Composite Treatment Module for Removing Acidity and Metal (Loid)S from Acid Rock Drainage

Louis AA[1] and Manoj M[2]*

[1]Department of Mining & Metallurgical Engineering, University of Nevada, USA

[2]Department of Mining & Mineral Resources Engineering, Southern Illinois University, USA

*Corresponding author: Manoj Mohanty, Department of Mining & Metallurgical Engineering, University of Nevada, 1664 N. Virginia St, Reno, NV 89557, USA, Tel: +1 6185598264; Email: mmohanty@unr.edu

Abstract

This study evaluates a two-stage approach for remediating acid rock drainage (AMD) generated from an abandoned coal mine site. The method combines the adsorption and chemical precipitation properties of drinking water treatment residue (WTR) in a continuous flow-through fluidized filter bed with hydroponic phytoremediation using Vetiver grass to remove dissolved metal(loid)s. In the first stage, the filter demonstrated exceptionally high removal efficiencies for several metals, including up to 99.67% of Al (<1 mg/L), 99.46% of Fe (<2 mg/L), and 92.48% of As. Significant removal efficiencies were also achieved for Zn (69.24%) and Cu (62.85%). However, relatively low removal efficiencies were observed for Mn (12.50%), Ni (26.74%), and Co (21.36%). Additionally, the system effectively reduced acidity, increasing the pH from 2.67 to a near-neutral level of 6.13. The second stage, a 30-day Vetiver grass treatment, further reduced Mn concentrations by 56.9%, improving upon the limited Mn removal observed in the fluidized filter column. This underscores the complementary role of hydroponic phytoremediation in enhancing the overall metal removal process. This innovative and cost-effective system demonstrates significant potential for AMD remediation, achieving simultaneous removal of metal(loid)s and neutralizing acidity in contaminated mine water.

Graphical Abstract
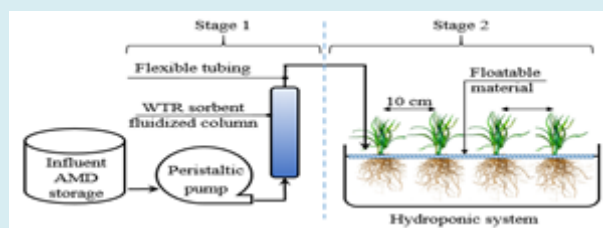
Highlights

- Acid rock drainage causes environmental pollution.
- Water treatment residues remove metals and reduce the acidity of contaminated solution.
- Vetiver plant has proven phytoextraction capabilities for metals in hydroponic systems.

Abbreviations

AMLs: Abandoned Mine Lands; AMD: Acid Rock Drainage; WTRs: Water Treatment Residues; EC: Electrical Conductivity; DO: Dissolved Oxygen.

Introduction

Centuries of unregulated coal mining have left behind thousands of abandoned mine lands (AMLs) worldwide, many of which generate acid rock drainage (AMD). This acidic effluent contains high concentrations of sulfates and metal (loid)s such as Al, Fe, Mn, Zn, Cu, and As, along with other co-occurring elements. When AMD discharges into surface water bodies, it significantly impacts aquatic ecosystems, reducing biodiversity and degrading water quality. Moreover, AMD contributes to the bioaccumulation of toxic metals in soils, leading to reduced soil fertility, plant toxicity, and health risks to humans and animals through the food chain [1,2].

Conventional AMD remediation methods, including reverse osmosis, ion exchange, electrodialysis, passive sulfate-reducing bioreactors, and adsorption, are often energy-intensive, labor-demanding, and associated with high costs. Among these, adsorption has gained popularity due to its operational simplicity and safety compared to other techniques. Commercial activated carbon and its derivatives have been widely studied for efficiently removing trace metal ions [3-6].

To improve cost-efficiency and sustainability, several researchers have focused on alternative adsorbents to replace activated carbon, including iron hydroxides, activated alumina, modified chitosan beads [7-9] and water treatment residues (WTRs) [10-12]. Despite their positive laboratory performances for metal removal, typically performed in a batch setup under controlled environment using synthetic acid rock drainage and in batch, they have mostly failed to demonstrate adequate effectiveness and long-term sustainability in a natural environment. This is because field AMD is a complex, dynamic mix of metals, acidity, sulfate, and organic matter which are difficult to replicate in synthetic solutions. Hence, the AMD solution used in this research was collected in the field to correctly capture the variability found across different mining sites and seasonal changes, providing more relevant and reliable data for evaluating the efficiency of the proposed treatment method in a continuous flow mode.

Relative to conventional adsorbents and other precipitants are WTRs - byproducts of coagulation-filtration and precipitative softening processes. It consists primarily of soil separates, organic matter, and (oxy) hydroxides of Al, Fe, and Ca. These residues can constitute 2–10% of a water treatment plant's throughput and are often readily available at minimal cost beyond transportation [13]. Their morphology, surface chemistry, availability, and low cost make WTRs a promising and sustainable alternative for metal removal in aqueous environments.
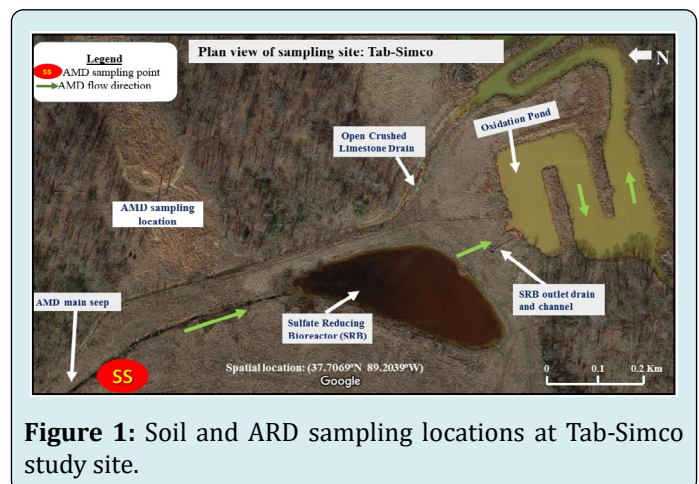
Phytoremediation, recognized for its cost-effectiveness, aesthetic appeal, and environmental sustainability, has gained considerable attention as a complementary method. Over 400 plant species, including Vetiver grass (*Chrysopogon zizanioides*), are identified as hyperaccumulators of metals [14,15]. Native to tropical and subtropical regions, Vetiver grass is well-suited for remediation due to its robust morphology, adaptability to diverse environmental conditions, and ability to tolerate and remove contaminants from water and soils.

Herein, the evaluation of the novel synergistic application of WTRs and vetiver to successfully attenuate metal (loid) concentrations in aquatic systems under natural environmental conditions, is reported. The goal of this research is to understand the biological and physicochemical mechanisms through which this system removes metals from acid rock drainage. Conducted at the Southern Illinois University Energy Park, the experiment also assessed the effectiveness and limitations of this integrated technique, highlighting its potential for scalable and cost-efficient AMD remediation pathways.

Material Sampling and Characterization

Acid Rock Drainage

ARD was collected from the Tab-Simco abandoned coal mine (Figure 1) in the Illinois Basin in the USA, identified in 1996 as one of the most heavily contaminated ARD sites in the mid-continent region, producing an average of 4.86 m3/hour (1,284 gallons/hour) [16,17].

Figure 1: Soil and ARD sampling locations at Tab-Simco study site.

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Representative samples were collected from a seep area (SS) where natural attenuation of contaminants is minimal to investigate its level of acidity and trace metal toxicity. A constructed weir was used to enhance the hydrologic head for sampling below the surface while avoiding sediment contamination from the bottom.

Field parameters, including pH, electrical conductivity (EC), dissolved oxygen (DO), and oxidation-reduction potential (ORP), were measured on unfiltered ARD samples immediately after collection using a portable HACH® multi-parameter water analyzer (Model HQ40D, HACH, USA). Calibrated probes that were employed included Intellical™ PHC101 (pH), Intellical™ MTC301 Ag/AgCl (ORP), and Intellical™ CDC401 (DO).

For dissolved metal analysis, aqueous samples were filtered using 0.45-micron filter paper and acidified with ultrapure nitric acid (HNO3) to lower the pH to <2.0, then stored at 4°C. Acidification was performed to prevent metal precipitation, minimize adsorption to container walls, and inhibit biological activity. Dissolved metal (loid)s were analyzed using an Agilent ICP-MS 7500, equipped with an ESI SC-4 high-capacity, fast-washout auto-sampler, and gallium as the internal standard. For quality control, duplicate acid blanks were analyzed, confirming that target metal (loid) concentrations in HNO3 blanks were below method detection limits (MDL). The concentrations of relevant metal (loid)s and additional parameters considered in the study are summarized in Table 1.

Analyte	Concentration [mg/L]	Chemical parameters	Value
Al	130	pH (std. units)	2.67
Mn	32.4		
Fe	327	ORP (Mv)	486
Co	0.426		
Ni	1.72	DO (mg/l)	16.8
Cu	0.035		
Zn	2.88	EC (μs/cm)	4884
As	0.003		

Table 1: Relevant properties and elemental analysis of the ARD.

Although the ORP and DO suggest the water is well-oxygenated with good oxidative capacity, the recorded extreme acidity and high concentration of metals suggest that the water is highly contaminated and severely toxic to most forms of aquatic life. The low pH increases the solubility and bioavailability of metals as corroborated by the recorded EC, thereby amplifying their toxic effects.

Vetiver Plant

Vetiver grass stolons used in this study were obtained from Agriflora Tropical in Puerto Rico. Vetiver stolons were potted and nursed in a greenhouse to ensure the development of new roots and shoots that indicate good health of the grass, hence, only stolon that developed new roots were used in the study.

Water Treatment Residue

Water treatment residue (WTR) sample was collected from various locations along the dewatering pond at the Saline Valley Conservancy District Water Treatment Plant in Saline County, Southern Illinois, USA. Composite (pooled) WTR samples were collected in labeled plastic buckets with lids to transport. To prevent high temperature drying (>120°F) from affecting the extractable cation analysis, the samples were air-dried at room temperature (~72°F). Once dried, the samples were crushed, thoroughly mixed, and sieved through a 2-mm mesh for storage and subsequent analysis. The samples were stored at ambient temperature and analyzed for pH and electrical conductivity (EC) within four weeks of collection. The WTR's physical and chemical properties were determined following standard procedures, as outlined in Table 2.

Particle Size Distribution (%)					
Ph	EC (μs/cm)	SG (g/cc)	Sand	Silt	Clay
9.1	321.3	2.47	12.5	76.5	11

Total Element Analysis by AR (mg/kg)								
Fe	Mn	Al	Zn	Cu	As	Pb	Ca	Ni
6100	203	400	9	1.4	18.1	0.4	3.30E+05	0.6

DTPA Extractable Micronutrients (mg/kg)					Exchangeable Cations (meq/100 g)			
Zn	Mn	Fe	Cu	Bo	K	Mg	Ca	Na
1.3	28	194	1.2	0.9	0.04	4.5	8.54	0.11

*pH = 1:2.5 (w:v; soil: water), EC = 1:5 (w:v; soil: water), AR: aqua regia digestion DTPA: diethylenetriaminepentaacetic acid (chelating agent)

Table 2: Selected characteristics of WTR used in this study.

Particle density was determined using a water pycnometer following ASTM D 854-00, while high-resolution particle size analysis was conducted with the patented Microtrac® Tri-Laser Technology Bluewave particle size analyzer (Pa, USA). The bulk mineralogical composition of randomly oriented WTR powder samples was characterized by X-ray diffraction (XRD) using a Rigaku Ultima-IV diffractometer. Intensity data were collected in the 2θ range

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of 2°–60°, with a scanning step of 0.02° using Cu-Kα radiation. Mineral phase identification was performed by comparing d-spacing values with published crystal structure data [18].

X-ray diffractograms Figure 2 revealed that the WTR was predominantly composed of calcite, with a significant presence of other non-crystalline, amorphous materials such as Fe or Al oxides mainly due to the use of coagulants such as alum [Al2(SO4)3.14H2O], ferric chloride [FeCl3], and iron (III) sulfate [Fe2(SO4)3] in drinking water treatment process.
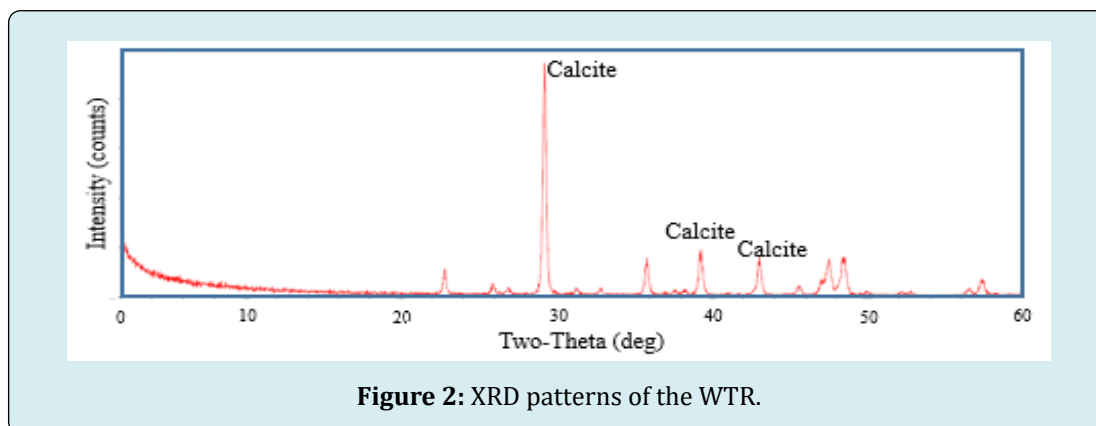

Figure 2: XRD patterns of the WTR.

Experimental Setup

Stage 1: Fluidized Column Filter

A transparent plastic tube measuring 5cm in diameter and 45cm in height, was loaded with 120 grams of WTR media to treat ARD at a consistent flow rate of 40 mL/min. Based on preliminary bench-scale experiments, the media was changed when the flow rate reduced to 15% of the initial flow rate mostly occurring after four (4) hours of the system operation. A reverse flow approach was used to maintain a fluidized layer to enhance contact and reduce short-circuiting. To minimize WTR material loss, the top cap of the column was packed with glass wool. A test run without any media indicated no significant impact of glass wool on pH or metal removal. A Teflon thread seal was used to control potential leakage from the system. Composite samples of the column effluent at the end of the experiment were collected and analyzed for pH and metal concentrations by ICP-MS. The staged setup of the experiment is illustrated in Figure 3.

Figure 3: Configuration of fluidized column (1st stage) and hydroponic phytoremediation (2nd stage).

Stage 2: Vetiver Hydroponic Unit

To assess the metal uptake capability of vetiver grass during the hydroponic treatment stage, approximately eight (8) gallons of column effluent were collected into a black open plastic container with a water depth of roughly 20 cm. The dark color of the container helped inhibit algae growth by blocking direct sunlight. Vetiver grass was placed in holes on floating platforms made of non-reacting Styrofoam, with plants spaced 10 cm apart (center-to-center), providing roughly 100 cm^2 of water surface area per plant. Cotton was used to support the plants at the base, as shown in Figure 4. The hydroponic study was conducted in batch mode over a 30-day period under ambient weather conditions in Southern Illinois. A transparent cover was erected over the setup to minimize dilution from precipitation, and the water level was regularly maintained by adding deionized water to compensate for evaporation losses. At the end of the 30-day study, the net growth of the vetiver plant root and shoot. Harvested vetiver was triple-washed with deionized water, and separated into shoot and root fractions. Plant tissue analysis was performed to evaluate metal uptake and translocation. The multi-element dry ash method [17] was employed to prepare the plant samples for total metal(loid) analysis using ICP-MS.
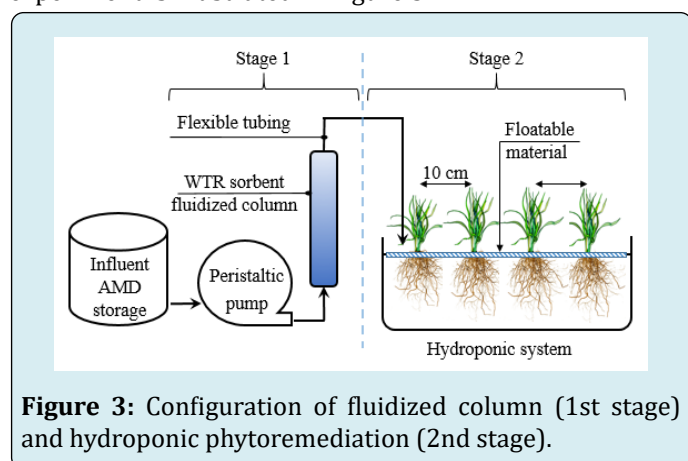

Figure 4: Vetiver floating platforms for hydroponic (2nd stage) treatment.

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Results and Discussion
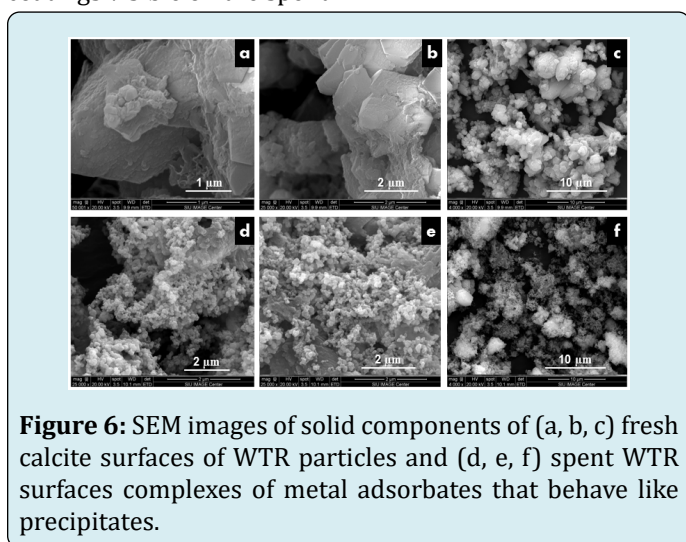
Metal (Loid)S Removal from Fluidized Column

The fluidized column filter effectively reduced the acidity of influent AMD, increasing the pH from 2.67 to 6.13. As shown in Figure 5, significant amounts of the target metals were removed, including Al (130 to 0.43 mg/L), Fe (327 to 1.75 mg/L), Mn (32 to 28 mg/L), and other co-occurring elements: Cu (62.85%), Zn (69.24%), Ni (26.74%), Co (21.36%), and the metalloid As (92.48%).

Figure 5: Metal (loid) concentration of column filter effluent by ICP-MS.
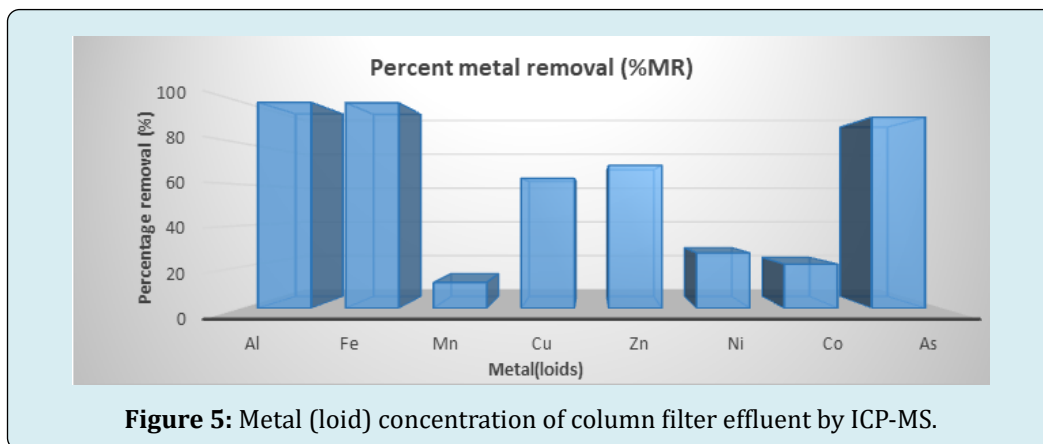
The multiple mechanisms driving the removal of the metal(loid)s can be attributed primarily to precipitation and nucleation resulting from the pH neutralization triggered by calcite dissolution, and adsorption on the amorphous surface structure of the WTR.

To further assess the occurrence of adsorption and/or precipitation, the surface structure of both fresh and spent WTR was analyzed using a FEI Nova Nano SEM 430 field emission scanning electron microscope (SEM), equipped with backscattered and secondary electron detectors. The clean calcite surfaces of the fresh WTR (Figure 6) were observed to have changed from the experiment, with surface coatings visible on the spent WTR.

Figure 6: SEM images of solid components of (a, b, c) fresh calcite surfaces of WTR particles and (d, e, f) spent WTR surfaces complexes of metal adsorbates that behave like precipitates.

The chemical nature of these surface complexes remains undetermined but could represent either a hydrated or dehydrated surface complex, or a phase that behaves similarly to a surface precipitate. This observation suggests that both adsorption and precipitation processes may have occurred, but further studies and analysis would be required to conclusively identify the nature of these surface modifications. Due to the complexity of surface sorption mechanisms and the roles of dissolution and precipitation, especially under conditions typical of natural systems, this study does not attempt to distinguish between adsorption and surface precipitation processes. The challenges of isolating these distinct metal removal mechanisms in aqueous environments were emphasized in a similar study by Al-Mahrouqi, et al. [18].

Hydroponic Phytoextraction of Manganese

During the initial stage of treatment, most metal ions present in the AMD were effectively removed, reducing their concentrations below regulatory discharge limits. The subsequent hydroponic phytoremediation stage was specifically designed to target and further reduce manganese levels in the column effluent. Elevated manganese concentrations cause operational and aesthetic challenges in water utilities [19] and may present health risks, including neurotoxicity [20]. At the end of the 30-day hydroponic stage, the concentration of manganese was further reduced, achieving an additional 56.9% removal, in addition to the 12.5% removal observed in the fluidized column filter. Although the recorded concentration is still above EPA's secondary maximum contaminant level (SMCL) of 0.05 mg/L for aesthetic considerations, the results reinforce the incremental purification capacity of the hydroponic system.

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Phytoextraction Assessment in Vetiver

After the experiment, it was observed that vetiver demonstrated significant growth, with additional root lengths averaging 11.6 ± 2.7 cm (≈ 0.38 cm/day), and net shoot length reaching 32.0 ± 11.67 cm (≈ 1.07 cm/day) (Figure 7). The growth of vetiver contributed to an increased surface area in both root and shoot tissues, which, in turn, enhanced photosynthetic activity and facilitated the uptake of manganese (Mn) and other dissolved metal(loid)s from the solution. This growth pattern supports its adaptability to the hydroponic environment.
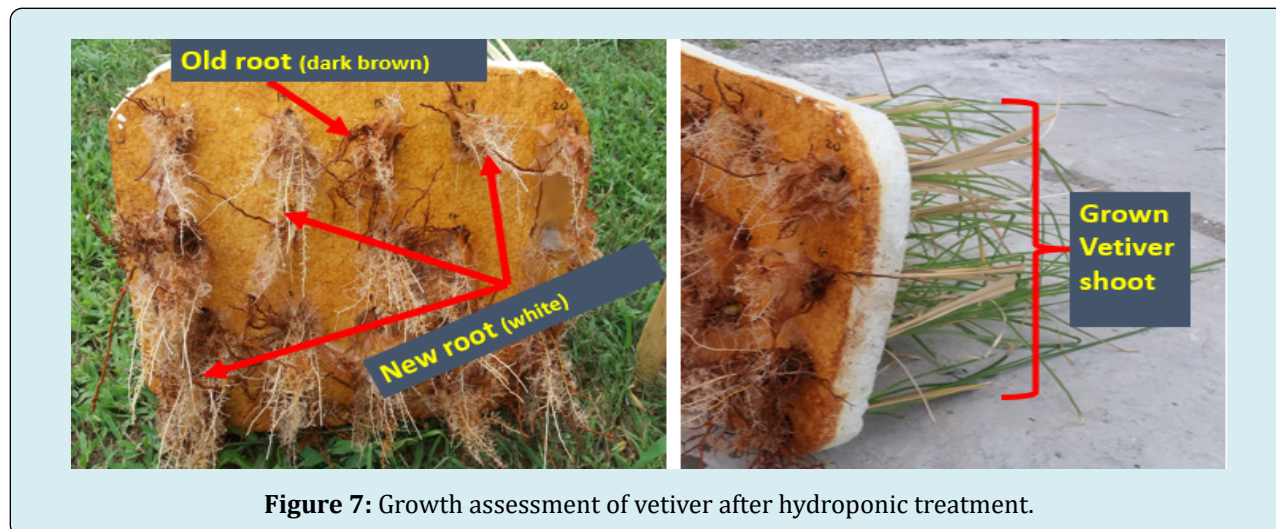

Figure 7: Growth assessment of vetiver after hydroponic treatment.

The tissue analysis of vetiver grass indicated a significant uptake of manganese (Mn). Concentrations of Mn in the shoot and root systems, measured on a dry weight (DW) basis, revealed that metal translocation within the plant was generally low, with higher Mn concentrations observed in the roots compared to the shoots. This finding aligns with the results reported by Mwegoha WJ (2008) Roongtanakiat N, et al. and Andra, et al. [21-24], who observed similar patterns of low translocation for Mn. In contrast, Ying O, et al. [25]. Reported high metal translocation as a key physiological characteristic of vetiver which suggests some variation in findings possibly due to differences in experimental conditions and duration. The mean Mn uptake per vetiver plant was estimated to be 0.059 mg on a dry weight basis. The 30-day experimental period in this study may not have provided adequate time for plant growth and subsequent metal uptake, particularly for manganese which was present at higher concentrations in the solution at the beginning of the experiment [26-28].

The researchers of this study acknowledge that a control setup of the hydroponic system was not included in this experiment, which represents a limitation of the study. The absence of control limits the ability to definitively attribute the observed metal reduction to phytoextraction by vetiver. Hence, the incremental metal reduction between the two stages should not be taken as a mass balance for the overall experiment. Future studies should incorporate appropriate control setups to validate these findings and ensure robust conclusions.

Acknowledgments

Conclusion

This study demonstrates the potential of using a two-stage treatment system involving WTR in a fluidized column followed by hydroponic phytoremediation with vetiver grass to effectively remove metal (loid)s from acid rock drainage (AMD). The first stage, utilizing WTR in the fluidized column, successfully neutralized the AMD and significantly reduced the concentrations of several metal ions, including Al, Fe, Cu, Mn, and As, through precipitation, nucleation, and adsorption. While manganese (Mn) removal was limited in the fluidized column stage, the hydroponic phytoremediation phase incrementally reduced Mn concentrations by 56.9%, demonstrating the ability of vetiver to uptake and translocate metal ions from the treated effluent. Future studies should explore the long-term effects of this approach, including desorption and regeneration to restore the WTR

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adsorbent to its original properties, as well as the potential for phytomining and the safe disposal or recycling of metal-saturated plant biomass.

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