

Assessment of leaching properties of solid residue from subcritical water extraction of *Zingiber zerumbet* rhizome

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Abstract. Landfilling is known as a popular way to get rid of municipal waste because it is easy to use and reasonably priced, but it also leads to environmental issues like leachate pollution. Thus, solid residues from *Zingiber zerumbet* rhizome subcritical water extraction (SWE) were assessed in this study to see if they met the criteria for municipal waste that could be disposed of safely in a landfill. The assessment comprises of four crucial properties; ignitability, corrosivity, reactivity, and toxicity and was conducted on a sample taken during a 15-minute SWE following the United States Environmental Protection Agency (USEPA) and Malaysian Department of Environment (DOE) criteria. The residue was found to be non-ignitable (flash point above 200°C), non-corrosive (pH of 6.65 ± 0.02), and non-reactive (no visible toxic gases, vapors, or fumes) in normal environmental settings. Meanwhile, in accordance with the Toxicity Characteristics Leaching Procedure (TCLP), the toxicity analysis results were considered at their maximum limits. Heavy metals such as barium showed the highest detectable concentration at

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< 0.8 mg/L, while cadmium followed at < 0.6 mg/mL, but both remained below the limits of ≤ 100.0 mg/L and ≤ 1.0 mg/L, respectively. The volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs) and herbicides detected in the sample were at remarkably low concentrations below the legal limit. Overall, these results provide convincing evidence that the solid residues of the rhizome of *Z. zerumbet* from the SWE can be categorised as municipal solid waste. Pre-treatment prior to landfill is therefore unnecessary. SWE promotes ecological waste management practises thus contributing to a safer and more sustainable future for all.

1 Introduction

In recent years, there has been increasing interest in pollution reduction measures, and this includes careful consideration of the environmental impacts and regulatory requirements at the design stage of chemical processes. Improper disposal of waste generated by industrial-scale agricultural, or crop extraction processes can lead to harmful consequences for the environment if the associated hazards are not adequately assessed. Industrial processes that use organic or inorganic solvents for extraction often generate solid residues containing hazardous compounds that can be flammable, corrosive, reactive or toxic to the environment. It is therefore vital to check the waste thoroughly before disposal.

There are various methods of waste disposal and management including incineration, landfill, recycling, reuse, pyrolysis, resource recovery, and composting [1]. As landfills are currently the most affordable, flexible, and easy way to dispose of waste, they are the preferred option for waste management [2]. Due to these, there are worries about possible environmental effects [3]. Contaminated leachate may seep directly into adjacent soil and water bodies, endangering both human health and the ecosystem [4]. Consequently, to meet the necessary standards prior to landfilling, the waste material must be properly evaluated or treated.

The Malaysia Environmental Quality (Scheduled Waste) Regulations 2005, Regulation 7(1), allows waste producers to request that their waste not to be treated, disposed of, or recovered at specific facilities [5]. But for these exclusions to be approved, the waste cannot have any dangerous characteristics like toxicity, ignitability, corrosivity, or reactivity. And this must be established through scientific research or testing that the waste has no harmful effects on people or other living things, is not carcinogenic, and is not toxic [6].

The United States Environmental Protection Agency (USEPA) initially developed a set of specific tests to evaluate the characteristics of waste materials. A number of nations including Malaysia, now had been using these tests as a standard procedure. Malaysia's Department of Environment (DOE) uses it extensively and is well-known for identifying materials that corrode at a specific acidic or alkaline pH, release harmful gases or vapors when ignited, or seep into groundwater at dangerous concentrations of known toxic substances.

The Toxicity Characteristics Leaching Procedure (TCLP) analyzes the mobility of organic and inorganic pollutants in various waste types, including liquid, solid, and multiphase materials, thus simulating the leaching of pollutants to determine the toxicity of

solid residues [7]. If waste contains organic compounds or specific metals that are more than allowed limits that can be inhaled, eaten, or otherwise harmed human health, it is categorized as hazardous waste. In the event that the leaching test reveals the extract has levels of heavy metals or other organic compounds above the permitted limits, the waste is classified as toxic [8].

Rhizome of *Zingiber zerumbet* (known as lempoyang in Malaysia) in this study is a medicinal plant famous for its anti-inflammatory and antioxidant properties. It was subjected to subcritical water extraction (SWE) where SWE is a green extraction technique which used water at high temperatures (100 to 374°C) and pressures (1 to 221 bar) to extract bioactive compounds from plant materials. During the SWE process, water is deliberately pressurised to keep it in a liquid state. This is achieved by applying pressure while ensuring that the temperature remains below the critical point [9].

The SWE process of plant material resulted in solid residues that were normally disposed of as municipal waste in landfills without prior treatment. However, prior to disposal, a comprehensive waste assessment is required to determine the hazardous nature of these solid residues. The analysis of solid residues is crucial for understanding their ecological risks, in providing valuable information on their behaviour and impact on the environment, thus improving the overall assessment of the waste management process.

2 Materials and Methods

Fresh rhizomes of *Z. zerumbet* were obtained from Naturemedic Laboratories Sdn Bhd, Chendering, Terengganu, and authenticated by the herbarium of Universiti Sultan Zainal Abidin with a voucher specimen labelled UniSZA/A/000000006. After cleaning and slicing, the rhizomes were subjected to an industrial-scale SWE process in a 70-litre stainless steel reactor from Natxtract, AM Zaideen Ventures Sdn Bhd, Malaysia, as shown in Figure 1. The resulting extract was frozen for subsequent quantitative and qualitative analysis, while the solid residues were collected for properties evaluation.



Figure 1. (a) *Z. zerumbet* rhizome (b) Subcritical water extraction.

Figure 2 outlines the procedure for analysing the properties of ground solid residues. The procedure begins with the preparation of the ground solid residues, followed by four key analyses specified by the USEPA and the DOE. The ground solid residues from the 15-minute SWE time serve as a representative sample of the broader range of residues produced at different extraction times. Ignitability was analysed using the American Society for Testing and Materials (ASTM) method. The analyses for corrosivity, reactivity, and toxicity were carried out according to the methods of the USEPA and the American Public Health Association (APHA). Each analysis is explained in more detail in the respective subsections.

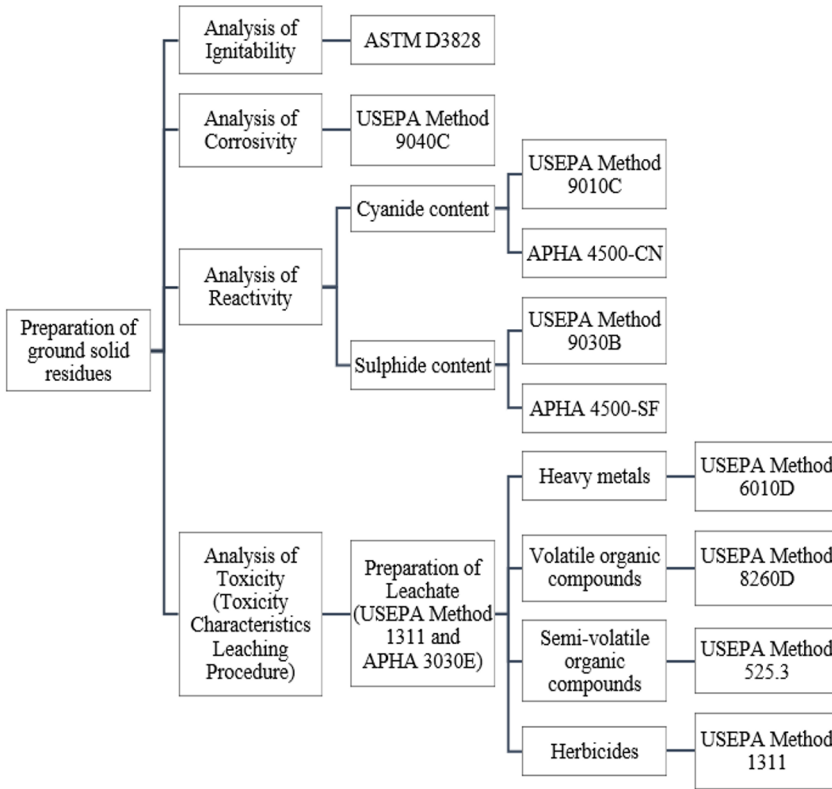


Figure 2. Flow chart of solid residues properties analysis.

2.1 Preparation of Ground Solid Residue

The solid residues from the 15-minute SWE (see Figure 3(a)) were taken and ground in a food processor. A small amount of reverse osmosis water was added as a solvent to convert the waste into a slurry-like form. The ground residue was sieved using a 2.0 mm sieve to keep the resulting sample as small as possible, as shown in Figure 3(b). The sample was then analysed for ignitability, corrosivity, reactivity, and toxicity.

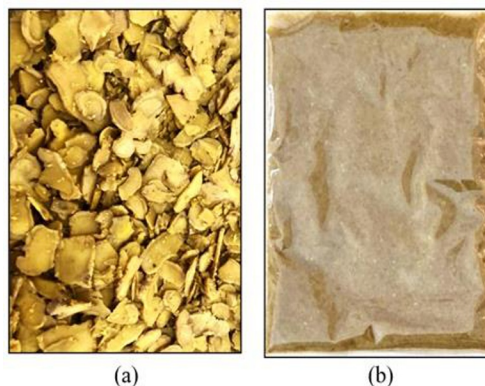


Figure 3. (a) Solid residue of *Z. zerumbet* rhizome (b) Ground solid residue with water as a sample.

2.2 Analysis of Ignitability

Ignitability was analysed using a Setaflash Closed Cup Tester according to ASTM D3828 standard [10]. The purpose of this assessment was to determine the flash point of the sample, i.e., the lowest temperature at which the sample could potentially ignite or catch fire when exposed to an open flame. The ignitability was determined when the sample shows a flash at a temperature below 60 °C, which is characterised by a sudden appearance of a short flame or spark [6]. If the determined flash point is above the specified minimum temperature, the sample is considered "non-ignitable" under the test conditions. The analysis began by carefully placing approximately 4 mg of the sample into the sample cup or vessel of the apparatus. The temperature of the sample was gradually increased at a controlled rate of 1 °C per minute. A flame was then introduced into the vapour space above the sample. Careful attention was paid to the presence of a flash, indicated a small flame or spark that appears out of the blue. The flash point of the sample was marked by a flame which immediately spread over its surface, and the flash point temperature was recorded. The procedure was carried out in duplicate.

2.3 Analysis of Corrosivity

Corrosivity of the sample was analysed using the standard test for electrometric pH measurement described in USEPA Method 9040 [11]. If the sample has an aqueous component and has a pH less than or equal to 2 ($\text{pH} \leq 2$) or greater than or equal to 12.5 ($\text{pH} \geq 12.5$), the sample is categorised as corrosive [6]. The pH analysis was performed using a pH metre with glass electrodes (Eutech™ PC 700 Multiparameter Metre, Thermo Scientific™). The analysis was performed at room temperature. The measurements were completed in triplicate and the results were expressed as mean values.

2.4 Analysis of Reactivity

Reactivity of the sample was analysed by measuring the cyanide reactivity using reflux distillation under acidic conditions and absorption in a scrubber with hydroxide solution. Total cyanide after distillation was then determined using the selective electrode method as described in USEPA Method 9010C [11] and APHA 4500-CN [12]. On the other hand, sulphide reactivity was determined according to USEPA Method 9030B [13] and APHA 4500-SF [14]. The presence of sulphide in the sample was confirmed by a distillation procedure, and reactivity was assessed qualitatively using a lead acetate paper and silver foil test. According to these qualitative test specifications, the result was classified as "reactive," meaning that cyanide or sulphide was present or not. Any type of specifications which can react violently, explode readily, or release harmful gases or vapours when they react are often contained residues in solid.

2.5 Analysis of Toxicity

Heavy metals, volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), and herbicides were among the chemical compounds whose presence was examined using the TCLP test. Before the sample was put through the TCLP test, it underwent a conversion procedure that turned it into leachate. All TCLP assessments were carried out twice to guarantee the accuracy of the results, and the maximum limits were reported as the outcome. The methods used in the toxicity assessment are explained in more detail in the following subsection.

2.5.1 Preparation of Leachate.

The leaching solution was prepared according to the guidelines of USEPA Method 1311 [15] and APHA 3030E [16]. In the fume hood, the standard leaching solution was prepared by combining 0.1 M acetic acid and 0.07 M NaOH extraction solution, and the pH was cautiously altered to 4.93 ± 0.05 . The prepared leaching solution was then mixed with the sample (Figure 2(b)) at a ratio of 20:1. Leaching was carried out over a period of 18 ± 2 hours at room temperature under continuous rotation at a speed of 30 ± 2 rpm in a rotary apparatus. The supernatant was then filtered through a glass fibre filter with a pore size of $0.7 \mu\text{m}$. The pH of the filtrate was determined and the leachate was acidified with nitric acid to a pH of less than 2 before the contaminants were measured [7].

2.5.2 Toxicity Characteristics Leaching Procedure Test.

Concentration of seven heavy metals in the sample was quantified using inductively coupled plasma optical emission spectrometry (ICP-OES) obeying the process explained in USEPA Method 6010D [17]. However, the presence of mercury (Hg) was specifically quantified using atomic absorption spectrometry (AAS) according to method APHA 3112B [18]. In addition, USEPA Method 8260D [19] was used for the determination of 10 VOCs. An Agilent Technologies 6890N GC and MS 5973, equipped with a DB-VRX capillary column ($20 \text{ m} \times 0.18 \text{ mm} \times 1 \mu\text{m}$) was used. The oven temperature was held at $40 \text{ }^\circ\text{C}$ for 3 minutes and then rapidly increased to $100 \text{ }^\circ\text{C}/\text{min}$ without holding time. The temperature was then further increased to $220 \text{ }^\circ\text{C}$ at a rate of $25 \text{ }^\circ\text{C}/\text{min}$ and held constant for 3 minutes. The sample was injected in split mode and helium was used as carrier gas. After obtaining the chromatogram, the mass spectrum of the unknown compounds was identified.

Concentration of 20 SVOCs was analysed using USEPA Method 525.3 [20]. A gas chromatography-mass spectrometry (GCMS) setup with a sample injection of $10 \mu\text{L}$ was used. The instrument used was an Agilent Technologies 6890N gas chromatogram coupled with mass spectrometry 5973 and a DB-5ms capillary column ($30 \text{ m} \times 0.25 \text{ mm} \times 0.25 \mu\text{m}$) was used to separate the compounds. The temperature was held at $70 \text{ }^\circ\text{C}$ for 1 minute and 50 seconds and then increased at a rate of $10 \text{ }^\circ\text{C}/\text{min}$ and held for 1 minute. The temperature was then increased to $280 \text{ }^\circ\text{C}$ at a rate of $7 \text{ }^\circ\text{C}/\text{min}$ and held constant for 10 minutes. The sample was too injected in splitless mode and helium was utilized once more as the carrier gas. After obtaining the chromatogram, the mass spectrum of the unknown compounds was identified.

Finally, USEPA Method 1311 [15] was used to analyse two herbicide compounds including 2,4-dichlorophenoxy acetic acid and 2,4,5- TP (Silvex). $10 \mu\text{L}$ of the sample was injected into a high-performance liquid chromatography (HPLC) system. The HPLC system, an Agilent Technologies 1200 series, consisted of a degasser, a quaternary pump, an autosampler, a UV detector and the LC1200 software. A Merck column ($150 \text{ mm} \times 4.6 \text{ mm} \times 5 \mu\text{m}$) was used to separate the compounds at $25 \text{ }^\circ\text{C}$ with a gradient elution programme at a flow rate of $1 \text{ mL}/\text{min}$. The mobile phase was a mixture of acetonitrile (ACN) (60%) and phosphate buffer solution (40%) (pH 7.0). The UV spectrum was recorded at a wavelength of 225 nm .

3 Result and Discussion

3.1 Ignitability, Corrosivity, and Reactivity Analysis of Solid Residues

Table 1 provides valuable information on three important properties of the sample: ignitability, corrosivity, and reactivity. The results of both test series consistently showed ignitability temperature of more than 200°C, which is above the legal standard. The results therefore confirmed that the sample had no ignitable properties and was not flammable or combustible under normal ambient conditions. Consequently, the pH of the sample was determined to be 6.65 ± 0.02 and was classified as non-corrosive as it was within the legal standard. The pH was only slightly acidic and relatively close to a neutral pH of 7, which reduces the likelihood of significant damage to metals and other materials. In addition, no toxic gases, vapours, or fumes were visible during the experiment. Therefore, the reactivity of the sample was classified as non-reactive, meaning that the likelihood of harmful reactions occurring, or gases being released is low.

Table 1. Assessment of ignitability, corrosivity, and reactivity.

Assessment	Test Parameter	Unit	Legal Standard	Result	Remark
Ignitability	Flash point	°C	> 60°C	> 200°C	Not ignitable
Corrosivity	pH	-	$2.0 < \text{pH} < 12.5$	6.65 ± 0.02	Non-corrosive
Reactivity	Cyanide and sulphide	mg/kg	Invisible of toxic gases, vapour or fumes when exposed to a pH 2.0 to 11.5	Invisible of toxic gas, vapour, or fumes	Non-reactive

In management of waste's perspective, solid waste is classified as hazardous by ignitability if it spontaneously ignites below 60°C and burns with a certain intensity under normal handling conditions. The waste is also classified as corrosive if it can corrode or dissolve metal, metals, or other materials due to its acidic or alkaline nature. They are also classified as reactive if they exhibit explosive behaviour under conditions of heat, pressure, exposure to water or typical handling. This leads to violent reactions and the formation of toxic vapours, gases or explosive mixtures [21,22]. With all these criteria, the waste is categorised as scheduled waste (not municipal waste). It is therefore imperative that the waste is treated and disposed of in accordance with certain regulatory guidelines to avoid environmental and health risks.

In this study, however, the sample met the criteria for municipal waste as it was non-ignitable, non-corrosive and non-reactive. The sample was categorised as non-hazardous and did not require treatment prior to landfilling due to its lower potential for environmental impact [23]. At the same time, the potential fire risk associated with handling, storing, and disposing of the sample in the landfill itself was also reduced. It was also less likely that the sample would cause significant greenhouse gas emissions through biochemical interactions [24]. Nevertheless, it is important to note that the decomposition of the sample in the landfill may generate heat that could lead to spontaneous combustion [25]. Harmful or hazardous substances would be released when exposed to the specified pH conditions. Therefore, the sample should remain safe and stable throughout its decomposition process.

3.2 Toxicity Analysis of Residue by TCLP

Table 2 shows the leachability of the compounds for the sample tested with the TCLP method. The results reveal that the total concentrations of the heavy metals were far below the legal limits (mg/L). For example, arsenic, cadmium, lead, mercury, selenium, and silver were detected in concentrations below the limits, indicating minimal contamination. The

highest concentration observed was barium at < 0.8 mg/L, followed by cadmium at < 0.6 mg/L, which was also well below the limit. In general, the presence of barium in acidic leachate from landfills is of concern and further precautions are needed for safe landfill practises [26]. However, its mobility is low under neutral or basic pH conditions (sample pH = 6.65 ± 0.02 , close to neutral), which would reduce the risk of leaching and potential health effects [27].

In addition, all VOCs in the sample such as benzene, chlorobenzene, 1,4-dichlorobenzene, 1,2-dichloroethane and 1,1-dichloroethane were detected at concentrations of less than 0.01 mg/L. The results also showed that all SVOCs detected in the sample were present at remarkably low concentrations. Both herbicides in the leachate; 2,4-dichlorophenoxy acetic acid and 2,4,5-TP (Silvex) were < 0.5 mg/mL, below the legal limit of ≤ 10.0 and ≤ 10.0 mg/L, respectively. Overall, the TCLP test results of the study proved that the concentration of all 40 contaminants present in the sample was well below the legal limit. This indicates that the sample were not hazardous and showed only minimal contamination.

Table 2. Leachability of solid residues by TCLP test.

DOE CW No. 1	CAS No. 2	Contaminant	Limit (mg/L)	Result (mg/L)
Heavy metal				
C004	7440-38-2	Arsenic	≤ 5.0	< 0.001
C005	7440-39-3	Barium	≤ 100.0	< 0.8
C006	7440-43-9	Cadmium	≤ 1.0	< 0.6
C007	7440-47-3	Chromium	≤ 5.0	< 0.03
C008	7439-92-1	Lead	≤ 5.0	< 0.007
C009	7439-97-6	Mercury	≤ 0.2	< 0.02
C010	7782-49-2	Selenium	≤ 1.0	< 0.007
C011	7440-22-4	Silver	≤ 5.0	< 0.005
Volatile organic compound				
C018	71-43-2	Benzene	≤ 0.5	< 0.01
C019	56-23-5	Carbon tetrachloride	≤ 0.5	< 0.002
C021	108-90-7	Chlorobenzene	≤ 100.0	< 0.01
C022	67-66-3	Chloroform	≤ 6.0	< 0.003
C027	106-46-7	1,4-dichlorobenzene	≤ 7.5	< 0.01
C028	107-06-2	1,2-dichloroethane	≤ 0.5	< 0.01
C029	75-35-4	1,1-dichloroethane	≤ 0.7	< 0.01
C039	127-18-4	Tetrachloroethene	≤ 0.5	< 0.001
C040	79-01-6	Trichloroethene	≤ 0.5	< 0.0001
C043	75-01-4	Vinyl chloride	≤ 0.2	< 0.00001
Semi-volatile organic compounds				
C012	72-20-8	Endrin	≤ 0.02	< 0.00002
C013	58-89-9	Lindane	≤ 0.4	< 0.00002
C014	72-43-5	Methoxychlor	≤ 10.0	< 0.00002
C015	8001-35-2	Toxaphene	≤ 0.5	< 0.0001
C020	57-74-9	Chlordane	≤ 0.03	< 0.00002
C023	95-48-7	o-cresol	≤ 200.03	< 0.0001
C024	108-39-4	m-cresol	≤ 200.03	< 0.0001
C025	106-44-5	p-cresol	≤ 200.03	< 0.0001

DOE CW No. 1	CAS No. 2	Contaminant	Limit (mg/L)	Result (mg/L)
C026	-	Cresol	≤ 200.03	< 0.0001
C030	121-14-2	2,4-dinitrotoluene	≤ 0.13	< 0.0001
C031	76-44-8	Heptachlor (and its oxide)	≤ 0.008	< 0.00002
C032	118-74-1	Hexachlorobenzene	≤ 0.13	< 0.00001
C033	87-68-3	Hexachlorobutadiene	≤ 0.5	< 0.0001
C034	67-72-1	Hexachloroethane	≤ 3.0	< 0.0001
C035	78-93-3	Methyl ethyl ketone	≤ 200	< 0.01
C036	98-95-3	Nitrobenzene	≤ 2.0	< 0.0001
C037	87-86-5	Pentachlorophenol	≤ 100.0	< 0.0001
C038	110-86-1	Pyridine	≤ 5.0	< 0.0001
C041	95-95-4	2,4,5-trichlorophenol	≤ 400.0	< 0.0001
C042	88-06-2	2,4,6-trichlorophenol	≤ 2.0	< 0.001
Herbicides				
CO16	94-75-7	2,4-dichlorophenoxy acetic acid	≤ 10.0	< 0.5
CO17	93-72-1	2,4,5- TP (Silvex)	≤ 1.0	< 0.5

¹Department of Environment characteristic waste number.

²Chemical abstracts service number.

'<' indicate the analyte was not observed above this limit

Creating a single, straightforward test that encompasses all potential scenarios of solid residue properties is challenging. Taking into account several tests for various scenarios can also be difficult [28]. Therefore, rather than measuring the amount leached, a practical method like TCLP is used to quantify the total concentration of contaminants. When the TCLP test is performed on a solid residue, a liquid called leachate is produced. This leachate resembles the fluid that would organically occur in the ground close to a landfill that produces the same kind of waste [21]. Numerous factors, including acidity, alkalinity, additives, redox characteristics, and landfill climate, affect the leaching behavior [29,30].

Next comes the testing phase, where each producer of solid residue is required to determine whether the leachate exceeds the specified limits in any of the 40 listed toxic chemicals. The residue is deemed toxicologically relevant (TC) if the leachate sample has an adequate concentration of any of these substances. The residue needs to be reviewed by the regulatory body and suitable treatment and disposal plans should be created if the overall concentration is higher than the designated limits. The USEPA established these concentration thresholds by utilizing groundwater modeling studies and toxicity data for several common toxic compounds and elements. Many of these thresholds were originally developed under the USEPA Safe Drinking Water Act (SDWA).

Comparing the TCLP results of contaminant concentrations in waste materials from mining and industrial processes, such as plant extraction, offers important information about how the contaminants leach. To evaluate their possible environmental impact, this information is essential. This comparison must consider the various types of contaminants that may be present, as they can differ greatly. Wastes from plant or agricultural extraction may contain natural materials, pesticides, or organic compounds [31]. Meanwhile, heavy metals, metalloids, and other inorganic pollutants are frequently found in mining wastes [8]. For instance, minimal bioavailable metal concentrations were discovered in *Ageratum conyzoides*'s waste (also referred to as rumput tali ayam in Malaysia), suggesting a decreased environmental risk [31]. In contrast, analysis of waste from copper mines revealed

significantly higher TCLP concentrations of aluminium, copper, cadmium, nickel, selenium, and zinc which were above the legal limits and classified as hazardous substances [32].

When the rhizome of *Z. zerumbet* is used as plant material, it is less likely to release harmful pollutants after the SWE process. Numerous extraction studies have been conducted using various methods showing that the rhizome of *Z. zerumbet* has no toxic effects on tested animals or cells. Instead, these studies emphasise its pharmaceutical benefits [33,34]. This inherent property means that only low levels of harmful substances are detected in the solid residues during testing. The results support its categorisation as municipal waste and proves that it does not need to be pre-treated prior to landfill.

Furthermore, this study has also proven that SWE is an environmentally friendly extraction method that allows safe extraction of valuable components from the plant material while minimising the generation of lethal waste. A notable aspect is the use of water as a solvent, which helps to ensure that the resulting residue is harmless [35,36] and offers distinct advantages for the extraction of herbs, vegetables and fruits [37]. In addition, the SWE process produces minimal liquid waste as a primary output, reducing the total amount of waste going to landfill.

4 Conclusion

The comprehensive evaluation of the solid residues from the rhizome of *Z. zerumbet* after SWE was successfully performed. The solid residues were found to be non-ignitable, non-corrosive, non-reactive, and non-toxic as all parameters tested were below the limits set by the USEPA and DOE. The natural composition of the plant reduced the probable risk of hazardous waste. The utility of water as a solvent during the SWE process ensured low pollution potential during disposal, contributing to a cleaner environment. The characteristics of the solid residues confirm their categorisation as municipal solid waste and eliminate the need for pre-treatment prior to landfill. This emphasises the environmental and compliance benefits of using subcritical water to process *Z. zerumbet*.

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