

Innovative Resource Recovery from Industrial Sites

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Global net-zero pledges are instigating a societal shift from a fossil-fuel-based economy to renewables. This change facilitates the use of batteries, solar photovoltaic (PV), wind turbines, etc., all of which are underpinned by critical metals. Raw metal extraction is not renewable and environmental pledges made by the government will not be met if this continues. Historic industrial sites contain vast waste stocks. These sites already have an established infrastructure for resource extraction. Applying green solvents and deep eutectic solvents (DES) to such sites for resource recovery alleviates pressure on existing raw extraction processes whilst generating more immediate stores of critical metal along with relatively insignificant environmental impacts.

Keywords: resource recovery ; critical metals ; green solvents

1. Introduction

Critical metal demand has increased exponentially due to rapid industrialisation and societal pressures. Reserves for critical metals are diminishing. Metals are in demand to provide services and goods which are essential to meet basic human needs ^[1]. European government-backed policies are setting targets to reduce greenhouse gas emissions by up to 95% (compared to 1990 levels) by 2050 ^[2]. In order to do so a societal shift towards renewable energies (e.g., solar PV, wind power, fuel cells, hydrogen storage) from fossil fuels is imperative. This change will be underpinned by the critical metal supply which is already under pressure ^[2].

A small portion of end-of-life lithium batteries are disposed of through inappropriate waste streams, and the majority are stored in landfills or collected in households ^[3]. The EU Battery Directive 2006/66/EC has inadequacies as there are no defined recycling efficiencies for specific elements or components nor does it reflect the CE concept ^[3]. Recent predictions highlight that lithium stores will deplete between 2060 and the end of the century ^[4]. Not only will this limit electric vehicle capacity but also solar PV, LED, electric vehicle, and nuclear power production ^[5]. The CE concept incorporating urban waste streams transforms industrial sites from a cost to society (contributes to global warming, occupation of valuable land and water contamination) to a resource recovery opportunity ^[6]. Solar PV relies on a supply of other critical metals; copper, cadmium, and aluminium ^[7]. In the last 30 years, the mining output for aluminium has grown 256% which is beneficial for solar PV outputs, copper output has been much slower, increasing by less than 2% since 2013 ^[8]. As a result of increased raw extraction costs and falling copper prices discouraging investments into new mines and ore extraction ^[9]. This provides an encouraging platform to invest and explore novel metal resource streams, e.g., metal recovery from past industrial sites. Solar power is expected to reach a global capacity of 8519 GW by 2050 ^[7]. Using historic production and consumption as a reference, the demand for indium, selenium and tellurium could limit PV panel production by 2030 ^[10]. This will limit the potential for mass PV panel production reducing the potential for energy generation. Wind turbines have a strong potential for a CE. Recycling pre-consumer products such as steel and post-consumer products to recover rare earth metals and metalloids from waste electrical and electronic equipment could provide vast quantities of metals for turbine generators ^[11].

Physical, economic, social, or environmental factors can limit critical metal supply ^[12]. 27 of 70 studies stated that physical factors will be the main limiting concern regarding metal supply ^[1]. To supplement the physical supply of metals, secondary production avenues should be explored for the recovery of metals from historic mines and other industrial sites. Studies suggest that even with recycling rates of 99% the supply of materials recovered from recycling will not meet the ever-increasing demand due to dissipative losses and long product lifetimes ^[13]. Therefore, resource recovery needs to be implemented to diversify critical metal sources from raw materials to secondary materials, i.e., wastes from past metallurgical sites ^[1].

To achieve metal recovery from wastes to meet net zero targets, novel metal extraction techniques are crucial. Bioleaching, green chemistry, urban mining and enhanced landfill mining are all emerging trends with novel metal extraction processes ^[14]. Green chemistry, including deep eutectic solvents (DES), is at the forefront of emerging "green

technologies” to recover metals [15]. It has been suggested that one single system alone will be unable to replace conventional extraction methods, however, a combination could retrieve up to 80% [16]. Using alternative extraction methods from historic metalliferous sites reduces dependency upon conventional mining processes. Historic metalliferous sites already have existing infrastructure and accessibility for resource recovery, providing additional economic and carbon savings.

For example, the lithium battery industry is predicted to grow from an annual production of 100 GWh in 2018 to 800 GWh in 2027 due to a trend shift from fossil fuel to electric vehicles [17][18]. Currently, it takes six years to commission a mine, this runs the risk of demand quickly outstripping the supply from raw metal reserves [9]. Metals, such as lithium, are required immediately to meet the demand for renewable and energy-saving technologies. There is the opportunity to find alternative lithium stores in past metalliferous sites which can be commissioned quicker than traditional mining processes. Up to 20,000 tonnes of lithium hydroxide could be sourced from historic mines in the south waste of England [19]. Applying novel green technologies, e.g., green solvents, to a historic site to recover metals from waste slag, relieves the need to commission traditional extraction mines. Reduced dependency on conventional mining processes alleviates all associated environmental impacts; soil, groundwater, and surface water contamination, land-use change resulting in a permanent scar in the environment, loss of biodiversity and habitat fragmentation. Using historic metalliferous sites minimises environmental degradation yet enhances opportunities for the circular economy (CE) whilst meeting growing metal demands.

2. Remediation of Contaminated Sites

The need to implement sustainable remediation technologies for the redevelopment of contaminated sites has been highlighted [20]. Several traditional remediation methods, including chemical, biological, and physical methods are well-established in the last two decades but still have shortcomings (**Figure 1**). When used independently of each other, chemical and physical methods generate by-products that can be sourced from secondary sources of pollution, and they are not always cost-effective. Further to this, biological methods can be very time-consuming (can take several months to years) [21]. Recent research development has shown that using a combination of remediation methods or also called treatment train approaches allow one to overcome these drawbacks. However, the treatment train approaches are often focused on the removal or destruction of the contaminants rather than on the recovery opportunities. Recently, there has been a growing interest in recovering metals and other added value elements from various contaminated solid matrices (i.e., soil, slag, municipal solid waste, sludge, sediments), and the use of green chemistry is becoming a promising emerging approach for resource recovery from contaminated sites [22].

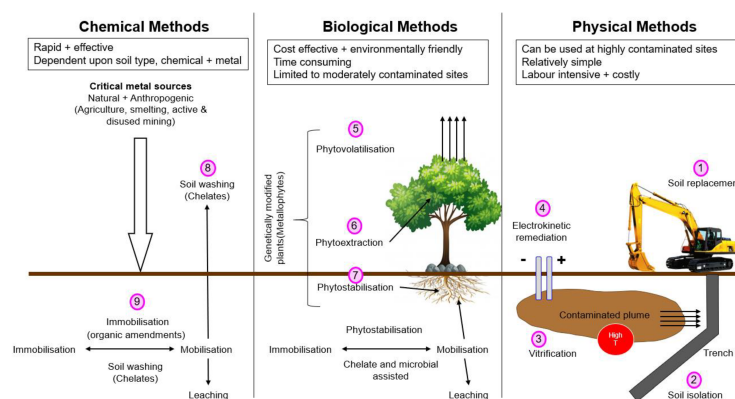


Figure 1. Different soil remediation techniques; physical, biological, and chemical.

3. Management of Contaminated Soil

3.1. Physical Remediation Approaches

Soil replacement involves either replacing or partly replacing contaminated soil with non-contaminated soil [23]. Replacing contaminated soil dilutes the concentration of metals in the soil, increasing soil functionality [24]. This method is costly due to the large working volumes and extensive machinery required so is most effective for remediating small sites with heavy metal contamination [24]. However, it is proven to increase soil environmental capacity and dilute pollutant concentration [25]. Removed soil is still heavily contaminated and should be processed correctly to avoid secondary pollution [24]. Soil isolation is the separation of contaminated soil using subsurface barriers to avoid the removal of contaminated soil [26]. Vertical barriers are used to restrict the lateral movement of groundwater, materials used include sheet piles, grout

curtains and slurry walls [23]. Soil isolation does reduce the trace metal pathway into groundwater; however, it is not a direct active remediation process [27].

Stabilisation of critical metals in soil can be achieved by applying high-temperature treatment (1400 °C–2000 °C) [23][25][28] with two main interactions: chemical bonding and encapsulation. Vitrification of industrial waste showed immobilisation of Cr, Cu, Fe, Mn, Ni, and Zn on the glass matrix [25][29]. The use of additives (clay, native soil, or sand) can improve the encapsulation of contaminants and leaching capacity [23][30]. Studies show small leaching ratios for Ag, Cr, and Cu (1.35, 0.02 and <0.01%, respectively) while Cd, Pb and Zn leaching ratios were higher (3.83, 2.46 and 0.36%, respectively) [31]. Temperature plays a vital role in metal recovery, showing low metal mobility at elevated temperatures [32]. Vitrification up to 1850 °C has been proven to efficiently stabilise critical metals (Cr, Cu, Ni, Pb, Zn, Al₂O₃, Fe₂O₃, MnO, MgO) from Pb and Zn-rich ceramic waste [29]. Using solar technology on waste from Ag-Pb mines in Spain, Fe, Mn, Ni, Cu and Zn were immobilised at 1350 °C whereas Zn, Ni, Mn, and Cu were immobilised at 1050 °C. Vitrification is ideal when working with small volumes but under field conditions where the soil has already lost its environmental function, or large scale (on-site) this approach is highly expensive [23][25].

Electrokinetic remediation operates on two established electric field gradients on either side of an electrolytic tank filled with contaminated soil [23]. Metals are removed through a combination of mechanisms; electromigration, electroosmosis, electrophoresis and electrolysis [23][27]. Remediation performs well in soils with low permeability achieving low soil function disturbance [25]. Applying chelators such as ethylenediaminetetraacetic acid (EDTA), ethylenediamine succinic acid (EDDS), nitrilotriacetic acid (NTA), and citric acid to metal-contaminated soils (As, Cd, Cr, Cu, Ni, Pb, and Zn) to enhance electrokinetic efficiency [33]. Results show that EDTA is more suited for enhanced Ni, Pb, and Zn removal in order of Pb > Ni > Zn with about 60% Pb recovered [33]. EDDs recovered 51% Cu and 26% Zn in the following order Cu > Ni > Cr > Cd ≈ Pb > As > Zn [33]. However, other research has shown that removal efficiencies are dependent upon the catalysts used and the metal remediated [23]. Additionally, the main limiting factor of electrokinetic remediation is soil pH fluctuations, as soil pH cannot be maintained thus an anionic complex cannot be formed [23][33]. Adding a buffer by using a complexant or ion-exchange membrane has been shown to control soil pH [34].

3.2. Phytoremediation Approaches

Phytoextraction uses the capability of plant roots to uptake, translocate and concentrate metals from soil into the plant biomass [23]. Phytoextraction has been shown to be the best approach for the removal of contaminants from the soil without destroying soil structure and fertility [35]. *Festuca rubra* L. is a commonly used grass, with recent research enhancing stabilisation through various soil additives (e.g., inherent mineral sorbents). Phytoextraction is suited to large quantities of low levels of contamination as most plants or hyperaccumulators cannot withstand heavily polluted sites [36]. Hyperaccumulator plant biomass is relatively easier to recycle, dispose of, treat, and oxidised than contaminated soil. The main limitation is that most hyperaccumulators are specialised to accumulate or tolerate only one metal at elevated levels with variable efficiency [36]. There are >320 Ni, 30 Co, 14 Pb, and 1 Cd hyperaccumulator species [35]. Subsequently, when trying to use phytoextraction as a remediation tool it is highly specific to the contaminating metal, with low efficiency resulting in a time-consuming approach. Though, one of the best multi-metal accumulators is *Thlaspi caerulescens* which can efficiently hyper-accumulate Ni, Cd, Pb, and Zn. Even with multi-accumulators, for this approach to be economically feasible the plants must be able to extract, translocate and produce large quantities of plant biomass [35][37]. Like any biomass production, it is limited by the growth rate, element sensitivity, disease resistance and harvesting method. Phytoextraction is only applicable to shallow contamination (up to 60 cm), and while there are low costs associated it is a time-consuming process [25]. Phytoextraction may remove contaminants from the soil, but the contamination remains in the plant biomass which must be disposed of correctly, this is the main limitation for commercial implementation [35].

Phytostabilisation aims to minimise metal mobilisation in soil and contaminants by using plants to enable stabilisation [25][38]. Phytostabilisation does not remove the metal contaminants but accumulates them within the root and results in their precipitation into the rhizosphere [36]. The isolated and adapted arbuscular mycorrhizal fungi are more adapted for phytostabilisation and are proven to be better than laboratory strains of fungi resulting in a promising avenue biotechnological remediation tool [39]. Additionally, phytostabilisation methods can be used on contaminated sites with regulatory restrictions as it limits off-site removal of contaminated soil [36]. However, because the metals remain onsite locked in plant roots, regular monitoring is necessary to ensure optimised stabilised conditions [40]. There is high public acceptance for phytostabilisation, but the method is time-consuming and limited efficiency so is better suited for large volumes of low-contaminated soil [25].

Phytovolatilisation is a highly specialised mechanism. Subsequently, phytovolatilisation exploits genetically modified plants to assimilate metal into volatile forms which are then released into the atmosphere through plant transpiration as

biomolecules [23][25]. Phytovolatilisation by *Pteris vittata* is the preferred biological remediation approach for As [41]. Phytovolatilisation focuses on Hg remediation as the gaseous form of Hg is significantly less toxic [35]. Once volatile compounds are released into the atmosphere, they become diluted and dispersed subsequently posing negligible environmental impact [36]. However, once in the atmosphere, the volatiles are likely to be recycled through precipitation and redeposited into the aquatic environment increasing the boundaries of environmental impacts past the contaminated site [42]. This technology is only applicable to volatile contaminants (Pb, Hg, As, etc.) thus application is limited [24].

3.3. Chemical Remediation Approaches

Soil washing uses extractants (e.g., surfactants and co-solvents) to leach metals out of the soil [36]. Soil washing is particularly effective at removing bioavailable and total concentrations of metals to be reused in horticulture and arboriculture [30]. Soil washing is one of the most cost-effective and powerful remediation technologies for ex situ remediation of contaminated soils [36]. Yet previous studies have reported that remediated soil was not fit for reuse in horticulture or arboriculture and suggests that soil washing should be used in conjunction with other remediation approaches, e.g., phytoextraction and phytostabilisation [30]. The efficiency of soil washing is dependent on the ability of the extractant to dissolve the metal contaminants; therefore, repeated rinsing of soils and targeted reagents for metal extraction improvement is the most efficient strategy [36]. EDTA is known to be the most effective reagent for cationic metals (Cd, Pb, Co, Li, Hg, etc.) [43]. While iron chloride (FeCl_3) is a highly effective extractant for soil washing Cd-contaminated paddy soils as it has high extraction efficiency (<70%) and low environmental impact making it highly cost-effective [44]. To further increase efficiency combining different reagents with multi-metal contaminated soils [30]. Using phosphoric-oxalic acid- Na_2EDTA for soil washing heavy with removal efficiencies of 41.9% for As and 89.6% for Cd [45]. Metals are immobilised in the soil through three different reactions: complexation, precipitation, and adsorption [36]. This is achieved by the addition of immobilising agents (clays, metallic oxides, biomaterials, etc.) to contaminated soil to limit metal transport and bioavailability in soil [24][36]. The use of lime-based agents, calcined oyster shells, eggshells and waste mussel shells is efficient at absorbing metals with minimal environmental impact and improves the soil quality and function making it a highly cost-effective treatment method [25][46].

After initial application, the effect of the amendment of metal bioavailability varies due to differences in the decomposition of organic matter [36]. Residual by-products of immobilisation contain metalliferous matter so must be processed appropriately to meet regulatory requirements. Such as, through advanced sewage treatment works which are successful in reducing metal concentrations in biosolids [36]. Although immobilisation processes are easy to implement with high initial results contaminants remain in the soil so long-term the effectiveness of this approach is relatively poor when compared to other remediation techniques [25].

4. Remediation vs. Recovery

Resource recovery is a more promising option for the sustainable management of contaminated sites rather than traditional remediation [47]. A shift from traditional remediation techniques could reclaim valuable urban land and recover minerals and metals to implement a circular economy [47].

Enhanced landfill mining (ELFM) can be defined as an “innovative transformation technology” and is a promising emerging technology [48]. In the first instance, ELFM concentrates materials (metals, combustibles, and inert materials) that can be used as secondary raw materials [48]. These materials are then reused to produce valuable products, e.g., inorganic polymers. Pyrometallurgical recycling avenues are well established, thus the metalliferous concentration can be sold at market value [49]. This makes it a key economic driver for ELFM. Numerous studies found that the recycling avenues for inorganic-non-metallic fractions are more challenging with low application aggregate as the preferred route [50][51]. Additionally, the combustible fraction is predominantly disposed of through traditional methods, e.g., incineration, which the supplier must pay for rather than obtaining revenue [48][52].

The absence of appropriate and sufficient recycling infrastructure has been highlighted as the main barrier to achieving a circular economy [53]. Using emerging green technologies for resource recovery will both improve sustainability and reduce the environmental impacts of processes involved in raw material extraction [54].

5. Emerging Extraction Techniques

Zeolites are naturally occurring, crystalline, hydrous silicates that can be modified depending on application [55]. The isomorphous substitution of silicon (Si^{4+}) by aluminium (Al^{3+}) results in a negative charge. The net negative charge within the zeolite is balanced by sodium (Na^+), cadmium (Ca^+) and potassium (K^+) ions. These cations are exchangeable with

critical metals cadmium (Cd^{2+}), lead (Pb^{2+}), copper (Cu^{2+}) and magnesium (Mg^{2+}) in solution [56]. Zeolites are highly suited to critical metal recovery due to high porosity, high sorption capacity, high ion exchange capacity, and exchangeable ions and are relatively innocuous [55]. Clinoptilolite has been proven to be a good absorbent to remove metals from wastewater to below-drinking water standards [57]. The ion exchange capacity of the zeolite is dependent upon the Si/Al ratio, where a higher Al content results in a higher number of charge-balancing cations, this subsequently increases the ion-exchange capacity of the zeolite [57]. Yet, studies show that zeolites are unable to perform direct ion exchange as negatively charged ions limit permeability to anions [58]. Similar limitations in both natural and synthetic zeolites have been found in other studies [59][60].

Zeolites have been applied in the recovery of lithium; studies have successfully recovered critical metals from spent lithium iron phosphate (LiFePO_4) batteries using zeolites [61]. This demonstrates a novel approach for reutilising spent batteries, further increasing resource efficiency, implementing a CE, and limiting environmental impact. The demand for LiFePO_4 batteries has increased exponentially since their first use in 1980. Subsequently, the number of decommissioned batteries has increased concurrently. The valuable metals and materials within the decommissioned battery are trapped in solid waste after expiration out of the material loop [62]. Resulting in diminishing stocks of such materials, including lithium [63].

Microbial technology shows promise to recover critical metals whilst producing electricity as microbial fuel cells [64]. Microbial fuel cells are bio-electrochemical systems that convert chemical energy within organic matter into electrical energy using the metabolic activity of microorganisms and have relatively low costs and are sustainable [65][66]. Metal removal using microbial fuel cells was through; bio-reduction, bioaccumulation, biosorption and biomineralisation [67].

Although promising, microbial fuel cells cannot treat all metal ions [68]. Reduction is only performed when thermodynamically favourable, i.e., the target metal has a high standard potential [69]. If metals have comparable or lower redox potentials such as Ni, Pb, Cd and Zn [70], an external power supply is required for reduction. To overcome this, Cr reduction was successfully used to power another reactor to reduce Cd simultaneously [71]. Other studies used either a Cr or Cu microbial fuel cell to successfully drive Cd reduction using no external energy supply [68][72]. Drawbacks for the recovery of critical metals using microbial fuel cells are the high impact and inhibitory effects of metal biotoxicity on microbes reducing removal efficiencies [68]. It has been found, with no exception, that all metals were proven to be toxic to *S. oneidensis* and at certain concentrations show inhibition of voltage [73]. Critical metals reduce anodic performance by inhibiting microbes due to metal toxicity [74]. Although $<100 \text{ ug/L}$ for Cu and $<1 \text{ mg/L}$ for Cd, voltage output was improved [73]. This is due to improved microbe attachment on the electrode increasing extracellular electron transfer.

Three of the twelve green chemistry principles are prevention, less hazardous chemical syntheses, and the use of renewable feedstocks. Microbial fuel cells not only treat chemical wastes but recover critical metals whilst generating electrical energy [68]. The scaling up of microbial fuel cells is limited due to wastewater capacity and the fuel cell architecture [68].

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