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The potential of *imperata cylindrica* for phytomining and soil remediation of red mud waste

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Abstract

As industrialization accelerates, the demand for metals, particularly aluminium, continues to rise. This growth has also led to an increase in by-products from the bauxite ore refining process, namely red mud. The highly alkaline nature of red mud, combined with its heavy metal content, poses significant environmental challenges. However, red mud also contains rare earth elements (REEs) that can serve as valuable secondary resources. An environmentally friendly approach to recovering these metals is phytomining, which utilizes plants and simultaneously contributes to land remediation. This study aims to evaluate the potential of *Imperata cylindrica* in metal recovery and the remediation of red mud waste. The research began by conditioning the pH of red mud through the addition of citric acid, fertilizers, and by adjusting the red mud composition to levels of 40% based on the optimum result using Response Surface Methodology. Phytomining was initiated once the pH of the substrate (a mixture of red mud and soil) reached an optimal range of 8.0–8.5. The results demonstrated that *Imperata cylindrica* was capable of absorbing several rare earth metals, including gadolinium (Gd), neodymium (Nd), and cerium (Ce), with concentrations of 119.5 mg/kg, 16.5 mg/kg, and 6.67 mg/kg, respectively, in its roots. Additionally, the plant showed the ability to absorb major components such as iron (Fe) and titanium (Ti), with the metals distributed throughout the plant's roots, stems, and leaves.

Keywords

Aluminum, Land restoration, Phytomining, Rare earth elements, Red mud

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Introduction

The problem of environmental pollution due to mining activities is increasingly becoming a serious problem. This is because by-products in the form of tailings, slag, and acid mine drainage from mining activities generally contain heavy metals and have extremely acidic or alkaline properties that can harm the environment and surrounding living things [1], [2]. One of the by-products of mining activities is red mud. Red mud is a by-product produced from the processing and refining of bauxite ore into alumina either through the sintering process or the bayer process [3]. Red mud waste is a problem due to its highly alkaline nature with pH values ranging from 10-13 [4], [5]. Apart from its alkaline nature, red mud also has another problem, namely in terms of the amount that continues to increase every year without any massive utilization of the waste. Every year the amount of red mud added worldwide is estimated to reach 150-200 million tons/year [6], [7]. This huge amount can occur because every 1 ton of alumina produced will produce 0.8 - 5 tons of red mud [3], [6], [8]. Therefore, to prevent the emergence of environmental problems caused as happened in Hungary in 2010 in the form of a broken red mud storage pond [9], nowadays research in the form of red mud utilization is intensified [10].

Currently, there have been many studies on the utilization of red mud. There are at least 3 main scopes of research on the utilization of red mud, including as secondary resources for the recovery process of valuable metals contained in it [11]. As it is known that although red mud contains heavy metals that have the potential to pollute the environment, red mud has its own blessings with the presence of valuable metal content in the form of rare earth elements, vanadium, yttrium, and scandium [12]. Currently, there are two methods that are widely used to recover these metals, namely pyrometallurgy and acid leaching [13]. In the research conducted by Zhang et al.[14], where he conducted a combination of acid leaching using hydrochloric acid followed by extraction using Aliquat 336 and P204 to recover Fe, Al, Ti, and REE, the results of the study stated that the leaching efficiency was above 82% and even reached 98.3% for Neodymium metal. Likewise, the extraction efficiency value using Aliquat 336 can reach 96%. Despite the high efficiency of metal recovery, acid leaching and pyrometallurgy methods have the disadvantage of producing acidic waste from the remaining leaching results which can be a new problem for the environment and also of course energy intensive [15]. Therefore, an alternative method that is more environmentally friendly is needed to recover valuable metals contained in red mud.

Phytomining or phytoextraction is one of alternative method that can be used. Phytomining is a method to perform metal uptake from a substrate using plants [16]. Not only in terms of metal accumulation, but this method can also improve substrate (soil) fertility [17]. Metals contained in a substrate will be absorbed by plant roots and then translocated to the aboveground biomass through the plant's transport mechanism [17]. Several types of plants such as *Brassica juncea*, *Medicago sativa*, and *Dicrapnoteris linearis* are reported to have successfully performed metal uptake of several types of metals such as cadmium (Cd) [18], [19], copper (Cu) [19], gold (Au) [20], and even REE [16] from various types of substrates. In the research conducted by Bali et al. [20], he used *Brassica juncea* and *Medicago sativa* plants to accumulate gold from artificial substrates with different concentrations and contact times. The results of the study stated that *Brassica juncea* and *Medicago sativa* were able to accumulate Au in the roots as much as 227 mg/g and 287 mg/g dry weight respectively. Another case conducted by Jally et al. [21], the results of observations he made on *Dicrapnoteris linearis* plants found in tailings waste proved that the plant was able to accumulate a total REE of 2.7 mg/g in the leaves. Based on the results of metal recovery ability by plants from the previously mentioned studies, the phytoextraction or phytomining method should be considered. However, research on phytomining in red mud waste is still very minimal. Therefore, the purpose of this study is to evaluate the phytomining ability of Imperata cylindrica plants on red mud waste. Due to the highly alkaline nature of red mud and the lack of nutrients in it, in this study adjustments were made first by adding NPK fertilizer, citric acid, and adjusting the composition of red mud.

Methodology

Materials

The raw materials used in this study were red mud sourced from Tayan, West Kalimantan, Indonesia, and soil. The digestion of raw materials (soil and red mud) and post-harvest substrates was conducted using 30% H_2O_2, 65% HNO_3, and 37% HCl (EMSURE for analysis, Merck Millipore). The materials were characterized to determine their physical and chemical properties before and after the experiments.

Characterization

The characterization of raw materials (red mud and soil) and post-harvest samples included an analysis of elemental composition and physical properties. Major elements were analyzed using X-Ray Fluorescence (XRF) while the elemental composition analysis of minor metals, including Rare Earth Elements (REE), was performed using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES, Optima 8300, PerkinElmer). The properties of the raw materials, such as pH and electrical conductivity were also determined. The results of the characterization are presented in Table 1, providing baseline data for the optimization and evaluation of phytomining performance.

Table 1. Initial characterization of red mud and soil media

Parameter	Value		Unit	Method
	Red Mud	Soil		
pH	10.91	8.43	-	Soil to water ratio of 1:5 (w:v)
Electrical Conductivity	5861	4556	(μs/cm)	
Fe	60	4.605	%	XRF
Ti	3.78	0.517	%	XRF
La	1.12	0	(mg/kg)	ICP-OES
Ce	3.87	0	(mg/kg)	ICP-OES
Nd	44.53	13.3	(mg/kg)	ICP-OES
Gd	407.24	108	(mg/kg)	ICP-OES
Sc	19.83	0	(mg/kg)	ICP-OES
Total Microbes	3.39×10^6	4.68×10^7	CFU	TPC

Method

The planting medium was conditioned to adjust the pH and optimize metal availability. Citric acid was added at a dose of 20.632 mg/g to lower the pH and act as a chelating agent. NPK fertilizer was applied at a uniform dose of 100 mg/kg to provide nutrients for plant growth. The planting media consisted of 40% red mud and soil, with a total weight of 1.5 kg per pot. The conditioning process was based on lab tests in the previous study to determining the optimal composition and pH adjustment [22]. After the medium is suitable, acclimatization was conducted for 60 days on soil media mixed with compost and livestock manure to observe plant growth performance. Flower blooming at the tops of plants was used as an indicator of successful acclimatization. After acclimatization, the plants were transferred to media containing red mud.

Plants were cultivated in the prepared substrate containing 40% red mud. During planting, NPK fertilizer was applied at 100 mg/kg on the first day, and watering was done daily with 150 mL of water, divided into two intervals (morning and evening). The experiment lasted 35 days, with harvesting conducted every 7 days. The plants were separated into roots, stems, and leaves for further analysis. Plant samples were dried at 100°C to a constant weight, ground into a fine powder, and homogenized. A total of 0.3 grams of the powdered sample was digested using aqua regia (HNO_3: HCl = 5:1) on a hot plate at 80°C for 6 hours. The digestion solution was analyzed for metal content using ICP-OES. Post-harvest substrates were analyzed for microbial content using the Total Plate Count (TPC) method to evaluate soil health.

Results and Discussion

Metal uptake profile based on different harvesting times

In this study, there are types of metals observed, which are valuable metals in the form of REE with the Ce, Gd, La, Nd, and Sc as well as major metals in the form of Fe and Ti. Observations were made on the roots, leaves, and stems at different harvest time intervals. For REE metals, metal uptake only occurs in the roots of the plant. It can be seen in Figure 1 that the uptake of REE metals began to occur on day 28 with the highest accumulation occurring on day 35. The highest accumulation occurred in Gd metal with a value of 119.5 mg/kg followed by Nd, Ce, La, and Sc with a value of 16.5 mg/kg, 6.67 mg/kg, 2.5 mg/kg, and 2 mg/kg respectively. This indicates that rare earth metals have a tendency to accumulate exclusively in plant roots, most likely due to the limited mobility of these metals in plant tissues and as a response to the resulting oxidative stress. Therefore, the plant will activate several protective mechanisms such as production of antioxidant enzymes in the form of superoxide dismutase (SOD) as well as an increase in non-enzymatic antioxidant compounds such as glutathione, ascorbic acid, and carotenoids [23], [24].

Unlike the rare earth metals, major metals such as Fe and Ti accumulated in all parts of the plant, including roots, stems and leaves. Fe and Ti uptake in the roots showed an increasing trend during the study period, with maximum accumulation occurring on day 35, amounting to 25,450 mg/kg and 888.50 mg/kg, respectively. However, in stems and leaves, the pattern of Fe and Ti absorption did not show a clear trend. For example, Fe uptake in leaves was only detected on day 7 (179.33 mg/kg) and day 21 (9.65 mg/kg), while Ti accumulated more consistently in stems and leaves at all harvest times with maximum values of 46.83 mg/kg (leaves) and 29.83 mg/kg (stems), respectively.

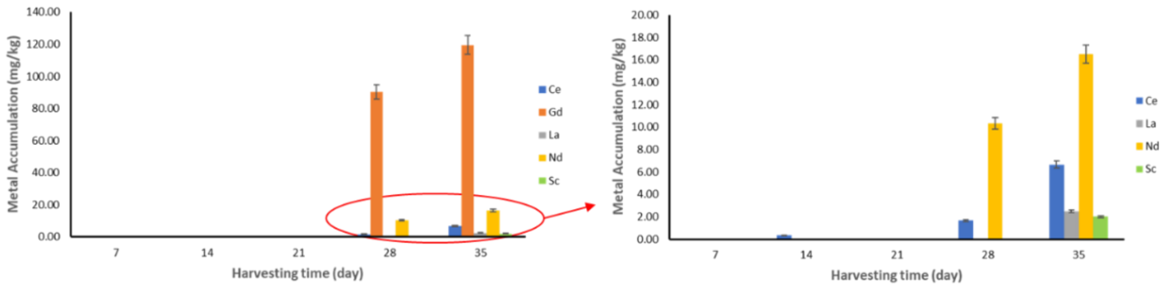

Figure 1. REE metal uptake profile at 40% red mud composition with different harvesting time in root part

These differences in uptake patterns reflect the chemical nature of these metals, where Fe and Ti are more easily mobilized in plant tissues than rare earth metals. Competition between metals also affects the uptake efficiency, as Ti and Fe compete to use the same transport pathways. Another factor that plays an important role in metal uptake is the interaction between metals with organic ligands in the substrate. Ligands such as citric acid can increase solubility of metals in the soil, but at the same time limit the mobility of metals to the upper part of the plant [25], [26]. The Fe and Ti uptake profiles can be seen in Figure 2.

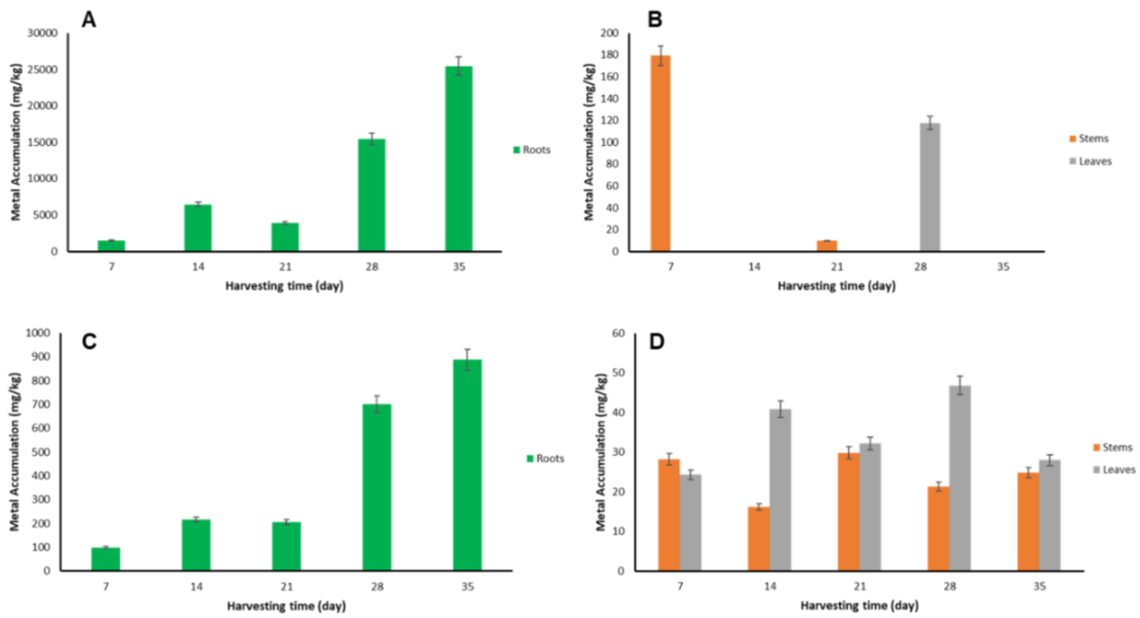

Figure 2. Fe (A and B) and Ti (C and D) metal uptake profile at 40% red mud composition with different harvesting time in roots, stems, and leaves

Soil health profile based on total microbes in different harvesting time

In this study, one of the variables evaluated as a fertility parameter is based on the total microbe's content. In Figure 3, it can be seen that the total microbes content experienced a downward trend from a value of 1.47 x 107 since day 7 to a value of 1.57 x

106 on day 28 of harvesting from a value of 1.47 x 107 to. This downward trend may be related to increased heavy metal content in the soil due to root exudation or metal remobilization processes from soil to roots. Accumulation of heavy metals in the soil can affect the activity of microorganisms, reducing microbial populations sensitive to metal toxicity.

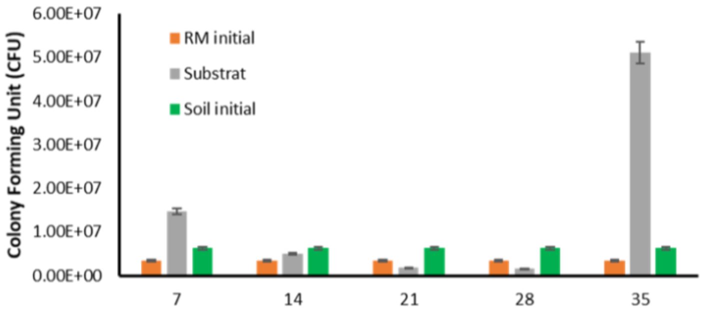

Figure 3. Total Microbes in Different Harvesting Time

Fortunately, a surprising thing happened where in a period of 1 week from harvesting day 28 to day 35, there was a very significant increase until it reached 5.11 x 107 on the last day of harvesting. This phenomenon can be attributed to several factors, such as the degradation of root exudates or changes in soil properties that favor the growth of certain microbes [27]. Another possibility is the adaptation of microorganisms to changing soil conditions, where more heavy metal-resistant microbial populations become dominant. The increase in total microbes at day 35 may also reflect a partial recovery of the soil microbial ecosystem. This provides an indication that phytoremediation practices, while having the potential to decrease microbial populations in the short term, may facilitate the regeneration of microbial communities in the long term. Further analysis is needed to understand the dynamics of microbial communities and their specific roles in improving soil health during the phytoremediation process.

Conclusion

The results showed that rare earth metals (REE) such as Ce, Gd, La, Nd, and Sc accumulated selectively in plant roots, with the highest accumulation on day 35. Major metals such as Fe and Ti were distributed throughout the plant, but with different accumulation patterns, where the roots showed a consistent increase in accumulation until the last harvest. Soil health as measured by total microorganisms showed significant changes during the harvest period. The decrease in microbial population at the beginning of the observation was most likely due to heavy metal toxicity. However, the sharp increase at day 35 indicates the potential for microbial adaptation to the contaminated environment, which may contribute to the regeneration of the soil ecosystem in the long term.

This study makes a significant contribution to the understanding of phytoremediation mechanisms, particularly in the context of metal accumulation and its impact on soil ecosystems. The findings add insight into how plants and soil microorganisms can interact in a phytoremediation system, as well as provide empirical data to optimize the technique. For further development, future research is recommended to explore specific microbial interactions that support improved soil health during phytoremediation. In addition, evaluation of additional parameters, such as soil enzymes or toxicity of metal residues in plants, will enrich the understanding of the sustainability of these methods.

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