<sup>2</sup> g<sup>−1</sup> [56,58]. In sawdust biochar the SSA varied depending on the pyrolysis temperature from 2.9 m<sup>2</sup> g<sup>−1</sup> at 300 °C to 595 m<sup>2</sup> g<sup>−1</sup> at 700 °C [59].

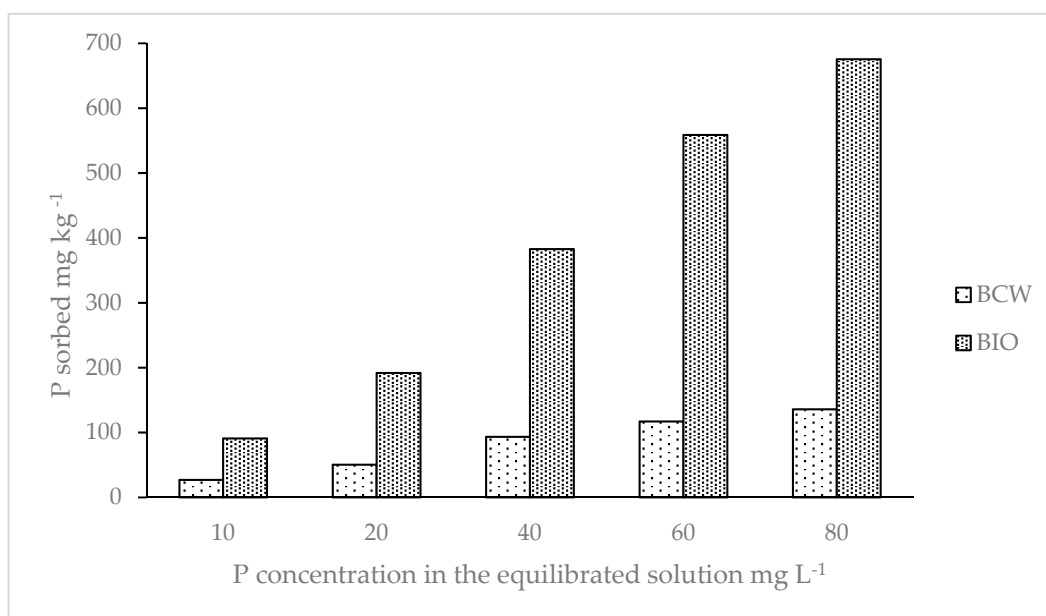
The pH of BIO (9.5) was significantly higher than of BCW (5.4). Carbonization results in reduction of acid surface functional groups, which combined with the enrichment of basic cations in the ashes may be associated with alkaline species, such as carbonates, oxides and hydroxides, causing that biochars particularly derived from wood are alkaline in nature [60]. The surface of BCW is dominated by oxygen-containing groups such as carboxylic, phenolic and carbonyl functional groups, whose deprotonation in solutions results in low pH [17,61].

The chemical composition of biochars depends primarily on the feedstock, and those derived from wood are inherently less mineral-rich compared to biochars from different agricultural feedstocks such as manure or crop residue. The chemical form of nutrients is mainly an effect of biochars' production temperature [33,42,62,63], while the chemical composition of BWC varies depending on geologic formation from which the lignite originates [57]. Compared to BIO, BCW had a higher content and higher portion of exchangeable/ available Ca<sub>M3</sub> and Mg<sub>M3</sub>. Uncrystallized forms of iron and aluminium active in P sorption processes were extracted from organic amendments using the Mehlich-3 solution. In each of the tested materials, the crystalline forms of Al and Fe which did

not pass into the Mehlich-3 solution dominated. A much higher content of  $Al_{M3}$  was in BCW, while  $Fe_{M3}$  was more in BIO. Available  $P_{M3}$  accounted for 0.5% of the total content of this element in BIO and was only  $5 \text{ mg kg}^{-1}$ . The same content of available P in pine wood biochars was found by Zhao et al. [58]. In BCW, however,  $P_{M3}$  constituted 41% and amounted to  $70 \text{ mg kg}^{-1}$ .

### 3.2. Phosphorus Sorption on Brown Coal Waste (BCW) and Conifer Woodchip Biochar (BIO)

Bath sorption experiments reveal that for both C-rich materials, sorption was increasing with the increasing concentration of P in the solution, which is generally common for materials with a low content of available P. With an increase of available P, the number of free sorption sites decreases, and in extreme cases desorption of P may occur [37,39,64]. Within the whole range of concentrations used, the sorption of P on BIO was in our study much higher than on BCW, and the difference was greater the higher was the concentration of P in the equilibrium solution (Figure 1). The amount of P that was desorbed to the solution of  $0.01 \text{ mol L}^{-1}$  ( $P_{CaCl2}$ ) was slightly higher for BCW ( $1.68 \text{ mg kg}^{-1}$ ) than for BIO ( $1.22 \text{ mg kg}^{-1}$ ), and it corresponded to the content of available P ( $P_{M3}$ ).



**Figure 1.** Phosphorus sorption on brown coal waste (BCW) and conifer woodchip biochar (BIO).

The data of P sorption on BCW and BIO were well fitted to the Langmuir and Freundlich equations, and the fitting parameters for both models are given in Table 2. The monolayer homogenous sorption, which is assumed by the Langmuir model, allows to determine the  $S_{\max}$  beyond which sorption ceases. The maximum sorption capacity was  $832.3 \text{ mg kg}^{-1}$  for BIO and much less, i.e.,  $250.4 \text{ mg kg}^{-1}$ , for BCW. Contrary to the Langmuir model, the Freundlich equation does not assume the existence of a maximum of sorption because on the surface of the adsorbent exists more than one sorption layer. The  $a_F$  constant determined from this model indicates the intensity of sorption and corresponds to the amount of immobilized P in the solid phase ( $\text{mg kg}^{-1}$ ), when the concentration of P in the solution is  $1 \text{ mg kg}^{-1}$ . A much higher value of this parameter for BIO ( $174.1 \text{ mg kg}^{-1}$ ) than for BCW ( $7.1 \text{ mg kg}^{-1}$ ) confirms the higher potential of this material to immobilize P. A significantly lower bonding energy in the case of the Langmuir model ( $k$ ) indicates greater susceptibility of BCW to desorption of previously retained P. The MBC of BIO ( $241.9 \text{ L mg}^{-1}$ ) shows much greater buffering capacity towards P than of BCW ( $4.4 \text{ L mg}^{-1}$ ).

**Table 2.** Phosphorus sorption parameters of brown coal waste (BCW) and conifer woodchip biochar (BIO).

| Property                                              | BCW   | BIO   |
|-------------------------------------------------------|-------|-------|
| $P_{CaCl_2}$ , mg kg <sup>-1</sup>                    | 1.68  | 1.22  |
| Langmuir model:                                       |       |       |
| P sorption maximum ( $S_{max}$ ); mg kg <sup>-1</sup> | 250.4 | 832.3 |
| Bonding energy (k); L mg <sup>-1</sup>                | 0.017 | 0.286 |
| Maximum buffering capacity (MBC); L mg <sup>-1</sup>  | 4.4   | 241.9 |
| Coefficient determination ( $R^2$ )                   | 0.986 | 0.981 |
| Freundlich model:                                     |       |       |
| Intensity of sorption ( $a_F$ ); mg kg <sup>-1</sup>  | 7.2   | 174.1 |
| Linearity constant ( $b_F$ )                          | 0.713 | 0.571 |
| Coefficient determination ( $R^2$ )                   | 0.992 | 0.979 |

Comparing the obtained results with the literature data, it can be seen that the tested BIO has relatively low P sorption properties compared to other wood biochars. Maximum P sorption obtained by Li et al. [65] for biochars produced at 500 °C was 2–3 times higher compared to our results. Zhao et al. [58] reported extremely high  $S_{max}$  of 14.484 g kg<sup>-1</sup> for pine biochars, but at high SSA (97.20 cm<sup>2</sup> g<sup>-1</sup>).

The difference in SSA between BIO (4.80 cm<sup>2</sup> g<sup>-1</sup>) and BCW (2.15 cm<sup>2</sup> g<sup>-1</sup>), which is usually accompanied by increased porosity, may be the cause of more than three times higher P sorption capacity of BIO compared to BCW. Studies on P sorption kinetics indicate that both rapid surface sorption and slower intra-particle diffusion through the adsorbent occur simultaneously [65]. As the SSA of the sorbent increases, the quantity of oxygen-containing groups increases as well, protonation and deprotonation of which create a net charge on the surfaces that decides on electrostatic interaction with ions [38,66]. In general, an increase in pH intensifies the deprotonation process and leads to an increase in the density of negatively charged surfaces, reducing their sorption properties towards anions, including phosphates [67,68]. The significantly lower pH of BCW (pH 5.4) than of BIO (pH 9.5) would indicate that the BCW surface had a greater electrostatic attraction to P than the surface of BIO. On the other hand, some research shows that biochar charge was negative at pH > 3.0–3.5, which makes sorption of P difficult in a broad range of pH [69]. It seems that the significantly smaller number of sorption sites ( $S_{max}$ ) and the lower bonding energy (k) are mainly due to the lower aromaticity and degree of aromatization of the naturally formed BCW compared to BIO. According to Fang et al. [70], in addition to electrostatic interactions, the main mechanism of anion retention by organic materials is attributed to hydrogen bonding to oxygen-containing groups. With an increase in pH and deprotonation of these groups, the role of carboxyl groups decreases and the role of hydroxyl groups increases with H-bonding donors. The strength of such anion binding is enhanced by aromatization and, consequently, depolarization of the structures to which these functional groups are attached. This is confirmed by the research of Eduah et al. [35], who showed significant linear relationships between P sorption parameters and O content, polarity, aromaticity and stable C, and readily labile OC of biochars. The low polarity and high aromaticity resulted in the high nonlinear sorption of P. Low generation temperature (280 °C), which limits the formation and condensation of aromatic structures and increases the content of the labile organic C pool may be the basic reason for the relatively low P sorption of the tested BIO. Low-molecular organic compounds of biochar can compete with P for sorption sites, effectively reducing  $S_{max}$  [35,67].

The share of the mineral fraction of biochars in sorption of P is ambiguous. A possible sorption mechanism in the presence of metal cations is the formation of weak hydrogen bonds (surface deposition) and strong chemical bonds (precipitation). On the other hand, the removal of the ash components frees up sorption sites in the pores. Dugdug et al. [37] observed an increase of P sorption capacity after washing out the minerals, mainly Ca

and Mg from hardwood and willow biochars. On the contrary, P sorption decreased after washing out of wheat biochar. According to some researchers, the high content of Al is an important component modifying the affinity of biochars for phosphorus [35].

The value of the  $b_F$  parameter from the Freundlich model for both tested amendments is above 0.4 and surpasses bonding energy from the Langmuir model ( $k$ ), which indicates a significant participation of chemisorption in sorption processes of P [71,72]. The higher  $b_F$  value for BCW (0.713) than for BIO (0.571) indicates a greater contribution of chemisorption of P in BCW, which had a much higher content of  $Ca_{M3}$ ,  $Mg_{M3}$  and  $Al_{M3}$  than in BIO. Despite the higher content of minerals that can immobilize P from the solution in BCW, the sorption indices of P are higher for BIO. Thus, it can be concluded that in both materials the P sorption properties are controlled mainly by the surface of their organic structures.

### 3.3. The Effect on Physicochemical Properties of Soil

The buffer properties of organic matter and the content of alkaline elements mean that the use of BIO and BCW generally increases the pH of the soil. Their liming effect depends on the dose but also on soil type and pH [31,32,62,73,74]. According to Jeffery et al. [75], addition of biochar with a pH that is two units higher than that of the soil, at the median biochar application rate ( $30 \text{ t ha}^{-1}$ ), is very likely to have increased the pH beyond the optimum for soil fertility. Wood biochars, due to the relatively low content of Ca, may show weaker liming effect than biochars from mineral-rich feedstock, including Ca. In clayey soil texture a significant increase in soil pH from 3.89 to 4.05 was observed with wood biochar application ( $20 \text{ t ha}^{-1}$ ) over 4 years, indicating its long-term beneficial effects [76]. Sandhu et al. [77] reported no increase in pH in sandy loam soil within two years from application of  $10 \text{ t ha}^{-1}$  of biochar produced from ponderosa pine (*Pinus ponderosa*) wood residues, justifying this by too low dose of biochar. In our long-term experiment 5 years after application of  $12.8 \text{ t ha}^{-1}$  of BIO, the soil pH (5.02) was higher compared to control (4.80) and with BCW (4.84) (Table 3). However, compared to the pH of the starting soil (5.07), it was lower in all combinations. The temperate climate in which the experiment was conducted with the predominance of precipitation over evaporation favors the acidification of low fertility sandy soil. Therefore, BIO, unlike BCW, mitigated the acidifying effect of crop production on the tested soil. Under similar conditions after 2 years from application of  $50 \text{ t ha}^{-1}$  woodchip biochar obtained from low-temperature ( $300^\circ\text{C}$ ) pyrolysis in sandy and weakly acidic soil, Gruss et al. [78] also observed a decrease in soil pH. Due to the low pH, the alkalizing character of BCW is less pronounced on acidic soils and, therefore, its application should be combined with liming [27].

**Table 3.** Properties of soil after application of amendments: brown coal waste (BCW) and conifer woodchip biochar (BIO) in the field experiment compared to the control (mean  $\pm$  standard deviation,  $n = 3$ ).

| Property                               | Amendment          |                   |                    |
|----------------------------------------|--------------------|-------------------|--------------------|
|                                        | Control            | BCW               | BIO                |
| pH <sub>KCl</sub>                      | 4.80 a             | 4.84 a            | 5.02 b             |
| C; g kg <sup>-1</sup>                  | 7.12 $\pm$ 0.11 b  | 6.73 $\pm$ 0.16 a | 7.02 $\pm$ 0.13 b  |
| N; g kg <sup>-1</sup>                  | 0.52 $\pm$ 0.04 a  | 0.56 $\pm$ 0.04 a | 0.52 $\pm$ 0.03 a  |
| Al <sub>ex</sub> ; mg kg <sup>-1</sup> | 1.89 $\pm$ 0.14 b  | 2.43 $\pm$ 0.19 c | 0.94 $\pm$ 0.12 a  |
| Mehlich-3 extraction:                  |                    |                   |                    |
| P <sub>M3</sub> ; mg kg <sup>-1</sup>  | 176.3 $\pm$ 2.5 c  | 143.7 $\pm$ 4.0 b | 128.7 $\pm$ 3.1 a  |
| Ca <sub>M3</sub> ; mg kg <sup>-1</sup> | 474.7 $\pm$ 6.1 b  | 445.3 $\pm$ 6.5 a | 487.0 $\pm$ 5.2 b  |
| Mg <sub>M3</sub> ; mg kg <sup>-1</sup> | 35.0 $\pm$ 1.0 a   | 33.7 $\pm$ 4.7 a  | 34.0 $\pm$ 4.0 a   |
| Al <sub>M3</sub> ; mg kg <sup>-1</sup> | 434.3 $\pm$ 13.6 b | 406.7 $\pm$ 6.7 a | 440.3 $\pm$ 11.2 b |
| Fe <sub>M3</sub> ; mg kg <sup>-1</sup> | 194.3 $\pm$ 9.0 a  | 186.0 $\pm$ 7.5 a | 183.0 $\pm$ 7.0 a  |

a, b, c letters indicate significant differences ( $p < 0.05$ ) between treatment means.

Numerous literature reports indicate that C sequestration potential of biochar and brown coal depends on soil characteristics, doses and impact time of amendments to soils [24,27–29,31,32,56]. After introducing the same amount of C from organic materials ( $10.2 \text{ t ha}^{-1}$ ), its content after 5 years in the soil amended with BIO was lower by 2.1%, and with BCW by 5.4% compared to the control. An important factor differentiating the content of C in the soil is not the amount of C introduced, but the degree of stability of the organic matrix of amendments. Decomposition of soil organic matter through alleviating energy limitation and stimulating microbial activities following the addition of fresh organic amendment into the soil known as the positive priming effect [79,80] could be the main reason for the loss of organic matter in the soil during the experiment. The longevity and intensity of this process largely depends on the amount of easily degradable organic matter [81]. The priming effect after application of biochars was so far observed under laboratory conditions, mainly for low fertility sandy soil and lower pH. It more often concerned biochars produced at lower temperatures with a higher content of easily degradable organic compounds [32,82,83]. Venture et al. [84] stated that the priming effect increases in the presence of plant roots. After introducing plant residues into the soil, a greater decrease in C content was found with the addition of humalite (a preparation made from lignite) than from biochar [57].

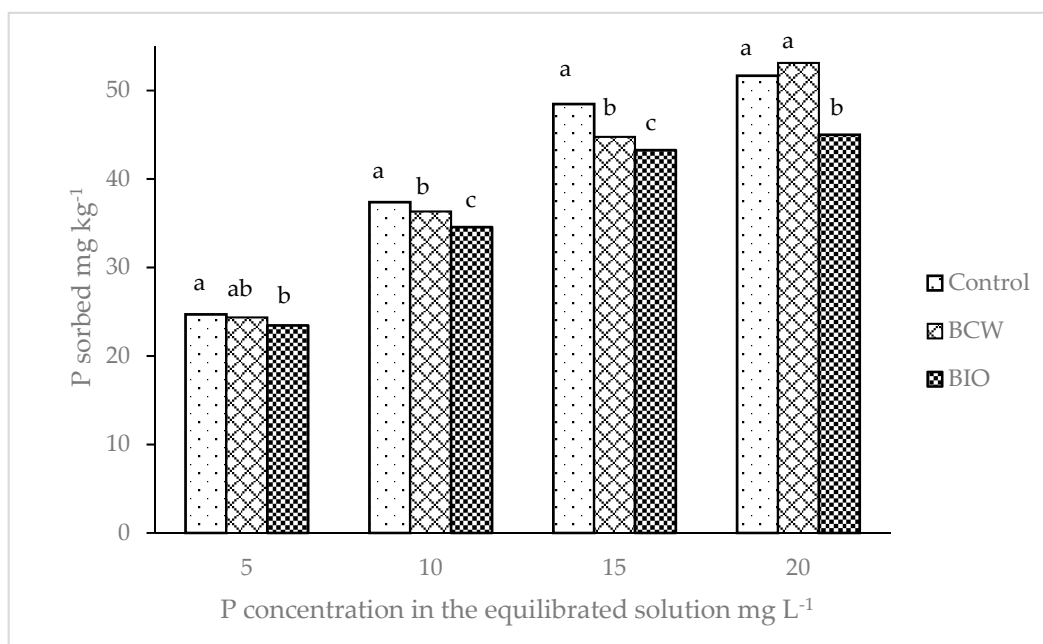
The content of exchangeable Al ( $\text{Al}_{\text{ex}}$ ) depends mainly on the pH of the soil, but it generally decreases with an increase of the organic matter content capable to complex it [85,86]. After using biochars,  $\text{Al}_{\text{ex}}$  reduction is observed not only due to the increase in pH but also as a result of its complexation by the organic functional groups on the porous surface of biochar [87]. Both of these reasons could cause the  $\text{Al}_{\text{ex}}$  content in the soil amended with BIO to be lower, and in the soil with BCW to be higher than in the control.

A slight increase in plant yield as a result of the use of BCW and BIO, which was observed in earlier studies conducted on this experimental field by Amoah-Antwi et al. [28], caused a significant diversification of the  $\text{P}_{\text{M3}}$  content. In both amended soils, the  $\text{P}_{\text{M3}}$  content decreased significantly compared to the untreated soil. The decrease of  $\text{P}_{\text{M3}}$  content was higher in soil amended with BIO than in the case of BCW. The very low content of  $\text{P}_{\text{M3}}$  in BIO and its lower dose than BCW meant that plants, when BIO was used, took up P mainly from the soil.

The increase in acidification and decrease of soil organic matter content after the BCW treatment resulted in a decrease of  $\text{Ca}_{\text{M3}}$  and  $\text{Al}_{\text{M3}}$  contents in relation to both: the soil with BIO and the control. Noble et al. [88] reported an increase of Al and Ca displacement to the subsurface as a result of applying coal-derived organic products. However, no major differences between the combinations were found for  $\text{Fe}_{\text{M3}}$  and  $\text{Mg}_{\text{M3}}$ . Based on the research, it can also be concluded that the effective improvement of acid sandy soil properties with low C content requires repeated BIO and BCW applications sooner than after 5 years.

### 3.4. Phosphorus Sorption Properties of the Amended Soil

The results of batch experiments indicate that the control soil generally sorbed larger amounts of P from the equilibrium solutions compared to soil amended with BIO and BC. However, for the highest concentration ( $20 \text{ mg L}^{-1}$ ) sorption of P was the highest in the soil amended with BIO. Despite the higher sorption potential of BIO, five years after its application, the soil showed lower P immobilization capacity within the entire tested range of concentrations used in balancing solutions than the soil to which BCW was introduced (Figure 2).



**Figure 2.** Phosphorus sorption of soil amended with brown coal waste (BCW), biochar (BIO) and untreated soil (Control). a, b, c letters indicate significant differences ( $p < 0.05$ ) between treatment means, separately for each equilibrium solution.

The results of batch tests for all treatments fit well both the Langmuir ( $R^2 \geq 0.985$ ) and Freundlich ( $R^2 \geq 0.966$ ) models, which indicates that both monolayer and heterolayer sorption participated in immobilization of P.

Clay and (hydro)oxide Fe and Al contents determine phosphate sorption in soil [89]. In Polish soils, however, the content of amorphous forms of Al and Fe is low in relation to crystalline forms, which results from light textural classes and low organic matter content, the presence of which delays the crystallization of (hydro)oxides Al and Fe [90]. Therefore, the sandy soil to which organic amendments were applied was characterized by low sorption properties towards P, while  $S_{\max}$  for the control soil was only  $70.1 \text{ mg kg}^{-1}$ , lower than  $S_{\max}$  for BIO and BCW themselves by 11 and 3.5 times, respectively. Such low values of  $S_{\max}$  for Polish sandy soils formed from heavily washed material deposited by glaciers are confirmed by other studies [91]. Five years after BCW application  $S_{\max}$  ( $73.6 \text{ mg kg}^{-1}$ ) was slightly higher than for the unamended (control) soil. The  $S_{\max}$  value of the soil to which BIO was added was  $58.3 \text{ mg kg}^{-1}$ , i.e., significantly ( $p < 0.05$ ) lower than for both the control and soil with BCW (Table 4). The opposite trend was observed for the energy bonding index ( $k$ ) from the Langmuir model. The soil amended with BCW was characterized by significantly lower bonding energy ( $0.159 \text{ L mg}^{-1}$ ) than the control ( $0.187 \text{ L mg}^{-1}$ ). A significant increase of  $k$  occurred in the soil with BIO ( $0.224 \text{ L mg}^{-1}$ ) in comparison to both: the soil amended with BCW and the control. Such differentiation of energy bonding may largely result from the properties of the organic amendments themselves, which significantly differed in the size of this parameter (Table 2). As a result of changes of the  $S_{\max}$  and  $k$  values, the MBC of the soil towards P after BCW application decreased compared to the soil with BIO and the control. In such conditions, the supply of P is greater, which may be beneficial for plants, but unfavorable for the environment. In both amended soils, despite the significantly lower content of  $P_{M3}$ , the content of dissolved P after extraction in  $0.01 \text{ mol L}^{-1} (\text{P}_{\text{CaCl}_2})$  was significantly higher than in the control. Both organic amendments made soils more susceptible to P losses. The increase in P losses from the soil by leaching after biochars application is confirmed also by some other studies [92,93].

**Table 4.** Phosphorus sorption parameters of soil amended with brown coal waste (BCW) and conifer woodchip biochar (BIO) from the field experiment compared to the control (mean  $\pm$  standard deviation,  $n = 3$ ).

| Property                                              | Amendment           |                     |                     |
|-------------------------------------------------------|---------------------|---------------------|---------------------|
|                                                       | Control             | BCW                 | BIO                 |
| $P_{CaCl_2}$ ; mg kg <sup>-1</sup>                    | 1.63 $\pm$ 0.03 a   | 1.87 $\pm$ 0.07 b   | 1.81 $\pm$ 0.03 b   |
| $\Sigma(Al_{M3}, Fe_{M3})$ ; mmol kg <sup>-1</sup>    | 23.1 $\pm$ 2.6 a    | 21.7 $\pm$ 1.5 a    | 23.3 $\pm$ 1.4 a    |
| Langmuir model:                                       |                     |                     |                     |
| P sorption maximum ( $S_{max}$ ); mg kg <sup>-1</sup> | 70.1 $\pm$ 2.0 b    | 73.6 $\pm$ 2.4 b    | 58.3 $\pm$ 0.4 a    |
| Bonding energy ( $k$ ); L mg <sup>-1</sup>            | 0.187 $\pm$ 0.070 b | 0.159 $\pm$ 0.011 a | 0.224 $\pm$ 0.010 c |
| Maximum buffering capacity (MBC; L mg <sup>-1</sup> ) | 13.0 $\pm$ 2.5 b    | 11.7 $\pm$ 0.4 a    | 13.4 $\pm$ 0.6 b    |
| Coefficient determination ( $R^2$ )                   | $\geq 0.966$        | $\geq 0.985$        | $\geq 0.994$        |
| Freundlich model:                                     |                     |                     |                     |
| Intensity of sorption ( $a_F$ ); mg kg <sup>-1</sup>  | 15.6 $\pm$ 0.5 a    | 14.8 $\pm$ 0.5 a    | 15.5 $\pm$ 0.6 a    |
| Linearity constant ( $b_F$ ); mg kg <sup>-1</sup>     | 0.460 $\pm$ 0.014 b | 0.469 $\pm$ 0.018 b | 0.408 $\pm$ 0.012 a |
| Coefficient determination ( $R^2$ )                   | $\geq 0.965$        | $\geq 0.992$        | $\geq 0.966$        |

a, b, c letters indicate significant differences ( $p < 0.05$ ) between treatment means.

The direction of changes in P sorption properties of the soil after adding BIO in our experiment is consistent with the results of incubation studies of Ghodszad et al. [38]. According to them, on acidic soils fertilized with biochar, sorption of P decreases and desorption increases, and the scale of these changes increases with the dose of biochar, revealing itself clearly only at a dose of 6% ( $w/w$ ). Other researchers, e.g., [66], reported the decrease of P sorption in acid soil already at a dose of 1% ( $w/w$ ). On the contrary, during incubation of acid black and acid brown soils amended with straw biochar at a dose of an equivalent of 1% ( $w/w$ ) of OC content, sorption capacity towards P decreased, while the dose of 5 and 10% (of OC content) resulted in its increase [36]. In a field experiment eight years after application of 0.5% ( $w/w$ ) biochar, Wakweya et al. [94] found only a marginal decrease in sorption and an increase in desorption of P in the acidic, highly weathered tropical soil. At the same dose of biochar used in this experiment, changes in P sorption on soil were more significant. In alkaline soils, biochar application generally increases P sorption properties [38,66]. The main reason for the increased mobility of P in acidic soils after the application of alkaline biochars is attributed to growth of electrostatic repulsion of phosphorus as a result of an increase in soil pH [38,67]. Due to the small changes in pH in our experiment, it seems that this factor was only of marginal importance, and only for the BIO amendment when the concentration of  $[H^+]$  ions was significantly higher and could increase the electrostatic repulsion of phosphates.

The  $k$  value from the Langmuir model was lower than the  $b_F$  value from the Freundlich model, which was greater than 0.4 in each of the tested soils. Such a distribution of these parameters proves the dominance of chemisorption processes in the immobilization of P from the solution [71,72]. The lowest  $b_F$  value was obtained for the soil amended with BIO, which may indicate a lower importance of chemisorption processes in P immobilization compared to soils with the addition of BCW and the control. In light soils with the low clay content, amorphous Al and Fe (hydro)oxides are of decisive importance in shaping the sorption potential of the soil, which immobilize phosphates by ligand exchange with OH groups (specific adsorption), and this process takes place regardless of the soil pH and the charge of OH groups [95,96]. The sum of amorphous forms of Al and Fe extracted in the Mehlich-3 solution is considered a good estimator of P sorption capacity, particularly in acidic soils [52,97]. The amount and reactivity of these forms largely depends on the soil pH and the content and quality of organic matter. Organic matter may chelate Fe and Al (hydro)oxides reducing the possibility of their crystallization and increasing their amount in the soil, and depending on the method of binding, they may or may not remain active in sorption processes [98]. In addition, dissolved organic matter can compete with phosphates for binding sites [67,96]. Despite lower  $P_{M3}$  content and comparable  $\Sigma(Al_{M3};$

$Fe_{M3}$ ) of the control, the  $S_{max}$  sorption of P in soil with BIO was significantly lower. It is, therefore, possible to block the sorption sites on Al and Fe (hydro)oxide by organic matter introduced with woodchip biochar. Eduah et al. [66] claim that due to the higher content of labile organic compounds, a decrease in sorption and an increase in desorption of P by limiting the activity of Al compounds to a lesser extent than Fe are more pronounced when the biochars generated at 300 °C were amended to the soils. According to some studies, the reduction of P sorption after application of biochar occurs mainly as a result of a decrease in content of exchangeable Al [99]. The comparable distribution of the  $Al_{ex}$  content and the  $S_{max}$  parameter values allow the possibility that Al-P precipitation in the presence of  $Al_{ex}$  could to a large extent determine the decrease in the number of sorption sites in the soil with the addition of BIO, and their slight increase in the soil with BCW. According to Xu et al. [36] a high dose of biochar with a significant content of minerals, i.e., Ca and Al, can lead to the improvement of soil sorption properties through the possibility of precipitation of Ca-P and Al-P. Our research shows that when applying mineral-poor woodchip BIO and BCW, the properties of the introduced organic matter and its impact on the amount of soil organic matter determine the reduction of soil sorption capacity and the possibility of greater P losses from the soil.

#### 4. Conclusions

The maximum sorption capacities of conifer woodchip biochar (BIO) generated at low (280 °C) temperature and brown coal waste (BCW) were 832.3 and 250.4 mg kg<sup>-1</sup>, respectively, and were controlled mainly by the organic surfaces of their structures.

Five years after the application of both organic materials, P sorption properties of soil decreased. The application of BIO significantly decreased maximum P sorption (by 17%) while BCW decreased the bonding energy of P by 15% compared to the untreated soil.

In the case of both amended soils, despite the lower content of available P, the amount of dissolved P increased significantly, which along with a surface runoff or through leaching, may ultimately get into waters.

The organic matter of BIO caused a decrease in the activity of Al, and Al and Fe (hydro)oxide, which reduced the number of sorption sites; however, the mechanism of decrease in binding energy found in the soil with BCW is unclear. Our field experiment confirmed the results of some previous laboratory tests indicating the negative impact of BIO on P sorption in the acidic soil.

An additional finding of our research is that a one-time application of BCW and BIO to acid sandy soil does not allow for a long-term effect towards healthy soil in terms of pH, carbon and macronutrients contents; therefore, this treatment should be repeated after less than 5 years.

The varied properties of brown coals depending on their origination, and biochars related primarily to their feedstock and the temperature of generation, do not exclude the possibility of such a selection of appropriate amendments for specific soil conditions, which can effectively limit the losses and dispersion of P into the aquatic environment. To achieve this, more research (preferably in field conditions) should focus on correlation of the influence of brown coal origin and biochar's feedstock and production temperature on the physicochemical properties responsible for increasing the retention of P in different types of soils.

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