



Effect of Chemical Fertilizers on the Efficiency of Biochar in Reducing Lead Mobility in Soil

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ABSTRACT

The objective of this research was to investigate the impact of ten different fertilizers on the mobility of lead in soils that had been treated with biochar. The soil used in this study was collected from Chanthaburi Province. To simulate the experimental conditions, this soil was artificially enriched with 550 mg/kg of lead. The synthetic soil was prepared by mixing it with 10% biochar and 0.04% of various chemical fertilizers. To assess the bioavailability of lead in the soil to plants, an extraction process using diethylenetriamine pentaacetate was performed. This allowed researchers to determine how these fertilizers affected the movement and availability of lead in the soil for plant uptake. In the study, it was observed that among the fertilizers tested, urea was the only one that increased the bioavailability of lead in the soil, making it more accessible to plants. Sequential extraction techniques were employed to analyze six different forms of lead in the soil. Interestingly, all fertilizers, except for urea, caused a transformation of lead from less stable forms to more stable forms in the soil. To further investigate the relationship between fertilizer variables and heavy metal uptake, a stepwise linear regression analysis was applied. The results indicated that the mobility of lead in the soil was primarily influenced by the nitrogen content, potassium levels, and sulfate ion concentration in the fertilizers.

Keywords: Acid soil; Biochar; Fertilizers; Lead

1. Introduction

Khlung and Na Yai Am Districts, situated in Chanthaburi Province, are prominent mining sites in Thailand, covering a total mining area of approximately 2,900 rai.

These mines contribute to the release of various toxic residues into the environment, with lead (Pb) being a notable heavy metal present in the vicinity of these mining sites. This situation has raised significant concerns among local farmers in these areas.

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Chanthaburi Province is renowned for its agricultural activities, with its primary products including durian, rambutan, mangosteen, and longkong. Plants growing in contaminated soils have the capacity to absorb heavy metals from the soil. Consequently, these heavy metals become distributed throughout various parts of the plants and are subsequently transferred to humans through the food chain [1].

Heavy metal toxicity affects the health of animals, plants, and humans. When humans are exposed to heavy metals, they may experience central nervous system dysfunction, fatigue, and an increased risk of cancer progression. Lead exposure during pregnancy can negatively impact the development of a baby [2]. Furthermore, heavy metals can impede the growth of certain plants and have adverse effects on essential plant components [3].

Biochar is an organic material that undergoes a slow pyrolysis process [4]. It has garnered significant interest in addressing the issue of heavy metal contamination in agricultural soils. Researchers have discovered that biochar possesses high porosity, surfaces with negative charges, and excellent adsorption capabilities for positive ions. When biochar is added to soils, it has the potential to reduce the uptake of heavy metals by plants [5, 6]. Hyacinth is an appealing organic material for biochar production due to its high carbon content, consisting of 20% cellulose, 48% hemicellulose, and 3.5% lignin [7]. Furthermore, hyacinth is a rapidly multiplying weed that can obstruct waterways [8]. By converting hyacinth into biochar, we can mitigate the adverse effects of this plant and utilize it to our advantage in addressing heavy metal contamination in agricultural soils.

Farmers commonly utilize chemical fertilizers to enhance plant growth. However, owing to the diverse chemical properties of these fertilizers, some may facilitate the mobility of heavy metals in the soil, while others may have the opposite effect. When heavy metals become more mobile in the soil,

plants are more likely to absorb them. The addition of nitrogen, phosphorus, and potassium to the soil has been observed to influence the distribution of heavy metals within the soil matrix [9].

In a research study, the investigator introduced both biochar and ten different chemical fertilizers into soil samples to assess their impact on the movement of lead in the soil. This evaluation aimed to understand how various fertilizers and biochar interacted with heavy metals in the soil and their potential influence on heavy metal mobility and plant uptake.

2. Materials and Methods

2.1 Preparation of soil, fertilizer, and biochar

The soil sample was collected from an undisturbed area in Chanthaburi Province, specifically at coordinates N13°50'32.1252" E101°9'5.6808, at a depth of 0-30 cm. This soil sample collection process involved sun drying, followed by grinding using a stone mortar. After grinding, the soil was passed through a 20-mesh sieve to achieve a consistent particle size. Subsequently, the soil was further dried in an oven at 105°C for a period of 24 hours. To create a controlled experimental environment, the soil was deliberately contaminated with lead at a concentration of 550 mg/kg. Synthetic soil contaminated with lead was marked with an S symbol. After the contamination, the soil was left at room temperature for a duration of three months to allow for natural processes and interactions to take place.

In this research, ten different fertilizer formulas were utilized. These formulas are represented by their N-P-K (Nitrogen-Phosphorus-Potassium) ratios and are as follows: 0-0-60, 0-52-34, 0-0-50, 0-3-0, 46-0-0, 16-20-0, 13-0-46, 13-13-21, 15-15-15, and 18-12-6. To prepare these fertilizers for the study, all of them, except for urea (46-0-0), underwent a process of crushing and sieving through a No. 20 sieve. This preparation ensured that the fertilizers were in a consistent

and uniform form for application in the research experiments.

The water hyacinth used in the study was sourced from the Lad Krabang area in Bangkok. To prepare it for further use, the leaves and stems of the water hyacinth were subjected to several rounds of washing with clean water. After washing, the plant material was dried under the sun. Following sun drying, it was then placed in an oven and dried at a temperature of 105°C for a duration of 24 hours. Once the water hyacinth was thoroughly dried, it underwent a pyrolysis process. During this process, the dried plant material was exposed to a temperature of 450°C for a period of one hour. Subsequently, the pyrolyzed material was sieved through a 35-mesh sieve, ensuring that it achieved a consistent particle size for use in the research.

2.2 Chemical characterization of soil, fertilizer, and biochar

The determination was carried out on particle distribution of soil by hydrometer method (ASTM No.1.152H), pH by pH meter (Consort model C860), electrical conductivity (EC) using a conductivity meter (Mettler model UM400). The chloride ion (Argentometric method), sulfate ion (Turbidimetric method), cation exchange capacity (CEC) (Ammonium acetate method), organic matter (Walkley-Black titrations), total nitrogen (Kjeldahl method), available phosphorus (Bray II method), and available potassium (Ammonium acetate extraction) were determined using the method developed by the Land Development Department [10]. Lead concentration in all samples was analyzed using acid digestion ($\text{HClO}_4/\text{HNO}_3$) before determination by an Atomic Absorption Spectrophotometer (AAS: Perkin Elmer model Analyst 200) [11]. Neutralization capability was examined by the titration method [12].

2.3 Experimental method

The experiment entailed thoroughly blending synthetic soil (S) with 10% biochar. Following this, the synthetic soil enriched with

biochar (SB) was combined with 0.04% chemical fertilizers. After this mixing process, the soil was allowed to sit at room temperature for a duration of two weeks to ensure proper interaction and stabilization of the components.

To evaluate the bioavailability of heavy metals in the soil for plant uptake, an extraction process was carried out using a 0.005 M diethylenetriamine pentaacetate (DTPA) solution [13]. The concentration of lead in the resulting extract was subsequently determined using an Atomic Absorption Spectrophotometer.

Furthermore, a sequential extraction procedure was employed to assess the distribution of lead within the soil into six different fractions: water-soluble fraction, exchangeable fraction, carbonate-bound fraction, Fe-Mn oxide-bound fraction, organically bound fraction, and residual fraction. This sequential extraction allowed for a detailed examination of how lead was distributed and associated with different soil components, providing valuable insights into their mobility and bioavailability in the soil.

2.4 Statistical analysis

The experimental process was conducted in triplicate for each set of experiments to ensure robust results. To analyze the data, the mean (average) and standard deviation (a measure of data dispersion) of the experimental data were calculated for each trial.

Statistical analysis was performed using the one-way analysis of variance (ANOVA) method to determine whether there were statistically significant differences between two or more groups of data. This analysis was conducted with a confidence level of 95%. The statistical software used for this analysis was SPSS version 23.

In addition to ANOVA, a stepwise linear regression program was applied to investigate the correlation between the chemical characteristics of fertilizers and the mobility of heavy metals. In this analysis, the

dependent variable was the concentration of heavy metals extracted using DTPA. The independent variables included pH, electrical conductivity, organic matter content, total nitrogen content, chloride concentration, available potassium content, total phosphorus content, and sulfate concentration. This stepwise linear regression aimed to identify which of these independent variables had a significant impact on heavy metal mobility in the soil.

3. Results and Discussion

3.1 Characteristic of soil, fertilizer, and biochar

Table 1 indicates that the synthetic soil was contaminated with lead (Pb) at concentrations that exceeded the acceptable limits for agricultural use. The acceptable limit for lead in agricultural soils is typically set at $Pb < 125$ mg/kg [14]. On the other hand, the concentration of heavy metals in all the studied

fertilizers did not exceed the standard values for use in agriculture. The standard values for these fertilizers are typically set at $Pb < 100$ mg/kg [15]. This means that the fertilizers used in the study met the standards for agricultural use in terms of heavy metal content.

Table 1. Lead concentration of samples.

sample	Pb concentration (mg/kg)
Soil	506.5 ± 0.42
Biochar	10.18 ± 0.19
0-0-60	35.63 ± 0.71
0-52-34	6.69 ± 0.14
13-13-21	16.32 ± 0.46
0-0-50	15.72 ± 0.41
0-3-0	39.4 ± 0.79
15-15-15	14.81 ± 0.44
46-0-0	8.41 ± 0.28
18-12-6	9.54 ± 0.32
16-20-0	15.32 ± 0.46
13-0-46	0.54 ± 0.03

Table 2 provides the chemical characteristics of both the soil and biochar used in the study.

Table 2. Chemical characteristics of soil and biochar.

parameter	soil	biochar
Soil texture		
% sand	81.52 ± 0.59	-
% silk	0.72 ± 0.10	-
% clay	17.76 ± 0.56	-
pH	4.97 ± 0.03	7.90 ± 0.03
Electrical conductivity (dS/m)	0.05 ± 0.01	0.55 ± 0.01
Cation exchange capacity (cmol/Kg)	16.71 ± 1.14	26.90 ± 1.95
Organic matter (%OM)	6.58 ± 2.80	16.19 ± 2.90
Total nitrogen (%)	0.10 ± 0.02	0.20 ± 0.03
Chloride ion (mg/kg)	2.71 ± 0.67	14.50 ± 0.50
Available phosphorus (mg/kg)	3.03 ± 0.02	19.05 ± 0.06
Available potassium (mg/kg)	11.11 ± 0.26	24.40 ± 1.90
Sulfate ion (mg/kg)	0.94 ± 0.78	0.49 ± 0.02
ANC	-	$4,255.32 \pm 134.21$

The soil studied in Chanthaburi Province is sandy loam, comprising 81.52% sand, 0.72% silt, and 17.76% clay (Table 2). This soil exhibits high acidity, with a pH range of 4.5-5 [10]. It is important to note that heavy metals tend to be highly mobile in acidic soil [16]. However, the levels of salinity and chloride fall within a range that does not adversely affect plant growth. Cation Exchange Capacity (CEC) values are informative indicators of a soil's ability to

either release or absorb nutrients. Several factors contribute to variations in CEC, including clay type, nutrient content, organic matter content, and soil pH. In the case of the studied soil, the CEC falls within the moderate range, specifically between 15 and 25 cmol/kg. This is noteworthy because the CEC value of most agricultural soils typically ranges from 2-20 cmol/kg. Soil organic matter is composed of various components, including humic substances, carbohydrates, proteins, and

humus. In this particular soil, organic matter content is high, exceeding 4.5%. This makes the soil well-suited for plant growth, as organic matter plays a crucial role in soil fertility and water retention. Despite the advantageous organic matter content, the soil falls short in terms of essential plant nutrients. Specifically, it lacks sufficient nitrogen, phosphorus, potassium, and sulfate concentrations to support plant growth effectively. As a result, the application of fertilizer is necessary to address these nutrient deficiencies. It's worth noting that in acidic soil conditions, phosphorus is typically present in the form of H_2PO_4^- , while in basic soil, it takes the form of HPO_4^{2-} . This distinction underscores the importance of considering soil pH when addressing nutrient availability and plant health.

The alkalinity of the biochar in question falls within the moderate range, with a pH level exceeding the standard value typically recommended for biochar used in soil improvement (standard value: pH 7.5) [14]. Notably, biochar with a higher pH can play a crucial role in complexing metal ions on its surface, leading to a reduction in the mobility of heavy metals [17]. The salinity of this biochar product is well within acceptable limits, measuring less than 7.30 ds/m [14]. This is an important factor as excessive salinity can have detrimental effects on soil and plant health. Moreover, this biochar boasts a high organic matter content, making it an effective means of enhancing soil fertility. Biochar with a high CEC can also be beneficial by limiting the movement of heavy metals [18]. However, it's worth noting that the biochar has relatively low concentrations of nitrogen, phosphorus, potassium, and sulfate. Consequently, when added to the soil, it doesn't significantly contribute to nutrient enrichment. One particularly noteworthy characteristic of this biochar is its Acid Neutralization Capability (ANC), which measures an impressive 4,255.32 meq/kg. To put this into perspective, Venegas reported ANC values of 4,280

meq/kg for municipal organic waste and 421 meq/kg for biochar derived from bark [12]. Biochar with a high ANC is particularly suitable for use as an absorbent since it has the capacity to increase soil pH.

In summary, this biochar exhibits characteristics that can make it valuable for soil improvement, particularly in terms of heavy metal immobilization and pH adjustment, but it should be used in conjunction with other nutrient sources when enriched soil is desired due to its relatively low nutrient content.

The chemical properties of ten studied fertilizers are displayed in Fig. 1. Urea (46-0-0) can undergo slow hydrolysis to produce ammonia and carbonic acid. The presence of ammonia can make the solution basic over time due to its alkaline nature. Carbonic acid can further decompose into carbon dioxide and water. Rock phosphate (0-3-0) comprises phosphorus, silica, clay, and limestone. The elevated pH of rock phosphate (as shown in Fig. 1a) is primarily attributed to the presence of limestone. The organic matter detected in rock phosphate (Fig. 1h) is derived from clay. Fertilizer 18-12-6 is composed of diammonium phosphate, which is slightly basic, ammonium sulfate, and potassium chloride, which is neutral. It's important to note that the addition of high pH fertilizers to soils can potentially lead to heavy metal precipitation. In contrast, both potassium chloride (0-0-60) and potassium sulfate (0-0-50) are considered neutral fertilizers. Among them, the highest chloride ion content is observed in fertilizer 0-0-60 (Fig. 1g). Fertilizer 16-20-0 contains mono-ammonium phosphate, which is acidic, along with ammonium sulfate. It's worth mentioning that hydrolysis of mono-ammonium phosphate can yield phosphoric acid and ammonium hydroxide, as described in Eq. (3.1).



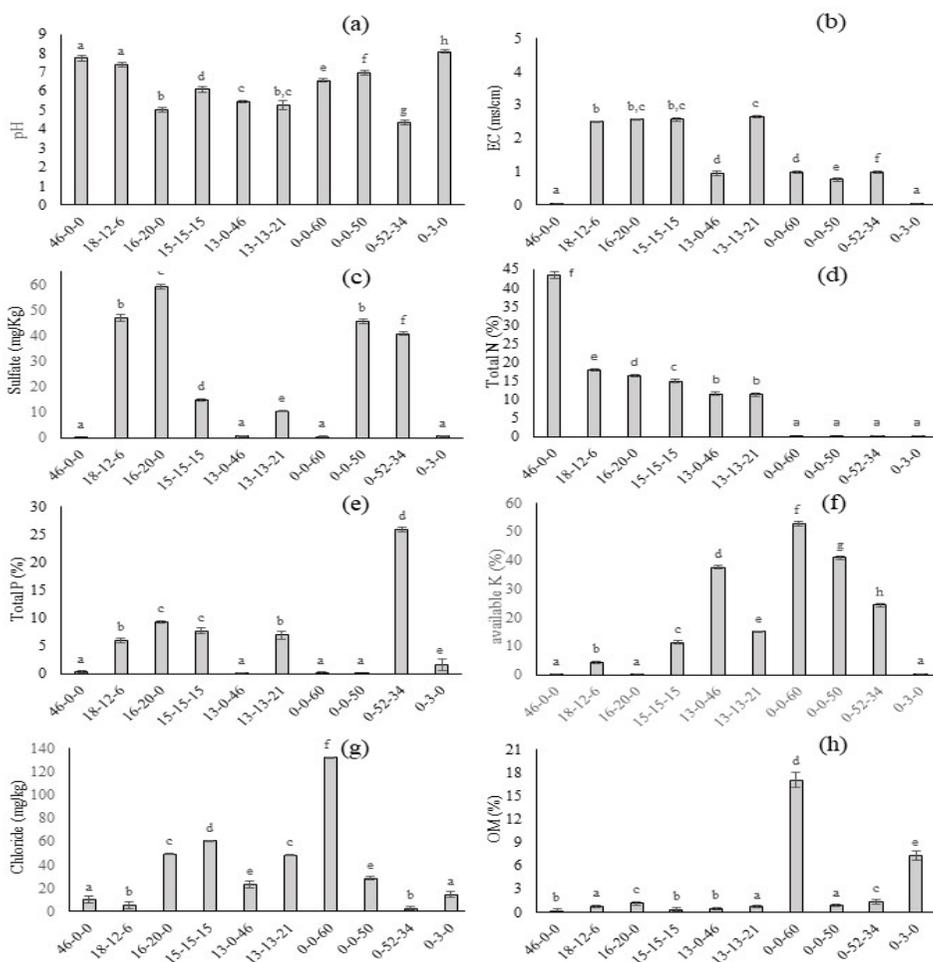


Fig.1. Chemical properties of fertilizers (a) pH (b) Electrical conductivity (c) Sulfate ion (d) Total nitrogen (e) Total phosphorus (f) Available potassium (g) Chloride ion (h) Organic matter.

Fertilizer 0-52-34 contains mono-potassium phosphate, which is classified as a strong acid, in addition to potassium sulfate. Notably, Fig. 1c illustrates a high sulfate content in fertilizers 18-12-6, 0-0-50, 16-20-0, and 0-52-34. The pH levels of fertilizers 16-20-0, 13-0-46, and 13-13-21 fall within the range of 5-5.5. It's essential to consider that adding acid fertilizers to soils can lead to a reduction in soil pH. A decrease in soil pH, as shown in some studies [19], can enhance the mobility of heavy metals, which may have implications for plant uptake. Furthermore, electrical conductivity is closely linked to soil salinity levels. Figure 1b demonstrates that the salinity of fertilizers 13-13-21, 15-15-15, 18-

12-6, and 16-20-0 is relatively high compared to others. Incorporating these fertilizers into the soil could contribute to increased soil salinity. It's worth noting that high soil salinity can affect the mobility of certain elements, such as cadmium, potentially impacting plant uptake [20]. Additionally, high salinity levels might decrease the yield of salinity-sensitive crops [21]. The total nitrogen content (Fig. 1d), total phosphorus (Fig. 1e), and available potassium (Fig. 1f) in the fertilizer formulations correspond to each respective type of fertilizer. Notably, the utilization of nitrogen fertilizers has been associated with an increased uptake of heavy metals by plants, as documented in previous research [22].

3.2 The influence of fertilizers on the mobility of lead

Lead extraction from soil was carried out using a 0.05 M diethylenetriamine pentaacetic (DTPA) solution for the purpose of evaluating the bioavailability of heavy metals in the soil to plants. The results of this assessment are presented in Fig. 2.

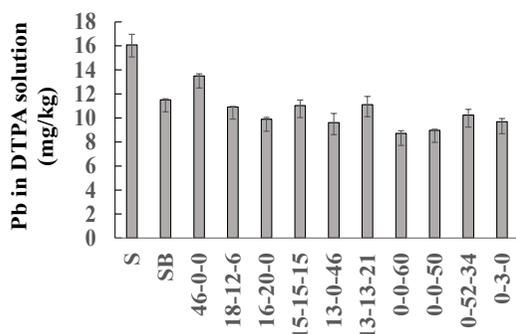
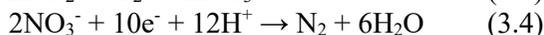
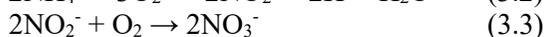
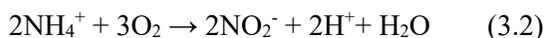


Fig. 2. The concentration of extracted Pb.

After the application of urea to the synthetic soil enriched with biochar (SB), it was observed that lead could be extracted by DTPA to a greater extent compared to samples without urea addition, as depicted in Fig. 2. This phenomenon can be attributed to nitrification processes, which may occur over time following urea application. As urea undergoes nitrification, it can lead to the release of H^+ and NO_3^- ions, as illustrated in Eqs. (3.2) and (3.3). Consequently, the soil tends to become more acidic, rendering lead more susceptible to extraction by the DTPA solution. Several researchers have reported that nitrate ions and ammonium ions can contribute to the accumulation of heavy metals in plants [22]. However, it's important to note that denitrification may also occur subsequently, resulting in a decrease in soil acidity, as demonstrated in Eq. (3.4).



The pH of all fertilizers, except for 16-20-0 and 0-52-34, was higher than that of the

soil. When higher pH fertilizers were added, they had the potential to raise the soil pH. Consequently, this increase in soil pH led to a reduction in the concentration of Pb in the DTPA-extracted solution, as illustrated in Fig. 2. Potassium content played a significant role in influencing the extraction of metals from the soil. Cations with higher valence are more effective at displacing and adhering to the soil surface compared to those with lower valence. Potassium had the ability to replace lead, and its addition contributed to improved heavy metal adsorption efficiency by increasing the porosity of biochar. Additionally, the addition of sulfate had a similar effect to potassium.

Fertilizers such as 18-12-6, 16-20-0, 0-0-50, and 0-52-34 were found to lower the lead concentration in the DTPA-extracted solution. Considering organic matter, fertilizer 0-0-60 contained a higher organic matter content compared to other formulations. As a result, the concentration of Pb in the extracted solution was lower than when no fertilizer was added to the soil. It is worth noting that cations, particularly transition metals, have the potential to form metal-organic complexes with organic compounds.

Furthermore, chloride exhibited an adsorption effect, where chloride ions could bind to lead ions, forming an inner sphere complex on the surface [23]. However, it is important to mention that fertilizers did not significantly affect the mobility of cadmium due to their low concentration.

The results obtained from the DTPA extraction were subsequently subjected to a stepwise linear regression analysis. The outcome of this analysis is presented below.

$$Y = 10.739 + 0.070(N) - 0.021(SO_4^{2-}) - 0.035(K) \quad (3.5)$$

where Y = Pb extracted by DTPA (mg/kg), N = Total nitrogen (%), K = Available potassium (%), SO_4^{2-} = Sulfate (mg/kg).

Based on the results derived from Eq. (3.5), it was observed that the total nitrogen content in the fertilizer was associated with

reduced lead adsorption in the soil. Conversely, the presence of sulfate and potassium in the fertilizer formulations was linked to increased lead adsorption.

The changes in the concentration of Pb in various forms were analyzed using a sequential extraction method, and the results are shown in Fig. 3.

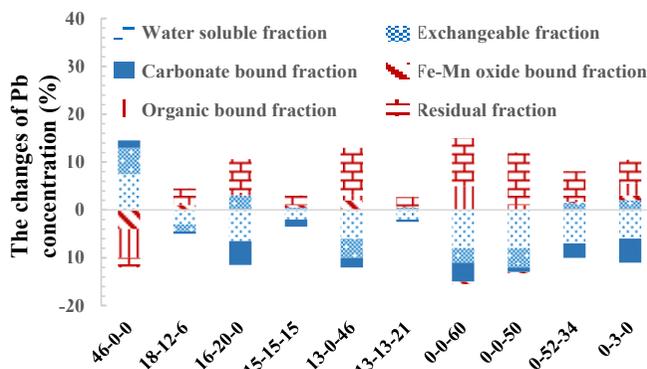


Fig.3. The changes of Pb concentration in various forms.

Heavy metals in water-soluble, exchangeable, and carbonate-bound forms are considered unstable, whereas the stable forms include Fe-Mn oxide-bound, organic-bound, and residual forms. As depicted in Fig. 3, the application of urea led to a reduction in Pb in stable forms, causing a shift towards unstable forms, resulting in an increase of approximately 14% in Pb in the unstable form. Consequently, Pb mobility increased as a result, and these findings are consistent with those shown in Fig. 2. Interestingly, the application of other fertilizers had the opposite effect, causing a transformation of Pb forms in the soil into more stable forms. Notably, the addition of fertilizer 0-0-60 resulted in the most significant change in the Pb form. Fertilizers 16-20-0, 13-0-46, 0-0-60, 0-0-50, and 0-3-0 caused Pb forms to shift from stable to unstable in the range of 10-15%. However, the change in Pb form was less than 10% after adding fertilizers 18-12-6, 15-15-15, 13-13-21, and 0-52-34.

4. Conclusion

The synthetic soil was contaminated with lead (Pb) at concentrations that exceeded the acceptable limits for agricultural use. It is worth noting that the concentration of lead in biochar and all the investigated fertilizers,

except for the "16-20-0" formula, adhered to the established standards for agricultural application. Upon the introduction of biochar to the acid sandy loam soil from Chanthaburi Province, the mobility of lead within the soil was notably reduced, indicating its potential to immobilize lead. Conversely, the addition of urea facilitated the movement of lead within the soil, making it more mobile. Other fertilizers had the opposite effect, restricting the movement of lead. Of significance, the nitrogen, potassium, and sulfate content within the fertilizers emerged as the most influential factors affecting lead mobility within the soil, surpassing the impact of other factors.

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