

Review

Chemical and Microbiological Techniques for Recovery and/or Removal of Elements from Incinerated Sewage Sludge Ash—A Review of Basic Methods

Monika Kasina *  and Kinga Jarosz 

Institute of Geological Sciences, Jagiellonian University, Gronostajowa 3a, 30-387 Krakow, Poland

* Correspondence: monika.kasina@uj.edu.pl

Abstract: In the face of material shortages, growing environmental awareness, and technical advancement, incinerated waste materials are being considered secondary resources. Especially incinerated sewage sludge ash is of interest due to its massive and increasing production and relatively stable composition with a satisfying concentration of elements of value. This paper revises the basic methods for recovery of valuable components from incineration sewage sludge and simultaneous removal or stabilization of elements that may negatively influence the environment with further ash usage or storage. The presented work focuses on collating and analysing the efficiency of currently used approaches as well as their limitations and perspectives for future development. Chemical methods analysed include acidic and alkaline leaching, chelating, and sequential leaching. Due to scarce examples from literature, the exploration of a microbiological approach focuses on the mechanisms and potential for application of different microorganisms for element extraction. The methods described are relatively efficient and affordable, yet still need further development. Specifically, microbiological approaches are rarely used for incineration sewage sludge treatment regardless of their potential advantages over other approaches. Constant mineral and chemical composition within one incineration plant can vary among plants due to many factors, so a well-established range of techniques and an individual approach are important.



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1. Introduction

1.1. Challenges in Sustainable Resources Supply

As a result of industrialization, globalization, digitalization, and ongoing technological development, humanity has consumed immeasurable amounts of natural resources [1]. A stable supply of raw materials, which are severely depleted in some regions, in order to maintain economic growth and to improve quality of life is one of the major challenges for economies.

Increasing consumption of everyday goods that require raw materials for their production and a constant decrease in natural resources is one of the largest problems of the Anthropocene. This challenge is notably present in EU countries. According to the Report on Critical Raw Material for the EU [2,3], EU countries can supply no more than 9% of the raw materials required to produce goods. The demand for raw materials in the EU in 2010 was approximately three billion tons [4]. According to predictions of the World Bank, the global materials demand will increase 500% by 2050 [5], thus it is expected that the competition for resources will remain unrestrained. The prices of industrially important materials are expected to grow and fluctuate, depending on the policy of the supplier. EU countries are highly dependent on supplies which are currently only extracted in a few countries worldwide.

The implementation of the principles of a circular economy, which focus on the preservation of mineral resources, the recycling of products, and reusing them for as long as

possible, leads to the point where waste management becomes one of the major social and economic issues in the development of a sustainable economy [6]. With growing concerns regarding mineral resources supplies on one hand, and the sustainable economy on the other, we are forced to search for alternative sources of economically important elements [7]. Waste stream materials are being more often considered novel anthropogenic waste-based substitutes for natural resources [8], allowing the maximizing of natural resource protection and returning elements into the production cycle by recycling, reusing, and—at the same time—fulfilling the ground rules of a closed-loop economy.

1.2. Risks Associated with Sewage Sludge Usage and Storage

One of the anthropogenic waste-based substitutes for natural resources is a municipal sewage sludge coming from municipal wastewater purification. Its composition, although varied and dependent on many factors such as wastewater technology or the wastewater source area characteristics, contains on average 50–70% of organic matter and 30–50% of mineral fraction, among which the IC accounts 1–4%, 3.4–4% of N is present, and around 0.5–2.5 P [9]. In addition, sewage sludge contains large amounts of heavy metals such as As, Cd, Co, Cr, Cu, Hg, Mn, Ni, Pb, and Zn [10,11]. Organic matter can easily decompose due to the low content of lignin and cellulose leading to the release of heavy metal contaminants and N. In addition, sewage sludge contains toxic organic substances [12] like PAH, PCB or AOX, pesticides, surfactants, hormones, pharmaceuticals, and pathogens [13] like bacteria, viruses, and parasites [14]. For this reason, prior to any application, sewage sludge requires stabilization (chemical or biological) and/or mechanical treatment (sewage-to-matter; sewage-to-energy) [15].

1.3. Resource Potential of Raw Sewage Sludge

Egle et al. [16] estimated that phosphorus from wastewater purification could, at least in theory, provide 40–50% of this element for agricultural purposes, with the annual production of 22.5 kg of dry sludge per capita in Europe [17] which gives around 14 million tons annually [18]. Recovery of valuable components directly from sewage sludge is inefficient. Even though the concentrations of elements are high, the large volume of waste makes the potential recovery reach levels not exceeding 50% [16,19]. In addition, the high concentration of both industrially important and unwanted components makes the handling of sewage sludge challenging. Options to properly manage increasing production of sewage sludge are related to population growth and industrial and technological development such as sea disposal, direct agricultural application, or landfilling, and are no longer supported by the assumptions of Agenda 2030 [20], where the need for a reduction in negative environmental impact is strongly connected to the banning of sea disposal and land spreading on one hand and the reduction in landfilling due to environmental side effects on the other [21,22].

1.4. Incinerated Sewage Waste as a Secondary Resource

Thermal processes are affiliated to the sewage-to-energy processes. Among many technologies, incineration seems to be the most popular. Sewage sludge incineration is a commonly used, well-established technology in developed countries. However, in other countries, it is not so widely applied due to its high cost of infrastructure and huge energy demands [23]. Regardless of the high cost, it is the best alternative to conventional disposal.

Incineration leads to volatile matter oxidation and ash production energy recovery, minimized odours and negative microbial influence. Volume reduction leads to a concentration of chemical contaminants.

Incinerated Sewage Sludge Generation by Autothermic Incineration of Sewage Sludge

In order to incinerate sewage sludge, the removal of moisture is required. Partly dewatered sludge (around 20% of a dry mass) is transported to the drying node, where, in the sludge membrane drying system, it is further dewatered down to around 30% of a dry

mass, under low negative pressure and using the heat recovered in the incineration process as a heating medium that usually forms a saturated steam. The incineration of dried sludge is commonly performed in the fluidized bed incinerator. The scheme of incineration is shown in Figure 1 and described in more detail in paragraph 1. This technique enables high turbulence with a constant intensity that provides stable temperature within 850–900 °C ranges. It allows for the complete decomposition of organic matter. The turbulence stream enables sludge disintegration [24], where heat-resistant components and volatiles are removed in the exhaust gases and organic matter is burned. The lighter components are removed, and purification products of exhaust gasses are collected into bag filters after the addition of a reactant (NaHCO_3) and are subjected to further processing, whereas heavier components and fraction that were not combusted are separated in the multicyclone, captured in the electrostatic precipitator, and stored in the ash silo.

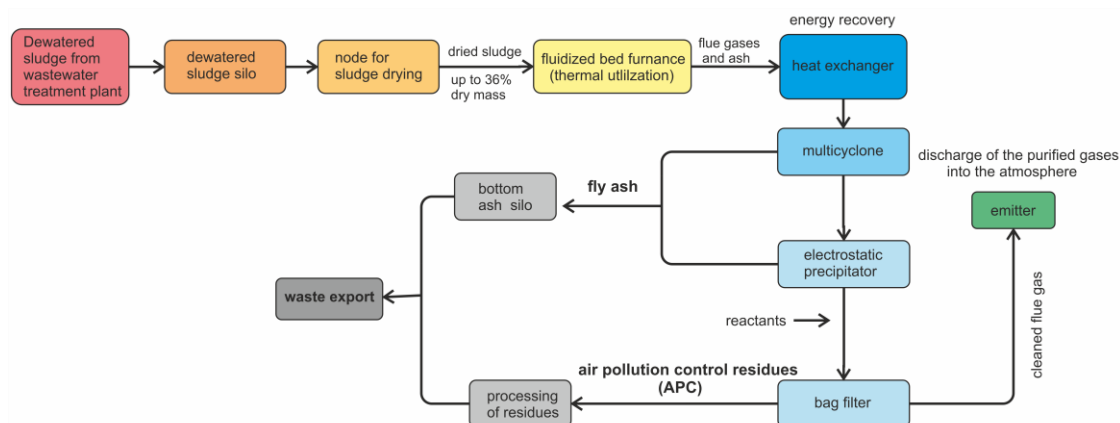


Figure 1. A scheme of sewage sludge incineration process using fluidized bed reactor.

Uncaptured suspended particles containing PM₁₀, CO, NO_x, NH₃, HCl, HF, C_{org}, and SO₂, Cd, Ta, Hg, Sb, As, Pb, Cr, Co, Cu, Mn, Ni, and W in the flue gases are released into the atmosphere. To mitigate this risk, the compounds are continuously monitored in order to control atmospheric emissions accordingly to the 2000/76/EC directive [25].

This autothermic reaction leads to a significant reduction in volume and mass of waste of 90% and 70%, respectively [26], and sanitization by destruction of toxic organic substances [27], deactivation of pathogens [28], and odorant removal [19]. As a result of sewage sludge incineration, the following residues can be created:

- Incinerated sewage sludge ash—ISSA
- Fly ash—FA
- Air pollution control residue—APC.

The amount of ISSA accounts for 90% of all solid residues produced during sewage sludge incineration, whereas FA and APC only account for 10% [29]. For this reason, most inorganic components such as phosphorus and elemental metals, which can either have industrial applicability or be hazardous and require further stabilization before any application, are concentrated in the ISSA [30–32].

1.5. The Purpose of the Manuscript

Since the ISSA has the largest share among sewage sludge incineration residues, the review focuses on the recovery of elements from this material. The chemical and biological methods for elements recovery are as described.

It is important to emphasize that even though there are numerous papers focusing on the recovery of elements directly from sewage sludge, especially regarding the recovery of phosphorus (e.g., [33,34]) or increasing the bioavailability of phosphorus by usage of microorganisms and then direct application in the agriculture (e.g., [35]), there is still a field for development in terms of recovery of important elements from ashes and/or sterilization

of ISSA by binding or removing from ash elements considered toxic, such as As, Cd, Cr, Cu, Hg, Ni, Pb, Zn.

As a result of sewage sludge incineration, organic compounds considered toxic (hazardous organic material, parasites, pathogens) are neutralized [36]. In this case, the main focus is to apply methods for inorganic components recovery including valuable components such as phosphorus, magnesium, Rare Earth Elements (REE), and those considered as potentially toxic, such as heavy metals.

The content of phosphorus in the sewage sludge is at a level of 1–5%, whereas in the ISSA, the content of a mentioned element ranges from 5 to 20% [8,37,38]. The concentration of potentially toxic elements is listed in Table 1. There are notable discrepancies in the elements' concentrations, which makes the individual approach to ISSA of great importance. The notable variations in the elements' concentration might be due to the level of industrial development of the sewage sludge source area or the technology used for the sewage sludge incineration or wastewater treatment

Table 1. Heavy metal concentrations in ISSA in ppm.

	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Min *	13	2.3	16	90	0.1	4	1.2	570
Max *	87	94	2100	5400	3.9	2000	2100	10,000

* [8,32,39,40].

Elements in ISSA can be released as a result of physical (due to large differences in approach, these methods are not included in this paper), chemical, and biological approaches.

The goal of the presented work is to collate and analyse the efficiency of the methods commonly used in terms of metal extraction, their drawbacks, and possible approaches for their improvement. The manuscript is divided into two main parts: chemical methods for elements recovery and microbiological approaches

2. Recovery of Elements from ISSA Using Chemical Approaches

Various elements can be extracted from ash with wet chemical extraction methods using acidic, alkaline, and chelating agents. The efficiency of the methods strongly depends on the molar concentration of chemicals and type of leaching agent, pH, L/S (liquid to solid) ratio, leaching conditions (time, temperature), and particle size [41,42]. The main goal is focused on the P recovery. The P concentration in the ISSA usually reaches a level of currently exploited medium-rich P ores [8], but it also leads to the leaching of pollutants such as heavy metals and metalloids (As, Cd, Co, Cr, Cu, Hg, Mn, Ni, Pb, Zn). Therefore, it is crucial to search and test methods that are effective in P extraction and simultaneously heavy metals recovery or stabilization within ISSA. The basic approach in chemical extraction is shown in Figure 2.

2.1. Acidic Extraction

Acid extraction is the most commonly used wet extraction method. It is an effective and proficient method for elements recovery. The most commonly used acids include both inorganic (hydrochloric acid HCl, sulfuric acid H₂SO₄, nitric acid HNO₃, phosphoric acid H₃PO₄) and organic (such as oxalic acid (C₂H₆O₆), citric acid (C₆H₈O₇), gluconic acid (C₆H₁₂O₇), formic acid (CH₂O₂), and acetic acid (C₂H₄O₂)) acids.

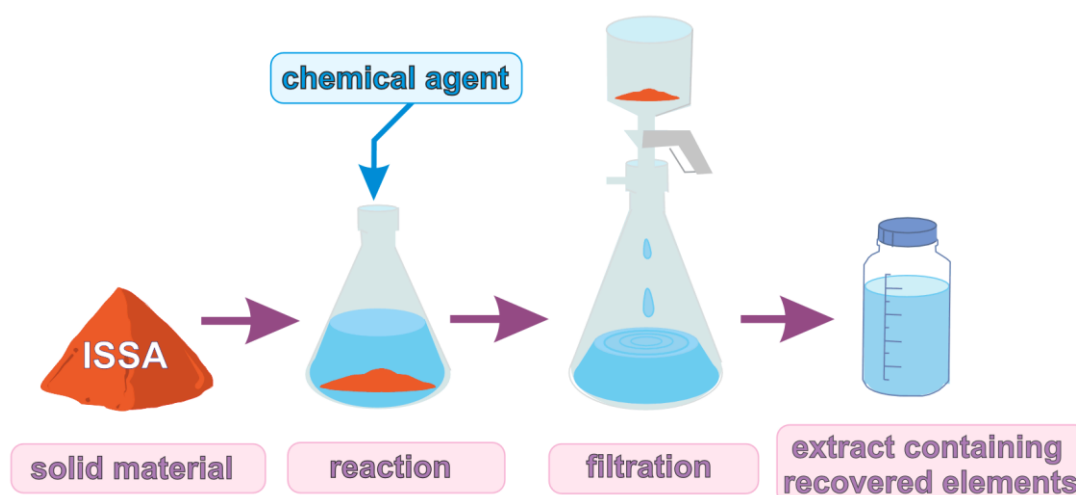


Figure 2. The standard steps in wet chemical extraction.

Inorganic acids show a high extraction capacity by alkali metal oxides dissolution, whereas organic acids are responsible for the release of metals and metalloids by inducing a chelating effect as suggested by [43]. Interestingly, the most commonly commercially used is H_2SO_4 due to its low costs [44]. The metal solubility is however dependent on the acid, and it can be shown as follows: aqua regia > HCl > HNO_3 > H_2SO_4 [45], where H_2SO_4 is considered as having a poor dissolving ability and being prone to form insoluble sulphates such as ash coatings, thus making the ash insoluble [46]. Liu et al. [19] indicated that if the H^+/P molar ratio is equal to 3, 100% of phosphorus should be extracted by acid solutions. Since ISSA is a complex material that contains other acid-soluble components the authors estimated that larger volumes of acids are required to achieve a similar result.

Both organic and inorganic acids show slight effectiveness in As, Cd, Cr, Cu, Ni, Pb, and Zn leaching from ISSA. HCl is slightly effective for the removal of almost all above-mentioned elements, however, H_3PO_4 and citric acid have also displayed slight efficiency. Usually, quite a low extractability of Pb is shown in H_2SO_4 extraction, whereas good results are obtained for HNO_3 and oxalic acid [28,47]. Leachability varies in a wide range depending on the type of ash and acid. For H_3PO_4 , H_2SO_4 , and citric acid, the result can reach from 80 to 100%, but for HCl and HNO_3 , the efficiency is lower than 25% [28,48]. Extraction of Ni can be justified for H_3PO_4 and citric acid only; in other cases, acid leaching is inefficient [28,49]. Recovery of Cu and Zn using acids can reach up to 60% [28,49]. Levels of Cr extraction are quite low [28,48,49]. In order to separate heavy metals from phosphorus, applying additional acid extraction treatment, which includes chemicals usage and energy inputs, is required to ensure the purity of the recovered products. Details concerning leaching efficiency with acids are shown in Table 2.

Table 2. Extraction efficiency using different extractant and various experimental conditions.

Extractant	Concentration [N]	L/S Ratio [mg L ⁻¹]	Contact Time [h]	Extraction Efficiency [%]								Source
				P	As	Cd	Cr	Cu	Ni	Pb	Zn	
Acidic extraction												
HCl	0.5	10	2	91	n.d	29	5	41	11	35	33	[49]
HCl	0.4	10	2	0.7	n.d	0.5	65	42	16	0.7	26	[50]
H_2SO_4	0.5	10	2	90	n.d	30	5	44	11	3	36	[49]
H_2SO_4	0.4	20	2	94	10	n.d	1	36	15	10	33	[51]
H_2SO_4	0.4	20	2	100	100	n.d	n.d	43	7	10	33	[43]
HNO_3	0.5	10	2	88	n.d	27	4.5	37	10	21	33	[49]
HNO_3	0.2	10	2	83	28	n.d	n.d	40	6	18	14	[43]

Table 2. Cont.

Extractant	Concentration [N]	L/S Ratio [mg L ⁻¹]	Contact Time [h]	Extraction Efficiency [%]								Source
				P	As	Cd	Cr	Cu	Ni	Pb	Zn	
H ₃ PO ₄	0.5	10	2	95	n.d	6	6	35	9	n.d	33	[49]
H ₃ PO ₄	0.4	10	2	96	91	n.d	n.d	35	6	21	23	[43]
C ₆ H ₈ O ₇	0.5	10	2	21	n.d	12	1	17	8	1	20	[49]
C ₆ H ₈ O ₇	0.5	10	2	75	99	n.d	n.d	43	9	4	31	[43]
Alkaline extraction NaOH	0.5	10	2	34	n.d	n.d	n.d	0.1	n.d	n.d	0.1	[49]

2.2. Alkaline Extraction

Alkaline extraction can lead to phosphorus recovery on one hand and no dissolution and release of heavy metals due to high pH on the other, which makes this method an alternative to acidic extraction. Strong bases such as sodium hydroxide NaOH or potassium hydroxide KOH must be used. Efficiency of phosphorus recovery is lower in comparison to acidic leaching [52–54] but the obtained product does not require application of any purification methods. The high pH and the high Ca/P ratio may negatively influence the precipitation of phosphate minerals over calcium carbonates [55]. In addition, the high content of Ca limits the phosphorus release [56]. In the mentioned study, 90% of phosphorus was leached from the ISSA containing 3% of Ca, whereas only 65% of phosphorus was leached from the ISSA containing 8% of Ca. Leaching behavior of other metals is dependent on their nature and form [57]. If divalent metal reacts with leachates, hydroxide simple metal (hydro)oxides can be formed, whereas metals like Pb, Cu, Ni, Zn, and V which are known for their amphoteric character will form complex bonds, and additionally will exhibit parabolic concentration curves in the leachates versus pH [58]. Additionally, some elements show cationic leaching patterns due to cationic properties [59], and thus elements' leachability decreases with increasing pH of the leachate. Details concerning alkaline extraction from ISSA are listed in Table 2.

2.3. Leaching with Chelating Agents

The chelating compounds like ethylenediaminetetraacetic acid (EDTA), ethylene diamine tetramethylene phosphonate (EDTMP), and nitrilotriacetic acid (NTA) usually exhibit a lower ability to extract phosphorus and quite a high ability to extract other metals. In the study of Kasina et al. [48], the Na-EDTA led to a release of around 35% of phosphorus. The EDTA enables the formation of complexes with Fe³⁺, which leads to the stabilization of iron in the solution and thus disables co-precipitation with phosphorus [60]. Leachability of other metals using chelating compounds can reach 50%; for this reason, EDTA is suggested to be used as one of the pre-treatment agents responsible for the removal of trace elements from ISSA, thus reducing contaminants prior to phosphorus extraction [17,43]. Additionally, it can be used for the complexation of metal ions to avoid re-precipitation of phosphorus [61]. Details concerning extraction using chelating agents from ISSA are shown in Table 2.

2.4. Sequential Extraction as an Alternative to Acid and Alkaline Leaching

To maximize extraction of expected components and to minimize extraction of unwanted elements, a combination of different leachates can be applied. In order to study the mobility of elements and their bioavailability, which are dependent on their bonds and chemical forms rather than their content, sequential extraction methods can be implemented. Sequential extraction methods exploit the fact that different solid phases exhibit different reactivity towards different solutions. Here, the material is extracted using a series of extractants selected to dissolve selectively chemical phases with similar characteristic [62]. The sequence of extractants is designed in the way that the most reactive phases and the intensity of extraction increase with each subsequent step. These methods enable us to determine possible pollution or contamination due to potentially toxic element

removal, as well as to estimate accessibility and bioavailability of elements of interest. Here, few sequential extraction methods can be introduced:

1. The Goltermann method [63] allows to distinguish four individual fractions of Phosphorus as shown in Figure 3.

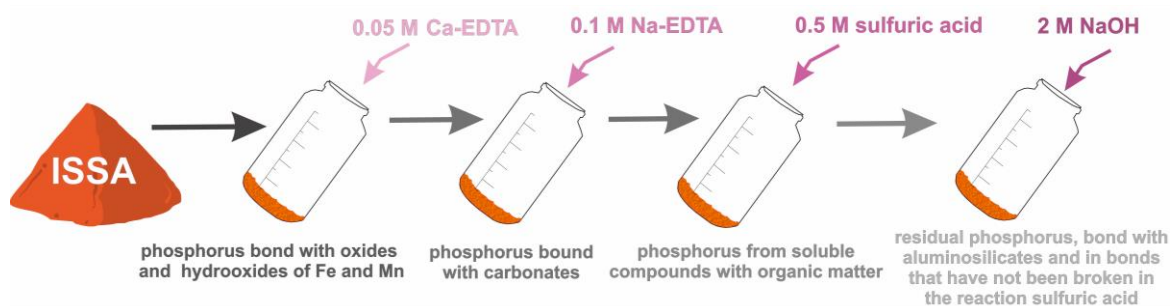


Figure 3. Details of Goltermann sequential extraction procedure.

2. Tessier sequential extraction [64] (Figure 4) is dedicated to trace and heavy elements where five fractions can be obtained. This method is a great asset to describing the contamination risk for the environment by the determination of mobility of environmentally toxic elements in various leaching conditions.

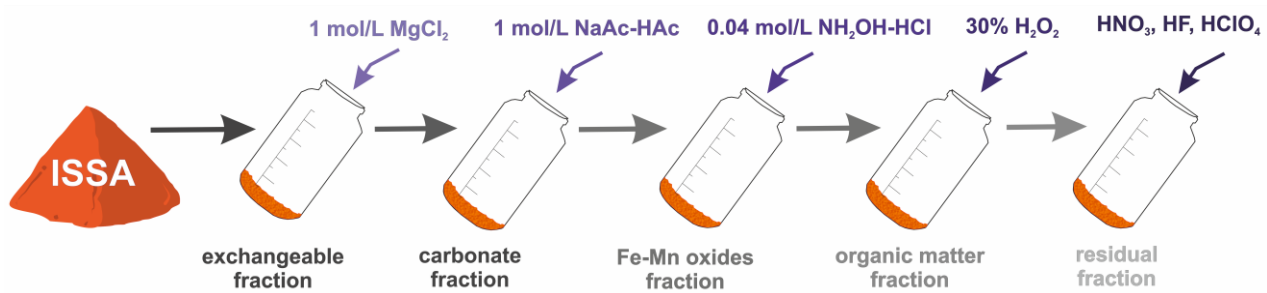


Figure 4. Details of Tessier sequential extraction procedure.

3. A three-step sequential extraction procedure in accordance with the Community Bureau of Reference (BCR, now Standards, Measurements and Testing Program) (Figure 5) enables to study trace element mobility [65].

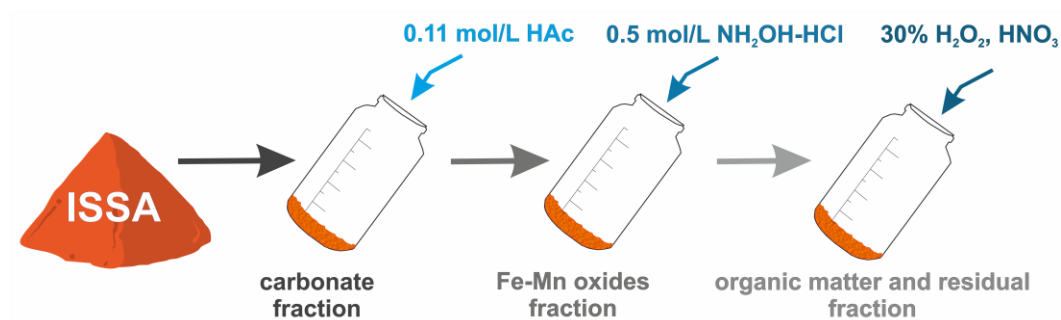


Figure 5. Details of BCR sequential extraction procedure.

4. A two-step sequential extraction following the procedure of Fang et al. [28] (Figure 6) where after heavy metal contaminants removal with EDTA, a high-purity phosphorus leachate using sulphuric acid can be obtained, and by addition of $\text{Ca}(\text{OH})_2$ in the last step, Ca-phosphate precipitation can occur. Even though this technique has great potential, it may also lead to gypsum precipitation, which negatively influences P precipitation

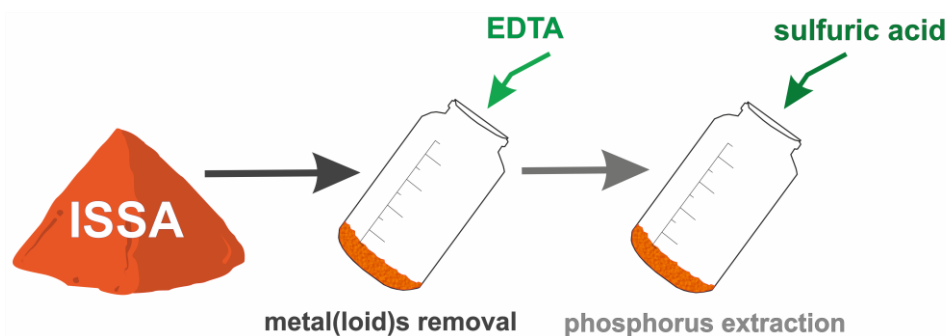


Figure 6. Details of a two-step sequential extraction procedure suggested by Fang et al. [28].

Sequential extraction methods seem to be the most complementary in P extraction and heavy metal and toxic elements removal for the ISSA, but are not without drawbacks. It is suggested that strong acids and alkaline extractants should be avoided since they are considered too aggressive and do not provide well-defined fractions. In addition, they lead to the co-dissolution of heavy metals. The methods still require optimization, which applies not only to chemical reagents used and their concentrations but also to duration time, liquid-to-solid ratio, and pH.

On the other hand, sequential extraction methods give us an overview of elements' behavior in the environment, but they need well-established reference material. In the case of the ISSA, it is necessary to know the details of chemical composition of the raw starting material in order to estimate heavy metal mobility and bioavailability routes.

3. Recovery of Elements from ISSA Using Microbiological Approaches

Chemical processing of secondary resources such as ISSA in order to extract valuable elements, even though effective [21], still produces effluents and other waste which require further processing [66] (generating additional costs and usage of resources) before discharge. Scientific and industrial interests, both driven by economic efficiency, and sustainability are being considered for application. As aforementioned, waste materials are seen as a valuable source of critical elements; however, apart from the secondary source itself, the attention is drawn towards limiting the negative impact of the technologies employed. This approach lines up with the green chemistry principles [67–69], especially the usage of safer solvents and auxiliaries, design for energy efficiency, and design for degradation. This is important both from a practical and ethical perspective. Chemicals, even if aggressive, are usually used in low concentrations and still mostly in small amounts and in highly controlled conditions; the problem starts to appear on an industrial scale implementation. A microbiological approach seems to be superior from an ethical perspective because the microorganisms used in the bioleaching are usually very common and do not pose a threat to human health or the environment, which makes this approach safe and conscientious. Numerous technological advances and solutions are inspired by naturally occurring processes or employ them directly [70,71]. Solutions based on microorganisms are recognized to limit those influences and improve or even replace chemical approaches. Over the last 20 years, the bioextraction of elements—especially via bioleaching—has developed drastically [72]. There are four basic biological techniques for recovery and removal of elements from waste material—biosorption, the use of bioelectrochemical systems (BESs), biomineralization, and bioleaching.

Biosorption results in the removal or recovery of elements by binding and in the resulting accumulation leading to a concentration of elements within biomass [73]. Biosorption requires a solid (biomatter) and liquid (solvent) phase containing the element. Biosorption is a physio-chemical interaction between the elements and cellular compounds [74]. No additional nutrients are required for this process to occur; therefore, the process itself is cost effective. As mentioned, in order for biosorption to occur, the elements must be present in the liquid phase. In the case of ISSA, only elements released into the solution

by leaching can be a subject of this process. The application of bioelectrochemical systems (BES) was based on the observation of the phenomenon of electricity generation during the oxidation of organic matter by microorganisms first made over a century ago [75]. The technique relies on the integration of electrochemical and microbial processes. Microbial communities with a bioanode (electron acceptor) or biocathode (electron donor) as a part of their metabolism might be used as BES [76]. In this technique, the external electrodes are employed to facilitate the development of differences resulting from microbial metabolism (electron donating or accepting). Cathodic reduction coupled with organic substrate oxidation had proven to be a promising and selective method for metals removal and recovery. Additionally, energy—some converted from chemical to electrical—can be recovered from the process. Biomineralization is the process induced by microorganisms resulting in the formation of minerals by the regulation of biomacromolecules [77].

Even though the mechanisms differ and the technological design focus is only placed upon one of them, they are often occurring at the same time [78]. Biological systems are complex and dynamic and therefore more difficult to design, but at the same time might be more effective as they rely on the amplification of naturally occurring processes and might even reach beyond planned results. The most common approach to biological element recovery is by bioleaching, however biosorption and biomineralization are often coupled with it as further steps.

The biotreatment of ISSA is not an established approach, and the bio-technique explored to some extent for this application is bioleaching. Bioleaching is a process of solubilization of metals from insoluble secondary wastes using naturally occurring microorganisms [79]. Via microbial extraction (bioleaching), the metals present in the form of sulphides and oxides are solubilized into the leaching medium in the form of metal cations [56]. Then, further processing of the metal-containing medium can be applied in order to recuperate valuable elements.

3.1. Metal Recovery from ISSA

Metal recovery efficiency from ISSA using biotreatment is yet to be explored, however it is possible due to both the material as well as microorganism characteristics. Microorganisms are widely used to extract valuable as well as toxic metals from different types of fly and bottom ashes [80–82]. The efficiency of removal of heavy metals from fly ashes by bioleaching varies widely between 15 and 100% depending on the metal and method applied [41]. The scope of metals which are being removed and then can be recovered from incinerated waste varies from valuable metals like gold, silver, nickel, and platinum, through most abundant metals such as aluminum, iron, or lead, to rare-earth elements (REE). Acidic bioleaching often assures satisfactory yields; however, microorganisms are used in this process mostly to maintain a low pH with a lower use of chemicals. Biotreatment in higher pH will lead to massive reduction in environmental impact and costs of metal recovery.

3.1.1. Mechanisms of Metal Recovery from ISSA

The most general distinction among bioleaching mechanisms is between the categories of contact and non-contact bioleaching [83]. The first type occurs when the microorganism cells are in direct contact with solid particles [84–86] as a result of microorganism metabolism, and the second occurs when the direct contact is not required [66] as a result of interaction with the products of their metabolism [87]. In case of the use of acidophilic bacteria, indirect bioleaching can be applied without the necessity to sustain the colony in alkaline conditions, applying only the products of their activity without direct inoculation.

In terms of mechanisms other than direct (primary) and indirect (secondary) bioleaching, other divisions can be made in terms of the reaction itself. There are three main reaction types: redox reactions (redoxiolysis), the formation of organic and inorganic acids (acidolysis), and the generation of complexing agents (complexiosis) [88,89]. Redoxiolysis can occur both in direct and indirect bioleaching, by electron transfer from solid phase to

the microorganism cell in the case of contact bioleaching and by oxidation of Fe^{2+} to Fe^{3+} for the latter (Figure 7).

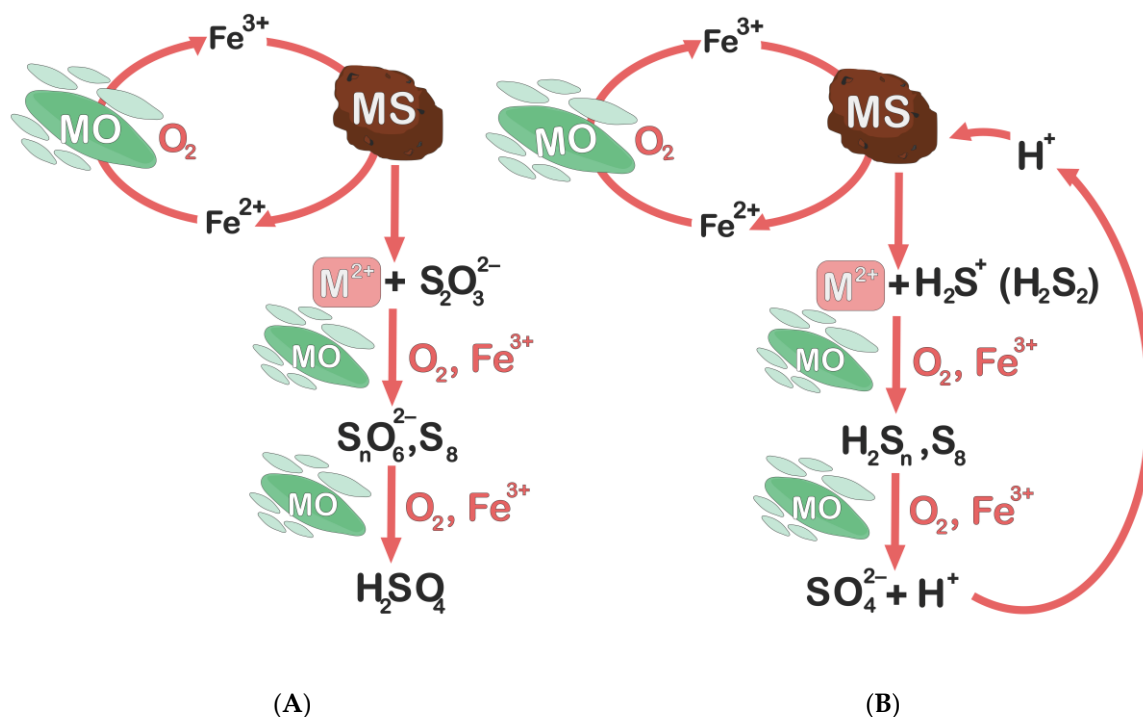


Figure 7. Schematic representation of thiosulfate (A) and polysulfide (B) pathways of metal sulfides bioleaching—MO—microorganism, MS—metal sulfide, M—metal.

Metal sulfides bioleaching is one of the most often used biohydrometallurgical techniques based on this phenomenon. The thiosulfate pathway (Figure 7A) starts with the oxidation of metal sulfides by Fe^{3+} produced by microorganisms. This results in the release of heavy metals and thiosulfate and in turn in oxidation of thiosulfate to sulfuric acid. It is applicable for minerals resistant to acidic leaching such as pyrite (FeS_2) or molybdenite (MoS_2). The polysulfide pathway (Figure 7B) is named after the main intermediate product of the process, which is polysulfides; the other main intermediate product is elemental sulfur from the oxidation of mineral phases soluble in acid. The proton attack by sulfuric acid is produced from the oxidation of reduced sulfur compounds on the metal sulfides [72,90,91].

Acidolysis occurs when microorganisms acidify the immediate environment. It results in the proton-mediated dissolution of insoluble metals. The acids produced can be organic or carbonic, and the production is powered by nutrient consumption, for example, elemental Sulphur in case of *At. Thiooxidans* [72,88]. Complexolysis is the formation of stable metal-chelating complex between metal ions and microbial metabolites [92].

The most important factors influencing bioleaching efficiency are waste characteristic (both mineralogical and physical) oxygen and iron concentrations, pH, temperature, pulp density, redox potential, and especially the microorganisms employed [88]. Often, multiple types of bioleaching occur simultaneously, especially when multi-microbial consortia are used.

3.1.2. Microorganisms Used in Bioleaching

The most often used microorganisms are sulfur-oxidizing bacteria, iron-sulfur oxidizing bacteria, and iron-oxidizing bacteria, which are classified as chemolithotrophs. Within chemolithotrophs, further division can be made depending on the growth condition caused by differences in metabolism requirements of different microorganisms (Figure 8).

Mesophiles grow at room temperature when moderate to extreme thermophiles require much higher temperatures (Figure 8).

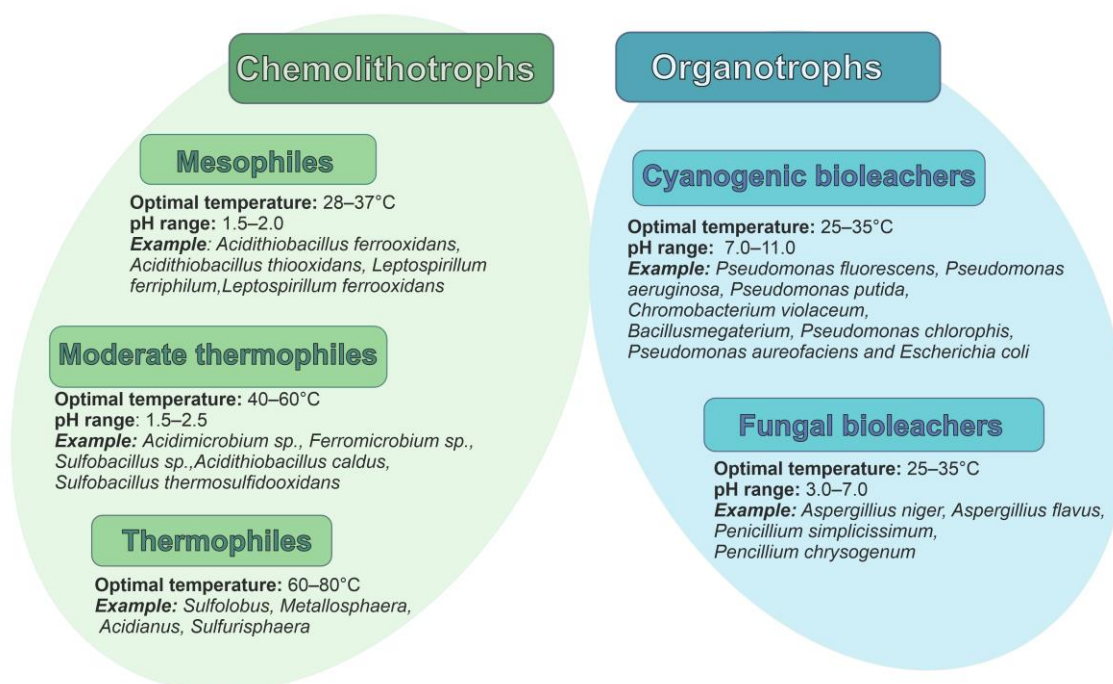


Figure 8. Microorganism types applied in bioleaching based on their metabolism requirements [79,93].

Organotrophs can be used in indirect bioleaching due to the production of organic acids (mostly by fungi) and hydrogen cyanide (by cyanogenic bioleachers, mostly bacteria). In comparison with bioleaching by chemolithotrophs, organotrophic bioleaching can occur in much higher pH—up to pH 11—and in lower temperature (approximately room temperature). This is an advantage for planning ISSA processing due to the alkalinity of its leachates. Optimal conditions for microorganisms' growth and activity can be obtained without adjusting the pH with additional chemical reagents.

Although bioleaching is rarely described as a method of treatment of ISSA, its applicability is being studied in different incinerated waste (Table 3).

Table 3. Examples of bioleaching implantation for incinerated waste materials.

No	Microorganisms Employed	Waste Type	Microorganism Type *	Reference
1	<i>Leptospirillum ferriphilum</i> , <i>Ferroplasma thermophilum</i> , <i>Sulfobacillus thermosulfidooxidans</i>	plant incineration ash (from soil remediation)	A B	[94]
2	Mixed culture <i>Marinobacter</i> sp., <i>Acidithiobacillus</i> , <i>Leptospirillum</i> , <i>Cuniculiplasma</i> , <i>Nitrosotenus</i> , and <i>Ferroplasma</i>	municipal solid waste incineration bottom ash	A B	[95]
3	<i>Acidithiobacillus ferrooxidans</i>	coal fly ash	B	[96]

Table 3. Cont.

No	Microorganisms Employed	Waste Type	Microorganism Type *	Reference
4	<i>Acidithiobacillus ferrooxidans</i> , <i>Acidithiobacillus thiooxidans</i> , and <i>Leptospirillum ferrooxidans</i>	ashes and slags from municipal waste incineration	B	[97]
5	<i>Bacillus cereus</i> and <i>Bacillus subtilis</i>	municipal and industrial wastes, incineration plants, power plant	B	[81]
6	Mixed culture of acidophilic bacteria	municipal solid waste incineration fly ash	B	[98]
7	<i>Pseudomonas</i> spp.	coal incineration fly and bottom ash	B	[98]
8	Sulfur oxidizing bacteria	municipal solid waste incineration fly ash	B	[99]
9	<i>Thiobacillus thiooxidans</i> and <i>Acidithiobacillus ferrooxidans</i>	municipal waste incineration fly ash	B	[100]
10	<i>A. ferrooxidans</i> and <i>A. thiooxidans</i>	sewage sludge incineration ash	B	[101]
11	<i>Aspergillus niger</i>	hospital waste incinerator bottom ash	F	[102]
12	<i>Aspergillus niger</i>	dry discharged incineration slag	F	[103]
13	<i>Aspergillus niger</i>	power plant residual ash	F	[104]
14	<i>Aspergillus niger</i>	fly ash from municipal solid waste incineration	F	[105]
15	<i>Aspergillus niger</i>	municipal solid waste incineration fly ash	F	[106]
16	<i>Aspergillus niger</i>	municipal solid waste incineration fly ash	F	[107]
17	<i>Aspergillus niger</i>	municipal solid waste incineration fly ash	F	[108]
18	<i>Fusarium oxysporum</i>	fly ash from thermal power plan	F	[109]
19	<i>Candida bombicola</i> , <i>Phanerochaete chrysosporium</i> and <i>Cryptococcus curvatus</i>	coal fly ash	F Y	[110]

Table 3. Cont.

No	Microorganisms Employed	Waste Type	Microorganism Type *	Reference
20	Autochthonous extremophiles	solid waste incineration fly ash	M	[111]
21	Indigenous community	fresh and aged bottom ashes from municipal solid waste incineration	M	[112]
22	Mixed culture of iron- and sulphur-oxidising microorganisms	fly and bottom ashes from municipal solid waste incineration	M	[113]

* A—archaea, B—bacteria, Y—yeast, M—mixed culture.

Although there is an abundance in microorganisms to be tested and in turn employed for bioleaching and other biotreatments, there is a visible reoccurrence of the same species in use (Table 3).

3.2. Phosphorus Recovery from ISSA

One of the most important perspective resources occurring in ISSA is phosphorus. Phosphorus-bearing minerals in ISSA are characterized by low solubility in water, therefore stronger agents and developed approaches are needed to release it from the phases. Phosphorus is mostly present in ISSA as calcium, aluminum, and iron phosphates [32]. For the acidic dissolution of the phosphates, not only does a sufficient amount of acid need to be provided, but due to the heavy presence of other acidic soluble compounds, an even higher acid concentration is also required. As the process is not selective, the final product—the leachate—contains a substantial concentration of dissolved metals. Additionally to bioleaching microorganisms, phosphate-accumulating bacteria might be involved in the phosphorus recovery procedure. Biotreatment by microorganisms can both induce bioleaching and recovery of phosphorus via biosorption or bioaccumulation and is often approached as a two-step process. After bioleaching, phosphates can be fixated in biomass by polyphosphate (poly-P)-accumulating bacteria. In this case, phosphate uptake and storage results in the formation of intracellular polyphosphates. This approach leads to the separation of the phosphorus from—often toxic—metals. In the first step, phosphates, along with other compounds, are dissolved by bioleaching, and in the second step bioaccumulation of phosphorus is taking place [114]. This mechanism has been employed for sewage sludge treatment since the year 2000 and is efficient in the recovery of biologically available phosphorus forms. Chemolithotrophs are capable not only of releasing metals from the solid phase to the solution but also to the phosphate ions by noncontact bioleaching [72].

Apart from phosphate accumulating, there are phosphate solubilizing microbes, which can be applied equally well. They might be efficient for P recovery from secondary sources of phosphorus as there are highly adaptable and efficient in providing P in soils—relatively less abundant with this element than ISSA. Those microorganisms are fairly abundant in soils (mostly rhizosphere) and responsible for the phosphorus supply of plants. They can also be isolated from phosphorus ores. The most common species among them are bacteria—*Pseudomonas* and *Bacillus*—and fungi—*Aspergillus* and *Penicillium* [115,116]. Tree microbial P solubilization mechanisms can be distinguished as follows:

- the release of mineral dissolving and complexing compounds such as siderophores, protons, or organic acid or hydroxyl ions,
- the biological P mineralization by liberation of extracellular enzymes,
- the biological mineralization (substance degradation) [117].

Phosphorus to be used as fertilizer must not only be separated from the harmful metals and pathogens but also must be transformed into bioavailable and stable forms. Applied biotreatment should therefore also be a solution for this challenge [118].

3.3. Efficiency of Microbiological Element Recovery

There are little to no published studies regarding a microbiological approach to ISSA treatment. Only a few available ISSA bioleaching and bioaccumulation efficiency studies are promising. Depending on the initial waste composition, targeted elements, procedure parameters such as time, liquid-to-solid ratio, and addition of substrate, and on the employed microorganisms, the efficiency varies.

3.3.1. Phosphorus Recovery

Maximum described releasing rates for P with *Acidithiobacillus* sp. going up to 93% P, which is higher than by chemical bioleaching performed as a part of the same study. The study was combined with simultaneous separation of released phosphorus by P-accumulating bacteria. The final form of recovered P by this microbiological approach was in the form of orthophosphate [101]. Other experiments were conducted with sulfur-oxidizing bacteria isolated and enriched from waste activated sludge. Optimisation of bioleaching parameters was performed in terms of duration, inoculum, and substrate amounts, and in the most efficient setting, the rate of P dissolution noted was 76% [119].

3.3.2. Metals Recovery

The studies from Section 3.3.1 also reported on metal recovery rates from ISSA. For the first one, *A. ferrooxidans* and *A. thiooxidans* mixed culture was applied and the maximum metal extraction yields were as follow: Fe-16%, Al-61%, Cu-41%, Zn-20%, Cr-13%, and Co-34%. For *Acidithiobacillus* sp. from digested sewage sludge from wastewater, metal recovery was comparable: Fe-13%, Al-55%, Cu-31%, Zn-45%, Cr-13%, and Co-34% [101]. In the second study employing sulfur-oxidizing bacteria isolated and enriched from waste activated sludge, the recovery rate was significantly higher: Ca-19.6%, Mg-70%, Fe-78.8%, Al-61.75%, Cu-98%, Zn-30.8%, and Ni-66% [119].

3.4. Challenges in ISSA Biotreatment

Bioleaching is a low-cost alternative or more often a process employed in addition to chemical methods. The efficiency of bioleaching is limited by the highly alkaline environment created by the dissolving material. Many among the microorganisms have proven to be efficient in bioleaching, as they thrive in low pH [120], but maintaining a low pH with highly alkaline ISSA can be impossible when relying solely on acid production by the microorganisms. The traditional bioleaching results employing *Acidithiobacillus thiooxidans* are far from being satisfactory, with a recovery efficiency up to 50%. Apart from adjusting the pH by acid addition, this issue can be resolved by adding a substrate, for example, elementary sulfur or another nutrient suitable for the microorganisms used. It is worth keeping in mind that many of the bioleaching methods explored for secondary resources originate in bio-hydrometallurgical solutions designed and explored for mining tailings, which are often acidic or extremely acidic conditions. The ISSA as well as other alkaline waste present the opportunity for new solutions to be explored and invented, among them being alkaline bioleaching with autochthonous extremophiles [111].

3.5. Sewage Sludge Biotreatment

Bioleaching, although rarely applied to ISSA, is a widely and long used and examined practice in the case of sewage sludge [121]. The microorganisms naturally occurring in the sludge are most commonly used for this purpose. Microorganisms take part both in dissolution of the metal containing compounds, and acidification of the environment. Sewage sludge has proven to be a valuable fertilizer, however, due to its high concentration of toxic elements, it cannot be applied directly to soil. Biotreatment leads to both safer storage and

processing and an increase in sludge applicability. Apart from microorganisms useful for biotreatment, there are often human pathogenic bacteria, viruses, protozoa, and helminths present, leading to epidemiological risk [122,123]. Apart from chemically neutralizing the sewage sludge, bioleaching is also efficient in the elimination of pathogens [124]. An important part of sludge treatment before incineration in numerous facilities is the process of enhanced biological phosphorus removal (EBPR). The very existence of bioreactors and the experience with microorganisms' application in sewage treatment industry create a solid ground for expansion toward the application of biotreatment techniques to ISSA.

4. Conclusions

Many aspects can influence leaching behaviour and elements' leaching efficiency. An individual approach is mandatory, since ISSA may differ in its final composition and in multiple factors such as incineration technology, locality (differences in sludge composition in industrial and rural areas), waste management system, level of development of a region, and even seasonal changes. The bulk chemical composition and mineralogy of ISSA influences its solubility and thus leaching efficacy as well as optimum leaching parameters such as L/S ratio, pH of the leachates, and contact time that might differ for individual ISSAs. There is a wide scope of well-established methods for the biotreatment of incinerated and other waste, however, these methods are rarely applied for ISSA. The solutions based on biotreatment—although more complex—can be very effective and allow to overcome chemical extraction shortcomings such as excessive production of effluents from the reactions, or to cut its costs. As described in the presented research, the experience and knowledge from other branches such as municipal or electronic waste biotreatment can be potentially adapted for ISSA treatment. Good understanding of mechanisms and optimisation of conditions needed for effective bioleaching of metals and phosphorus and bioaccumulation is the gateway for the development of new solutions.

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Glossary

Abbreviations

APC	Air pollution control residue
BCR	Community Bureau of Reference (BCR, now Standards, Measurements and Testing Program)
EU	European Union
FA	Fly ash
ISSA	Incinerated sewage sludge ash
PM10	Particulate Matter 10
ppm	Parts Per Million

Elements and compounds

AOX	adsorbable organohalogens
Al	Aluminum
As	Arsenic
C _{org}	Organic carbon
Ca(OH) ₂	Calcium hydroxide
Cd	Cadmium
CH ₂ O ₂	Formic acid
C ₂ H ₄ O ₂	Acetic acid
C ₂ H ₆ O ₆	Ascorbic acid
C ₆ H ₈ O ₇	Citric acid
C ₆ H ₁₂ O ₇	Fructose
Co	Cobalt
Cr	Chromium
Cu	Copper
EDTA	Ethylenediaminetetraacetic acid
EDTMP	Ethylene diamine tetramethylene phosphonate
H ₂ SO ₄	Sulfuric acid
H ₃ PO ₄	Phosphoric acid
HAc	Acetic acid
HCl	Hydrochloric acid
HF	Hydrofluoric acid
Hg	Mercury
HNO ₃	Nitric acid
H ₂ O ₂	Hydrogen peroxide
IC	Inorganic carbon
MgCl ₂	Magnesium chloride
Mn	Manganese
N	Nitrogen
NaAc-HAc	Sodium acetate/acetic acid buffer
NaHCO ₃	Sodium bicarbonate
NH ₂ -OH-HCl	Hydroxylamine hydrochloride
NH ₃	Ammonia
Ni	Nickel
NO _x	Nitrogen oxides
NTA	Nitrilotriacetic acid
P	Phosphorus
PAH	Polycyclic aromatic hydrocarbons
Pb	Lead
PCB	Polychlorinated biphenyls
Sb	Antimony
SO ₂	Sulfur dioxide
Ta	Tantalum
Zn	Zinc
V	Vanadium
W	Tungsten

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