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Availability of Phosphorous on Ex-Gold Mining Soil Ameliorated with Sub-Bituminous Coal & Activation of Sub-Bituminous Coal with NaOH

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ABSTRACT

Mercury (Hg) contamination of ex-gold mining soils is of particular concern as it affects the availability of nutrients in the soil such as phosphorus (P). The interaction of Hg with P in soil needs to be studied quantitatively through amelioration technology that is expected to control Hg in the soil system. The purpose of this research is to study and analyze the correlation of Hg and P in ex-gold mining soil ameliorated with sub-bituminous coal and the activation of sub-bituminous coal with NaOH. The experimental design used in this study was a completely randomized design (CRD) with three (3) replications. The treatment I (Sub-bituminous coal) and II [activation of Sub-bituminous coal with 10% NaOH) was implemented in the pot with equivalent doses, respectively: (A) 0 [0g 100g^{-1} soil], (B) 10 [0.5g 100g^{-1} soil], (C) 20 [1g 100g^{-1} soil], (D) 30 [1.5g 100g^{-1} soil], and (E) 40 t ha^{-1} [2.0g 100g^{-1} soil]. The correlation and equation of linear regression between total Hg with Available P on ex-gold mining soil ameliorated with sub-bituminous coal [$r = -0.922^{**}$ and Hg = -0.206 (PO_4^-) + 7.0068; R^2 = 0.9052] and activation of sub-bituminous coal with 10% NaOH [$r = -0.862^{**}$ and Hg = -0.0265 (PO_4^-) + 6.2774; R^2 = 0.7851]. Amelioration technology on ex-gold mining soil with sub-bituminous coal and activation of sub-bituminous coal with 10% NaOH can reduce the total Hg by 2.50 and 2.84 mg kg^{-1} and also increase the available P by 11.88 and 76.91 ppm PO_4^- respectively, compared to control.

Keywords: Ex-gold mining soil; phosphorus; NaOH; Mercury; Sub-bituminous coal

INTRODUCTION

Gold mining is one of the metal contaminations that enter the food chain, mostly through plant uptake (Nnaji *et al*., 2023). On the other hand, the mobilization of metals in soil for plant uptake and leaching into groundwater can be reduced by adsorption and complex/ chelate reactions through chemical immobilization (Kumar *et al*., 2022). Soil is a major chemical contamination reservoir. Heavy metals enter the soil environment as a result of pedogenic and anthropogenic activities (Awad *et al*., 2021). The majority of metals are naturally present in soil parent material (paedogenic processes), mainly in unavailable forms. Metals introduced by anthropogenic activities, on the other hand, are usually highly available. Anthropogenic activities are a significant source of metal enrichment in soil due to direct contamination into the soil such as mercury (Hg) in amalgamation activities in gold mining that occurred in Dharmasraya Regency, West Sumatra. The traditional gold mining process with the amalgamation method uses Hg as a gold binder, which is the basis for Hg pollution in gold mine soils, where 25-30% of the Hg added in this process is lost to the environment (Esdaile & Chalker, 2018). Concentrations of heavy metals, such as Hg, have been found in high amounts in the soil around mine sites. The distribution of Hg in the Batanghari watershed in river water ranges from 0.001 - 0.07 mg kg^{-1}, while in the soil it is around 0.01 - 0.42 mg kg^{-1} (Ratnaningsih *et al*., 2019). Hg in soil amounted to 5.20 ppm in Nagari Batu Bakauik, Dharmasraya (Sahara & Puryanti, 2015). Hg pollution in this area is at a critical threshold for Hg pollution, where it is proven that there is an interaction between Hg and total N in gold mine soils ameliorated with sub-bituminous coal and activation with NaOH (Maulana *et al*., 2023). Therefore, it is also necessary to prove that the availability and solubility of P in gold mine waste soil is due to the fixation of P by Hg.

The P is among the macro-nutrients in plants that are most taken up during metabolism (Hafiz *et al*., 2022). The main source of P for plants is PO_4^- or can be in the form of P_2O_5, both of which can be obtained naturally in the soil or the application of P fertilizer, as well as enzymatic activity in the transformation of inorganic P into organic P, which is necessary for plant biomass accumulation, growth, and production (Kumar *et al*., 2021; de Bang *et al*., 2021). **Figure 1** shows the modification of the P cycle (Mullen, 2019) in soils contaminated with Hg, such as those of ex-gold mine soils. It would be interesting to investigate the relationship between P in ex-gold mining soils contaminated with Hg and the use of sub-bituminous coal-based amelioration technologies and the activation of sub-bituminous coal with 10% NaOH for both. The influence of organic matter (Mabagala & Mng'ong'o, 2022) and pH (Kicińska *et al*., 2022), as well as the presence of metals (Lin *et al*., 2020), greatly affect the availability of soil P (Neina, 2019), where the availability and solubility of phosphorus are regulated by the breakdown of organic matter which produces organic acids that have the property of binding metal ions in the soil (Khoshru *et al*., 2023; Silva *et al*., 2023). Complex reactions that are difficult to dissolve will develop in the soil. The concentration of free metal ions will decrease, and more phosphate will be accessible (Ahmed *et al*., 2022). This relates to the mechanism of metal mobilization by P compounds as well as the impact of P compounds on metal transformation in the soil (Li *et al*., 2022). If ex-gold mining land is used as productive land, especially for afforestation, Hg will slow down the availability of P by reducing the absorption of PO_4^-. There are several impacts of Hg on P metabolism that are interesting to study if ex-gold mining soil is used as productive land. Increasing nutrient requirements can be done through the application of amelioration technology based on sub-bituminous coal and its activation with 10% NaOH.

The use of Indonesian sub-bituminous coal (SC) as an ameliorant material stems from its high potential as humic compounds (HS) (Herviyanti *et al*., 2019) when compared to other organic materials (e.g. peat) extracted with various types of alkaline materials (Rezki *et al*., 2007). Chemical activation methods involving alkaline materials are also used to optimize the use of SC. NaOH is the optimal alkaline material for SC activation as a soil improver in various experiments (Herviyanti *et al*., 2021). On marginal lands, SC with alkaline materials has been used (Herviyanti *et al*., 2019; Prasetyo *et al*., 2021). However, it has never been used on ex-gold mining soil contaminated with Hg. This is of interest for the potential development of SC application as well as activation of sub-bituminous coal with 10% NaOH on ex-gold mining soil contaminated with Hg. The chemical properties of SC, such as pH (5.34 - 12.65) and CEC [24.39 - 148.20 cmol(+)kg^{-1}], can be improved by activating it with 10% NaOH. There is an increase in the number of O-H, C=O, and CH_3 functional groups and can also improve the chemical properties of Ultisol soil such as pH H_2O, CEC, organic C, available P, and total N in order of 49 units, 28.08 cmol(+)kg^{-1}, 1.63% C, 2.37 ppm P, 0.06% N, and reduced Al of 1.17 cmol(+)kg^{-1} as well as sodium adsorption ratio (SAR) of 0.03% and exchangeable sodium percentage (ESP) of 0.82%, compared to the control (Herviyanti *et al*., 2018). Application of 40 t ha^{-1} of Indonesian sub-bituminous coal to the ex-gold mining soil increased the soil surface charge (ΔpH) by 0.60, including increases in pH H_2O, EC, CEC, and OM. Meanwhile, 40 t ha^{-1} SC-NaOH was applied to the ex-gold mining soil, it could change the soil surface charge by increasing the pH H_2O EC, CEC, and OM. The soil surface charge was supported by soil minerals quartz, graphite, and pyroclastic, and also decreased the transmittance at the OH group (0.18%), which resulted in an increased adsorption capacity of the soil towards Hg (Prasetyo *et al*., 2023). The objective of this study was to examine the interaction of P and Hg in ex-gold mining soil that had been ameliorated with sub-bituminous coal and the activation of sub-bituminous coal with 10% NaOH.

MATERIALS AND METHODS

Experimental design

This study was conducted in the Laboratory of Chemistry and Soil Fertility, Faculty of Agriculture, Andalas University, Padang from July 2024 to September 2024. This study's experimental design was a completely randomized design (CRD) with three replications and two experiments. Experiment I (sub-bituminous coal) and II [activation of sub-bituminous coal with 10% NaOH) was implemented in the pot with equivalent doses, respectively: (A) 0 [0g $100g^{-1}$ soil], (B) 10 [0.5g $100g^{-1}$ soil], (C) 20 [1g $100g^{-1}$ soil], (D) 30 [1.5g $100g^{-1}$ soil], and (E) 40 t ha^{-1} [2.0g $100g^{-1}$ soil].

Ameliorant and soil sampling

Sub-bituminous coal

Sub-bituminous coal (SC) was taken from the Bonjol Pasaman, West Sumatra, Indonesia at a depth of 1-2 meters from the ground soil, then cleaned, smoothed with Disc Mill model FFC 23, and sieved with a sieve of 500µm electromagnetic sieve shaker EMS-8 for 10 minutes, after SC is obtained.

Activation of Sub-Bituminous Coal with 10% NaOH

The SC was weighed based on the dosage and percentage of the formulation used (sample weight). Activation of SC is carried out by giving 10% NaOH (SC-NaOH) as the activating agent dissolved with H_2O based on the field capacity of SC, and the calculations are shown in Formula 1.

$$\text{NaOH (g)} = 10 / 100 * \text{coal weight} \qquad \text{(F.1)}$$

Furthermore, the composition is stirred evenly, allowed to stand for 1*24 hours in a 250 mL beaker, and covered with aluminum foil with plastic wrapping. The results of SC-NaOH are dried using an oven at a temperature of 70^0C for 1*24 hours to homogenize the water content in the ameliorant (Herviyanti *et al*., 2018).

Ex-Gold mining soil

Soil samples from ex-gold mining soil were collected in a composite from Dharmasraya in West Sumatra at depths ranging from 0-20 cm. The soil was dried, crushed, and sieved through a 2 mm sieve before being homogenized and placed in the experiment pot. The treatments of SC and SC-NaOH are then incubated into the soil (100g) for one week, and soil samples are taken for analysis of changes in soil chemical characteristics following incubation.

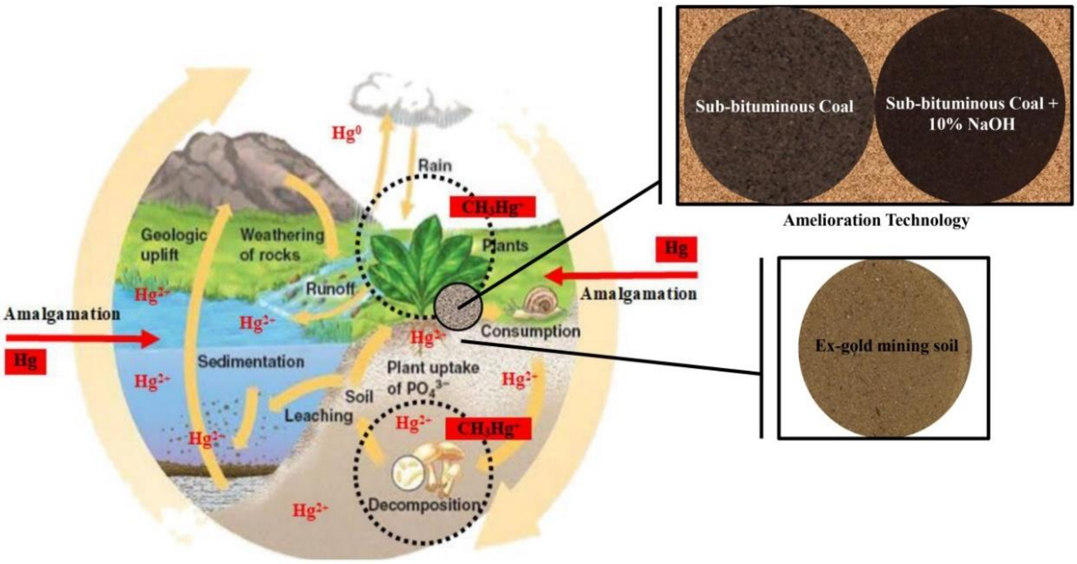

Figure 1: Modification of cycle P on ex-gold mining soil contaminated with Hg ameliorated with sub-bituminous coal and activation of sub-bituminous coal with 10% NaOH (Mullen, 2019)

Soil and statistical analysis

Soil analysis includes pH H_2O by the glass-electrode method, CEC by the leaching of NH_4OAc pH 7 method, organic carbon by the Walkley-Black method, available P by Bray II method, and Hg by cold vapor atomic absorption spectrometry (CV-AAS) (Soil and Fertilizer Instrument Standard Testing Center, 2023). The statistical analysis has carried the software Exel 2016, SPSS 16.0, and Statistics 8 to analyze soil. Bivariate correlations using a Pearson correlation coefficient and a two-sided significance test were used; the coefficient R^2 and p-values [** = Correlation is significant at the 0.01 level (2-tailed); * = Correlation is significant at the 0.05 level (2-tailed)].

RESULTS AND DISCUSSION

The Hg contamination in ex-gold mining soil in Dharmasraya is the main problem in this study that needs to be controlled against its interaction with nutrients, especially P (**Figure 2**). The Hg concentration in ex-gold mine soil was 6.88 mg kg^{-1}, higher than the Hg contamination limit. The WHO sets the limit for Hg levels in soil at 0.05 mg kg^{-1} (Chiroma *et al*., 2014), the European Union at 0.08 mg kg^{-1} (Horvart *et al*., 2019), and the US EPA at 1 mg kg^{-1} and 0.3 mg kg^{-1} (Alloway, 2012). This shows that the concentration of total Hg in the soil of the ex-gold mining soil exceeds the predetermined threshold, so it requires the use of special technology in the remediation process to reduce total Hg levels in the soil. The technique that can be offered in the research is amelioration technology using SC and SC-NaOH.

The effect of SC on the ex-gold mining soil was significant in reducing Hg, where the rate of 30 and 40 t ha^{-1} resulted in the greatest reduction in Hg, namely 2.10 and 2.50 mg kg^{-1}, compared to the control. Meanwhile, the effect of SC-NaOH resulted in a decrease in Hg in the former gold mine soil which was almost the same as the application of SC, where at the rate of 40 t ha^{-1} resulted in the largest Hg reduction of 2.84 mg kg^{-1}, compared to the control. The Hg is reduced with SC and SC-NaOH application, and organic compounds such as humic acid and Hg cations (e.g. Hg^{+} and Hg^{2+}) interact. The interaction between humic acid and clay micelles also contributed to the decrease. The decrease in Hg in ex-gold mining soil after SC and SC-NaOH application can also be influenced by several variables that affect whether or not Hg adsorption occurs in the soil, such as the amount and type of Hg chemical compounds injected, the proportion of clay, soil pH, redox potential, and organic matter (Velásquez Ramírez *et al*., 2021). There was a negative relationship between Hg and pH (r = -0.767** and -0.933**), OC (r = -0.863** and -0.883**), and CEC (r = -0.833** and -0.741**) in the ex-gold mining soil (**Table 1**). The application of SC and SC-NaOH amelioration technology on ex-gold mining soils can improve soil nutrient status, especially available P. Available P in the former gold mine soil is very low (0.04 ppm PO_4^-) which is inversely proportional to Hg in the soil which exceeds the safe threshold (6.88 mg kg^{-1}).

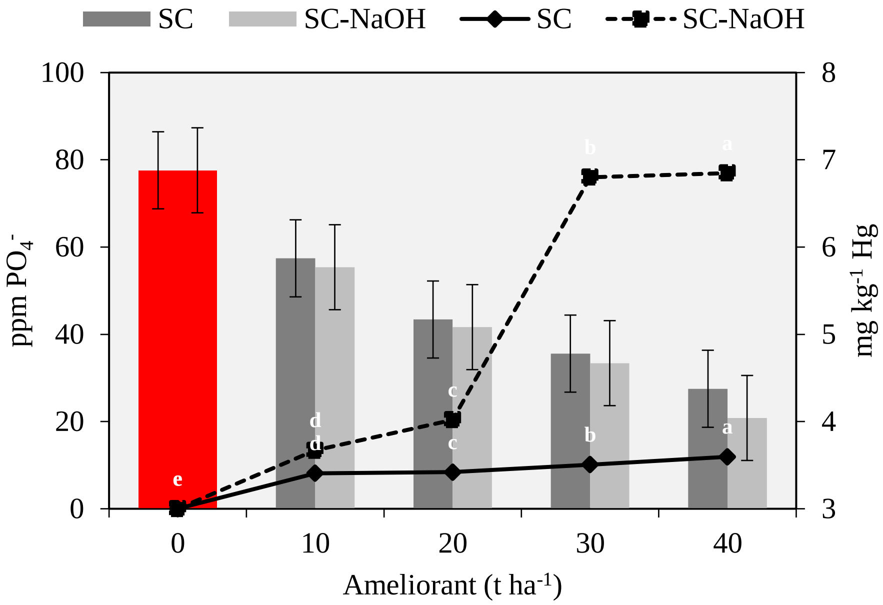

Figure 2: Available P and Hg on ex-gold mining soil ameliorated with sub-bituminous coal and activation of sub-bituminous coal with 10% NaOH

The effect of SC and SC-NaOH on the ex-gold mining soil resulted in an increase in the availability of P in the former mine soil to 11.88 and 76.91 ppm PO_4^- which meets the criteria of moderate to very high. This is due to the decrease in Hg levels in the soil after the optimum application of 40 t ha^{-1} SC and SC-NaOH, and its relationship with the pH, OC, and CEC of the ex-gold mine soil (**Table 1**). The chemical makeup of the ex-gold mining soil affects Hg-P interactions. The amount of CEC value is determined by soil OC and pH, which affect the increase of P availability and Hg in the soil due to variations in soil negative charge. Cation exchange capacity (CEC) is an important chemical property of the soil support system. Exchange reactions regulate the balance between the solid and liquid phases of the soil, reducing fluctuations in ion concentration. An increase in pH and OM will result in an increase in soil CEC for adsorption complexes, as well as close contact between humic acid with Hg cations in the formation of clay-organic complexes as a result of the complex evolution of the characteristics and distribution of soil components that are predicted to vary (Gumbara *et al*., 2019). Meanwhile, OC, pH, and CEC all have an impact on increasing soil available P, which in turn will also affect the decrease of total Hg. The correlation results between CEC values depending on SOC (r = 0.965** and 0.857**) and pH (r = 0.804** and 0.694**), which will affect soil available P (r = 0.743** and 0.796**) after SC and SC-NaOH application (**Figure 3**).

Table 1: Correlation of available P and Hg of ex-gold mining soil ameliorated with sub-bituminous coal and activation of sub-bituminous coal with 10% NaOH.

Pearson Correlation	Available P	pH H_2O	OC	CEC	Hg
A. Sub-Bituminous coal					
Available P (ppm)	1	0.660**	0.769**	0.743**	**-0.922****
pH H_2O		1	0.816**	0.804**	**-0.767****
OC (%)			1	0.965**	**-0.863****
CEC [cmol(+)kg^{-1}]				1	**-0.833****
Hg (mg kg^{-1})					1
B. Activation of Sub-Bituminous coal with 10% NaOH					
Available P (ppm)	1				
pH H_2O	0.772**	1			
OC (%)	0.980**	0.761**	1		
CEC [cmol(+)kg^{-1}]	0.796**	0.694**	0.857**	1	
Hg (mg kg^{-1})	**-0.862****	**-0.933****	**-0.883****	**-0.741****	1

Remarks: OC = organic carbon; CEC = Cation exchange capacity; **. Correlation is significant at the 0.01 level (2-tailed) and n = 15 samples/ experiment.

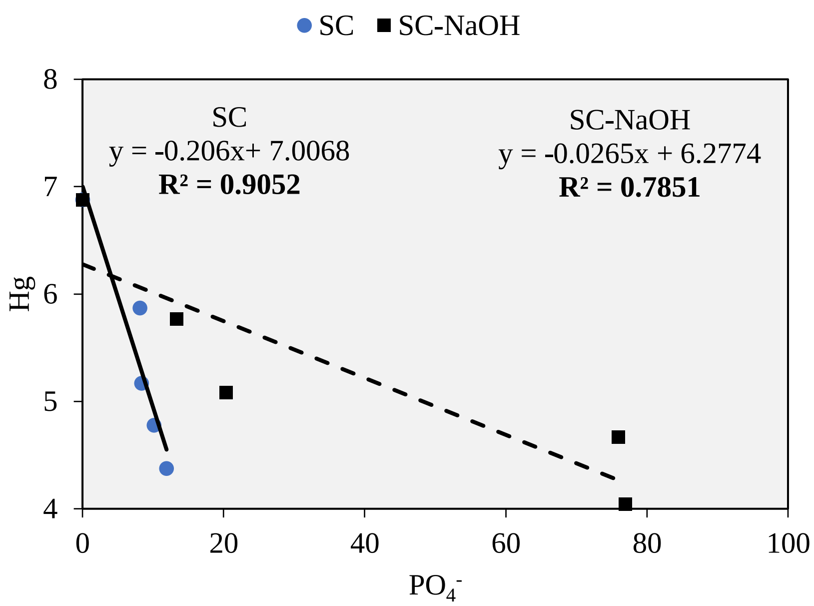

Figure 3: Linear regression equation of Hg with available P of ex-gold mining soil ameliorated with sub-bituminous coal and activation of sub-bituminous coal with 10% NaOH

The effect of SC and SC-NaOH, the interaction of Hg with available P has a correlation value of (r = -0.922** and -0.862**). The Hg can be retained in the soil through sorption, precipitation, and complexation reactions, and removed from the soil through plant uptake, leaching, and volatilization (Seeda *et al*., 2020). The degradation of organic compounds that occurred after the application of SC and SC-NaOH on the ex-gold mining soil is the main source of nutrients for plants. This SC has a P_2O_5 content of 0.30% (Anon, 1981) or 600-1200 mg kg^{-1} P (Akinyemi *et al*., 2012). The correlation and linear equation between Hg and P in the ex-gold mining soil ameliorated with SC ($y = -0.206x + 7.0068$; $R^2 = 0.9052$) and SC-NaOH ($y = -0.0265x + 6.2774$; $R^2 = 0.7851$). The level of relationship between Hg and available P after treatment of SC > SC-NaOH. Phosphate compounds are related to Hg mobilization in the soil-plant system through various processes, which are influenced by metal source; P-micronutrient imbalance in plant nutrition; direct metal adsorption by P compounds;

anion-induced adsorption and desorption of Hg; direct precipitation of Hg with P solution as Hg-phosphate; precipitation through the liming action of PRs; and other (Teng *et al*., 2020). The interaction between P and Hg is particularly relevant due to the coexistence of two fundamental aspects namely (1) P deficiency is common in acid soils and (2) Hg solubility increases especially in acid soils, with an increased risk of causing environmental pollution that may even affect the food chain. The interaction of P with Hg can affect the mobility and transport of Hg, where 3 main types of interactions between P and Hg can occur in soil: (1) Hg interacts with P-containing minerals; (2) Interaction through soluble forms present in the soil solution; (3) Interaction of Hg with P adsorbed on soil colloids (Geizer *et al*., 2021).

The practical implications of the interaction between Hg and P in ex-gold mining soil ameliorated with sub-bituminous coal and activation of sub-bituminous coal with 10% NaOH are significant, especially in the context of soil remediation and fertility enhancement. Reduction of Hg contamination through the application of sub-bituminous coal proved effective in lowering Hg concentrations, which is important for reducing health risks to humans and ecosystems. The reduction of Hg by 2.50 mg kg^{-1} for sub-bituminous coal application and 2.84 mg kg^{-1} for activation of sub-bituminous coal with NaOH shows the potential of amelioration technology as a form of ex-gold mining soil management. The increase in P availability from each ameliorant by 11.88 ppm for sub-bituminous coal and 76.91 ppm for activation of sub-bituminous coal with NaOH indicates that amelioration technology not only cleans the soil from Hg contaminants but also improves soil fertility. Higher P availability can increase crop productivity, which is important for the rehabilitation of ex-gold mining lands.

Correlation between P and chemical properties ex-gold mining soil has a strong relationship between P and Hg, OC, CEC, and pH which explains that P management in ex-gold mining soil can contribute to the reduction of Hg contamination. This suggests the need for an integrated approach in soil management by considering elemental interactions through the recommendation of sub-bituminous coal-based amelioration technology can be recommended as a solution to transform ex-gold mine soils into productive land. This is important not only for environmental aspects but also for improving community welfare through sustainable agriculture and appropriate soil management by improving soil fertility and reducing contamination, can provide opportunities for sustainable and safe land rehabilitation for agricultural activities.

CONCLUSION

The interaction between Hg with P on ex-gold mining soil ameliorated with sub-bituminous coal [$r = -0.922^{**}$ and linear equation Hg = -0.206 (PO_4^-) + 7.0068; $R^2 = 0.9052$] and activation of sub-bituminous coal with 10% NaOH [$r = -0.862^{**}$ and and linear equation Hg = -0.0265 (PO_4^-) + 6.2774; $R^2 = 0.7851$]. Amelioration technology on ex-gold mining soil with sub-bituminous coal and activation of sub-bituminous coal with 10% NaOH can reduce the total Hg by 2.50 and 2.84 mg kg^{-1} and also increase the available P by 11.88 and 76.91 ppm PO_4^- respectively, compared to control. The correlation between available P and soil chemical properties in ex-gold mining soil ameliorated with sub-bituminous coal (P with Hg > OC > CEC > pH) and activation of sub-bituminous coal with NaOH (P with OC > Hg > CEC > pH). Sub-bituminous coal-based amelioration technology can reduce the impact of Hg contamination and can also increase the availability of P, so it can be recommended in the remediation process on ex-gold mining soils as productive land.

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