FORMULATION OF FISH FEED USING ESSENTIAL ELEMENTS FROM PLATINUM MINERAL TAILINGS AND GROUNDNUT OIL PROCESSING WASTE: A CIRCULAR ECONOMY INITIATIVE

by

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DECLARATION

I, Crispin Kudakwashe Mandimutsira , hereby declare that this dissertation is my work with the assistance of my supervisor, technicians, and fellow students. The report has not previously been submitted to any tertiary institution for any related degree. The undersigned, hereby certify that they have read and agree to submit this final version of the dissertation as the official submission.

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DEDICATION

This dissertation is dedicated to the memory of my late parents, who always supported and encouraged excellence in academics.

ABSTRACT

The high cost of imported fish feed ingredients in Zimbabwe has become a concern. Given that fish feed primarily consists of essential elements and proteins, this study explored the potential of platinum mineral tailings, an underutilized byproduct of ore flotation, as a source of essential elements for fish feed formulation. Platinum mineral tailings may cause negative environmental effects through acid drainage caused by uncontrolled leaching of elements. The platinum mineral tailings were characterized using X-ray fluorescence (XRF) and X-ray Diffraction (XRD) to determine their elemental and mineralogical composition, respectively. XRF revealed the presence of Fe, Ca, Si, Mg and Cr. XRD results concurred with the XRF analysis showing key mineral phases including taenite, pyroxene, landauite, enstatite ferro and kyanite.

To recover essential elements, comparative leaching studies were carried out using inorganic acids 0.5 M sulfuric, hydrochloric, and organic acids: acetic, and citric acids. Citric acid exhibited higher extraction efficiencies compared to acetic acid. A combination of 0.5 M acetic acid and 0.5 M citric acid was used in the ratio 1:1 to improve extraction efficiency. Optimization studies were carried out using response surface methodology to determine the effects of contact time, acid concentration, temperature and pH. The optimized conditions were pH = 2, contact time = 5 hrs, and a temperature of 70 °C. The resultant leachates were analyzed using Inductive Coupled Plasma- Optical Emission Spectroscopy (ICP-OES). ICP-OES showed percentage recoveries of Ca (42. 42 %) , Cu (0.97 %) , Fe (0.05 %), K (45.61 %), Mg (35.48 %) ,Mn (27.43 %) , Na (64.42 %) and Zn (16.62 %).

The leachates were then combined with groundnut oil processing waste. Groundnut oil processing waste was used as both an organic binder for the extracted elements, as well as a source of protein for the formulation. The resultant mixture was processed into pellets using optimized parameters of a volume of 25 ml of the mineral solution, 37.5 g of groundnut oil processing waste with 11% moisture content. The proximate and elemental analysis produced the nutritional content (%) as carbohydrates (56.6 ± 0.38) , protein (10.79 ± 0.12), lipids (15.91± 0.05) , ash (4.26 ± 0.10) , fiber (2.18 ± 0.04) and moisture (9.07 ± 0.05). Elemental concentrations (mg/kg) were, Ca (961.49 ± 0.46) , Cu (6.54 ± 0.069) , Fe (79.71 ± 0.29), K (5118.06 ± 1.4) , Mg (1090.10 ± 0.49), Mn (6.46 ± 0.061), Na (1183.07 ± 0.81) and Zn (0.296 ± 0.0029).

The results showed potential of using waste from from metallurgical processing of platinum mineral as a source of essential elements combined with waste of processing oil from a local available indigenous crop Arachis hypogaea (groundnut). These are cheap to obtain as they are local however cost arise from purification and analysis to ascertain safety and quality. This study validates the technical feasibility of extracting elements from platinum mining waste for use as nutritionally essential elements to enrich groundnut oil processing waste. Comprehensive analytical characterisation and formulation optimisation were carried out. The results demonstrated nutrient recovery efficiency, toxic element removal, providing the essential baseline data required for designing further research on safety assessments, in vivo feeding trials and economic feasibility assessments.This approach has potential to offer a sustainable, locally sourced alternative to conventional feed ingredients, contributing to waste valorization and economic resilience in Zimbabwean aquaculture.

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CHAPTER 1

INTRODUCTION TO THE STUDY

1.1 Background of the study

The global fish supply requires an increase in production by 2050 to meet rising food demand, driven by the rise in population and shifting dietary preferences (Habimana, 2024a). Fish enriches diet by providing nutrients which include omega-3 fatty acids, iron, zinc and high-quality proteins (Lupton and Cross, 1994). Fish consumption has been projected to decline due to production constraints (Obiero et al., 2019; Habimana, 2024b). These constraints are largely due to high cost and limited availability of essential raw materials required for feed formulation; raw materials like fish meal and fish oil derived from marine resources. Fish meal and mineral supplements are part of about 60 – 70% of fish production costs (Pangestika and Putra, 2020; Silva et al., 2021). These have been reported to lead to overfishing, ecosystem degradation and supply chain vulnerabilities, and fish meal prices have been on rapid increase since the early 2000s (Tacon et al., 2009; Velichkova et al., 2024). For the production of feed and mineral supplements, aquaculture depends on terrestrial mineral resources such as phosphorus rock. Phosphate rock is a scarce resource that is primarily found in a few countries (Pandit et al., 2023).

For sufficient growth in the shortest amount of time, fish need a diet that is high-quality and nutritionally balanced (Tacon et al., 2009). Most of farmers depend on imported feeds however some nations are seeing a rise in investments in domestic feed production in order to benefit from more affordable and readily available raw materials as well as improved accessibility for fish farmers (Bhosale et al., 2010; Ragasa et al., 2022).

1.1.2 Fishmeal dependency and fish feed raw material challenges

Fishmeal made from waste fish and wild fish is a common source of protein in fish feed formulations. The meals consisted of amino acids, minerals, vitamins and omega-3 fatty acids. There has been concern of exploitation of wild fish combined with the high price of fish meal (Yohana et al., 2023). Some other common fish feeds are formulated with fishmeal combined with plant sources, but, in some instances, may not provide adequate levels of certain essential elements. Some plants may contain antinutritional factors that may limit availability of minerals for fish absorption (Bhosale et al., 2010). This leads to some fish producers utilizing supplements with essential elements to further enrich the dietary requirements of fish (Manyala et al., 2015).

1.1.3 Essential elements in fish diets

Essential elements are important for the various physiological make-up of fish they are critical for processes such as enzymatic functions and formation of vital components such as bones. These processes require for instance Ca, P and Mg. Deficiencies in some of these elements can cause physiological disorders and reduce fish production (Craig and Helfrich, 2002; Lall and Dumas, 2015). Then also for pigmentation, oxygen transport, and immunity, fish require Cu, Fe and Zn respectively (Lall and Kaushik, 2021a) Cu is mainly sourced from copper sulfate, fish meal, and some plant proteins. Fe is sourced from ferrous sulfate, ferric citrate, fish meal, and blood meal. Zinc commonly provided as zinc oxide, fish meal, and plant proteins (Lall and Dumas, 2022).

1.1.4 Platinum mineral tailings

The mining industry generates an estimated of over 14 billion tons of tailings annually, with platinum mineral tailings producing about 10 tons of tailings per ton of ore (Adiansyah et al., 2015b; Gibson et al., 2023a). The tailings have been reported in literature to contain a significant composition of elements which include Cr, Pb, Ca, Mg, Fe and Zn within their mineral matrices. Interaction of these elements with the environment poses risks such as acid mine drainage and heavy metal leaching (Schindler et al., 2018; Godirilwe et al., 2021). Generally, platinum tailings are managed by containment rather than recovery of associated elements as according to circular economy principles. Platinum mineral tailings have been investigated for potential repurposing, for example, in road surfacing and recovery of critical metals; the platinum group metals (PGMs) compared to recovery of metallic minerals essential for biological systems (Araya et al., 2021; Schueler et al., 2024).

Figure 1.1 is a flow diagram showing the various stages which result in the production of platinum mineral tailings during the platinum extraction process.

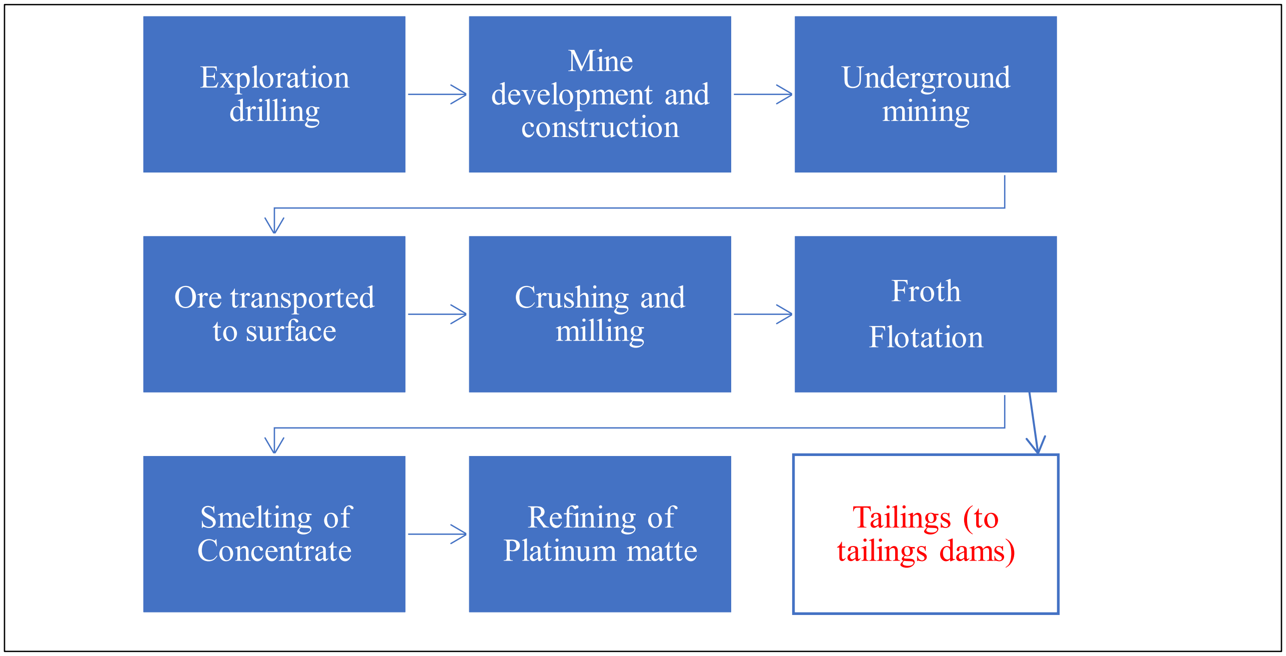

Figure 1.1: General process flow diagram of platinum extraction

Tailings are ground rock and mineral processing waste composed of unrecoverable metals, minerals, chemicals, organics, and process water wastes, produced by chemical and mechanical processes (Adiansyah et al., 2015a). After thorough process recoveries, the tailings are used in applications such as road surfacing, and some similar studies in the region have reported utilizing the tailings to synthesize geopolymers (Abbadi and Mucsi, 2024).

1.1.5 Recovery of elements from tailings

Hydrometallurgical processes by leaching are used to recover elements or solutes from an ore or tailings matrix by dissolving in a liquid solvent (Habashi, 2001). There are several leaching agents which include water, inorganic acids and organic acids. Use of inorganic acids such as sulfuric acid and hydrochloric acid is highly reported due to their effectiveness in dissolving a wide range of minerals (Teir et al., 2007; Lim et al., 2009; Muravyov et al., 2012). However, they are associated with high corrosivity, hazardous to the environment, and require further treatment and are not suitable for food formulation application (Gergoric et al., 2018; Cruz-Rodríguez et al., 2022). There has been growing interest in the use of organic acids due to some having a lower environmental impact compared to inorganic acids (Gerold et al., 2022). In addition, organic acids have complementary characteristics which can be sustainably useful for other applications; characteristics such as antimicrobial activity, chelating properties and biodegradability (Goyne et al., 2010; Larba et al., 2013). However, some organic acids are more expensive than some inorganic acids. The tailings linked to the food processing systems add to circular economy theory.

1.1.6 Groundnut oil processing waste as an organic binder

A binding agent is another crucial component of fish diets because it stabilizes the pellet and lessens nutrient leaching into the water. Popular binding agents also include carbohydrates like starch, cellulose and a variety of extracts or derivatives from plants and animals. Binding agents are included into fish feed formulations to optimize their physicochemical properties. Properties which include aquatic stability, pellet firmness, and minimal fine particle generation during processing and handling (Karim et al., 2024). Examples of binders common in commercial applications, include sodium and calcium bentonites, lignosulfonates, hemicellulose, carboxymethylcellulose and alginate (Paolucci, Fabbrocini, Volpe, Varricchio, 2012). Fiber, notably cellulose and hemicellulose, is included in fish diets, and provides physical bulk and binding properties. Research has shown that fiber influences gastric emptying rates and improves growth and protein efficiency (Dhingra et al., 2012; Colombo et al., 2020;).

Groundnut oil processing waste also called groundnut seed cake or groundnut meal is a byproduct after processing oil from the indigenous crop of the legume species, *Arachis hypogaea*, commonly known as peanut or groundnut. Its proximate composition has been alluded to in literature to consist of about 40 – 45% crude protein, fiber and other essential nutrients, making it serve as a valuable ingredient for feed formulations (Purohit and Rajyalakshmi, 2011; Nguyen et al., 2022). The current study investigates the use of groundnut oil processing waste as an organic binder of the essential elements, simultaneously enriching the nutritional profile of the feed.

1.1.7 Formulation of fish feeds

Feed formulation at the commercial level is the process of finding out how much of each ingredient and additives are required to make compound feeds that satisfy the known nutrient needs of the species being targeted and meet production targets at the lowest possible cost (Lall and Dumas, 2022). During the early 1900s, fish feeds incorporating animal byproducts were formulated according to the proximate composition, availability, cost, and palatability of the raw materials (Lall and Dumas, 2015). Since then, several other raw materials have been investigated for potential application in fish feeds, including industrial and agricultural byproducts. Fish nutritional requirements include amino acids, protein, lipids, vitamins and minerals. The nutritional requirements are derived and utilized in formulation from various sources which include animal fats, grains and supplements (Tacon et al., 2009). During fish feed formulation; raw materials used are also enriched for other nutrients by the addition of essential nutrients through the use of vitamin and mineral premixes. An example of a premix is the Deutsche Vilomix GmbH, which was included as 2% of a reported fish feed made of ingredients; noug cake, brewery waste and wheat bran. Table 1.3 shows the composition of the Deutsche Vilomix GmbH premix

Table 1.1: Composition of the premix per kg of premix

Vitamins	**Amount**	**Minerals**	**Amount**
Vitamin A	4,000,000 IU	Iron	25,000 mg
Vitamin E	40,000 mg	Copper	1,200 mg
Vitamin D3	400,000 IU	Manganese	4,000 mg
Vitamin B1 (Thiamine)	3,000 mg	Zinc	6,000 mg
Vitamin B2 (Riboflavin)	4,000 mg	Iodine	400 mg
Vitamin B6 (Pyridoxine)	3,000 mg	Selenium	20 mg
Vitamin B12 (Cobalamin)	8,000 µg	Cobalt	200 mg
Vitamin K3 (Menadione)	1,200 mg		
Nicotinic Acid	30,000 mg		
Calcium Pantothenate	10,000 mg		
p-Amino Benzoic Acid	20,000 mg		
Folic Acid	1,000 mg		
Biotin	300,000 µg		
Vitamin C	50,000 mg		

Source: (Yimer et al., 2023)

As shown in Table 1.3, the premix was incorporated into the formulation to provide vitamins and essential minerals. The 2 % inclusion of the vitamin and mineral premixes is also shown (Table

1.4) in the typical formulation done during a project on a feed for Clarias macrocephalus fry fish by the National Inland Fisheries Institute, Thailand.

Table 1.2: Typical formulation for *Clarias macrocephalus* fry fish

Ingredients	**Diet No. 1 (% by weight)**	**Diet No. 2 (% by weight)**
Fish meal	60	60
Peanut meal	10	10
Fine rice bran	8	8
Alpha starch	15	15
Fish oil	5	5
Vitamin and mineral premix	2	0
Carboxymethyl cellulose	0	2

Source: (Taechajanta and Sitasit, 1981)

As shown in Table 1.4, from their formulation in diet No. 1 which included vitamin and mineral premix, Taechajanta and Sitasit (1981) observed that the inclusion improved the survival and weight of the fish compared to use of diet No. 2 (Taechajanta and Sitasit, 1981). Other similar innovative formulation studies include the following: Pangestika and Putra formulated two varieties of fish feed from the sludge of dairy waste water and other ingredients (Pangestika and Putra, 2020). Arisa et. al researched on the utilization of organic waste as a raw material for fish feed production (Arisa, Fadli, Anwar, Nizamuddin, and Parmakope, 2018). Wong et. al reviewed the feasibility of recycling food waste into fish feeds for safe and high quality fish production (Wong et al., 2016).In a novel study on feed supplementation; the effects of the Artemisia(A) plant species as a feed supplement on the growth performance of seabream were investigated. Among other biological characteristics, Artemisia (A) is known to have neuroprotective, digestive and antimicrobial activities. In their study they concluded that A. arborescens may have a negative effect on the growth of the seabream (Status et al., 2024).

1.1.8 Circular economy synergy between platinum mineral tailings, agriculture, and aquaculture

The concept of the circular economy is to increase product efficiency while reducing waste and recycling resources. In addition to extending material life and lowering waste production, this strategy aims to maximize resource utilization and minimize energy consumption (Suárez-Eiroa et al., 2019). This concept is opposite to the conventional linear economic model, which is based on extraction, production, consumption, and disposal. Circular economy initiatives encourage the recovery, recycling, repair, and reuse of already-existing resources while reducing adverse environmental effects and generating new business opportunities (Lèbre et al., 2017; Ancuţa and Sonia, 2020). This current study links two sectors, mining and fish farming, by investigating the extraction of essential minerals, which include Ca, Mg, Zn and Fe from platinum tailings (a mineral processing waste) and combining them with groundnut oil processing waste (an agro-process byproduct). This aims to provide an alternative to replacing mineral supplements such as calcium carbonate with tailings-derived elements combined with groundnut processing waste, which contains other important nutrients required by fish: protein, crude fiber and lipids.

1.2 Statement of the problem

Feed costs and feed ingredients, including vital minerals, are challenges to aquaculture production's economic feasibility and environmental sustainability. At the same time, Zimbabwe's platinum mineral tailings management poses a challenge to the environment as well as a possible source of valuable elements. Similarly, groundnut oil processing waste produces huge amounts of nutritious waste which remains underutilized despite its known value as a source of animal feed. The underexplored circular economy niche of the availability of locally generated mineral-rich tailings and agricultural by-products represents limits both sectors contribution to national food security and economic development.

1.3 Justification of the study

Investigating the feasibility of recovering essential elements from platinum mineral tailings combined with agricultural waste for use in animal feed supports circular economy initiatives. These waste-based ingredient sources provide a link between the mining sector and the aquaculture industry. Therefore, this research attempts to address these two issues by offering a sustainable source of vital nutrients for formulating fish feeds. This will add circular economy aspects and support the sustainable development goals (SDG) of eradicating food insecurities by 2030. The SDG 2 and SDG 12 of eradicating food insecurities and sustainable production by 2030 (Hwang and Kim, 2017). The study addresses a knowledge gap over the safety and optimal effective application of mining by-products in aquaculture nutrition. There have been technical recoveries of elements from tailings in some previous studies, but none have assessed the nutritional quality and safety of these materials to aquatic animals. Likewise, though groundnut oil cake is applied to formulate fish feeds, it has not yet been reported as a binder for essential minerals extracted from mineral waste.

1.4 Research aim

The research aims to formulate a fish feed through the valorization of platinum mineral tailings as a source of essential elements, and combine with groundnut oil processing waste as a source of protein and an organic binder.

1.5 Research objectives

To achieve the aim of this research, objectives of the study were to:

1. characterize the composition of platinum mineral tailings and identify the essential elements present using XRF and XRD techniques.
2. optimize the recovery efficiency of essential elements from platinum mineral tailings through organic acid leaching.
3. formulate fish feed by combining the extracted elements with ground nut oil processing waste as an organic binder.
4. characterize the formulated feed for proximate composition and elemental composition using AOAC methods and ICP-OES, respectively.

1.6 Ethical consideration

The research was carried out ethically with findings initially based on laboratory analysis until concluded on toxicity.

1.7 Impact on the environment

The research intends to support environmentally friendly utilization by proposing to recover elements from tailings before disposal, thereby reducing risks of acid drainage. Although mineral recovery from tailings minimizes environmental impacts such as acid drainage, effects occur if not properly handled in the recovery process. Mild alkaline pretreatment involving environmentally benign reagents and low energy input is utilized in this study to avoid causing such impacts. Future research should incorporate environmental studies to ensure the process is sustainable.

CHAPTER 2

LITERATURE REVIEW

2.0 Introduction

The aquaculture industry is seeking for potential raw materials for fish feed manufacturing to replace common traditional options like fish meal and soymeal which cause environmental issues including deforestation and overfishing (Goglio et al., 2022; Kumar et al., 2025). The mining industry is key in producing essential raw materials for the modern society. Mineral processing involves mechanical particle reduction processes and chemical processes to extract valuable minerals (Grewal, 2006). This target mineral is separated and the byproducts produced after the extraction are mining tailings (Edraki et al., 2014; Kossoff et al., 2014). These vary in chemical composition depending on the ore type and extraction processes used (Pryor, 2012; Edraki et al., 2014). Globally, a considerable volume and variety of mine tailings are produced daily, many of which contain heavy metals, chemicals, and toxic substances that pose potential environmental risks (Weng et al., 2022; Manaviparast et al., 2024). The general composition of most mineral tailings includes silica, and some compounds such as carbonates, oxides and sulfur (Maltrana and Morales, 2023). These tailings are stored in dams and or landfills (Marín et al., 2022a). According to Azapagic, 2004, and Moran et. al, 2014 cited in Marin et.al 2022; depending on the extraction efficiency or selectivity of the target mineral, tailings may contain valuable elements (Marín et al., 2022b). Similarly, Juutinen et al. (2023) reported that tailing storage facilities may contain useful trace minerals that have potential applications as secondary raw materials (Juutinen et al., 2023).

2.1 Occurrence and classification of mining waste

Mineral waste consist of solids and liquids produced during processing of minerals (Kalisz et al., 2022). Wastes can be grouped into three types; mining waste, metallurgical waste and, processing waste (Matinde et al., 2018). Mining waste is all waste rock and tailings generated during the mining process, Metallurgical waste is produced during metallurgical processes such as smelting, refining, and alloying, and processing waste is produced in various stages of mineral processing, such as crushing, grinding, concentration, and chemical treatment (Lottermoser, 2007; Nwaila et al., 2021; Villa Gomez et al., 2024a). There is a need for the development of effective strategies for managing mineral waste to maintain a sustainable equilibrium between the projected increase in demand and the availability of metals and industrial minerals (Mateus and Martins, 2019). Processes that produce tailings include comminution, liberation and concentration of ore via processes such as froth flotation (Mondal et al., 2021; Bilal et al., 2022). There is a global production of mining tailings that is ever increasing and only a minimal fraction of metals is extracted from each ton of processed ore. There is a niche for sustainable resource utilization of the ever increasing tailings (Woźniak and Pactwa, 2018; Cacciuttolo and Atencio, 2022). In a similar context, tailings are commonly regarded as low-grade deposits therefore recycling and reusing these tailings would be effective in providing economic benefits (Ndlovu et al., 2017; Faramarz et al., 2024).

2.2 Fate of tailings in the environment

Mineral processing stages produce a large quantity of tailings. Without proper storage and management, tailings may result in serious waste of resources and environmental pollution (Ge and Su, 2020). If a large amount of rainwater accumulates in tailings reservoir, it causes serious contamination of water resources (Kossoff et al., 2014; Wang et al., 2019; Ge and Su, 2020), the oxidation of tailings can produce large amounts of acids and metals thus posing potential health risks (Chen et al., 2018; W. Li et al., 2024).

A study by Van Rensburg and Morgenthal (2004) on the rehabilitation of platinum tailings; a platinum mine in South Africa revealed that tailings contain high levels of metals. Analysis using Atomic Absorption Spectrometer (AAS) revealed that it consisist of composition of Ca (3.51 mM), Mg (3.45 mM), K (1.25 mM), and Na (6.54 mM) and ion chromatograph revealed composition of SO_4 (3.21 mM), NO_3 (2.93 mM), and Cl (9.50 mM) (Van Rensburg and Morgenthal, 2004) compared with standards proposed by the South African Department of Water Affairs and Forestry (Department of Water Affairs and Forestry, 1996). This typical composition of tailings poses possible negative environmental impacts including air and groundwater pollution (Maboeta et al., 2008). Woodall (2021) postulated that tailings from PGM mines will be very relevant in the years to come. They further numerically suggested that, when forecasting the production of PGMs, values increase to 25 Mt and 5 kt per year , which results in the co-production of up to 3.5 Gt/year of ultrabasic mine tailings (Woodall et al., 2021). Previous studies by Boshoff and Hamman (2011) highlighted that platinum tailings facilities are often viewed as future resources for remining with improved technology, militating against closure expenditures during operation (Boshoff and Hamman, 2011). According to Gibson et.al(2023) in their study of the valorization of platinum group metals from flotation tailings; platinum tailings are a potential mineral resource, considering the significant historic tailings deposits and considerable annual production (Gibson et al., 2023b).

2.3 Potential uses of tailings in fish feed formulations

Extensive research is being carried out, to identify innovative solutions that emphasize the sustainable management of tailings. Research has aimed to shift from the linear production approach to a circular economy concept, so as to minimize waste and maximize resource recovery (Suárez-Eiroa et al., 2019; Araya et al., 2021; Marín et al., 2022b). The circular economy model considers mining waste as an opportunity for environmental stewardship and sustainable mining (Yu et al., 2024). Mining tailings contain various inorganic elements, some are essential dietary minerals such as Fe, Zn, Mn and Cu and some contain some toxic heavy metals such as cadmium (Lim et al., 2009; Manaviparast et al., 2024). The specific elemental composition varies depending on the type of ore mined and the extraction processes used (Bernadette and Ogbonnaya, 2023; Nakhaei et al., 2025).

The global fish farming industry is affected by limited supply of quality feeds. Approximately 70% of fish farming expenses are attributed to feed, with protein being the most costly component (Munguti et al., 2024). The high cost of fishmeal results in high aquaculture production expenses and leads to resource competition with human fish consumption (Davies and Ezenwa, 2008; S. Ovie and Ovie, 2010; Ramotar-John, 2014). In an investigation of fish seed and feed value chains in aquaculture in Tanzania by Shoko et al. (2023); it was concluded that third world nations fish production is affected by inadequate fish feeds among other factors. The need to increase feed production through the collaboration of researchers and investors has been highlighted (Shoko et al., 2023). Partnerships between researchers and investors enable substantial funding for infrastructure, technology, and equipment, as contemporary aquaculture requires a significant capital investment (Habimana, 2024b).

Fish serve as an important source of protein, as they are more easily digestible and complement the carbohydrate-rich diets common in many parts of Africa. Fish consist of essential amino acids, minerals, and micronutrients such as iron, zinc, omega-3 fatty acids, and vitamins and is often found in highly bioavailable form (Vilain and Baran, 2016; Muhammad and Selamoglu, 2023). Given the documented composition of certain mineral tailings, which contain essential nutrients such as zinc and iron, a potential synergistic relationship exists between the use of these tailings as a source of such elements and the formulation of nutritionally enriched fish feed.

2.4 Characterization of mineral tailings

Mining tailings are characterized on the basis of targeted needs for example for assessing potential environment risks through mineralogical characterization (Mancini et al., 2024). Table 2.1, shows the classification of characterization methods on the basis of five important characteristics:

Table 2.1: Classification of characterization methods for mineral tailings

Class/Category	Principle	Examples	References
Geotechnical characterization	in-depth analysis of mining waste to investigate behavior and stability.	Parameters such as density and shear strength	Azam et al. (2020); Coelho and Camacho (2024)
Physical characterization	carried out to suit physical properties of material with tended application	Mechanical compressive test on bricks made from various metal tailings	Ally et al. (2021); Benahsina et al. (2021); Vazquez et al. (1991)
Petrographic characterization	optical microscopy techniques for identification of mineralogical composition and grain sizes	Scanning Electron Microscopy (SEM), Petrographic microscopy	Aladejare (2021); Lapakko (2002)
Mineralogical characterization	for identifying and analysis of various minerals present including their properties	Scanning electron Microscopy (SEM), X-ray diffraction (XRD), X-ray Fluorescence (XRF)	Faulstich et al. (2011); Juutinen et al. (2023); Molahid et al. (2023).
Geochemical characterization	techniques to analyses content of elements in samples.	IXRF for major elements and Inductive Coupled Plasma Mass spectrometry (ICP-MS) for trace elements	Bevandić et al. (2021); El-Taher et al. (2023); Marguí et al. (2022).

2.4.1 Characterization techniques for the analysis of tailings and extracts

Characterization techniques are employed for the purpose of examining the physical properties, chemical properties and elemental composition of tailings.

2.4.2 Elemental analysis using X-ray fluorescence

X-ray fluorescence (XRF) is a nondestructive, near real-time method for simultaneous multielement analysis of various sample types, including liquids, powders, solids, and thin films (Kanngießer et al., 2007; Lemière, 2018).

When a material is subjected to high-energy X-rays, the atoms within the material may undergo ionization, resulting in the excitation of an electron from an inner shell (Kanngießer et al., 2007). Typically, when the emitted radiation has a lower energy than the absorbed radiation, this phenomenon is termed fluorescence (Zofka, 2013). Each element displays a typical X-ray fluorescence (XRF), and the intensity of each fluorescence is linearly related to the concentration of the respective element within the material. This technique identifies and quantifies specific elements in a sample (Bosco, 2013).

XRF is chosen for its ability to perform multielement analysis and to apply sample matrix corrections. The typical target metals analyzed using this technique include Pb, Zn, Cu, Cr, Ni, Cd, As, and Ba (Bernick et al., 1995).

For elemental recovery from PGM tailings via thermochemical treatment, Mohamed et.al, 2016, used an X-ray fluorescence (XRF) with a 4 kW Rh tube to identify the major oxides of Al, Mg, Fe, Cr, Si and Ca. They successfully extracted some key elements from PGM mine tailings (Mohamed et al., 2016a). XRF techniques have been used to improve the flotation of refractory phosphate ore which is difficult to separate because of the high amount of impurities present (Xu et al., 2021). XRF with a rhodium-anode tube revealed the presence of impurities as oxides of Fe, Al , Mg and Si (Xu et al., 2021). The recovery of iron and copper from copper slag was investigated by Li et. al, 2021 who identified elements including Cu, Fe, Zn and Mn present in tailings using X-ray fluorescence (XRF). Copper and iron are recycled from the slag (Li et al., 2021).

During analysis of the elements in gold mine tailings using X-ray fluorescence (XRF); silica, alumina, iron oxide, potassium oxide, calcium oxide and sulfur trioxide were revealed. The researchers concluded that some of the oxides have the potential to be used in glass making (Balbin et al., 2024). During studies to recover silver from high-sulphur mining waste, XRF showed the presence of various elements including Fe, S, Mg, Zn and Al (Muñoz Hernández et al., 2025).

2.4.3 Crystal morphology and phase identification using x-ray powder diffraction

X-ray powder diffraction (XRD) is widely implemented for both qualitative and quantitative analyses of crystalline compounds. Thorough XRD, minerals can be identified with precision, allowing for quantitative assessments, and determination of crystal structures (Duyvesteyn et al., 1994; Iwasaki et al., 1988; Rabatho et al., 2010; Cui et al., 2018; Pöllmann and König, 2021;; Trifunović et al., 2024).

XRD has been the method of choice for qualitative phase analysis for nearly a century, following the discovery of X-ray diffraction in 1912 (Chipera and Bish, 2013). The quantitative phase analysis of multicomponent mixtures utilizing X-ray powder diffraction data has evolved from the Rietveld computer program developed by Wiles and Young (Bish and Howard, 1988; Chipera and Bish, 2013).

The effectiveness and ease of XRD is due to its simplicity and speed, as well as its applicability to various types of materials (Bish and Post, 1993). In 1936, Hannawalt and Rinn reported a phase identification method in which XRD was used to compare experimental and reference X-ray powder diffraction patterns. XRD has evolved over the years for the determination of periodicity and symmetry, examination of crystal structure, phase identification and measurement of the physical properties of crystals (Chung and Smith, 1999). The development of methods for quantifying X-ray diffraction data has occurred more gradually, advancing through theoretical approaches but, being more crucially linked with the increase in computational power (Chipera and Bish, 2013). Diffraction is a general characteristic of all waves where the behavior of light is modified by interactions with an object (Suryanarayana and Norton, n.d.). XRD analysis offers insights into the types and nature of the crystalline phases present, with analytical technique elucidates the characteristics of the crystalline phases, the relative proportions of predominant mineral phases, the macrostrains, crystallite dimensions, and the patterns of isomorphic substitution within these phases (Aylmore, 2019).

.During a feasibility study on the recovery of PGMs from oxide ores of the Great dyke, Zimbabwe, XRD was employed to analyse raw ore during roasting. Major peaks associated with chlorite and talc were observed (Duyvesteyn et al., 1994). Penberthy et. al, (2000) alluded to the recovery of PGMs from a UG-2 chromitite ore, and phase composition studies with XRD identified chromite, interstitial orthopyroxene, clinopyroxene, biotite, chlorite, quartz, serpentine and calcite (Penberthy et al., 2000). In a study on the potential for mineral carbonation of PGM tailings by Vogeli et .al, (2011), XRD was used to quantify the relative abundances of the crystalline phases in the PGM tailings, resulting in the identification and quantification of plagioclase, orthopyroxene, talc, enstatite, and serpentine (Vogeli et al., 2011). During studies XRD was used to study the phase composition of ceramics produced from iron tailings. XRD revealed that the ceramic consist of akermanite, gehlenite and diopside. These three compounds were consistent with the results of the XRF analysis (Hua et al., 2023). A case study of Mauritania on copper tailings, and phase composition analysis using X-ray diffraction revealed that the samples contained silicates, iron oxides, various sulfides, arsenides, koutekite, tellurides and various carbonates. It was concluded that these tailings consist of secondary minerals (Ba et al., 2024).

2.4.4 Inductively coupled plasma–optical emission spectrometry and inductively coupled plasma–mass spectrometry

Inductive coupled plasma (ICP) techniques involve the use of very small amounts of samples compared with other techniques to identify major and trace elements (Al-dousari et al., 2021). The detector, is capable of detecting metals and several nonmetals in very low concentration in part per quadrillion (ppq) (Sudhakar et al., 2016). ICP techniques which include inductively coupled plasma-optical emission spectrometry (ICP–OES) and inductively coupled plasma–mass spectrometry (ICP–MS) are key for qualitative and quantitative analysis of trace element analysis in samples (Mancini et al., 2024). Compared with atomic absorption techniques, these techniques have higher speed, better precision, and higher sensitivity (Sudhakar et al., 2016).

The application of OES using ICP as an emission source originated in 1964 (Greenfield et al., 1964). OES is based on the excitation of atomic spectra with wavelengths between 150 and 800 nm (Hou and Jones, 2000). It measures the electromagnetic radiation emitted when electrons transition between the outer energy levels of an atom. These electronic energy levels are defined by various quantum numbers. The energy differences between energy levels is described in terms of those numbers (Alfassi, 2008) .

ICP–OES has been popular since its full launch in 1974, and has been recommended throughout the years because of its analytical characteristics which include freedom from chemical interferences compared with FAAS, multielement analysis, a wide linear dynamic range , simplified calibration, better precision and better sensitivity (Beauchemin, 2020; Makonnen and Beauchemin, 2020). ICP–OES is highly stable and can be used to analyse multiple elements simultaneously (Li et al., 2021; Balaram, 2023).

ICP–MS, the hyphenated technique involving the use of a mass spectrometer as a detector evolved in the 1980s, significantly improving analytical throughput, detection sensitivity, and isotopic resolution capabilities. Although ICP–MS is prone to polyatomic and isobaric interferences compared with ICP–OES, it has a greater capacity for simultaneous multielement analysis, ultra-trace detection limits, and an extensive linear dynamic range compared with ICP-OES (Winefordner et al., 2004, Beauchemin, 2020; Makonnen and Beauchemin, 2020;).

In an environmental assessment of sulfide-rich tailings: major and trace element quantification was conducted using an ICP–OES system. The detection limits (LODs) were defined as follows: $LOD = 3\sigma$ of ten replicate measurements of a reagent blank. The analysis they carried out revealed higher concentrations of As, Cu, Fe and Zn than the acceptable regulatory limits (Bhattacharya et al., 2006).

Ilieva et. al (2019) carried out estimation studies of soil and tailing dump toxicity using ICP-OES techniques. The limits of quantification (LOQs) were determined as: $LOQ = 10\sigma$, from three replicate measurements of blank solutions (Ilieva et al., 2019). Elevated concentrations of Fe, Cu, Pb, and Zn, with recoveries between 97% and 105% along with trace levels of Mn, Cd, Ni, and As, with recoveries between 4.7% and 11%, were identified in the analysed samples (Ilieva et al., 2019).

During studies in which vanadium tailings were utilized for synthesis of foamed concrete; quantitative analysis of the metal content in the raw materials was performed using ICP-OES (X. Cui et al., 2021). PGM mine tailings were characterized using ICP-OES as part of measuring the dissolution rates of cations during carbon mineralization. Quantitative analysis using ICP-OES revealed that most cations, excluding Ca^{2+} and Al^{3+}, were below detection limits (Woodall et al., 2021).

In a study on the release of heavy and rare earth metals into the environment, ICP–MS was used (Krasavtseva et al., 2021). The contents of trace elements were detected and used to study leaching conditions of the heavy and rare earth metals from tailings (Krasavtseva et al., 2021). In similar studies Cherkasova et. al., (2021) successfully determined the elemental content of rare metals in ash and slag using ICP–OES and ICP–MS. They established that ICP–OES identified the major elements, and the trace elements were revealed by ICP–MS. ICP–MS is preferred for trace element analysis, but requires complete sample dissolution for accurate results, emphasizing optimized chemical preparation (Cherkasova et al., 2021).

2.4.6 Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) elucidates the molecular structure of analytes by analyzing the interaction between matter and infrared radiation (Alvarez-Ordóñez and Prieto, 2012). The FTIR technique is used to analyze the organic and inorganic components of the samples. Its nondestructive nature and analytical flexibility make it a valuable tool for the chemical characterization of a wide range of materials such as silicate glasses, minerals, and microfossils (Chen et al., 2015).

The principle of FTIR is based on the absorption of specific infrared frequencies, as a function of molecular vibrational modes (Griffiths, 2007). These molecular vibrations, ranging from simple diatomic oscillations to complex polyfunctional motions, provide structural information (Griffiths, 2007; Tkachenko and Niedzielski, 2022).

Investigations through vibrational spectroscopy provide valuable insights into hydration properties, interlayer cations, and moisture levels in samples (Saikia and Parthasarathy, 2010). The resulting transmission or absorption spectrum provides information regarding the constituent molecular bonds (Alvarez-Ordóñez and Prieto, 2012; Moore Emily, 2016).This technique's ability to elucidate is further enhanced by the pronounced absorption of infrared radiation within the mid-infrared region, which is exhibited by many molecular compounds (Smith, 2011).

Several studies have been conducted on the analysis of minerals and mineral tailings using infrared spectroscopy to determine the nature of chemical bonds. Kaolinite–pyrite flotation tailings were characterized using FTIR spectroscopy before and after the production of the 4A zeolite. Compared with those of commercial 4A zeolite the characteristic bands confirmed that the synthesis was successful (Cui et al., 2018). Artisanal and small-scale gold mine tailings were characterized by ATR-FTIR during an investigation of the use of tailings for the synthesis of geopolymers. The spectra showed characteristic bands for Fe–O, O–H and S–S absorption. The Fe–O stretch was because of the exposure to air (Opiso et al., 2021). During the synthesis of scorodite from a acid mine drainage: FTIR spectroscopy confirmed the chemical changes as the Fe-sludge was converted to scorodite (Kimura and Okibe, 2025). According to Negrao et.al (2021) FTIR among other techniques can be used to validate and refine X-ray powder diffraction analysis (XRPD). Mineralogical evaluation of bauxite mineral waste for use in ceramic production was carried out using FTIR. Like XRPD, FTIR confirmed the characteristic OH stretching bands of Kaolinite, gibbsite and goethite (Negrão et al., 2021).

FTIR analysis of cement made from Iron tailings revealed the characteristic bands of hydroxyl, Si-O-Si and AlO_4. These findings confirmed the extent of the polymerization of the silica-oxygen tetrahedra (Hu et al., 2024). Similar conclusions were reported for (i) recycling and application of mine tailings in alkali-activated cements and mortars (Cristelo et al., 2020) and (ii) optimization of ultrahigh–performance concrete using a clinker–free binder and iron tailings aggregate (Heraiz et al., 2025).

2.4.7 Fourier transform-near-infrared analysis

Fourier transform-near-infrared (FT-NIR) spectroscopy is a popular modern technique used for simultaneous quantitative determination of fat, protein, and dry matter contents. FT-NIR spectroscopy serves as a valuable tool for molecular characterization, offering insights into the presence of functional groups through the identification of characteristic absorption bands (Benešová et al., 2022).

Badaro et. al (2022) on the application of NIR in wheat-based products, postulated that despite the applicability and wide use of the classical method in the determination of composition. Some of these techniques have disadvantages, including the need for chemical reagents, which are used in most classical analytical methods (Badaró et al., 2022). For example, Fodor et. al (2024) echoed that Kjeldahl method for protein analysis is a destruction process with concentrated sulfuric acid at a high temperature (Fodor et al., 2024). Nielsen and Carpenter (2017) highlighted that determination of fat content by solvent extraction involves the use of large amounts of organic solvents, such as petroleum ether, and hexane (Nielsen and Carpenter, 2017).

FT-NIR is used for the rapid and nondestructive determination of proximate chemical compositions, including protein, dry matter, fat, and fiber, across diverse food ingredients and products (Cozzolino, 2021). FT-NIR is a molecular vibrational techniques probing internal degrees of freedom (bond stretching/deformation) through electromagnetic radiation interaction, resulting in characteristic absorption bands at specific frequencies (Beć et al., 2020). NIR is a type of high-energy vibrational spectroscopy performed in the 750 to 2500 nm range (13,333 – 4000 cm^{-1}) (Pasquini, 2018). FT-NIR spectroscopy is widely used in food safety, offering superior resolution and range over traditional methods for rapid, accurate analysis of key food components such as proteins and polysaccharides (Luo et al., 2025).

The essential dietary requirements include water, proteins, lipids, carbohydrates, vitamins, and minerals (Manam, 2023). Additionally for fish; carotenoid pigments are commonly included in the diets of salmonid and ornamental fish for their flesh and skin development (Velasco-Santamaría and Corredor-Santamaría, 2011; Prabu et al., 2017; Idenyi et al., 2022).

2.5 Extraction of elements from mineral tailings

Mineral tailings are being explored as secondary sources of important elements for sustainable recovery and applications in the formulation of fish feeds.

The extraction of metals from mine tailings has gained interest over the years through the use of hydrometallurgical methods, which include acid leaching. The evolution of hydrometallurgical processes has generated knowledge on the extraction of metals from mineral raw materials (Trujić et al., 2025). Various methods for recovering metals from mineral waste have been explored including the following:

(i) acid leaching both organic and inorganic,
(ii) salt leaching which involves alkali metals,
(iii) base leaching,
(iv) bioleaching methods,
(v) flotation, and

(vi) oxidation methods

(Gunarathne et al., 2022; Gulliani et al., 2023;; Jiang et al., 2024; Schueler et al., 2024; Villa Gomez et al., 2024b).

Compared with inorganic acids the use of organic acids has greater advantages in overcoming some of the drawbacks of using inorganic acids (Mends et al., 2024). The following table shows several reported successful studies of the recovery of metals from tailings:

Table 2.2: Recovery of various elements from various potential tailings**.**

Source of tailings/low grade ore	**Leaching agent(s)**	**% recoveries**	**Inference of the leaching process**	**References**
Low grade manganese ore	Citric acid, oxalic acid	Mn = 60% with oxalic acid, Mn = 44% with citric acid	Oxalic acid had a higher recovery for manganese than citric acid due to its affinity for manganese.	Das et al. (2012)
Low grade platinum concentrate	Bioleaching using mesophilic and thermophilic microorganisms	Cu = 52%, Ni = 95%, Co = 85%	Microorganisms have potential for recovering metals from low-grade flotation concentrates	Mwase et al. (2012)
Copper slag and Copper Flotation tailings	Sulfuric acid + ferric sulphate/hydrogen peroxide	Cu = 13%, Fe= 48%	Best recovery was achieved with combination of sulfuric acid and hydrogen peroxide	Urosevic et al., (2015)
Oxidized PGM ore	Hydrochloric acid, nitric acid, aqua regia	Pt= 48%, Pd=24.5%	High reagent consumption	Sefako et al., (2019)
Gold tailings	Cupric chloride, Sodium chloride	Cu = 98%, Ni = 93%, Zn = 83%, Co = 76% and Fe = 80%	Pure chloride can be utilized in metal extraction of metals in trace amounts	Altinkaya et al., (2019a)

2.6 Response surface methodology approach to maximize leaching of elements

Response surface methodology (RSM) is a combination of statistical methods used for application in process optimization, product design and improvement (Lamidi et al., 2023). According to Bai et. al (2018), the RSM uses appropriate quadratic regression models and planned experiments to optimize processes and a popular RSM technique for examining nonlinear effects and interactions to identify ideal factors with the least amount of resource consumption is the central composite design (CCD) (Bai et al., 2018). The RSM model is designed to forecast experimental results, and uses a statistical approach to determine how independent input variables and dependent outputs relate to one another (Zahid et al., 2018). The traditional single-factor optimization approach is a limitation for a complete and systematic analysis of various factors (Simate et al., 2009; Teimouri et al., 2020) and is expensive and limited for understanding complex factor interactions (Teimouri et al., 2020). The RSM determines the minimum optimum experimental parameters and how they are related to each other (Demir and Derun, 2019).

Compared with single factor methods, conditions optimized by RSM have been reported to improve efficiency and reduce costs by minimizing the number of experiments that can be run for a particular experiment (Lamidi et al., 2023). RSM optimizes the leaching of metals from mineral tailings by experimentally determining the optimum conditions for maximum metal recovery (Mwanat et. al 2021, Kolbadinejad, 2024). RSM approach involves the following key stages:

a) Identifying key parameters that affect the leaching process from literature and preliminary experiments; such as acid concentration and temperature (Zhang et al., 2015; Lucay et al., 2021;).
b) Statistical planning of experiments using methods such as the central composite design to systematically plan the variation of factors in the experiments to be carried out (Pereira et al., 2021).
c) Experiments are carried out to measure the recovery percentages of the metals. The collected data is analyzed to determine the relationships between the input parameters and response variables (Aydar, 2018).
d) A mathematical model, second-degree polynomial is used for the prediction of the response variables based on the input parameters which gives insights into the interactions of various factors involved in the experiments (Santos et al., 2015; Bai et al., 2018).

Previous studies based on RSM approach on recovery of elements have been reported which include:

Zakrzewska-Koltuniewicz et.al (2014) used optimization technique based on response surface methodology (RSM) to identify the best leaching conditions for the simultaneous extraction of multiple metals from Polish ores, including uranium, lanthanum, vanadium, molybdenum, ytterbium, and thorium (Zakrzewska-Koltuniewicz et al., 2014). Alaoui (2015) used the Box–Behnken to model the following variables: potassium oxalate concentration, solid–to–liquid ratio and sulphuric acid concentration. The Mn recovery conditions were 80%; the optimum recovery conditions were solid to liquid ratio of 50 g/L–70 g/L, sulfuric acid concentration is 1.1–1.3 M an oxalate concentration in range 0.35–0.45 M, and a time and temperature of 30 min and 25°C respectively (Alaoui et al., 2015). Malemba et .al (2022) used RSM to study and optimize the desliming impact of flotation recovery of platinum group metals from mine sludge. A recovery of 79% of PGMs was reported after desliming at an optimum pH of 10.29 and a pulp density of 25.08 Wt.% (Malenga et al., 2022). Pengwei et. al (2022) using RSM proposed an environmentally friendly recycling process for recovering metals from spent lithium-ion batteries (LIBs) using optimized mixture ratios of sulfuric acid and malonic acid as leaching agents. They recovered high percentages > 96% for Li, Ni, Co, and Mn within 81 min with H_2SO_4, malonic acid, and at a temperature of 70 °C and with 5 vol % of 30% H_2O_2, (P. Li et al., 2022). Rautela et. al (2024) optimized Li recovery from used waste batteries via the use of glycine as a leaching agent, and optimum temperature, glycine concentration, and pulp density were established by RSM approach (Rautela et al., 2024). Table 2.3 shows the extracted references for potential variables for response surface methodology in this study.

Table 2.3: Optimization variables used for leaching experimental design

Factor	Low (–1)	High (+1)	Literature Reference
pH	2.0	9.0	Mn, Zn recoveries at pH 7–10 Sayılgan (2022); Zn, Cu, Cr, Ni, Cd at pH 1.98–3.32 Zaleckas et al. (2013); acetic/citric acid leaching at pH 2.9–5.0 Motha et al., (2023).
Temperature (°C)	30	90	Effective leaching reported from 30–90°C Motha et al., (2023); Sayilgan et al., (2010) studies at 30°C Nagarajan and Panchatcharam, (2023) and up to 60°C Motha et al., (2023)
Contact Time (h)	1	48	Ranges from 1–5 hours Sayilgan et al. (2010), up to 48 h Crane and Sapsford (2018) and 6–72 hours Woodall et al., (2024b),
Acid Concentration (M)	0.1	2.0	Leaching with 0.01–2 M Crane and Sapsford (2018); 0.5–2.5 M Nagarajan and Panchatcharam, (2023); 0.1–1.0 M for organic acids Woodall et al. (2024b).

2.6.1 Formulation of Fish feed via the RSM Approach

RSM is essential to the design and development of both new and improved products. It can identify the ideal factors required to produce the best outcome (Manuel Pais-Chanfrau et al., 2021, Lamidi et al., 2023;). Tumuluru (2013) maximized the properties of aqua feed pellets through RSM. The RSM determines extrusion process variables to improve characteristics of the aqua feed: "water stability, true density, and expansion ratio" (Tumuluru, 2013). Irung et. al (2018) applied RSM to optimize the effects of temperature, die diameter and feed preconditioning time on the properties of fish feed pellets which include expansion ration, bulk density and floatability (Irungu et al., 2018). During a study to improve nutritional value of a soyabean based aqua feed, soyabean meal fermentation was optimized by RSM to establish conditions for achieving a higher degree of protein hydrolysis and crude protein (Zhang et al., 2021). The liquefaction and "sweetening" properties of germinated flours from two new types of maize , were examined in a study to optimize to various malting parameters, including different durations of soaking, temperature, germination, maturation and salt concentration (Tambo Tene et al., 2023). A formulation including groundnut cake, milk powder, lemongrass and corn cobs was optimized after proximate analysis of each ingredient. The formulation was adjusted for maximum protein content using response surface methodology. Significant mineral and protein levels in the feed ingredients demonstrated their suitability for fish feed development (Monday et al., 2024). Response surface methodology was used to explore how extrusion parameters such as moisture levels (12–18%), temperature (150–175 °C), screw speed (200–280 rpm), and changes in feed composition affect the characteristics of the extruded snack food extruded from rice grit combined with fruit waste, durum clear flour, and partially defatted hazelnut flour (Yagci and Gogus, 2008). The following Table 5 shows potential variables to consider during formulation for response surface methodology in this current study.

Table 2.4: Variables used for formulation experimental design

Factors	**Low (–1)**	**High (+1)**	**Literature Reference**
Mineral Solution (% of feed)	0.5	5.0	(Dawodu et al., 2012; Lall and Kaushik, 2021b; Olapade and Saboleh, 2022; Riaz et al., 2021; Zafar and Khan, 2019)
Groundnut oil processing Waste (% of feed)	40	90	(Davies and Ezenwa, 2008; Ekeocha et al., 2023; Purohit and Rajyalakshmi, 2011; Shukla et al., 2018)
Moisture Content (%)	5	15	(Adedeji et al., 2020; Fregene et al., 2020; Manam, 2023)

2.7.1 Common raw materials used for production of fish feeds

Raw materials include the nonconventional fish feeds, which involve feed ingredients that are not typically found or used in standard commercial fish feed formulations and are common among small holders (Agbo, 2022). These resources include waste materials from agricultural activities and processing industries that are unsuitable for human consumption. These methods are cost-effective and considered part of waste management (Kolawole and Mustapha, 2023).The fish feeds commonly accessed by smallholder farmers, lack some quality aspects. The provision of high nutritional requirements for fish is still a problem. There is a need for improvements in the quality of fish feeds to increase production in the aquaculture industry (Munguti, 2021). Table 2.4, shows the typical composition of some of the common ingredients used in the fish feed formulations

Table 2.5: Typical composition of commonly used aquaculture feed ingredients

Ingredient	**Typical % in Feed**	**Dietary value**	**References**
Fish Meal	20-50%	proteins, essential amino acids, lipids and omega–3 fatty acids	Naylor et al., (2000), Stephenson and Smedbol (2001), Chen et al. (2009), Adibrata et al. (2022)
Soybean Meal	10-50%	proteins, essential amino acids, fiber and carbohydrates	Sørensen et al., (2009), National Research Council, (2011a), Liu et al., (2021),Zhang et al.(2023)
Other Plant based Meals	10-30%	Include sunflower meal and groundnut meal provide proteins, carbohydrates, and fiber.	Sanz et al. (1994), Agbo et al. (2011), Chanda et al. (2015,
Wheat Flour	0-20%	Carbohydrates and can act as a binding agent in feed formulation	Apper-Bossard et al. (2013), Draganovic et al. (2013), Onuoha et al. (2021)
Fish Oil	0-15%	Essential fatty acids, energy source, and improves palatability	Pickova and Mørkøre, (2007), Glencross, (2009), Sargent et al., (1999)
Vitamins and essential elements	0-5%	Essential vitamins and minerals required for the growth and health	Oliva-Teles, 2012), Gasco et al., (2018), Lall and Kaushik, (2021a); Manam, 2023
Other functional Additives	Variable composition	Include antioxidants, preservatives and binding agents	Gatlin (2003), Maqsood et al. (2013), Encarnação (2016), Yadav et al. (2021)

Aquaculture practices are increasingly prioritizing the development of affordable alternatives to fish meal derived from terrestrial animal and plant-based protein sources, driven by economic and sustainability considerations (National Research Council, 1993; Millamena, 2002).Common sources of nutrients for fish feeds include; fish meal, bagasse, shrimp meal, brewery waste, squid meal, brine shrimp, soybean meal, spirulina and groundnut cake meal (Kari et al., 2023; Kolawole and Mustapha, 2023).

Fishmeal made from waste fish and wild fish is a common source of protein in fish feed formulations. The meals consisted of amino acids, minerals, vitamins and omega-3 fatty acids. There has been concern of the exploitation of wild fish combined with the high price of fish meal (Yohana et al., 2023).

2.7.2 Groundnut oil processing waste

Generally, some groundnut oil processing waste may naturally contain some elements as part of their composition. Table 1.2 (a) and (b) shows general reported proximate and elemental analysis of groundnuts for nutritional evaluation and a fish leaf meal, which was used in a typical fish feed formulation incorporating fish meal, corn starch, fish oil, soy oil, mineral premix and vitamin premix.

Table 2.6 (a): Proximate analysis of ground nuts

	Groundnuts
Proximate Composition (% dry weight)	
Crude Protein	38.61
Crude Fat	47
Carbohydrate	1.81
Crude Fiber	3.70
Ash	3.08
Moisture	5.80

Sources: (Garduño-Lugo and Olvera-Novoa, 2008a; Atasie et al., 2009)

Table 2.6 (b): Elemental analysis of ground nuts

Elemental Composition (mg/kg)	**Groundnuts**
Iron (Fe)	6.97±1.62
Zinc (Zn)	3.20±0.11
Calcium (Ca)	2.28±1.94
Magnesium (Mg)	3.98±0.04
Sodium (Na)	42.00±0.71
Potassium (K)	705.11±0.86
Phosphorus (P)	10.55±0.68

Sources: (Garduño-Lugo and Olvera-Novoa, 2008a; Atasie et al., 2009)

Depending on the processing method, the groundnut oilcake can have limitations due to the potential for aflatoxin contamination, but these can be mitigated by fermentation and good hygiene storage conditions (Waliyar et al., 2013).

2.7.3 Antinutrients

Plant based raw materials may contain naturally occurring antinutrients that can negatively impact fish performance. According to Abbas (2020) antinutrients are substances which work to decrease nutrient ingestion, digestion, absorption and/or consumption and may cause other undesirable effects (Abbas, 2020). Antinutrients include phytic acid, trypsin inhibitors and pyrrolizidine alkaloids (Francis et al., 2001). The following is a synopsis of the aforementioned antinutrients:

a) Plant-derived feedstuffs may contain phosphorus primarily in the form of phytic acid. Phytates, which act as potent chelators, form complexes with proteins and essential minerals such as zinc, calcium, magnesium, and iron, thereby reducing their bioavailability and necessitating supplementation to ensure adequate nutrients (Manikandan et al., 2020; Ma et al., 2024). Groundnuts processing oil waste has potential of having phytin antinutrients (Francis et al., 2001).

b) Raw soybeans contain trypsin inhibitors, which are crystalline globular proteins. While thermal processing effectively inactivates these antinutritional factors, excessive heat application may compromise the bioavailability of specific amino acids, notably lysine (Liener, 1994; Vagadia et al., 2017; Kohli and Singha, 2024)
c) Pyrrolizidine alkaloids and known hepatotoxins present in certain plants produce toxic pyrrole compounds when metabolized by hepatic enzymes. However, these alkaloids are not typically prevalent in the majority of plant species commonly utilized in fish feed formulations, unless they are introduced through contamination(Watson, 1998; Pan et al., 2023) .

Several detoxification techniques, including postharvest processing, chemical detoxification, leaching (low molecular weight compounds), thermal treatment/decortication (oxalates), thermal inactivation (lectins, protease inhibitors in legumes), and genetic modification, reduce the effects of antinutritional factors (Abbas, 2020).

2.7.4 Categories and processing of fish feeds

Fish feeds can be categorized based on preparation method, structure and on the basis of nature. On preparation these are defined by the method used to combine the ingredient for example dry feed prepared by baking to produce granules and medicated feed prepared by including active pharmaceutical ingredients (Ranjan, 2017; Onomu and Okuthe, 2024). The basis of the structure is the shape and form of the resulting prototype for example the most common pellets are ground, mixed and extruded as pellets of various sizes. A formulation that is nature based, refers to those characteristics displayed by the feed as applied to the target environment (Joshi et al., 2021). Some pellets are made to float and some to sink (Forte and Young, 2018).

During the formulation of fish feeds the pelleting qualities of the mixtures are important similar to their nutritional value (Sørensen, 2012; Hardy and Brezas, 2022). Factors such as cost and stability are important. It is cost-effective in terms of manufacturing, shipping, storage, and delivery to the fish. The stable pellets should then be stable in water until they are consumed by the fish without leaching (Hardy and Barrows, 2003; Mohamed et al., 2023).

The processing of fish feeds requires specific processing techniques to achieve physical characteristics that improve feeding in water and the differences in feeding between different species (National Research Council, 1993). In fish farming, the interaction between the aquatic environment and the prepared feed may pose unique difficulties not found in the nutritional management of land animals (Sørensen, 2012). According to Hemre et al. (2002) and Liu et al. (2022) certain species of fish require feeds with special properties such as feeds with a soft texture (Liu et al., 2021). The preferred physical properties of pellets include hardness, size of the pellet, and the pellet durability index (Wan Nooraida et al., 2023). Fish feeds are also classified into life cycle stages including starter feeds, fry feeds, fingerling feeds, grow-out feeds and broodstock feeds (Hervé et al., 2024). The basic production of manufacturing fish feeds requires grinders, mixers, elevators, cookers or extruders, coolers and steam boilers (Silva De and Anderson, 1995; Robinson et al., 2001).

2.7.5 Types of fish feeds currently on the market and their composition in Zimbabwe

Nile tilapia (*Oreochromis niloticus*) followed by rainbow trout (*Oncorhynchus mykiss*) are common in Zimbabwean aquaculture (Mabika and Utete, 2024) . This is due to their greater growth performance, demand, and economic benefits than those of other species (Mabika and Utete, 2024). Novatek is one of the companies based in the SADC region. Novatek produces fish feeds using the extrusion processes. They produce different products including fry mash, starter pellets, and grower pellets. Key raw materials used by the company for feed formulation are imported fishmeal, bone meal, and soy-based additives (Musuka et al., 2023). Another popular company is Profeeds which, through joint ventures with Aquafeeds produces tilapia feeds. Tilapia feeds similar to those of Novatek are produced through extrusion. The feeds are fortified with OMEGA-3 fatty acids, which include fiber, fatty acids, minerals, vitamins, and amino acids. Their products include; Juvenile 1, grower, broodstock and pond.

2.7.6 Trends in the production of fish feeds

Fish farming is an industry that has expanded globally and is being considered an alternative source of income even for small-scall farming (de Barros et al., 2025). This expansion is based on its role in addressing the global concern of the ever-increasing population. An increase in population calls for high demand for food, employment and economic development (Alobaidy and Jbara, 2021; Konstantinidis et al., 2021; Willer et al., 2022). Fish farming as a subsector of agriculture contributes immensely to the development of the national economy (Taufikurahman et al., 2024). The Food and Agriculture Organisation (FAO) postulated that although there is increase in demand of fish there is still need to improve food and nutritional security. A major challenge facing most fish farmers especially smallholder farmers is the inadequate availability of high-quality feeds. Among other production expenses for aquaculture, fish feed is the most critical and costly input (Klon et al., 2023).

2.8.0 Characterization of Fish Feeds

Characterizing fish feeds is important for ensuring that the feed contributes to optimal fish health, and performance, and has minimal environmental impact. Characterisation includes proximate analysis and elemental composition analysis.

2.8.1 Proximate analysis

Proximate analysis is the macro quantitative analysis of molecules in food (Cortés-Herrera et al., 2021). According to Hynd (2019), proximate analysis refers to 'proxies' for the actual entities of chemical entities being defined, it describes the feeds in terms of nutrient proportion relative to the adequate amounts required by the animal (Hynd, 2019). Proximate analysis includes fractions in mg/kg of dry matter or standardized assays of , crude protein, crude fiber, lipid, ash and digestible carbohydrates (Hynd, 2019; Mahmud et al., 2012). Components determined by proximate analysis are important in the food industry for innovation, quality control and regulatory compliance (Thangaraj, 2016).

The nutritional assessment of feed formulations prioritizes protein content, which is subsequently balanced with dietary energy to achieve optimal ratios. The following is the typical range of proximate compositions of fish feeds:

Table 2.7: Typical proximate composition of the fish feeds

Component	**Average Composition (%)**
Crude protein	35 – 70
Crude fibre	1.0 – 15
Crude lipid	4.0 – 30
Ash content	4.0 – 35
Moisture	5.0 – 15

Sources: (Adewolu et al., 2010; Khan et al., 2012; Ayuba and Iorkohol, 2012; Yen et al., 2015; Asimi et al., 2018; Isah et al., 2021;; Mishra et al., 2023; Omasaki et al., 2024;)

Crude protein quantification is a routine analytical procedure in food science (Hasan et al., 2014). According to Jia et al., (2023) moisture, crude protein, crude fat, and crude ash are the most common indicators of the quality of feed products (Jia et al., 2023).

2.8.2. Classical methods used for proximate analysis

The classical methods are based on the Association of Official Analytical Chemists (A.O.A.C) standard methods for dry matter, ash content, crude fat, crude fiber, and proteins (Horwitz, 2005; Suffo Kamela et al., 2016). These are traditionally analyzed by Kjeldahl and oven methods (Torquato et al., 2017). Moisture is estimated gravimetrically through sample drying at elevated temperatures (Horwitz, 2005; Raugel, 1999). Ash determination involves sample incineration at 600–700 °C, with the resulting residue quantified as ash (Masum Billah et al., 2018; Tacon et al., 2009). Lipid content is determined quantitatively by solvent extraction using for example chloroform-methanol (2:1) extraction, followed by solvent removal and gravimetric assessment of the extracted lipids (Asimi et al., 2018; Sarker et al., 2022). The protein content of each sample was determined by the Micro-Kjeldahl method (Raugel, 1999; Nalwanga et al., 2009). The crude fiber content is determined by stages which include; digesting a ground sample with hot acid and then hot alkali. Then the remaining residue is dried, weighed, and then incinerated. The weight lost during incineration represents the crude fiber content (Nayeem et al., 2019).

2.8.3 Essential elements for fish nutrition

Most of the elements on the periodic table are classified as metals, of which only a few are considered essential for living organisms because of their physiochemical functions in living organisms (Remick and Helmann, 2023). According to Chanda et.al (2015),essential elements are dietary nutrients required because their absence or insufficiency in the diet causes deficiency syndromes, and in some instances, supplementation may be necessary to restore normal health (Chanda et al., 2015). Aquatic organisms universally require the essential inorganic elements for normal growth and metabolism (Chanda et al., 2015). Table 2.7 shows the mineral requirements per kg of dry matter.

Table 2.8: Typical essential element composition of fish feeds

Mineral	**Dietary Requirement (mg/kg)**	**Physiological/Biochemical Functions**	**Fish Species Studied**	**Source of Mineral**
Cu	3–12	Superoxide dismutase, cytochrome c oxidase, melanin production	Rainbow trout, Coho salmon, Channel catfish	Copper sulfate, fish meal, plant proteins (limited by phytate)
Fe	30–170	Hemoglobin synthesis, oxygen transport, electron transfer	Rainbow trout, Hybrid tilapia, Common carp	Ferrous sulfate, ferric citrate, fish meal, blood meal
Zn	20–202	Enzyme systems (alkaline phosphatase), immune function, wound healing	Atlantic salmon, Rainbow trout, Hybrid tilapia	Zinc oxide, nano-ZnO, fish meal, plant proteins (phytate-bound)
Se	0.15–0.3	Glutathione peroxidase, thyroid hormone metabolism	Rainbow trout, Coho salmon, Atlantic salmon	Sel-Plex, fish meal, marine ingredients
Ca	5–7 g/kg (FW species)	Bone formation, blood clotting, nerve transmission	Tilapia, Catfish, Salmonids	calcium carbonate, fish bone meal
P	5–8 g/kg	ATP synthesis, nucleic acids, bone mineralization	Rainbow trout, Common carp	Monocalcium phosphate, fish meal, dicalcium phosphate
Mg	400–700	Enzyme activation, osmoregulation, ATP metabolism	Rainbow trout, Atlantic salmon	Magnesium oxide, dolomite, plant ingredients
Mn	2.5–15	Antioxidant systems, bone development, reproduction	Channel catfish, Common carp	Reef Factory Manganese, plant ingredients, fish meal
I	0.5–1.5	Thyroid hormone synthesis	Salmonids, Tilapia	Potassium iodide, marine algae, fish meal

(Tacon et al., 2009; Saha, Abdul Mottalib, et al., 2020; Lall and Kaushik, 2021a; Lall and Dumas, 2022)

Compared with terrestrial animals, fish can absorb inorganic elements from both the diet and the surrounding aquatic environment (Hilton, 1989). According to Terech-Majewska et al., (2016) elements such as Ca, Mg, Na, and K can be acquired by fish through aqueous uptake depending on the composition of the aquatic environment. However, phosphates and sulfates are predominantly derived from the diet (Terech-Majewska et al., 2016) . Regulatory mechanisms associated with fish anatomy govern mineral uptake, storage, and excretion, enabling fish to maintain equilibrium with their aquatic habitat. Ions such as Na^+, K^+ and Mg^{2+} are crucial for osmotic and ionic regulation within fish fluids (National Research Council, 1993).

2.9. Examples of innovative applications of tailings aligned with circular economy initiatives

Several studies have reported more on the use of tailings in road surfacing and geopolymers which include for example; the manufacture of bricks (Chinyama, Wenga and Gwenzi, 2023), road construction (Segui et al., 2023), and the synthesis of geopolymers (Mabroum, 2020). Adiguzel, Tuylu and Eker (2022) reviewed the utilization of tailings in concrete products and iterated that tailings have been investigated and shown potential use as concrete. Manjarrez and Zhang (2018) investigated copper mine tailings for potential use in road surfacing through geopolymerization. Zinc tailings have been studied for utilization as cement substitutes in concrete. The results led to the conclusion that this approach is feasible (Agrawal, 2022). In addition soil rehabilitation and the manufacturing of fertilizers for example; abandoned coal mine waste utilized in greenhouse farming by Technosols (Walmsley et al., 2022) and evidence on the potential of mine waste to support biodiversity was investigated (Ouédraogo et al., 2024). Sigvardsen (2022) , reported the use of mine tailings as a replacement for cement (Sigvardsen et al., 2017).

Mining waste is evolving into valuable raw materials for other production sectors. Sectors which include construction, glass manufacturing, synthesis of zeolites and recovery of critical elements. Two forms of glass were formulated from fluorite mine tailings (Alfonso et al., 2020). Copper mining tailings with a high composition of carbonate-associated elements were utilized in a case study to neutralize extremely acidic lake water. In their study after treatment of tailings, NaOH was applied to remove residual Mn and Ag (Petronijević et al., 2020).

2.10 Summary

The literature reported no link between mine tailings production trends by the mining sector and aquaculture nutritional demands. As the mining sector produces around 3.5 Gt/year of mine tailings and agro-processing industries generate enormous amounts of organic waste streams, the products are yet to be comprehensively explored for aquaculture utilization despite being extremely nutritious for fish feeding.

There are no established procedures exist for tailings characterization for nutritional content, and their usability as feed components. Platinum tailings have diverse elements required for fish nutrition such as Fe, Zn, Mn, and Cu). There is little critical debate on the topic of bioavailability, the key factor affecting nutritional value. Much reported Work is conducted on the basis of overall elemental composition rather than bioavailable fractions.

Groundnut oil production is linked with substantial waste in the forms of hulls, meal, and shell waste. Groundnut meal is generally 45-50% crude protein and therefore can be a quality source of protein. It does contain antinutrients such as phytic acid that can chelate dietary minerals and lower their bioavailability. Regulated phytic acid content in groundnut waste might facilitate mineral complexation, thereby possibly enhancing mineral stability and bioavailability.

Current uses of tailings in circular economy are mainly in construction products and not in biological processes. The suggested integration is an innovative way of realizing circular economy through creating sources of nutrients among metallurgical processing industries and agriculture, reduction of reliance on environment-harming traditional feed supplies and value creation from waste streams

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CHAPTER 3

MATERIALS AND METHODS

3.0 Introduction

This chapter describes the experimental procedures used to investigate the extraction of essential elements from platinum mineral tailings and their utilization in the formulation of fish feed pellets. Materials and experimental procedures are outlined for (1) collection, sample preparation and characterization of platinum mineral tailings, (2) pretreatment of mineral tailings to remove toxic elements (3) extraction of essential elements from platinum mineral tailings and (4) subsequent incorporation with groundnut oil processing waste as a binder to formulate mineral-enriched fish feed pellets.

3.1 Materials, instruments, chemicals, and reagents

Details of reagents, chemicals and materials used in this study are presented (Table 3.1) for extraction of elements from platinum tailings and for calibration of XRF , XRD and ICP-OES.

Table 3.1: List of materials, chemicals, and reagents used

Name	**Formula**	**Supplier**	**Purity/Conc.**
Deionized Water	H_2O	In–house	
Acetic Acid	CH_3COOH	Sigma–Aldrich	≥99.7%
Citric Acid	$C_6H_8O_7$	Sigma–Aldrich	≥99.5%
Hydrochloric Acid	HCl	Merck	37% w/w
Sulfuric Acid	H_2SO_4	Merck	98% w/w
Nitric Acid	HNO_3	Merck	65% w/w
NIST standard reference material 2711a	various elements	Sigma- Aldrich	–
NIST Standard reference material 640G	Si	Sigma- Aldrich	99.9%
Sodium Hydroxide	NaOH	Sigma Aldrich	97 %
Sodium hydrogen carbonate	$NaHCO_3$	Merck	99%
Whatman Filter Paper No. 41	-	Cytiva	-
Yttrium Oxide (Internal Std)	Y_2O_3	Sigma–Aldrich	99.99%
ICP Multi-Element Std. IV	-	Merck	1000 mg/L
Groundnut Oil Processing Waste	-	Local supplier	-

Table 3.2 shows instruments used for sample preparation and characterization of raw platinum tailings, leachates, residues of platinum mineral tailings. Then for proximate and elemental studies of the formulated fish feed pellets.

Table 3.2: Instruments used for sample preparation, characterization and analysis

Instrument	**Make and Model**	**Key Parameters**
X-ray Fluorescence (XRF)	Olympus Innov X, USA	4 kW, Rh anode, 20–60 kV
X-ray Diffraction (XRD)	BTX, Japan	Cu Kα, 30 kV, 40 mA, 0–80° 2θ
ICP-OES	Thermo Fisher iCAP 7000, USA	Qtegra software, axial view
Orbital Shaker	Cole-Parmer, Ireland	30–300 rpm
Centrifuge	BIOBASE, China	4000 rpm, 20 min, 4°C
Magnetic Stirrer/Hotplate	IKA C-MAG HS 7	Variable, up to 85°C
Ballmill	Retsch, German	Particle size < 75µm
Laboratory Oven	UCHEN	100-200°C
Grain Moisture meter	BMCA-1G, Infitek, China	3 – 35%

3.2 Platinum mineral tailings sample collection and preparation

Platinum mineral tailings were collected using a simple random sampling technique from the surface of the tailings dam of the Ngezi platinum mine, Zimbabwe. The sampling area was demarcated under the guidance of the accessible surface of the tailings dam. Inside this demarcated area, three sampling points were chosen randomly. At every chosen point, a scoop of tailings slurry was collected from approximately 10 cm layer below the surface for the top layer sample and 1 meter below the surface using a stainless-steel container. A total of six subsamples were mixed well to provide approximately 10 kg of composite sample and were packed in a stainless steel 20 liter size container. The composites, which were in slurry form containing fine-grained solids, were washed with 20 L of distilled water to remove all surface contaminants as well as soluble salts, followed by air drying at room temperature for approximately 5 days. After drying, the sample was ground with a Retsch planetary ball mill to achieve a grading below 75 µm. The powdered sample was dried in a UCHEN oven at 100°C for 24 hours to remove any residual moisture and thereafter stored in a desiccator (Cole-Parmer, USA) containing silica gel.

3.3 Characterization of the platinum mineral tailings

The main characterisation techniques used for platinum mineral tailing were XRF, XRD and ICP-OES, to determine different parameters as discussed in subsequent sections.

3.3.1 Characterization using X-ray Fluorescence

A hydraulic press set to 10 tons for three minutes was used to prepare a pellet by combining a mass of 5.00 g of dried, powdered platinum tailings with 0.25 g of analytical-grade starch, which formed 13 mm-diameter tailings pellets for elemental analysis. A mounted Olympus Innov-X XRF spectrum analyzer was used to perform the analysis at room temperature. The standard reference material 2711a was used for calibration.

3.4 Alkaline pretreatment of tailings to remove toxic elements

The method of Ginter-Kramarczyk et al. (Kostrzewa et al., 2022) was adopted with some change: CaO was not used for pH adjustment, for the treatment of toxic elements from platinum mineral tailings. A mass of 50 g of tailings was combined with a solution of 1 M NaOH and 0.5 M $NaHCO_3$. The pH was adjusted to 8.5 ± 0.1 using a pH meter (Brbooti et al., 2011; Kostrzewa et al., 2022; Rozaimah et al., 1999). The alkaline mixture was stirred continuously at 200 rpm for 2 hours at room temperature (23 ± 2°C) (Kostrzewa et al., 2022). After reaching equilibrium, the solution was allowed to settle for 1 hour to encourage the maturation and agglomeration of the precipitate (Brbooti et al., 2011; Kostrzewa et al., 2022). After alkali treatment, the resulting solution was separated by centrifugation at 3000 rpm for 15 minutes, after which the supernatant was analyzed using ICP-OES. The residue from platinum mineral tailings was subsequently sonicated at approximately 40 kHz and 150 W for 15 to 30 minutes, and maintained at room temperature (23 ± 2°C). Thereafter, the mixture was stirred with a magnetic stirrer at 300 rpm for 20 minutes to achieve uniformity. The resulting slurry was then separated through centrifugation, and the washing process was repeated two to three times until the pH of the filtrate approached neutral Then the residue was dried at 60°C, to prepare for the leaching of essential elements.

3.5 Leaching of essential elements

Comparative leaching using different acids was carried out with the inorganic acids hydrochloric acid (HCl) and sulfuric acid (H_2SO_4), organic acids, citric acid ($C_6H_8O_7$) and acetic acid (CH_3COOH). The fifth leaching solution was made up of a 1:1 ratio of citric acid to acetic acid, the 1:1 ratio was adopted from literature (Nagarajan and Panchatcharam, 2023). Different acids: HCl, H_2SO_4 , $C_6H_8O_7$ and CH_3COOH were used to determine the acid or acid mixture with the highest leaching capacity, as previously reported (Nagarajan and Panchatcharam, 2023; Xiao et al., 2022; Yan et al., 2014). All the leaching solutions were prepared with modification of the volume to tailings proportion; a volume of 100 ml was combined with 5 g of the platinum tailings (Kumkrong et al., 2022; Schueler et al., 2021). A mixture of $C_6H_8O_7$ and CH_3COOH was combined at a 1:1 volumetric ratio. The agitated solution was filtered using centrifugation at the respective rpm. The experimental procedure is shown in Table 3.3.

Table 3.3: Experimental procedure with inorganic and organic acids

Mass of tailings/g	**Acids**	**Initial pH**	**Concentration (M)**	**Temperature (°C)**	**Mixing rate (rpm)**	**Reaction time (hrs)**
5 g	HCl	1.1	0.1	r.t.p. (23±2)	250	48
	H_2SO_4	1.2				
	CH_3COOH	2.8				
	$C_6H_8O_7$	2.3				
	CH_3COOH + $C_6H_8O_7$	2.6				

The filtrate was stored at a temperature of 5 °C to avoid any potential further chemical reactions before ICP–OES analysis. Figure 3.1 shows a summary of the sequential leaching process. A mixture of 1:1 acetic acid (CH_3COOH) and citric acid ($C_6H_8O_7$), which had high leachate concentration recovery, was used for process optimization.

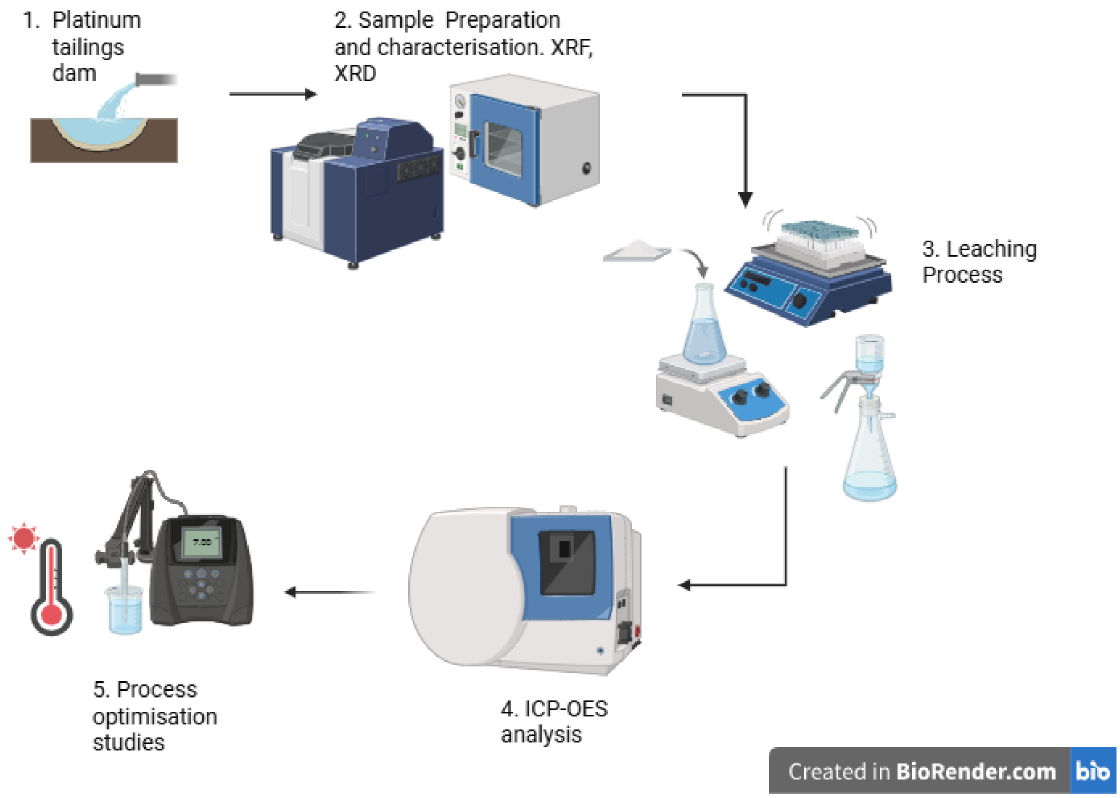


Figure 3.1: Steps of the leaching process to recover essential elements created using Biorender.com online

In preparation for optimization to establish possible factors, a basic leaching was carried out by adopting conditions reported by Yogurtcuoglu (2023) and Yogurtcuoglu (2024) with modifications; concentration of 0.5 M, temperature at 60 °C for a contact time of 1 hour (Yogurtcuoglu, 2024; Yoğurtcuoğlu, 2023) with a pH of 2 (Geng et al., 2020).

3.5.1 RSM Optimization of leaching process using organic acid mixture of acetic acid and citric acid

A mixture with a 1:1 ratio of acetic acid, to citric acid was used to recover essential elements from platinum mineral tailings through a response surface methodology (RSM). The Box–Behnken design was used to optimize four independent variables.

The low (-1) and high (+1) values were selected on the based on the literature and the preliminary leaching experimental data as shown in Table 3.3. The independent factors in this study were pH

(2.0–9.0), contact time (30–2880 minutes), temperature (30–90°C), and leaching reagent concentration (0.1–0.5 M). These four factors will be assigned as A, B, C, and D, respectively.

Table 3.4: Independent variable experimental ranges for optimization

Factor	**Variable**	**Low (-1)**	**High (+1)**
A	pH	2	9.0
B	Contact time (minutes)	30	2880
C	Temperature (°C)	30	90
D	Acid concentration (M)	0.1	0.5

The leaching experiments were carried out based on an RSM, using the Box-Behnken experimental design. A total of 29 experimental runs were generated with the four variable factors. The expected responses targeted the highest element recovery, while the process variables were pH, contact time, temperature and acid concentration. The mathematical models were obtained in terms of coded and actual values. The experiments were performed randomly to avoid systematic error. Twenty-nine (29) runs were carried out for the leaching as shown in Table 3.5 for the factors (Table 3.4) and for responses of elemental composition of: Ca, Cu, Fe, K, Mg, Mn, Na and Zn

Table 3.5: Process optimization experimental design for the 29 runs

Run	pH	Contact time	Temperature	Acid Concentration
	H+	hours	°C	M
1	5	3	70	0.3
2	5	3	75	0.1
3	2	3	70	0.5
4	2	5	70	0.3
5	5	3	75	0.5
6	5	1	75	0.3
7	5	5	65	0.3
8	2	3	75	0.3
9	8	3	70	0.5
10	5	1	70	0.1
11	5	3	70	0.3
12	8	3	70	0.1
13	5	3	65	0.5
14	5	5	75	0.3
15	8	5	70	0.3
16	5	1	70	0.5
17	5	3	65	0.1
18	5	5	70	0.5
19	8	1	70	0.3
20	8	3	65	0.3
21	5	3	70	0.3
22	5	3	70	0.3
23	8	3	75	0.3
24	5	3	70	0.3
25	2	1	70	0.3
26	2	3	70	0.1
27	5	5	70	0.1
28	2	3	65	0.3
29	5	1	65	0.3

The RSM experiments were each carried out as follows: A 100 mL Pyrex borosilicate glass reactor was filled with 5.00 g of platinum mineral tailings for each experiment. The reactor was loaded with 100 ml of the acid mixture and stirred with a Teflon-coated stirrer bar at 250 rpm whilst placed in an oil bath, at 65°C and 70°C as per Table 3.5. The reactor was removed from the oil bath and allowed to cool to room temperature after each contact time.

3.5.2 Analysis of leachates of suggested RSM runs

An inductively coupled plasma optical emission spectrometer (ICP–OES), model iCAP 7000 series (Thermo Fisher Scientific, Massachusetts, USA) with Qtegra software, was used to measure the concentrations of metal elements in the leachate samples. The leaching efficiency for each metal was calculated as according to equation (3.1):

$$\text{Leaching Efficiency (\%)} = \frac{C_{\text{leachate}} \times V}{M_{\text{tailings}} \times C_{\text{initial}}} \times 100 \qquad (3.1)$$

where:

$-C_{\text{leachate}}$ = metal concentration in leachate (mg/L)

$-V$ = volume of leachate (L)

$-M_{\text{tailings}}$ = mass of tailings (g)

$-C_{\text{initial}}$ = initial metal content in tailings (mg/g)

3.5.3 Analysis of the results of actual experiments

The experimental data were fitted to a second-order polynomial model:

$$Y = \beta_0 + \sum_{i=1}^{4} \beta_i X_i + \sum_{i=1}^{4} \beta_{ii} X_i^2 + \sum_{i=1}^{3} \sum_{j=i+1}^{4} \beta_{ij} X_i X_j + \varepsilon \qquad (3.2)$$

From equation (3.2), Y is the leaching efficiency, X_i are the coded factors, and β are regression coefficients. Analysis of variance (ANOVA) was used to assess model significance and fit. Response surface and contour plots were generated to visualize the effects and interactions of the variables. Multi-response optimization using desirability functions was applied to identify conditions maximizing the simultaneous extraction of all target metals (Bai et al., 2018; Hamza et al., 2022; Kolbadinejad and Ghaemi, 2024).

3.6 Preparation of groundnut oil processing waste

The groundnut oil processing waste was bought from a small-scale groundnut oil processing plant. Groundnut oil processing waste was chosen as it possesses inherent binding properties due to its starch content, protein structure, and oil residues (Nguyen et al., 2022).The samples were washed, sun dried, and ground using a pulverizer after being obtained from a processing unit, as shown in Figure 3.2.

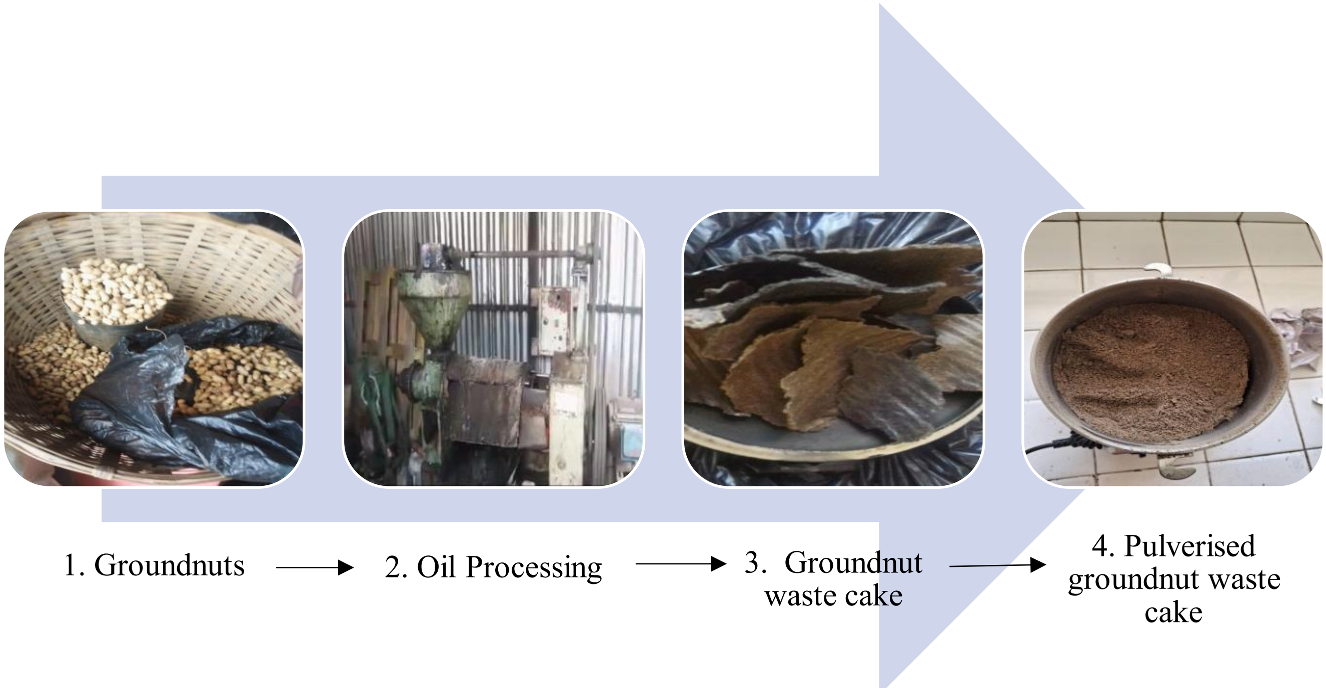


Figure 3.2: Process flow of the generation of groundnut oil processing waste

The groundnut oil processing waste was analyzed for moisture content using a BMCA-1G moisture detector meter (Inteck, China) with measurement range of 3 – 35% and ≤ ±0.5% measurement error.

3.6.1 Proximate analysis of groundnut oil processing waste

The groundnut processing oil waste was characterized systematically using proximate analysis according to the AOAC official methods: moisture content by oven drying at 105 °C during 24 hours (AOAC 930.15), crude protein through the Kjeldahl method (AOAC 984.13), crude fat with the ether extraction (AOAC 991.36), ash content with the muffle furnace incineration during 550 °C (AOAC 942.05), and carbohydrates by calculation using equation (3.3):

Carbohydrates (%) = (100% - (% Moisture + % Protein + % Fat + % Ash + % Fiber) (3.3)

3.6.2 Elemental analysis of groundnut oil processing waste

ICP-OES was used to analyze the elemental composition of the groundnut oil processing waste. Each sample was mixed with a volume of 7 ml of concentrated nitric acid and 1 ml of hydrogen peroxide and digested at 180 °C for 15 min. A mass of 0.5 g of sample was used. The digested products obtained were diluted to 50 ml of deionised water, and they were filtered.

3.7 Formulation of fish feed

The formulation was carried out in two steps first the ingredients were fermented then next the ingredients were extruded.

3.7.1 Fermentation of each feed formulation

The two ingredients: groundnut cake and the solution with extracted elements were combined. After mixing ingredients for each formulation, the mixture was fermented to enhance nutrient bioavailability and reduce potential antinutritional factors. Each feed formulation was combined with 0.5 g of yeast incubated at 30°C for 6 hours.

3.7.2 Feed formulation optimization

The RSM was applied using the Box-Behnken. A total of 17 experimental runs were generated with three variable factors (Oghenerukevwe et al., 2023). The process variables were the percentage composition of the moisture content, volume of mineral solution, and groundnut waste. The mathematical models were obtained in terms of coded and actual values. The models in terms of coded and actual factors were used to make predictions about the response for given levels of each factor. The high-level factors were coded as +1, and the low-level factors were coded as -1 (Table 3.6). The high and low values were obtained from the consulted from literature with modifications. The experiments were performed randomly to avoid systematic error. The optimum inhibition parameters were also obtained.

Table 3.6: Independent variable experimental for the feed formulation

Factors	Low (–1)	High (+1)
Mineral Solution (ml)	15	60
Groundnut oil processing Waste (g)	10	40
Moisture Content (%)	10	15

Seventeen runs (17) were carried out for the pellet formulation as shown in Table 3.7 for the factors in Table 3.6 for responses of proximate and elemental composition: Carbohydrate, Protein, Lipid, Ca, Cu, Fe, K, Mg, Na, Mn and Zn.

Table 3.7: Experimental formulation percentages for the 17 runs

Run	Mineral Solution	Groundnut oil processing Waste	Moisture Content
	ml	g	%
1	60	40	12.5
2	60	25	15
3	60	10	12.5
4	37.5	40	10
5	15	10	12.5
6	37.5	40	15
7	60	25	10
8	37.5	25	12.5
9	37.5	25	12.5
10	15	25	10
11	37.5	25	12.5
12	37.5	25	12.5
13	37.5	10	15
14	37.5	10	10
15	15	25	15
16	15	40	12.5
17	37.5	25	12.5

Fish feed pellets in this study were prepared with the use of a microextruder. The dry raw materials were subsequently dried in an oven to vary the moisture content as per RSM suggested runs to prepare groundnut waste samples with 10% - 15% moisture content before mixing with the mineral solution. The volume of the mineral solution was added, and the resultant paste was heated at 50 °C for 5 minutes. The hot paste was then transferred to a single screw laboratory microextruder, and a knife cut the extrudate into pellets. Pellets were dried in an oven containing hot air at 60 °C to a point where the moisture level was less than 10 % and then cooled to room temperature and stored in a dessicator.

3.7.3 Proximate analysis of the fish feed pellets using AOAC methods

Proximate analysis of carbohydrate, crude fat, protein, fiber, and moisture was performed using the Association of Official Analytical Chemists (AOAC) International for determining the moisture, fat, ash, crude protein, and crude fiber contents of the feed sample. The moisture content was determined by drying 2.0 g of ground pellets at 105 °C to a constant mass as per AOAC 934.01. The crude protein content was determined using the Kjeldahl method, with the nitrogen content converted to protein content by a factor of 6.25 per AOAC 984.13. Crude fat was extracted via a Soxhlet apparatus with petroleum ether over 3 hours. Crude fiber AOAC 962.09 was assessed through sequential acid (1.25% H_2SO_4) and alkali (1.25% NaOH) digestion, followed by gravimetric correction for ash. The total carbohydrate content was calculated using equation (3.3). The ash content was measured via the AOAC 942.05 method by incinerating samples at 550°C for 6 hours in a muffle furnace.

3.7.4 Elemental analysis of the fish feed pellets

The pellets were analyzed by ICP-OES to determine the content of Ca, Cu, Fe, K, Mg, Na, Mn, and Zn in the fish feed pellets using the methodology as per section 3.5.2.

3.7.5 X-ray Diffraction (XRD)

XRD was conducted to determine the crystalline phases of the tailings. A BTX benchtop XRD instrument with X-powder 12 software was used. The instrument parameters are presented in Table 3.2. The XRD instrument was calibrated with a silicon standard, NIST SRM 640G. The analysis was conducted by loading 1.0 g of the powdered sample into the sample holder and scanning it over a 0–80°, 2θ range.

3.7.6 Physical characteristics of the formulated pellets

Three tests were carried out via the following methods with slight modifications: nutrient leaching test and floatability tests. Floatability test and nutrient leaching test are very important when formulating fish feed to know the quality of the feed and also its efficiency (Felix and Oscar, 2018). Nutrient leaching tests evaluate the proportion of nutrients in a feed that can dissolve in water, and floatability tests the duration that feed pellets can remain above water, which affect feeding efficiency and reduces wastes (Zaabwe et al., 2020).

3.7.6.1 Nutrient leaching test

Three pellets were soaked in 250 ml of distilled water for one hour. After that, the water samples were analyzed using ICP-OES to measure any potential released elements among Ca, Cu, Fe, K, Mg, Na, Mn, and Zn (Saalah et al., 2010).

3.7.6.2 Floatability test

Floating experiments were performed to assess the buoyancy properties of fish feed pellets made from groundnut oil processing waste mixed with mineral tailings solution. A total of 20 pellets with 11% moisture content were randomly chosen and carefully placed into a 1000 ml glass beaker filled with a volume of 800 ml of distilled water at room temperature. The floating behaviour of the pellets was monitored over a total duration of 15 minutes, with the number of floating pellets recorded at intervals of 5 minutes. This method was repeated five times. The mean percentage of floating pellets was calculated using equation (3.4) the formula adapted from Solomon et al. (2011):

$$\% \text{ Floating Pellets} = \left(\frac{\text{Number of Floating Pellets at Time } t}{\text{Total Number of Pellets}}\right) \times 100 \quad (3.4)$$

(Solomon et al., 2011).

CHAPTER 4

RESULTS AND DISCUSSION

4.0 Introduction

In this chapter the results and discussion are presented for the methodology carried out for (1) platinum mineral tailings characterization, then toxic element pretreatment and extraction and optimization of the essential elements, (2) sample preparation of the groundnut oil processing waste, proximate and elemental analysis and (3) formulation, optimization and extrusion using a combination of the mineral solution obtained from platinum mineral tailings with groundnut processing waste.

4.1 Characterization using X-ray Fluorescence

The results of the XRF analysis of the raw platinum mineral tailings are shown as oxides in Table 4.1:

Table 4.1. Chemical composition of raw platinum mineral tailings samples

Chemical	**Percentage composition (%)**
Fe_2O_3	19.611
SiO_2	50.811
Al_2O_3	2.490
CaO	11.136
MgO	13.376
Cr_2O_3	2.576
Total	100

Shown in Table 4.1, is the oxide composition of the raw platinum mineral tailings. This was carried out to find out the elemental composition of the tailings so as to pave way for extraction eperiments and to reveal the availability of potential elements relevant for fish feed formulation. The mineralogical composition of the platinum mineral tailings agrees with that reported in the literature. These oxides are related to the mineral abundance, which includes forsterite

($(Mg,Fe)_2SiO_4$), plagioclase ($NaAlSi_3O_8$–$CaAl_2Si_2O_8$), and chromite ($FeCr_2O_4$) (Baloyi et al., 2023; Kilbride et al., 2006; S. Mohamed et al., 2019; Pamato et al., 2019; J. Pan et al., 2021; Sixhuta et al., 2024).

4.2 Alkaline pretreatment of raw platinum mineral tailings

The raw platinum tailings were treated with NaOH (1 M) and $NaHCO_3$ (0.5 M). The alkaline treatment with 1 M NaOH and 0.5 M $NaHCO_3$ systems resulted in different levels of composition of various elements in the raw platinum mineral tailings. Table 4.2 shows the calculated removal efficiencies for each element.

Table 4.2: Extraction efficiency of toxic elements by alkaline treatment prior to extraction of essential elements.

Element	Raw Platinum mineral tailings (mg/L)	Mineral solution After Treatment (mg/L)	Leaching Efficiency (%)
Al	105.66 ± 0.41	21.03 ± 0.08	80.09
As	0.11 ± 0.01	ND	100
Ca	1274.52 ± 4.37	509.86 ± 1.36	59.99
Cr	149.86 ± 0.70	41.29 ± 0.30	72.44
Cd	1.12 ± 0.03	0.01 ± 0	99.11
Cu	82.71 ± 0.31	8.54 ± 0.07	89.67
Fe	4388.68 ± 2.71	1151.17 ± 2.68	73.77
K	850.61 ± 1.67	478.72 ± 1.58	43.72
Mg	3470.23 ± 2.00	938.82 ± 2.45	72.95
Mn	1372.49 ± 2.52	295.87 ± 1.31	78.44
Na	1242.17 ± 2.52	1647.69 ± 2.52	-32.64%
Pb	70.66 ± 0.35	0.74 ± 0.02	98.95
Zn	88.1 ± 0.30	9.61 ± 0.08	89.09

The extraction of toxic elements that is elements which are not safe for fish feed formulation is presented and discussed in Table 4.3.

Table 4.3: Extraction analysis for each element

Element	Precipitation Equations	Justification
Al	$Al^{3+} + 3OH^{-} \rightarrow Al(OH)_3\downarrow$ $Ksp = 1.8\times10^{-33}$	The 80.09% (Table 4.2) removal of Al indicates removal by effective precipitation. Al $(OH)_3$ has a very low solubility product. Precipitation was favoured at pH 8.5. This aligns with the >99% removal efficiency reported in similar precipitation findings (Loughlaimi et al., 2024). Furthermore, on the simultaneous precipitation of other essential metals, $Al(OH)_3$ creates a gelatinous precipitate that improves coprecipitation of other metals (Chernyaev et al., 2023; Rene et al., 2017).
Cr	$Cr^{3+} + 3OH^{-} \rightarrow Cr(OH)_3\downarrow$ $Ksp = 6.3\times10^{-31}$	The 72.44 % (Table 4.2) removal to ND limits indicates precipitation of Cr. The best pH range for Cr precipitation reported is 8.0-9.5, which matches the current experimental condition of pH 8.5 (Kostrzewa et al., 2022). Similar studies have findings of 99% Cr removal efficiency at pH greater than 8.0 through hydroxide precipitation mechanisms (Nowicki et al., n.d.).
Fe	$Fe^{3+} + 3OH^{-} \rightarrow Fe\ (OH)_3\downarrow$ $Fe^{2+} + CO_3^{2-} \rightarrow FeCO_3(s)$	The 73.77% (Table 4.2) removal reflects Fe's complex precipitation behaviour. Fe forms multiple hydroxide precipitates and can also precipitate as carbonate or hydroxycarbonate compounds.

	$K_{sp} = 4.0\times10^{-38}$	According to Li et al (2025), the process improves due to the oxidation of Fe^{2+} to Fe^{3+} in alkaline conditions. Fe (III) easily precipitates as $Fe(OH)_3$, while Fe(II) stays soluble over a wider range of pH levels (S. Li et al., 2025).
Pb	$Pb^{2+} + 2OH^- \rightarrow Pb(OH)_2\downarrow$ $Pb^{2+} + CO_3^{2-} \rightarrow PbCO_3\downarrow$ $K_{sp}(PbCO_3) = 7.4\times10^{-14}$	The removal of Pb at 98.95% (Table 4.2) to 0.74 is justified by two possible precipitation mechanisms. $Pb(OH)_2$ forms best at pH 8.0-8.5 (Karimi, 2017). $PbCO_3$ precipitation ($K_{sp} = 7.4\times10^{-14}$) improves with bicarbonate ions. Studies show over 95% Pb removal efficiency using carbonate precipitation at pH 8-11 (Khalati et al., 2022).
Cd	$Cd^{2+} + 2OH^- \rightarrow Cd(OH)_2\downarrow$ $Cd^{2+} + CO_3^{2-} \rightarrow CdCO_3\downarrow$ $K_{sp}(CdCO_3) = 5.2\times10^{-12}$	The 99.11% (Table 4.2) indicates high removal of Cd considering the trace amount to ND. Cd precipitation occurs through combined hydroxide and carbonate precipitation. $CdCO_3$ forms easily when carbonate ions are present ($K_{sp} = 5.2\times10^{-12}$). Previous findings Lin et a.l (2023) and Zhu et. al (2024) revealed that Cd solubility decreases as carbonate concentration increases at treatment pH values below 11 (Lin et al., 2023; Zhu et al., 2024); thus, Removal efficiencies reach nearly 99.9% at pH levels between 8 and 11 (Loughlaimi et al., 2024).
As	$As^{3+} + 3OH^- \rightarrow As(OH)_3\downarrow$ $AsO_4^{3-} + 3Fe^{3+} \rightarrow FeAsO_4\downarrow$ (co-precipitation)	Complete As removal of 100% (Table 4.2) is consistent with effective alkaline precipitation mechanisms. Pinisakul et al. (2002) postulated that the precipitation can also occur by As being incorporated and adsorbed on Fe_2O_3 sludge, they

		achieved an efficiency of 64.9% to 94.9% (Pinisakul et al., 2002). On the same phenomenon; studies on alkaline wastewater indicate that As removal rates can reach 90.16% through magnesium arsenate formation in alkaline conditions (Y. Wang et al., 2022). The "ND" (Not Detected) result for arsenic it may indicate a 100% removal efficiency due to aforementioned effective alkaline precipitation chemistry or As levels dropped below the detection limit of the ICP-OES since As is a weak emitter in ICP-OES, which makes it hard to detect at very low levels (Neubauer and Magarini, 2021).
Mn	$Mn^{2+} + 2OH^{-} \rightarrow Mn(OH)_2\downarrow$ $Mn^{2+} + CO_3^{2-} \rightarrow MnCO_3\downarrow$	The Mn percentage of 78.44 % (Table 4.2) indicates a reduction of Mn content due to effective precipitation. Mn forms precipitates at a pH greater than 9.0 for the highest percentage removal (Schaefer et al., 2020). The bicarbonate buffer system helps create mixed Mn hydroxide-carbonate species. Higher pH levels significantly improve Mn^{2+} adsorption and oxidation processes (Y. Li et al., 2019)
K	$K^{+} + OH^{-} \rightarrow$ Limited precipitation	The K percentage, 43.72% (Table 4.2), indicates a reduction of K content due to moderate precipitation. Direct precipitation was minimal as its hydroxide is known to be very soluble. The precipitates are likely to have occurred through coprecipitation in the gelatinous matrix of Al and Fe

		hydroxide precipitate (Collins et al., 2023; Nowicki et al., n.d.).
Na	$Na^{+} + OH^{-} \rightarrow$ No direct precipitation	The negative percentage of -32.64% (Table 4.2) is because of excess Na from the treatment reagents NaOH and $NaHCO_3$, which increases the overall Na concentration. However, reduction may occur through co-precipitation within hydroxide precipitates. Research shows that Na ion concentrations usually rise when NaOH is added (Nowicki et al., n.d.; Rao and Raju, 2010).
Cu	$Cu^{2+} + 2OH^{-} \rightarrow Cu(OH)_2\downarrow$ $Cu^{2+} + CO_3^{2-} \rightarrow CuCO_3\downarrow$	The high percentage of 89.67% (Table 4.2) indicates a reduction of Cu content due to effective Cu precipitation. The precipitation of Cu is improved due to both hydroxide and carbonate mechanisms. Carbonate precipitation has been reportedly preferred over hydroxide precipitation, depending on pH (Q. Chen et al., 2018; Shadrunova and Orekhova, 2017).
Zn	$Zn^{2+} + 2OH^{-} \rightarrow Zn(OH)_2\downarrow$ $Zn^{2+} + CO_3^{2-} \rightarrow ZnCO_3\downarrow$	The high percentage of 89.09 % (Table 4.2) indicates a reduction of Zn content due to effective Zn precipitation. The bicarbonate buffer system improves the formation of $Zn(OH)_2$ and $ZnCO_3$ precipitates (Shadrunova and Orekhova, 2017). Some reported findings have indicated removal efficiencies of approximately 99.9% for Zn at a pH range of 8-11 using alkaline precipitation (Loughlaimi et al., 2024).

4.3 Preliminary leaching experiment

A preliminary leaching stage was done with organic acids: acetic acid and citric acid and inorganic acids: hydrochloric acid and sulfuric acid. This was done for comparative study and key interpretation of the efficiency of the organic acids.

4.3.1 Comparative analysis of inorganic acids and organic acids

The results in Figure 4.1 show a comparison of the recovery of elements using inorganic acids: hydrochloric acid (HCl), sulfuric acid (H_2SO_4), and organic acids; citric acid ($C_6H_8O_7$), acetic acid (CH_3COOH) and a mixture of acetic acid and citric acid.

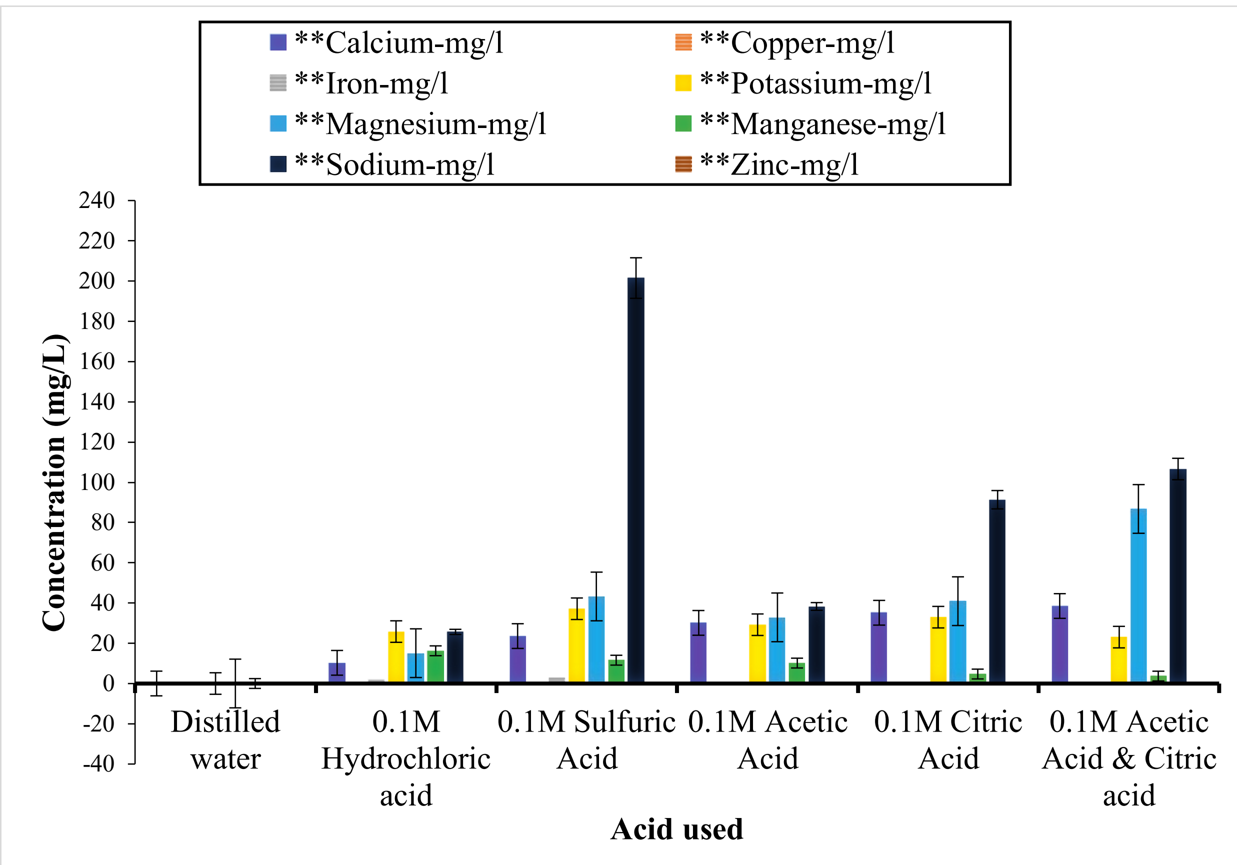


Figure 4.1: Preliminary leaching comparing various acids and acid mixtures

Leaching was carried out at room temperature for 72 hours. The concentrations of the elements, Ca, Cu, Fe, K, Mg, Mn, Na, and Zn extracted using various leaching agents are shown in Figure 4.1. The variations in the concentration of the extracted elements depend on the strength of the acid. Acid leaching is necessary for efficient recovery, as evidenced by the zero extraction of all the elements in distilled water. This confirms earlier research by Mohamed (2016), who noted that while efficiency varies by acid type and target element, acid leaching is required for cation extraction from platinum group metal tailings (S. Mohamed et al., 2016a). In line with earlier studies (D. Das et al., 2021; Nasab et al., n.d.; Xiao et al., 2022; Yan et al., 2014) on the mechanisms underlying mineral dissolution, inorganic acids (HCl and H_2SO_4) generally showed better extraction efficiency than organic acids. The combination of acetic and citric acids produced encouraging results, revealing the effect of two acids compared to one organic acid for the selective recovery of specific elements, as similarly reported in similar studies of the effects of combining acids (Bassi et al., 2000; Kong et al., 2014). According to Astuti et al.(2016) a greater recovery of the acetic-citric acid combination suggests that these organic acids work in synergy to extract these elements (Astuti et al., 2016). Citric acid is a tricarboxylic acid; through its carboxyl and hydroxyl functional groups, it enhances the effective removal of cations such as Ca^{2+} and Mg^{2}+, and acetic acid provides additional sites for reactions with the metals (Fatima et al., 2024; Roshanfar et al., 2024; Woodall et al., 2024a).

4.3.2 Effect of concentration of mixture of acetic acid and citric acid

The organic acid mixture was used to study the effect of varying concentration from 0.1 M to 0.5 M for 72 hours, and the results are shown in Figure 4.2.

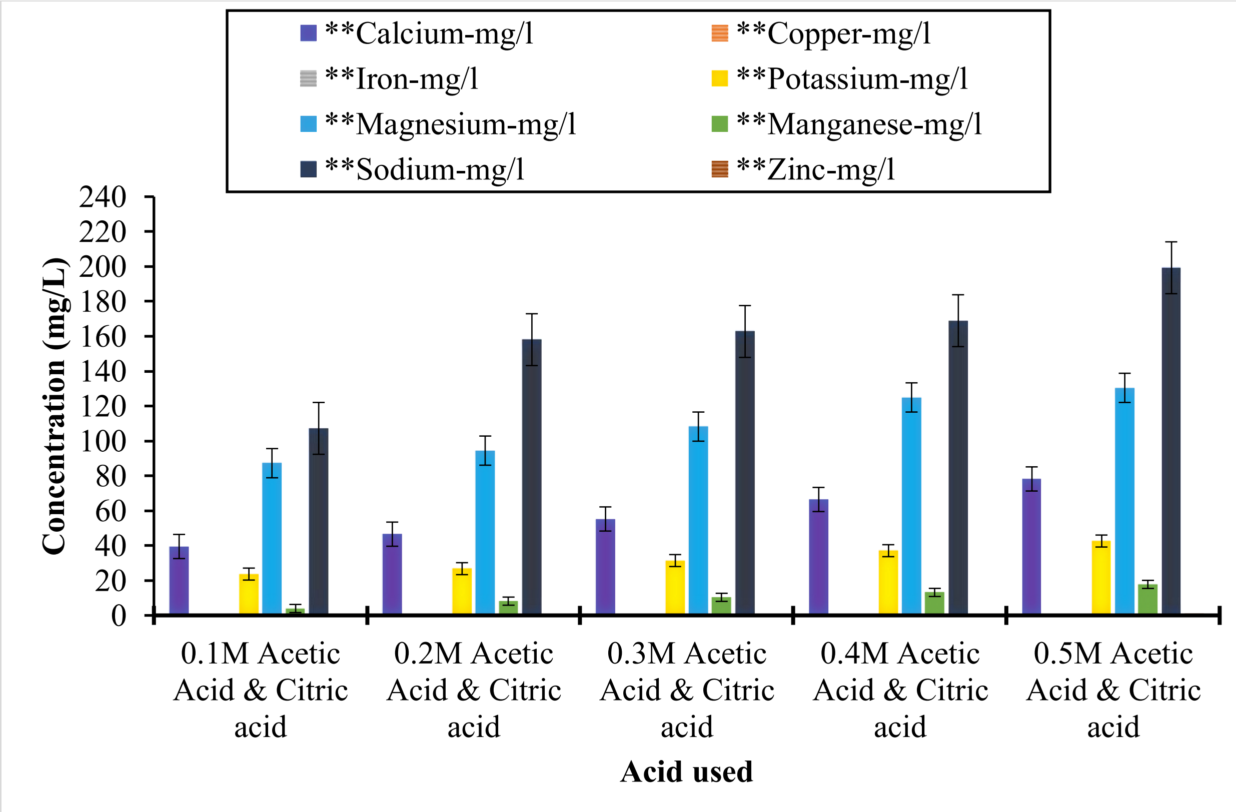


Figure 4.2: Different concentrations of mixtures of acetic acid and citric acid

Figure 4.2 shows an increase in the leaching efficiency with increasing acid concentration between 0.1 M and 0.5 M, with Na and Mg recovered more than the other elements, with peak concentrations of approximately 199.26 mg/L and 130.42 mg/L, respectively, at 0.5 M acid concentration. Ca, Cu, Mg, Na, K, Zn, Mn, and Fe show a trend of extraction as Na > Mg > Ca > K > Mn > Zn > Cu ≈ Fe. The trend shows the nature of compounds associated with the elements as part of the platinum tailings matrix, where readily accessible elements trapped in soluble fractions (most notably sodium in plagioclase lattices) possess better leaching kinetics than the elements trapped in refractory minerals (Gibson et al., 2023a). As was reported in similar studies, the increase in concentration indicates the direct proportionality of the strength of the acids and decomposition of minerals, releasing the elements into solution (Goyne et al., 2010; Munyai et al., 2016; Pokrovsky et al., 2009). Table 4. 4 shows leaching results at 60 °C, at pH 2, for 1 hour, as was adopted with modifications.

Table 4.4: Leaching efficiency of recovering essential elements from the platinum mineral tailings residue using a mixture of 0.5 M acetic acid and citric acid

Element	Metal in residue (mg/L)	Metal in leachate (mg/L)	Leaching efficiency
Al	10.57 ± 0.025	ND	0
As	ND	ND	0
Ca	318.12 ± 0.20	52.6 ± 0.10	16.53
Cr	11.2 ± 0.05	ND	0
Cd	ND	ND	0
Cu	4.14 ± 0.02	0.02 ± 0.001	0.48
Fe	1558.32 ± 0.66	0.23 ± 0.01	0.015
K	212.5 ± 0.10	31.51 ± 0.02	14.83
Mg	836.65 ± 0.55	102.17 ± 0.05	12.21
Mn	157.8 ± 0.20	23.59 ± 0.04	14.95
Na	693.03 ± 0.28	210.43 ± 0.03	30.36
Pb	0.35 ± 0.01	ND	0
Zn	4.4 ± 0.025	0.24 ± 0.008	5.45

ND means Non detected

The leaching process using a 0.5 M mixture of acetic and citric acid to recover essential elements from the platinum mineral tailings residue produced a remarkable variation in the different elements using the adopted and modified conditions. This trend revealed the complex mineral content and chemical behavior of the tailings. Sodium recovery (30.36 %) is attributed to the presence of highly soluble sodium-bearing phases and the effectiveness of organic acids in mobilizing alkali metals from silicate matrices (Guo et al., 2022) and from the alkali pretreatment process. The leaching efficiency and elemental recovery percentage values shown in Table 4.4 reveal negligible extraction (0%) for elements such as Al, Cr, As, and Pb. Although Cr and Al were present in significant amounts, they were not detected in the organic acid leachate. This may be due to their complex bonding with the tailings matrix, which could incorporate them into highly insoluble compounds that require extreme conditions for dissolution (Rivera and Garbers-Craig, 2023; Ross et al., 2022). The Al, ND value, is much lower than the average LOD of 0.07 µg/L (axial mode) of the iCAP 7000 series. Although Al was present in the extract of the residue at 10 mg/L, the mixture of organic acids was not able to record a substantial amount of Al extraction.

This could be due to the tendency of Al to form stable complexes with silicate matrices under mildly acidic conditions (Hanzlicek and Perna, 2011; Pokrovski et al., 1996). On ND for As (Table 4.4), it has weak emission properties for analysis with concentrations below the iCAP 7000 LOD of 3.2 µg/L and hence the low ND of the leachate, which indicates low emission of As and confirm extraction during pretreatment stage. The ND outcome for Cr (Table 4.4) is attributed to the level of 11.2 mg/L in the residue and rather low LOD of 0.50 g/L (axial mode). The refractory nature of chromium in organic acids is attributable to the close association of chromium with spinel minerals in platinum tailings (S. Mohamed et al., 2019). The ND for Cd, (Table 4.4) indicate that the level of cadmium is less than the LOD of 0.25 µg/l (axial mode). This is either the good extraction during pretreatment or just low Cd levels in the platinum mineral tailings. The ND of Pb (Table 4.4) in leachate, despite its residual concentration of 0.35 mg/L, is lower than the LOD of 2.5 µg/l in axial mode. The extraction efficiency of Pb using acetic-citric acid mixture is low, with organic acids being less effective at extracting Pb than more powerful mineral acids (Ash et al., 2016; Jadhav et al., 2018a). The essential minerals recovered showed moderate to high recoveries: Ca (16.53%), Mg (12.21%), Mn (14.95 %), and Na (30.36 %). Trace elements; Fe, Cu and Zn, had low extraction rates (0.015 %, 0.48 % and 5.45%, respectively), suggesting these elements may be incorporated with resistant sulfide minerals that are not readily soluble (S. Mohamed et al., 2016b). Zn extraction is higher than copper maybe due to its occurrence in more soluble oxide and carbonate phases (Ash et al., 2016; Kundu et al., 2014). Toxic elements such as Cd and As were below detection limits in the leachate, suggesting their reduction or low solubility under the conditions used, since they may be incorporated into a complex compound requiring extreme conditions. Similar findings revealed that As and Cd can form ternary bridging complexes and precipitate with iron and aluminium oxides, further reducing their availability under the experimental conditions (Gunadasa et al., 2023; M. Lin et al., 2024).

The moderate leaching of Ca and Mg corresponds with their typical occurrence in more soluble mineral forms such as carbonates and silicates, in which weak organic acids, such as acetic and citric acids, can easily dissolve (Musariri et al., 2019; Woodall et al., 2024b). Citric acid, a tricarboxylic acid, can form stable complexes with divalent and trivalent metal ions using its multiple carboxyl functional groups, thereby improving dissolution kinetics (Yang et al., 2011). The Fe recovery is 5%, indicating that Fe largely exists in resistant mineral forms such as chromite and hematite, which require minimal dissolution in mildly acidic organic acid environments. Nevertheless, organic ligands such as citrate can facilitate iron dissolution via ligand-enhanced processes and complexation, although this method is less effective than more intense leaching conditions (Mukherjee and Gao, 2016).

4.4. Optimization results for essential element extraction from platinum mineral tailings using RSM Box-Behnken design using the RSM Box-Benken

A Box-Behnken Design (BBD) was employed to optimize four variables in the leaching of essential elements from platinum mineral tailings using a 0.1–0.5 M mixture of acetic and citric acids.

4.4.1 Factors and experimental results

A Box-Behnken Design (BBD) was employed to optimize four variables in the leaching of essential elements from platinum mineral tailings using a 0.1–0.5 M mixture of acetic and citric acids. The design matrix included 29 experimental runs, 5 of the runs are displayed in Table 4.5. The factors are represented with the letter F, and actual responses represented with the letter R, measured in mg/L by use of ICP-OES. All the other values are displayed on appendix B.

Table 4.5: The BDD experimental design actual results

Std	Run	F1	F2	F3	F4	R1	R2	R3	R4	R5	R6	R7	R8
		H+	hr.	T°C	M	Ca	Cu	Fe	K	Mg	Mn	Na	Zn
28	1	5	3	70	0.3	123	0.03	0.63	70.6	240	25.2	351	0.65
6	2	5	3	75	0.1	105	0.02	0.54	63.8	190	19.2	256	0.54
11	3	2	3	70	0.5	132	0.04	0.69	75.9	290	39.5	442	0.71
3	4	2	5	70	0.3	128	0.04	0.68	74.2	260	28	370	0.7
8	5	5	3	75	0.5	129	0.02	0.69	90	290	40	440	0.71

The experimental data were fitted to a second-order polynomial model according to equation (3.2).

The model polynomial equations (Equations (1) – (8)) below are for binomial fitting regression equations that describe the relationships between influencing factors and response values.

(1) $Ca\ (mg/L) = -106.20574 + 5.24352A + 0.670417B + 5.20900C + 157.32917D - 0.116667AB - 3.29602 \times 10^{-16} AC - 0.916667AD + 0.0375BC + 1.5BD\text{-}0\text{-}0.0892407A^2 + 0.423542B^2 - 0.035267C^2 - 127.97917D^2$

(2) $Cu\ (mg/L) = -0.225521 - 0.000694A + 0.001875B + 0.007C + 0.1375D - 1.39429\times10^{-17}AB + 2.90113\times10^{-19}AC - 0.004167AD + 9.32813\times10^{-19}BC + 3.00966\times10^{-18}BD + 9.54481\times10^{-18}CD - 0.000278A^2 - 0.000313B^2 - 0.00005C^2 - 0.1875D^2$

(3) $Fe\ (mg/L) = -0.132685 - 0.03537A - 0.028958B + 0.0215C + 0.564583D + 0.000417AB + 0.000333AC + 0.0125AD + 0.0005BC - 0.025BD - 1.53783\times10^{-16}CD - 0.001019A^2 + 0.000521B^2 - 0.000167C^2 - 0.385417D^2$

(4) $K\ (mg/L) = 111.30294 + 1.47546A - 1.32958B - 1.42933C - 28.10833D - 0.05AB + 0.033333AC + 0.875AD + 0.005BC + 0.625BD + 0.05CD - 0.561852A^2 + 0.326458B^2 + 0.010233C^2 + 116.08333D^2$

(5) $Mg\ (mg/L) = 195.88975 - 9.48611A + 1.29167B + 0.291667C + 208.75D - 0AB - 0AC - 0AD - 0BC - 0BD - 0CD - 0A^2 + 0B^2 + 0C^2 + 89.6875D^2$

(6) $Mn\ (mg/L) = 12.47431 + 2.9375A - 2.28542B - 0.015833C + 16.6875D - 2.53752 \times 10^{-15}AB - 6.95365 \times 10^{-16}AC - 4.79167AD + 0.0275BC - 3.59515\times10^{-15}BD - 0.05CD - 0A^2 + 0B^2 + 0C^2 + 0D^2$

(7) $Na\ (mg/L) = 244.67179 - 12.09444A + 1.66667B + 0.475C + 400.375D + 0AB + 0AC - 0.016667AD + 0.0275BC + 0BD - 0.0025CD - 0A^2 + 0B^2 + 0C^2 + 0D^2$

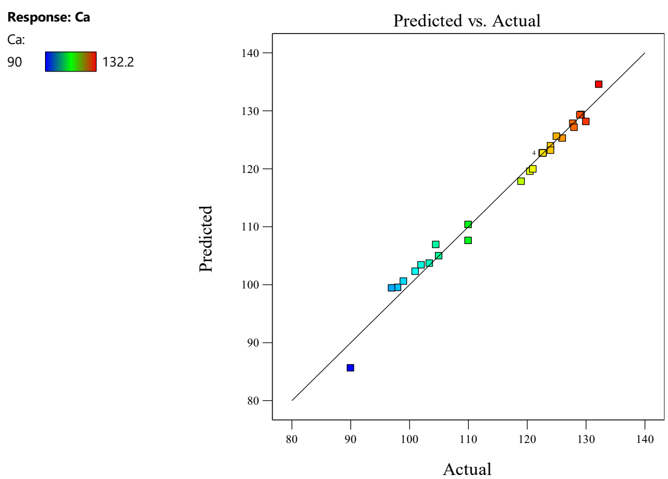
(8) $Zn\ (mg/L) = -1.18306 - 0.008333A - 0.010625B + 0.048C + 0.964583D - 1.23128\times10^{-16}AB + 0.0005AC - 0.016667AD - 0.00625BD - 0.0025CD - 0.004861A^2 - 8.83282\times10^{-17}B^2 - 0.00035C^2 - 0.5625D^2$

The models reveal some variation in the extent of extraction of various metals. This is due to different chemical interactions in terms of affinity to the organic acids (Cano-Lamadrid et al., 2023; Zabiszak et al., 2018). Concerning equations (1), (4),(5) and (7) for Ca, K, Mg and Na respectively, the metals have significant intercept terms because of their ability to form stable complexes with organic acids, and facilitated by the combination of the organic acids used (acetic acid and citric acid) considering the much availability carboxylate groups (Horakova et al., 2024; Zabiszak et al., 2018). For Cu, Fe, Mn and Zn, a trend with smaller coefficients was observed. Equations (2), (3), (6) and (8) respectively; Cu and Zn their extraction depends on combined interactions of factors rather than individual effects, which has been reported before that Cu and Zn form stable complexes affected by multiple environmental factors simultaneously (Ding et al., 2014; Zabiszak et al., 2018). Equation 3 for Fe extraction, the negative variables for A and B reveal that an increase in the factors may decrease extraction maybe due to the possibility of precipitation of Fe, depending on whether the concentration or the acids are sufficient enough to dissolve the Fe from the mineral matrix. According to Balintova et al. (2011), although precipitation is more noticeable at pH values above 6, studies show that some iron precipitation can start around pH 3.5 to 5, especially when Fe^{2+} is oxidised to Fe^{3+}, forming insoluble hydroxides or oxyhydroxides (Balintova and Petrilakova, 2011). Mn has the positive coefficient for D (16.6875) compared to Fe, Zn and Cu, demonstrating that concentration is an influential factor in manganese extraction; According to Granholm et al (2010) manganese forms exceptionally stable chelate complexes with citric acid compared to other transition metals (Granholm et al., 2010). The many coefficients close to zero in the equations for Mg, Mn, and Na indicate that these metals follow simpler extraction methods and are influenced by individual process variables rather than their combined effects. Thus, there are minimal synergistic effects between the process variables (Mohrazi et al., 2024).

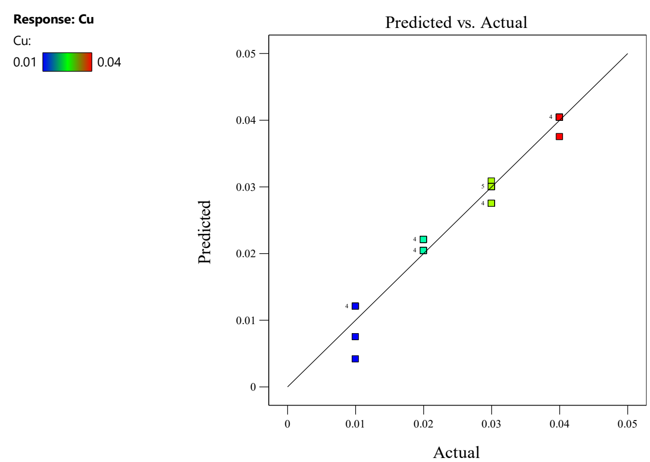
The polynomial models reveal differences in coefficient sizes between alkali and alkaline earth metals (Ca, Mg, K, and Na) and transition metals (Cu, Fe, Zn, Mn), highlighting the basic differences in how they interact with organic acids. This is consistent with studies showing that transition metals usually have slower complexation rates with organic acids than main group metals (Okonkwo et al., 2021; Fatima et al., 2024). Organic structures with their coordination sites tend to bind more strongly to alkaline and alkaline earth metals than to softer transition metals (Zaleckas et al., 2013; Zabiszak et al., 2018).

4.4.2 Comparison of actual vs predicted values of response models for the various elements

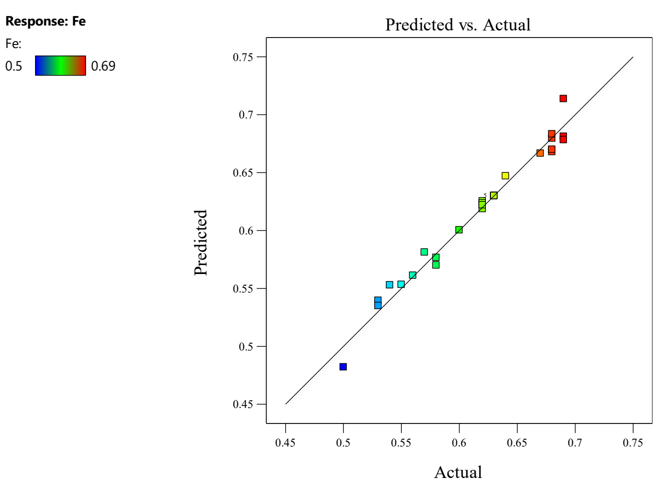
Predicted vs. actual plots, Figures 4.3 (a) to (h), fit statistics showing the validity and predictability of the model in maximizing major element recovery from platinum tailings by acetic-citric acid leaching. The predicted vs actual plots graphically compare the model's predicted responses with the data points observed during the experiments (Sai et al., 2023) . When points closely align along the 45-degree line, a good fit of the model is shown, confirming that the model can reliably predict responses within the experimental range (NIST, 2003,Bezerra et al., 2008; Myers et al., 2009).



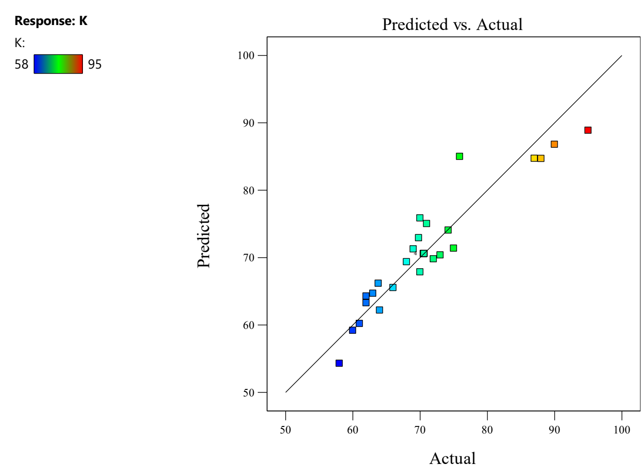
(a)



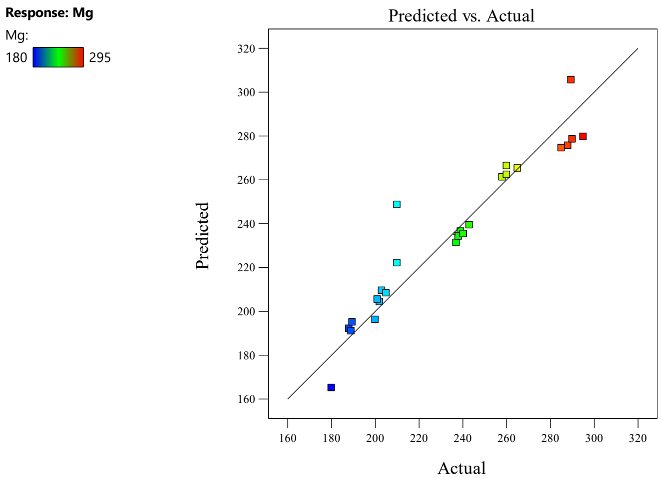
(b)



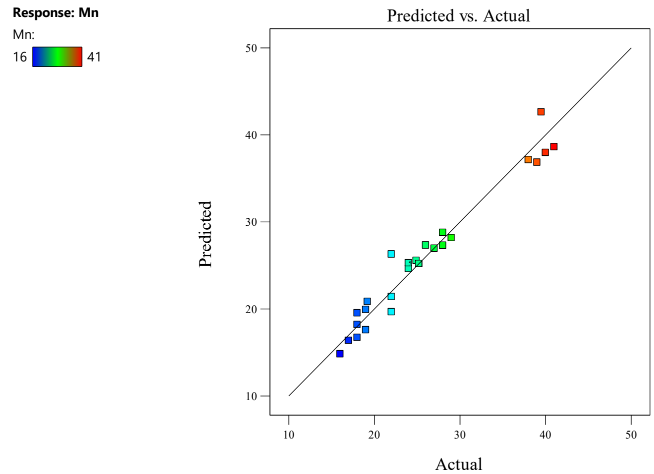
(c)



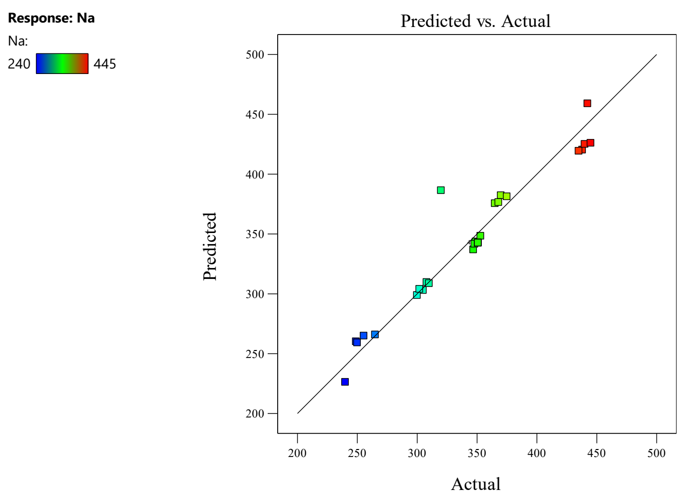
(d)



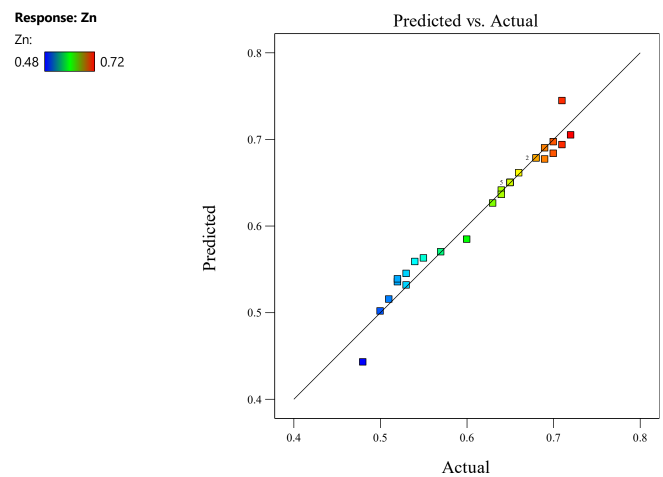
(e)



(f)



(g)



(h)

Figure 4.3.: Plots of the predicted vs actual for response values of (a) Ca, (b) Cu, (c) Fe, (d) K, (e) Mg, (f) Mn, (g) Na, and (h) Zn

When the predicted vs actual plot shows a high correlation and minimal deviation, the optimization results are relevant for further research (Xiong et al., 2023). The scatter plot configurations for the eight essential elements shown in Figure 4.3 (a) – (h) for Ca, Cu, Fe, K, Mg, Mn, Na, and Zn demonstrate predictive accuracy across the eight analyzed elements, with data points generally clustering around the theoretical 1:1 diagonal line that represents perfect prediction (Myers et al., 2009; Xiong et al., 2023). Elements with higher concentrations generally demonstrate better predictive performance, which is consistent with analytical precision limitations at trace levels and the inherent signal-to-noise ratio advantages of higher concentration measurements (G. F. Silva et al., 2018).

Table 4.6: Fit statistics of the experimental data and regression model

Element	R^2	Adjusted R^2	Predicted R^2	Adeq Precision	Lack of Fit (p-value)
Ca	0.9858	0.9688	0.9498	32.86	0.525
Cu	0.9630	0.9259	0.7866	18.12	0
Fe	0.9760	0.9519	0.8615	26.25	0
K	0.8792	0.9584	0.9446	10.80	0.580
Mg	0.9076	0.8736	0.8578	29.61	0.679
Mn	0.9559	0.9518	0.9459	17.73	0.623
Na	0.9278	0.9157	0.8885	32.28	0.536
Zn	0.9678	0.9355	0.8143	21.89	0

Differences between adjusted R^2 and predicted R^2 were observed below 0.2, for the elements Ca, K, Mg, Mn and Na, confirming good predictive accuracy (Fazeli Burestan et al., 2020). For all the elements, the adequate precision values are well above the threshold of 4, ranging from 10.8 – 32.86, indicating acceptable signal-to-noise ratios. The non-significant lack of fit, with p values greater than 0.05 for Ca, K, Mg, Mn and Na, further supports the model's reliability (Myers et al., 2009; Azeem et al., 2024). However, the response model for the Cu, Zn and Fe levels and their lack of fit values was found to be zero. This suggests that the average value of the data serves as a more accurate predictor than the model itself (Arfini et al., 2025).

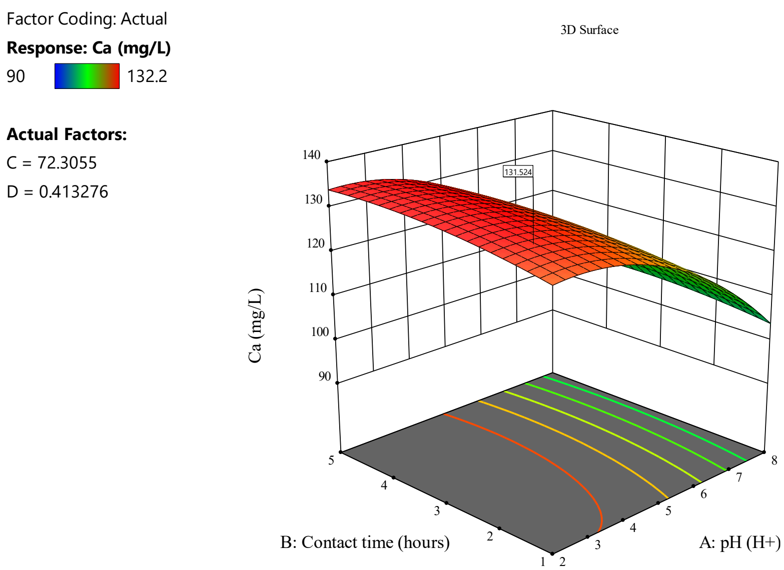
4.4.3. 3D response surface plots

Figures 4.4 – 4.11 (a) and (b), are a series of 3D response surface plots showing the interactive effects of pH, contact time, temperature, and acid concentration on the extraction of the essential elements from platinum mineral tailings with a combination of acetic and citric acid lixiviant.

4.4.3.1 Ca recovery

Figure 4.4 shows surface plots of the variables and 3D response, showing the impact of the key factors on Ca recovery

(a)



(b)

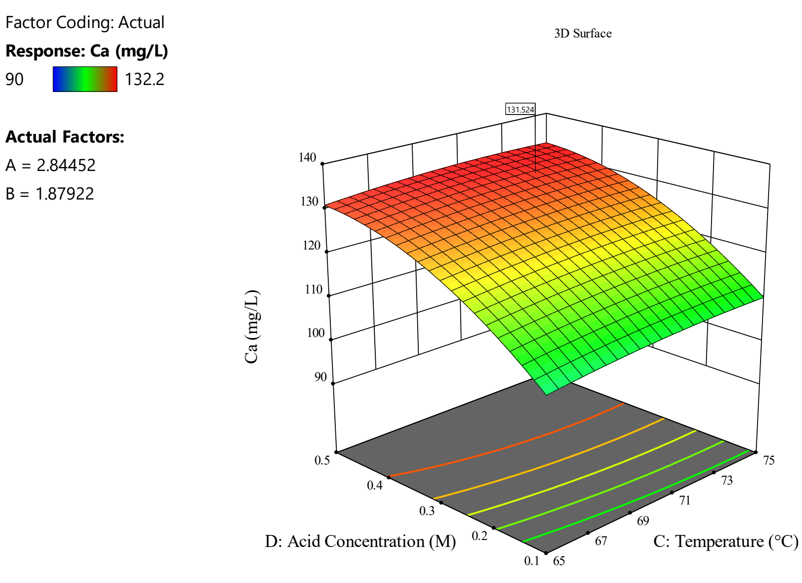


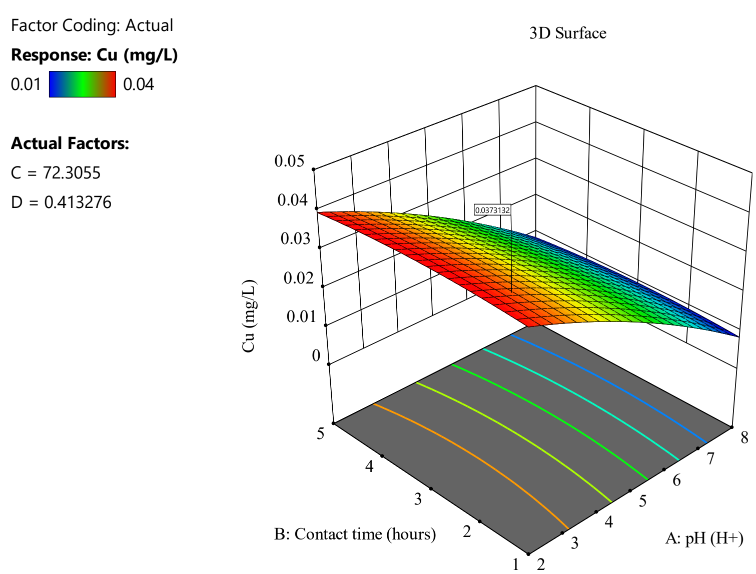
Figure 4.4 (a) and (b): 3D surface response plots showing the impact of two variables on Ca recovery (mg/L): (a) pH and contact time; (b) temperature and acid concentration

As shown in Figure 4.4 (a), the optimal response was observed at around pH 3 and 4-5 hours of contact time, yielding a concentration of approximately 132.2 mg/L. The extraction of Ca was inversely related to the pH, possibly because Ca-bearing minerals may involve compounds that can be broken down by protonation, leading to the formation of soluble Ca ions (Xia et al., 2015). According to Lee et. al (2021); in general, Ca leaching generally increases significantly with decreasing pH, with optimal extraction occurring at pH < 3 (Lee et al., 2021). The contact time of the extraction of Ca is characterized by quick initial dissolution followed by a stable phase. This behavior is related to the dissolution of Ca-bearing minerals, where Ca is present within readily accessible mineral surfaces (Deng et al., 2017). As shown in Figure 4.4 (b) concerning the polynomial model and the 3D model of temperature vs concentration, the concentration had an influence, ranging from 0.3 M to 0.5 M at temperatures ranging from 60–70 °C. Ca is obtained from the dissolution of calcium-containing compounds associated with the platinum tailings. The higher mg/L obtained at higher temperatures indicate a diffusion-controlled mechanism according surface chemical reaction kinetics models of reported earlier in similar studies (Wang et al., 2023; Romano et al., 2024).

4.4.3.2 Cu recovery

Figure 4.5 shows surface plots of the variables and 3D response, showing the impact of the key factors on Cu recovery

(a)



(b)

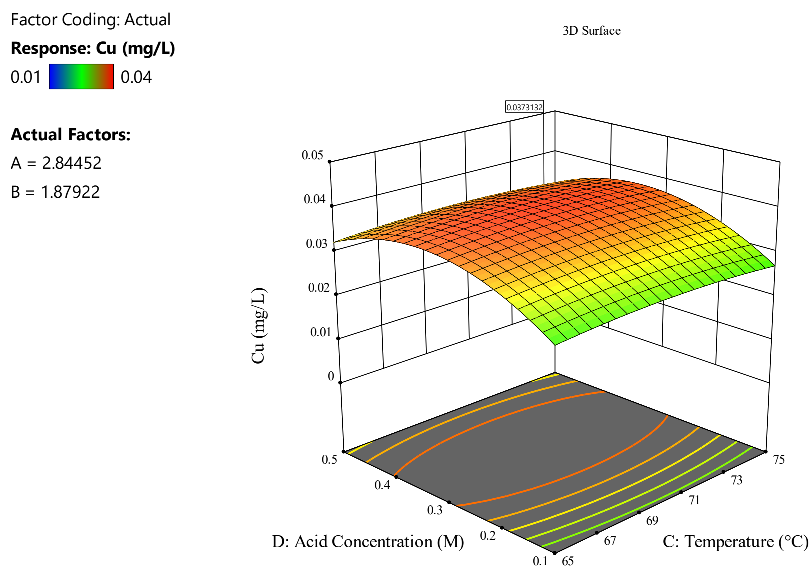


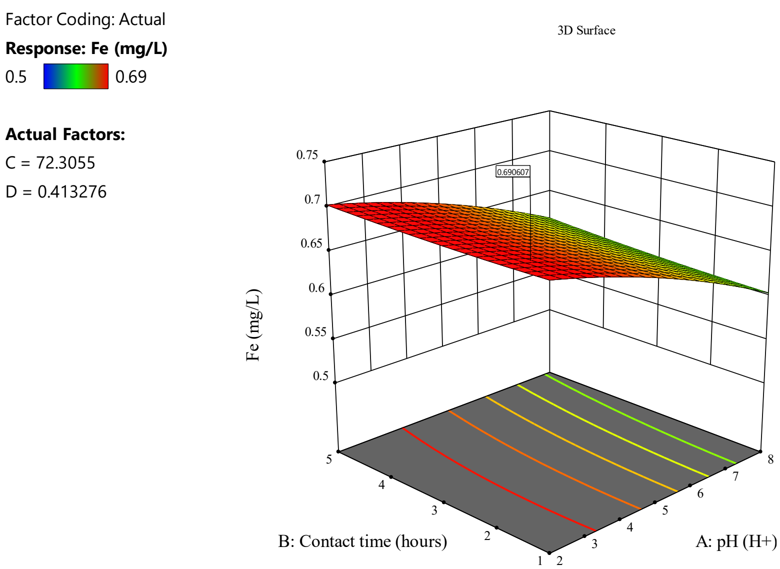
Figure 4.5 (a) and (b): 3D surface response plots showing the impact of two variables on Cu recovery (mg/L): (a) pH and contact time; (b) temperature and acid concentration

Cu was extracted more at low pH values of 1–2 and high contact times (4–5 hours), with a recovery of approximately 0.04 mg/L at temperatures ranging from 73–75 °C and acid concentration ranging from 0.3 – 0.4 M acid. As shown in Figure 4.5 (a), the pH range shows a balance between low concentration of Cu and free Cu ions, and as the pH increases, Cu forms stable complexes with organic acids; the citrate and acetate ligands (Yan et al., 2014). In terms of the contact time, Cu extraction requires an extended time to achieve maximum recovery, which may be attributed to the formation of Cu-organic complexes, which require time for ligand exchange and complex formation (Nagarajan and Panchatcharam, 2023). Figure 4.5 (b) shows the effect of temperature and acid concentration; Cu shows an optimization trend which is consistent with similar literature findings that copper dissolution from various mineral compositions is enhanced by high temperatures, where such high temperatures allow for oxidation reactions that are required for copper solubilization (Altinkaya et al., 2019b). The low extracted Cu (0. 01–0. 04 mg/L) confirms its low composition in the platinum tailings matrix (Kumari et al., 2016; Liddell and Adams, 2012).

4.4.3.3 Fe recovery

Figure 4.6 shows surface plots of the variables and 3D response, showing the impact of the key factors on Fe recovery.

(a)



(b)

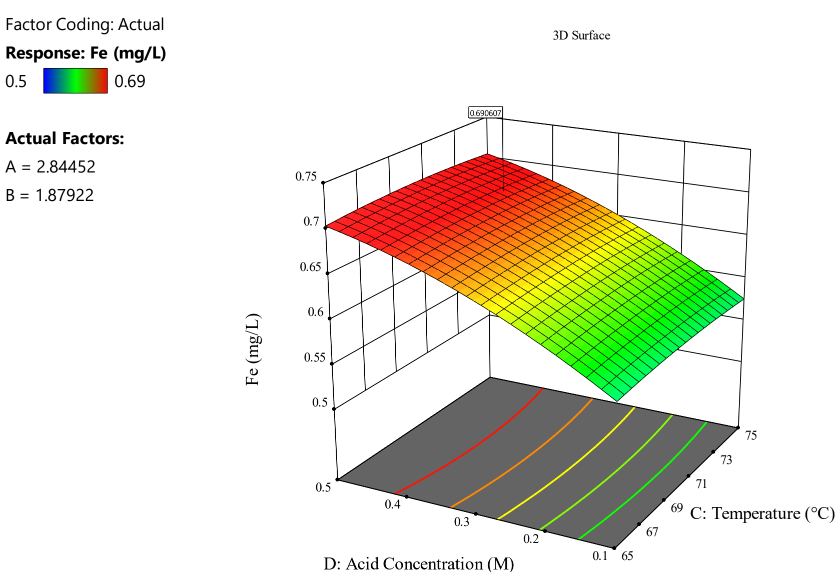


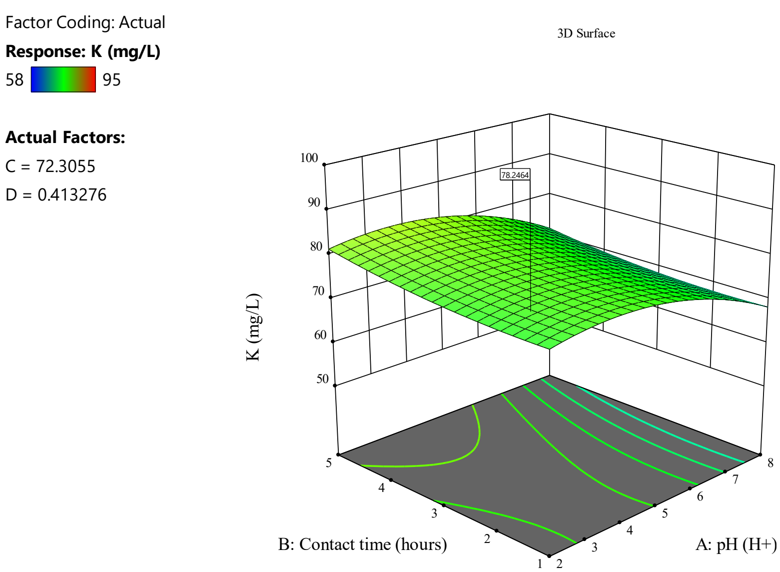
Figure 4.6 (a) and (b): 3D surface response plots showing the impact of two variables on Fe recovery (mg/L): (a) pH and contact time; (b) temperature and acid concentration

The Fe extraction ranges from 05 to 0.69 mg/L, optimized at pH values of 1–3 and 3–5 hours. Figure 4.6 (a) shows that the extraction efficiency decreases for pH > 3, and it has been observed that Fe ions predominate at low pH values and that as the pH increases, Fe may precipitate depending on the solution matrix (Deng et al., 2018). Concerning contact time, Fe extraction was rapid, followed by a slower rate, suggesting the presence of multiple Fe-bearing minerals with different dissolution rates (Deng et al., 2018). Figure 4.6 (b) shows that, with increasing temperatures, the dissolution of Fe increases. In similar studies, they noted that Fe may be associated with complex silicate or other mineral phases requiring aggressive agents, thus explaining why temperature has a greater impact on iron dissolution than does acid concentration (Alfassi, 2008; Teimouri et al., 2020).

4.4.3.4 K recovery

Figure 4.7 shows surface plots of the variables and 3D response, showing the impact of the key factors on K recovery.

(a)



(b)

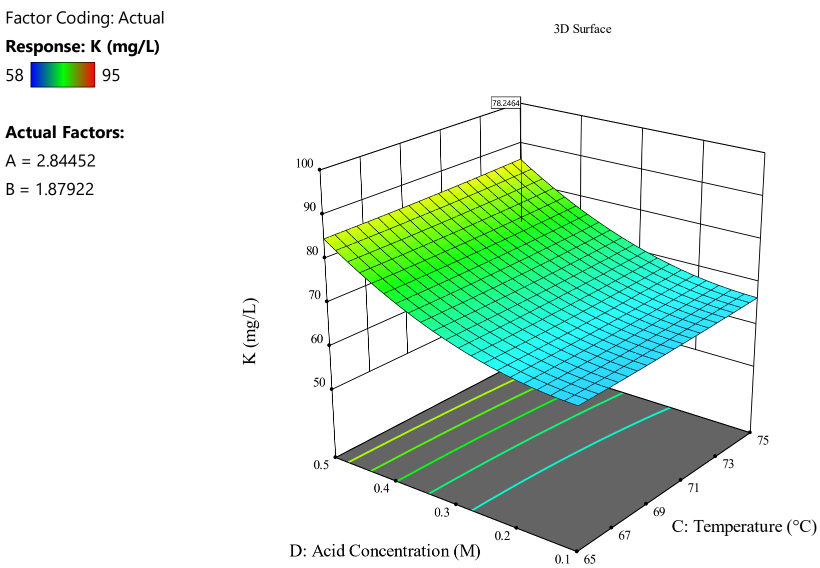


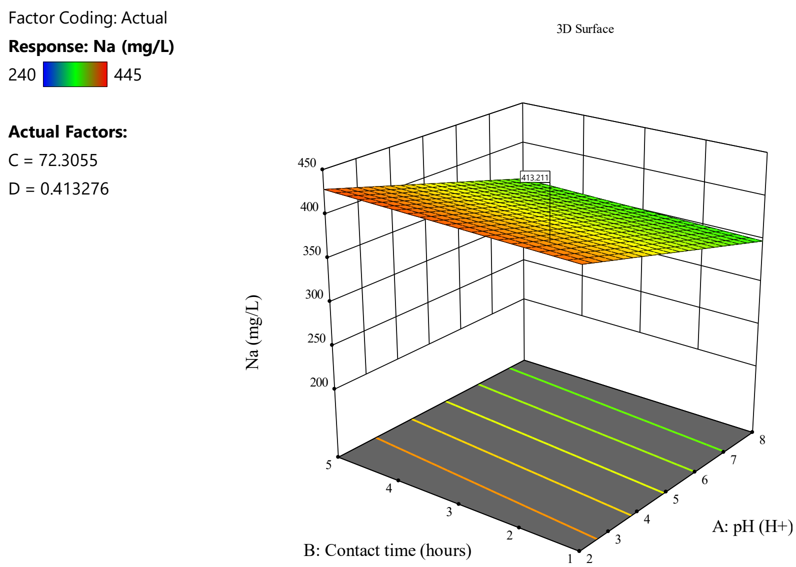
Figure 4.7 (a) and (b): 3D surface response plots showing the impact of two variables on K recovery (mg/L): (a) pH and contact time; (b) temperature and acid concentration

K have maximum extractions of 95 mg/l, with pH values of 1–3 and contact times of 4–5 hours., a temperature of 70–73 °C and a concentration of 0.4 – 0.5 M acid. Figure 4.7 (a) shows that K extraction results in moderate pH changes in the acidic range, possibly because the dissolution of K-bearing minerals is the rate-limiting step rather than pH (Zhu et al., 2023). K is favoured by a high contact time to achieve maximum recovery, K extraction was observed to continue to increase with increasing contact time, and longer reaction times may be beneficial for maximizing recovery (Zhu et al., 2023). Figure 4.7 (b) reveals that the acid concentration and temperature effects increase up to the optimum value, suggesting the dissolution of K is thermally activated and facilitated by acid strength (Mohamed et al., 2016a). The relatively high concentration of Ka in the leachate is indicative of its abundance in the platinum minerals tailings, which is in line with the ion exchange processes in which hydrogen ions in acid exchange positions replace K ions from aluminosilicate matrices (Hamza et al., 2022; Lamidi et al., 2023).

4.4.3.5 Na recovery

Figure 4.8 shows surface plots of the variables and 3D response, showing the impact of the key factors on Na recovery.

(a)



(b)

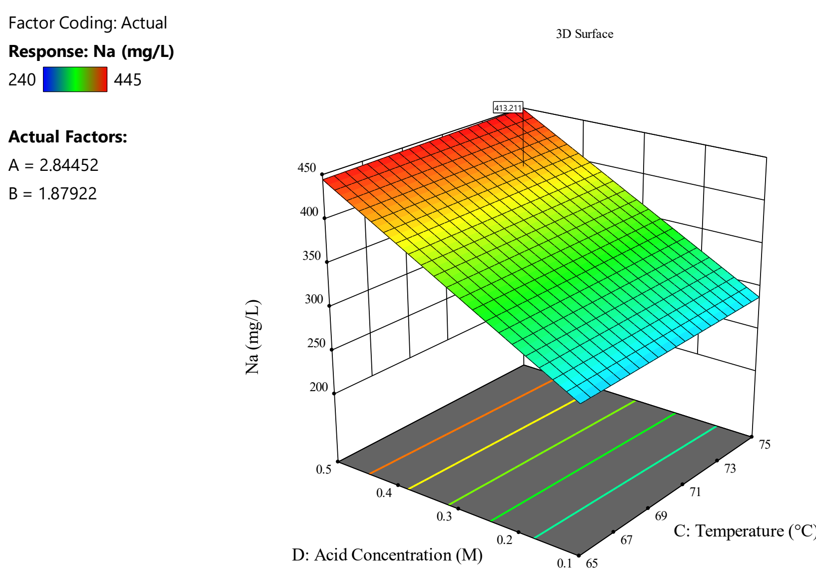


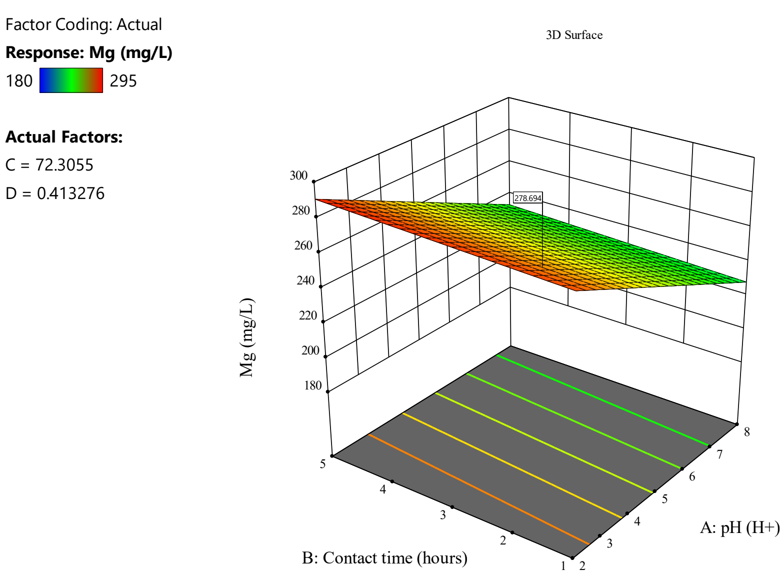
Figure 4.8 (a) and (b): 3D surface response plots showing the impact of two variables on Na recovery (mg/L): (a) pH and contact time; (b) temperature and acid concentration

Na has a maximum extraction of 445 mg/l, respectively, with a pH of 2–3 and prolonged contact times (4–5 hrs). The recovery was at a maximum at 70–73 ° C and a concentration of 0.4 – 0.5 M acid. As shown in Figure 4.8 (a), Na extraction was less pH dependent due to the availability of Na in the tailings matrix, suggesting the high solubility of Na compounds across a wide pH range (Jadhav et al., 2018b). The contact time was relatively short, possibly due to rapid dissolution of Na-bearing compounds, though extended contact time may improve total recovery of Na associated with complex mineral components (Kassab et al., 2023). Figure 4.8 (b) shows that the temperature and concentration were directly proportional to the extraction efficiency, with relatively high concentration exhibiting great mobility under acidic conditions. This is in line with the fact that with the reactivity of Na, there are many ion exchange processes in which hydrogen ions in acid exchange positions replace Na ions from aluminosilicate matrices (Hamza et al., 2022; Lamidi et al., 2023).

4.4.3.6 Mg Recovery

Figure 4.9 shows surface plots of the variables and 3D response, showing the impact of the key factors on Mg recovery.

(a)



(b)

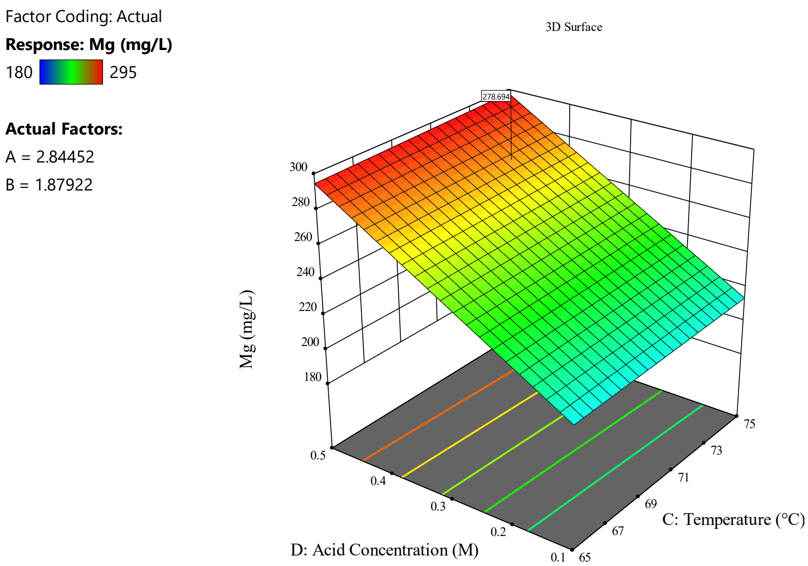


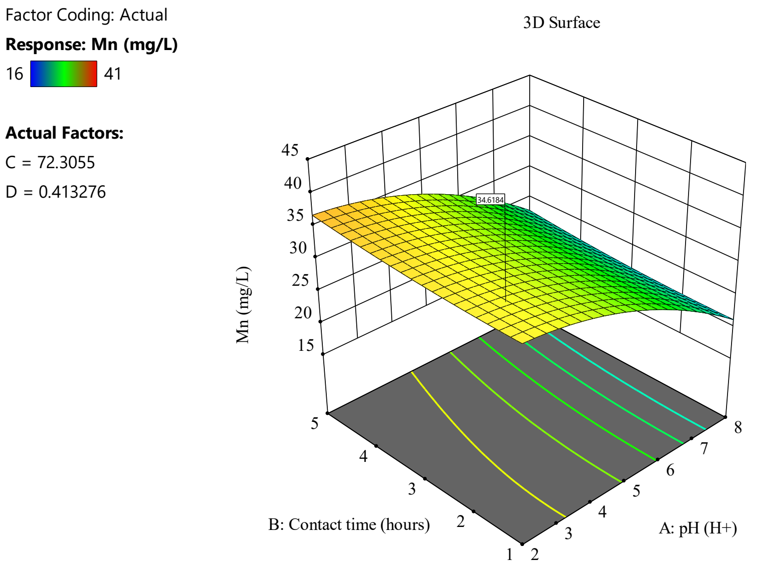
Figure 4.9 (a) and (b): 3D surface response plots showing the impact of two variables on Na recovery (mg/L): (a) pH and contact time; (b) temperature and acid concentration

Mg recovery was optimal at 295 mg/l, pH 2–4 and 4–5 hours, and relatively higher temperatures of approximately 71–75 °C and acid concentrations of 0.3 – 0.4 M. As shown in Figure 4.9 (a), Mg extraction is favoured by pH < 4, at pH > 4, Mg can precipitate, and there is reduced mineral dissolution (Tan and Choi, 2023). Mg extraction was low, suggesting the reactivity of some Mg-bearing minerals under acidic conditions, and the interaction between pH and contact time shows that an extended time might provide minimal benefit (Tan and Choi, 2023). Figure 4.9 (b) 3D plot showing the relationship between temperature and acid concentration; the recovery considering acid concentration and temperature, the behaviors observed are those attributed to reported dissolution mechanisms, thus producing high levels of Mg within the platinum tailings matrix typical of ultramafic platinum deposits (Malenga et al., 2022).

4.4.3.7 Mn recovery

Figure 4.10 shows surface plots of the variables and 3D response, showing the impact of the key factors on Mn recovery.

(a)



(b)

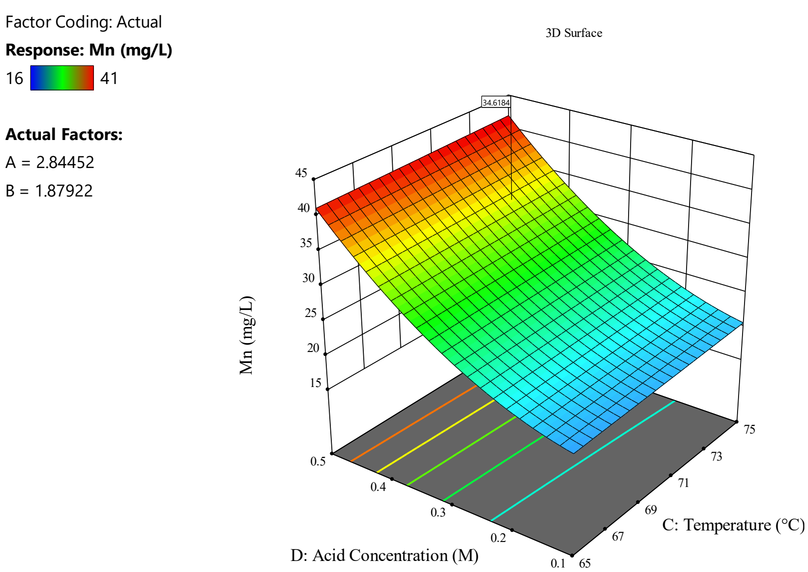


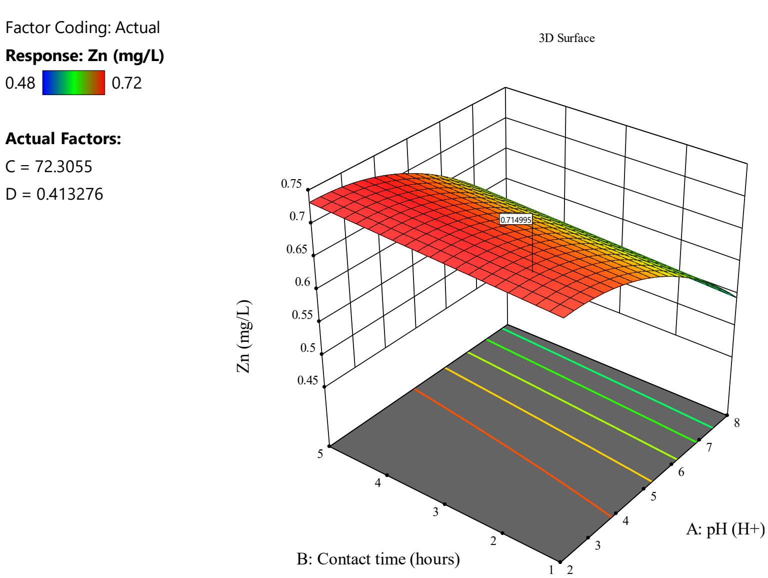
Figure 4.10 (a) and (b): 3D surface response plots showing the impact of two variables on Na recovery (mg/L): (a) pH and contact time; (b) temperature and acid concentration

Mn recovery is highest at 41 mg/l at approximately pH 2–3, a contact time of 4–5 hours, and a temperature of 71–75 °C with 0.2 – 0.4 M acid. Considering Figure 4.10 (a), Mn shows a pH-dependent extraction, which is controlled by the dissolution of the Mn-bearing minerals within the tailings matrix. At low pH, Mn exists as many soluble ions, and as the pH increases, it tends to form insoluble precipitates (Skrzekut et al., 2022). The effect of contact time shows that some Mn may be associated with a complex mineral phase that may require an extended contact time for maximum recovery (Skrzekut et al., 2022). Figure 4.10 (b) shows that the pattern observed for Mn was consistent with the mechanisms of reductive dissolution of Mn oxides under acidic conditions and high temperature, and the synergistic effect of the acids was attributed to enhanced recovery of Mn, as similarly reported in the literature (Aromaa et al., 2013; Fosso-Kankeu and Munyai, n.d.).

4.4.3.8 Zn recovery

Figure 4.10 shows surface plots of the variables and 3D response, showing the impact of the key factors on Zn recovery.

(a)



(b)

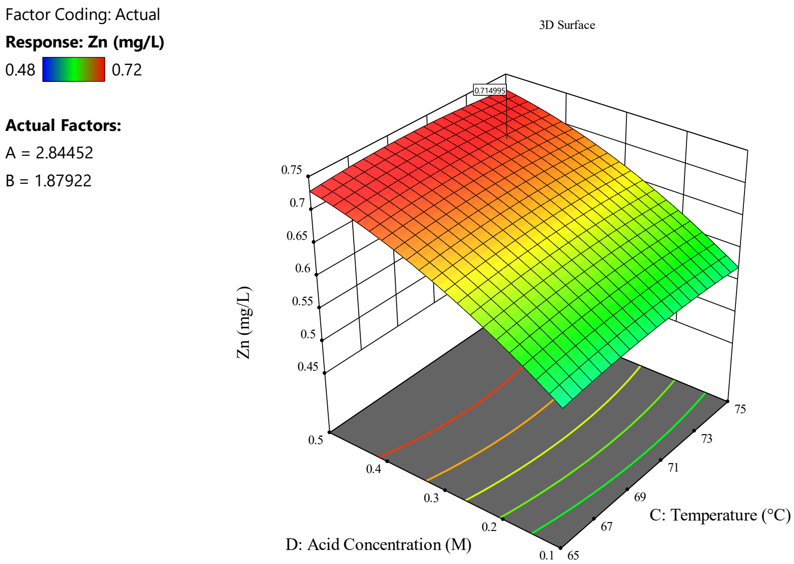


Figure 4.11 (a) and (b): 3D surface response plots showing the impact of two variables on Na recovery (mg/L): (a) pH and contact time; (b) temperature and acid concentration

Zn extraction reaches a maximum at approximately 0.72 mg/L, at pH values 4–5 and contact times of 2–4 h, under optimal conditions of 69–72 °C and concentrations of 0.1 – 0.3 M acid. As shown in Figure 4.11 (a) Zn showed much solubility at low pH. This may be attributed to the behavior of Zn, with increasing pH depending on the solution matrix, Zn may be present in precipitates, and pH values of less than 3 have been recommended (Ghosh and Kartha, 2025; Yan et al., 2014). In terms of contact time, Zn requires a long contact time to achieve maximum recovery and time is required for interactions with the acid ligands (Skrzekut et al., 2022). Figure 4.10 reveals that Zn requires a high temperature and moderate concentration, although quite low concentrations of Zn (0.48 - 0.72 mg/L), suggesting Zn is present in a minor composition in the tailings' matrix (Altinkaya et al., 2019b; Baibatsha et al., 2021).

4.4.3.9 Optimum extraction parameters of elements from platinum mineral tailings

Table 4.7 shows the optimum extraction parameters, which, on the basis of the comprehensive analysis of the 3D surface plots and experimental data from the BBD, for element recovery from platinum mineral tailings were determined through a statistical evaluation of 29 experimental runs.

Table 4.7: The optimum extraction parameters for leaching essential elements from platinum mineral tailings

Parameter	Optimum Value
pH	2.0
Contact time	5.0 hours
Temperature	70 °C
Acid concentration	0.5 M

The optimum pH of 2.0 was similar to that reported by Dacera and Babel (2006) who reported >90% extraction for Cr, Cu, Ni, and Zn at pH 2.33 using citric acid (del Mundo Dacera and Babel, 2006), and Gaber et al reported 88–96% removal of heavy metals at pH 2–3 during comparative studies with citric acid and oxalic acid, with the contact time stretching from 4 hrs to days (Gaber et al., 2011). A notable study from 2025 on rare earth element (REE) leaching from phosphogypsum using citric acid reached a 90.11% dissolution efficiency under optimized conditions: 2 mol/L citric acid, 343 K temperature, and 180 minutes duration (Ray et al., 2025). These finding supports the effectiveness of organic acid leaching systems and support the optimization conditions found in the current study. Similarly, a 2024 study on recovery of Ca and Zn from low-grade waste via RSM-CCD methodology reported a 75.05% recovery for Cd and 86.13% for Zn under optimal conditions: pH 2.5, 70°C, and 2.5 hours. (Kolbadinejad and Ghaemi, 2024). Table 4.8 shows results obtained using optimized conditions.

Table 4.8: Leaching efficiency of recovering essential elements from the platinum mineral tailings using optimized parameters

Element	Platinum mineral tailings after treatment to reduce heavy metals	Result obtained after leaching residue with 0.5 M acetic acid and citric acid (mg/L)	Leaching efficiency (%)
Al	10.57 ± 0.025	ND	0
As	ND	ND	0
Ca	318.12 ± 0.20	134.38 ± 0.13	42.24
Cr	11.2 ± 0.05	ND	0
Cd	ND	ND	0
Cu	4.14 ± 0.02	0.04 ± 0.001	0.97
Fe	1558.32 ± 0.66	0.71 ± 0.01	0.05
K	212.5 ± 0.10	96.92 ± 0.08	45.61
Mg	836.65 ± 0.55	296.88 ± 0.20	35.48
Mn	157.8 ± 0.20	43.29 ± 0.21	27.43
Na	693.03 ± 0.28	446.46 ± 0.15	64.42
Pb	0.35 ± 0.01	ND	0
Zn	4.4 ± 0.025	0.74 ± 0.01	16.82

As shown in Table 4.8, the extraction efficiency of the organic acid mixture of acetic acid and citric acid was Ca (42.42%), Cu (0.97%), Fe (0.05%), K (45.61%), Mg (35.48%), Mn (27.43%), Na (64.42%) and Zn (16.62%). The leaching efficiencies achieved show the potential feasibility of converting platinum mine waste into valuable mineral supplements for aquaculture. Platinum mineral tailings may reduce fish farming cost by eliminating the need for expensive commercial mineral premixes in fish feed formulation. Extraction of these essential elements prior to using tailings for other applications like road surfacing has positive environmental impact. These recovered essential elements are not lost into the environment thus reducing negative environmental impact of acid drainage. This has potential to create a new revenue stream from previously considered waste material.

Sefako et. al (2019) improved platinum group metal recovery with H_2SO_4, with percentage recoveries of Cu (73.3%), Ni (82.9%) and Fe (8.1%) from oxidized platinum ores (Sefako et al., 2019). Woodall et. al (2024) explored the use of a pH swing process to optimize the extraction of Ca and Mg from platinum group metal tailings using various acids; the highest extraction efficiency obtained was with citric acid: Ca (10.29%) and Mg (81.73%) (Woodall et al., 2024b). Mohamed et. al (2016) investigated the extraction of elements via thermochemical treatment followed by leaching with $(NH_3)_4SO_4$, and reported Al (60%) , Fe (35%), Si (32%), Cr (27 %) and Mg (25%) (Mohamed et al., 2016a). The variations in leaching efficiencies may be attributed to the different mineralogical compositions of the tailings. Elements bound with carbonates may be readily soluble even with weak acids and facilitated by increasing acid strength (Gunarathne et al., 2022). Some elements may be bound to complex bonds that are difficult to separate using acid leaching; for example according to Terry (1983), some elements may be associated with crystalline phases of silica, with strong Si-O bonds that require aggressive conditions for dissolution to occur (Terry, 1983; Sanchez-Segado et al., 2022).

4.5 Formulation of fish feed pellets

Fish feed pellets were formulated by combining groundnut oil processing waste and solutions containing various elements extracted from platinum mineral tailings. The mineral solution taken from platinum tailings uses a mixture of organic acids; acetic-citric acid mixtures show great potential as a mineral supplement for aquaculture feeds. Table 4.9 shows the elemental analysis of the mineral solution.

Table 4.9: Elemental analysis of the mineral solution

Parameter/Element	**Result (mg/L)**
Arsenic (As)	ND
Calcium (Ca)	132.41 ± 0.62
Chromium (Cr)	ND
Cadmium	ND
Copper (Cu)	0.04 ± 0.001
Iron (Fe)	0.69 ± 0.007
Potassium (K)	95.23 ± 0.31
Magnesium (Mg)	295.64 ± 0.59
Manganese (Mn)	41.09 ± 0.29
Sodium (Na)	445.91 ± 0.45
Lead (Pb)	ND
Zinc (Zn)	0.72 ± 0.009

As shown in Table 4.9, the elemental composition of the mineral solution included high concentrations of sodium (445.91 mg/L), magnesium (295.64 mg/L), and calcium (132.41 mg/L). There are also moderate levels of potassium (95.23 mg/L) and manganese (41.09 mg/L), with low levels of copper (0.04 mg/L), iron (0.69 mg/L), and zinc (0.72 mg/L). The raw platinum tailings were pretreated with a mixture of NaOH and $NaHCO_3$ to reduce harmful elements in the solution (Junuzović et al., 2019; Zendelska et al., 2022). Heavy toxic metals were not detected, and this elemental composition (Table 4.9) is good for aquaculture feed formulation. It lowers the risk of heavy metal toxicity while providing the essential minerals needed for fish growth and development (Lall and Kaushik, 2021b; Jamil Emon et al., 2023). Heavy metal toxicity can harm fish growth, reproduction, and overall health, posing a risk to the sustainability of the aquaculture industry (Lall and Kaushik, 2021b). Essential minerals such as calcium, magnesium, sodium, potassium, iron, zinc, copper, and manganese are necessary for bone formation, maintaining bodily systems, balancing acids and bases, enzyme functions, and other vital processes in fish (Muralisankar et al., 2022). Fish generally can absorb microminerals from water in their natural environment and feed (Hossain and Yoshimatsu, 2014). They can take in microminerals through their gills or the surface of their bodies. However, the amount of minerals that fish may absorb from the environment is not enough to meet their dietary needs. Therefore, they require additional supplements to their feed (Chanda et al., 2015). The mineral solution (Table 4.9) directly addresses mineral dietary requirements for aquaculture. The extracted solution provides: Na (445.91 mg/L) meeting osmotic regulation requirements, Mg (295.64 mg/L); supporting enzyme activation and ATP metabolism, Ca (132.41 mg/L); contributing to bone formation (though requiring supplementation) and the absence of toxic metals ensuring feed safety.

4.5.1 Proximate and elemental analysis of groundnut oil processing waste

Many authors have reported that groundnut oil processing waste, is a suitable plant protein source for replacing fishmeal (Garduño-Lugo and Olvera-Novoa, 2008b; Waliyar et al., 2013; Jannathulla et al., 2017; Kamuhu et al., 2019; Mokolopi, 2022; Ndebele-Murisa et al., 2024). It is known to be very tasty and has good binding properties, and contains a high crude protein content of 40 – 45% (Siddiqua and Khan, 2022). Table 4.10 shows the results of raw groundnut processing oil waste before formulation compared with results in the literature (Ajah and Asuquo, 2018). It has substantial carbohydrate (58.23%), lipid (15.91%), and protein (11.07%) contents. The mineral content of groundnut waste has K (5051.34 mg/kg), high among other elements and moderate amounts of Mg (962.67 mg/kg), Ca (892.51 mg/kg), and Na (892.00 mg/kg) are present.

Table 4.10: Proximate analysis of groundnut oil processing waste

Parameter	**Groundnut oil processing waste (Arachis hypogaea L)**	**Proximate and mineral compositions of Arachis hypogaea L. cake by Ajah and Asuquo (2018)**
Carbohydrates (%)	58.23 ± 0.69	53.016 ± 0.166
Protein (%)	11.07 ± 0.09	9.47 ± 0.435
Lipids (%)	15.91 ± 0.27	16.04 ± 0.264
Ash (%)	3.35 ± 0.09	10.72 ± 0.384
Fibre (%)	2.18 ± 0.05	3.583 ± 0.219
Moisture Content (%)	3.78 ± 0.02	10.40 ± 0.435
As (mg/kg)	ND	–
Ca (mg/kg)	892.51 ± 0.25	3662.760 ± 7.540
Cr (mg/kg)	ND	–
Cd (mg/kg)	ND	–
Cu (mg/kg)	6.27 ± 0.017	–
Fe (mg/kg)	77.36 ± 0.895	341.460 ± 9.610
K (mg/kg)	5051.34 ± 0.80	1768.800 ± 2.880
Mg (mg/kg)	962.67 ± 0.35	531.330 ± 2.720
Mn (mg/kg)	2.33 ± 0.030	–
Na (mg/kg)	892.06 ± 0.25	1892.160 ± 2.400
Pb (mg/kg)	ND	–
Zn (mg/kg)	0.186 ± 0.0035	–

The comparison reveals important differences groundnut seed cake K content (5051.34 mg/kg) vs. literature (1768.80 mg/kg) which is significantly higher, then groundnut seed cake protein content (11.07%) vs. literature (9.47%), which is moderately higher and groundnut seed cake Ca content (892.51 mg/kg) vs. literature (3662.76 mg/kg) which is lower. These variations reflect processing methods, groundnut variety, and regional soil conditions, emphasizing the potential need for mineral supplementation to improve the mineral content.

These results are also comparable to those of Nankya et. al (2021) who analyzed nutrient contents of peanut varieties, and reported similar mineral profiles in groundnuts. The varieties with the highest mineral contents were 420.34 mg/kg (Na),527.20 mg/kg (Mg), 510.13 mg/kg (Ca), 44.96 mg/kg (Fe) and 18.79 mg/kg (Zn). The proximate analysis results were 48.17% (Protein), 8.20% (lipid) and 3.86% (ash) (Nankya et al., 2021). The relatively low fibre content (2.18%) of the groundnut cake is beneficial for aquafeed, as high fiber can lower digestibility and nutrient absorption in many fish species. Likewise, the moderate ash content (3.35%) indicates a suitable mineral load that can be enhanced by the mineral solution from the tailings to reach optimal mineral profiles in the final feed formulation. The lack of detectable levels of harmful elements such as As, Cd, and Pb (marked as ND in Table 4.10) further confirms that this material has potential for use in aquafeed. This combination addresses the challenge of mineral supplementation in plant-based aquafeeds, which often need additional nutrients to meet the dietary demands of farmed fish. In the following sections, the interaction of the ingredients and their respective nutritional responses during feed formulation and processing is discussed.

4.5.2 Formulation of feed using RSM methodology

The experimental design employed a Box–Behnken approach with three independent variables: mineral solution concentration (A), groundnut oil processing waste percentage (B), and moisture content (C). This design is widely recognized in formulation because of its effectiveness in optimizing multiple process variables simultaneously while minimizing the number of experimental runs (Das et al., 1993;Ma et al., 2021; Manuel Pais-Chanfrau et al., 2021; Tumuluru, 2013;Zhang et al., 2021). The statistical analysis of the experimental data involved developing second-order polynomial models to describe the relationships between the independent variables and the response variables (Szpisják-Gulyás et al., 2023).

Table 4.11, shows the coded factors and the experimental results observed for the suggested seventeen (17) runs.

Table 4.11: Coded factors and proximate and elemental analysis of formulations (F1 – F17)

F	*MS (ml)*	*GW (g)*	*MC (%)*	*C (%)*	*P (%)*	*L (%)*	*Ca (mg/kg)*	*Cu (mg/kg)*	*Fe (mg/kg)*	*K (mg/kg)*	*Mg (mg/kg)*	*Na (mg/kg)*	*Mn (mg/kg)*	*Zn (mg/kg)*
1	60	40	12.5	24.8	1.39	2.95	100.58	0.34	8.52	2961.89	323.17	1008.95	0.863	0.019
2	60	25	15	26.2	1.82	3.5	991.81	6.74	90.13	5340	1144.11	1180.33	8.997	0.195
3	60	10	12.5	57.8	11.2	15.5	100.66	0.32	8.25	2982.63	325.34	1009.12	0.919	0.018
4	37.5	40	10	57.2	11.1	15.2	557.93	3.68	8.42	4209.85	767.64	1095.48	4.579	0.112
5	15	10	12.5	54.6	10.3	14.3	999.43	6.21	79.73	5071.18	1111.27	1178.45	8.04	0.202
6	37.5	40	15	39.4	5.74	8.51	547.41	3.49	1.63	4353.92	711.85	1103.72	4.511	0.12
7	60	25	10	39	5.62	8.35	998.16	6.55	71.38	5113.34	1144	1172.95	8.475	0.188
8	37.5	25	12.5	26	1.76	3.41	548.32	3.58	0.72	4333.73	758.69	1092.84	4.956	0.1
9	37.5	25	12.5	41.2	6.28	9.2	544.76	3.77	5.12	4344.81	743.98	1096.13	4.988	0.1
10	15	25	10	41.7	6.41	9.36	100.09	0.29	7.98	2974.42	326.72	1008.18	0.79	0.017

11	37.5	25	12.5	38.9	5.58	8.3	552.22	3.62	4.03	4248.76	739.06	1106.44	4.724	0.105
12	37.5	25	12.5	26.3	1.84	3.51	553.7	3.66	11.84	4192.92	741.24	1104.56	4.654	0.104
13	37.5	10	15	42.2	6.57	9.56	553.67	3.35	5.12	4365.76	734.17	1101.73	4.506	0.096
14	37.5	10	10	54.9	10.4	14.4	547.72	3.67	4.03	4309.52	704.93	1105.44	4.429	0.1
15	15	25	15	39.5	5.77	8.55	993.81	6.24	80.29	4989.18	1063.67	1189.84	8.352	0.201
16	15	40	12.5	39.5	5.78	8.55	98.78	0.28	7.45	2971.18	321.04	1008.47	0.918	0.018
17	37.5	25	12.5	40	5.92	8.74	548.86	3.87	11.84	4238.47	749.78	1105.92	4.583	0.098

4.5.2.1 Fermentation of each formulation

Research shows that fermentation can improve the nutrient availability and bioavailability of feed. Fermentation can help increase the digestibility of nutrients, minerals and phytochemicals available to digestive enzymes (Nkhata et al., 2018). Furthermore, several studies have shown that fermentation lowers the levels of antinutritional factors, crude fiber, and harmful substances (Jannathulla et al., 2017). Nutrients include elements such as Ca, Fe, Cu, Mn, and Zn (Chen et al., 2022). Yeasts have been reported to improve the immune and antioxidant systems of fish, mainly because of their components such as β-glucan, mannoproteins, and nucleic acids (Siddik et al., 2024; Sultana et al., 2024).

4.5.2.2 Lack of fit and model assessment

The validity of the prediction models and analysis was described using R^2, adjusted R^2, Predicted R^2, predicted R^2, adequate precision and lack of fit. The statistical analysis is shown in Table 4.12.

Table 4.12: Model validation of proximate and elemental analysis

	C	**P**	**L**	**Ca**	**Cu**	**Fe**	**K**	**Mg**	**Na**	**Mn**	**Zn**
Std. Dev.	1.21	0.3622	0.4567	3.81	0.2196	4.47	7.33	4.59	6.54	0.1984	0.0066
Mean	40.54	6.08	8.94	549.29	3.51	23.91	4176.56	730.04	1098.15	4.66	0.1055
C.V. %	2.97	5.96	5.11	0.6943	6.26	18.69	1.76	3.55	0.5957	4.25	6.27
R^2	0.9898	0.9897	0.9899	0.9999	0.9917	0.9918	0.9961	0.9931	0.9907	0.9956	0.9912
Adjusted R^2	0.9875	0.9873	0.9876	0.9999	0.9898	0.9812	0.9911	0.9915	0.9885	0.9946	0.9891
Predicted R^2	0.9826	0.9823	0.9828	0.9998	0.9857	0.9174	0.9602	0.9863	0.9833	0.9921	0.9873
Adeq Precision	53.7504	53.3548	53.9369	485.746	58.5489	25.1022	41.9445	64.0534	54.5099	82.8038	57.1684
Lack of Fit (p-values)	0.6958	0.6887	0.7011	0.4733	0.4819	0.2708	0.2583	0.0924	0.1429	0.5687	0.9671

As shown in Table 4.12, the closeness of values between R^2, adjusted R^2, and predicted R^2 values across all parameters suggests that the models are valid without overfitting (Burestan et al., 2020). This is important in feed formulation studies, where obtaining accurate predictions is crucial for commercial use. The adequate precision values, from 25.10 for Fe to 485.75 for Ca, significantly exceed the minimum of 4.0 usually needed for reliable model predictions in experimental design studies. These values confirm that the signal-to-noise ratio is strong enough for the models with confidence, allowing for precise optimization of the feed formulation parameters (Azeem et al., 2024; Fazeli Burestan et al., 2020). All the parameters have p-values well above the conventional minimum $\alpha = 0.05$, indicating that the models effectively represent the true relationships between factors and responses without a significant lack of fit. The general lack of fit statistics across all the parameters provides confidence that the RSM models can be reliably used for feed optimization and prediction within the experimental design model (Azeem et al., 2024).

4.5.2.3 Normal plots of residuals

Normal probability plots of residuals are important in the RSM for checking whether model errors follow a normal distribution. According to Myers, Montgomery, and Anderson-Cook (2009), normal probability plots help identify potential problems in the model, such as outliers, nonconstant variance, or the need for data transformation (Myers et al., 2009) When residuals closely follow a straight line in these plots, the errors are random and symmetric. Normal plots describe the residuals plotted against the expected normal order statistics. Possible wide deviations from linearity in these plots can affect the reliability of the results and predictive accuracy. Normal plots, used with other diagnostic tools, help identify these issues and confirm that the fitted model is adequate (Sai et al., 2023; Wasan et al., 2023). S–shaped curves suggest deviation from normality and may require remeasurements (Sai et al., 2023), and too many points extremely far from normality indicate outliers that should be investigated (NIST, 2003). The normal plots of the residuals are shown in Figures 4.12 (a) – (c) for carbohydrates, proteins and lipids and in Figures 4.13 (a) – (c) for the elements.

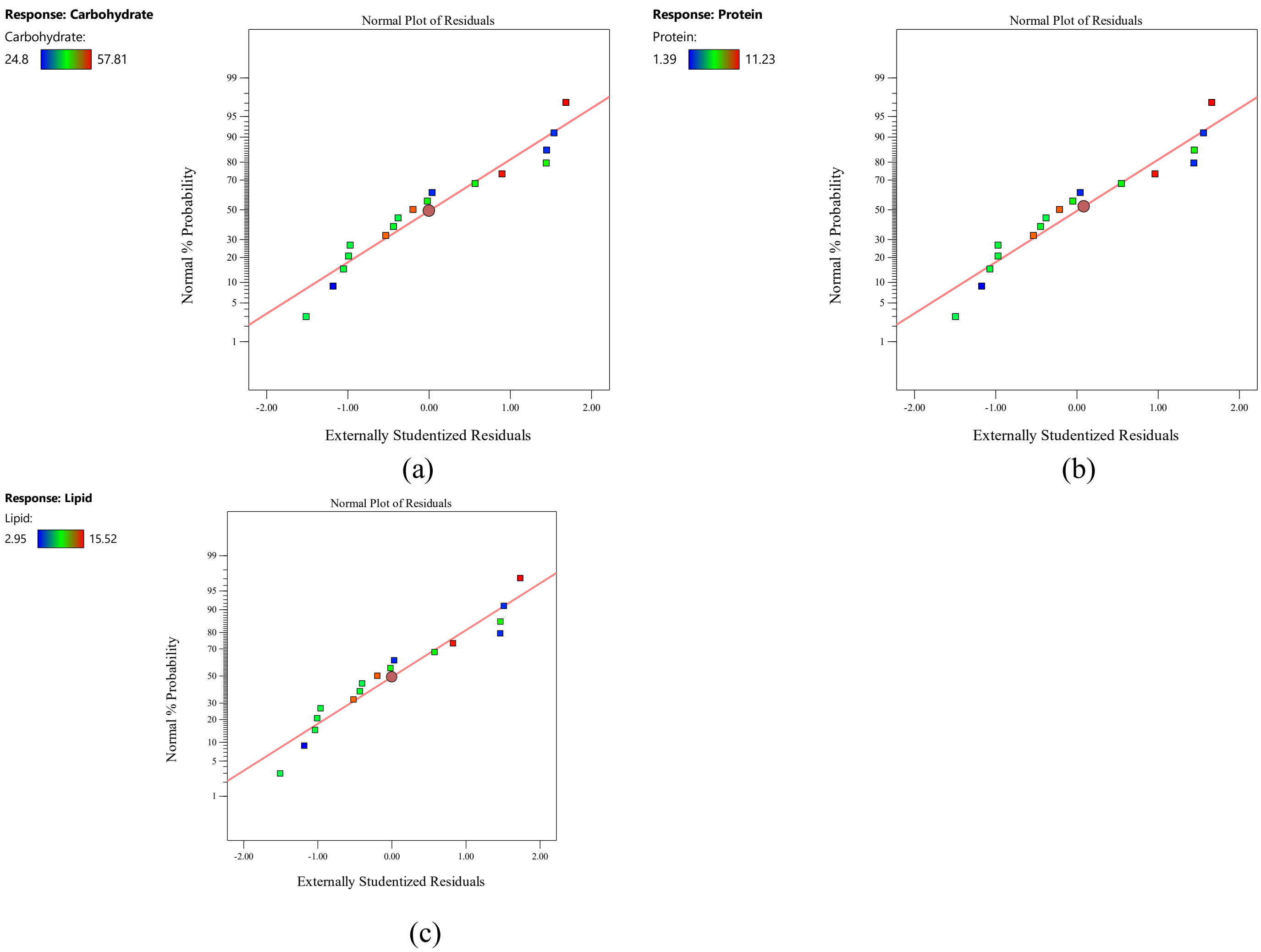
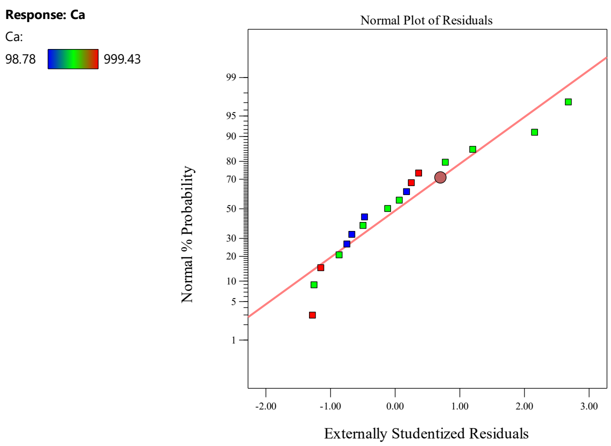


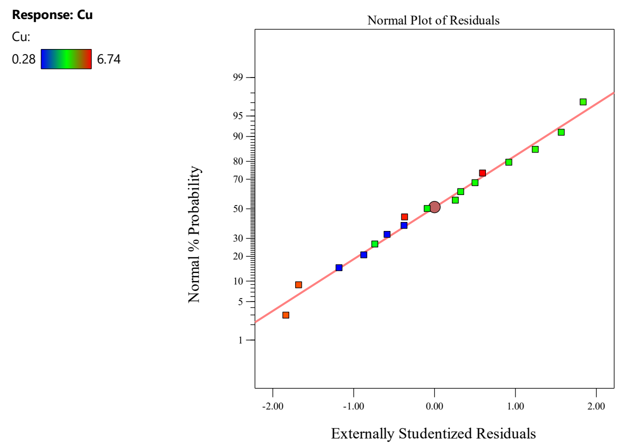
Figure 4.12 (a) – (c): Normal plot of residuals showing the distribution of the response variables (a) carbohydrate, (b) protein and (c) lipids.

The normal probability plot for carbohydrate content, Figure 4.12 (a), shows that the model fits well, with externally studentized residuals spanning from -2.00 – 2.00. These residuals closely follow the theoretical normal distribution line. This strong linear relationship means that the RSM model effectively captures the variation in carbohydrate response, which ranges from 24.8% to 57.81% across the experimental design space. Therefore, following the normality assumption supports the reliability of confidence intervals and statistical conclusions for carbohydrate optimization (Sai et al., 2023; Wasan et al., 2023). Similarly, for protein content, the residuals showed acceptable normality, Figure 4.12 (b), with externally studentized residuals falling within acceptable limits of -2.00 – 2.00. This finding indicates that the model performs well across the protein response range of 1.39 – 11.23%. The narrow protein range suggests that the experimental factors mainly affect other nutritional components instead of the protein content directly. Nevertheless, the normal distribution of residuals confirms the model's ability to predict protein variations accurately (Danbaba et al., 2015). The lipid content normal probability plot, Figure 4.12 (c), shows residuals ranging from -2.00 – 2.00, and it closely follows the normal distribution line. This confirms that the model effectively predicts lipid responses (2.95 – 15.52%) (Myers et al., 2009; Sai et al., 2023).

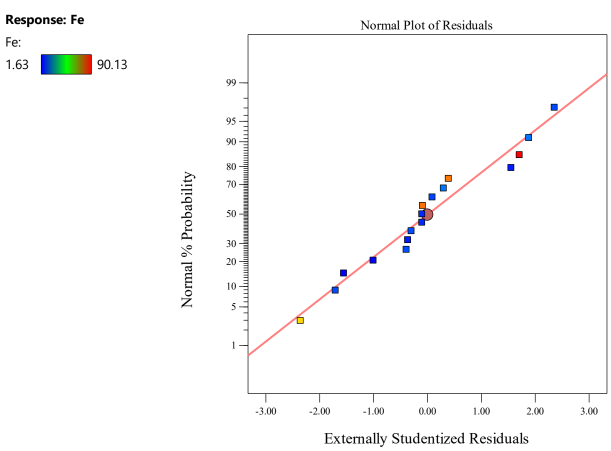
Figure 4.13 (a) – (h) shows the normal plots of the residuals for the elements.



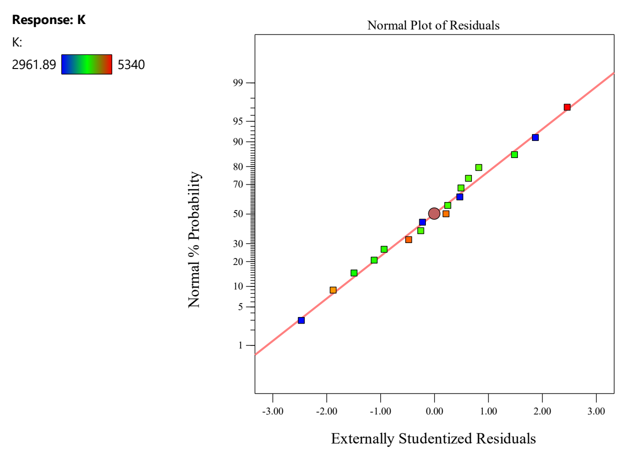
(a)



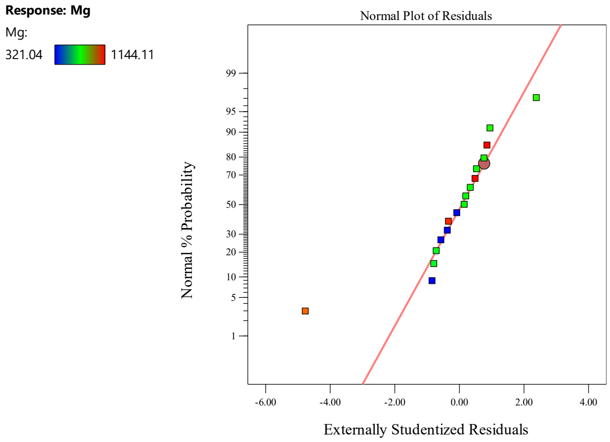
(b)



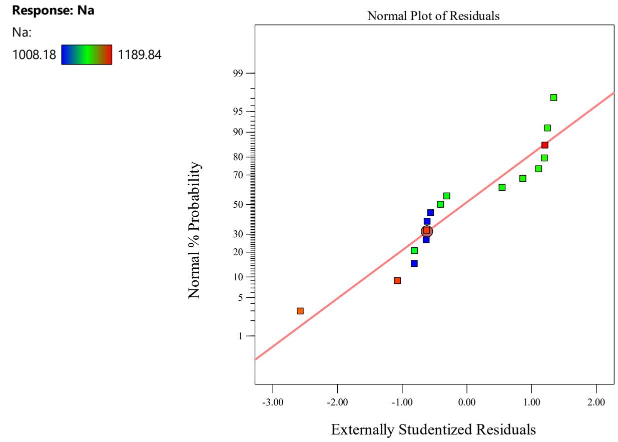
(c)



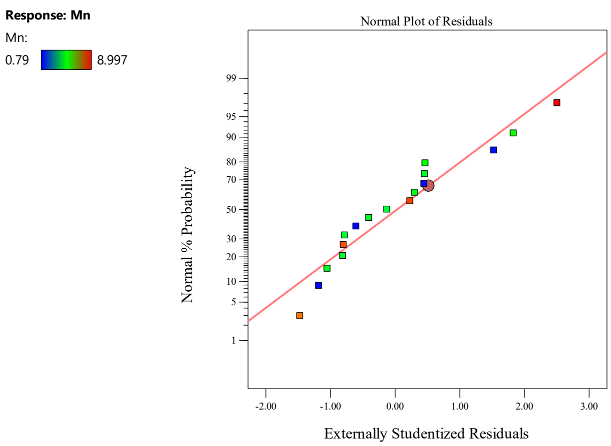
(d)



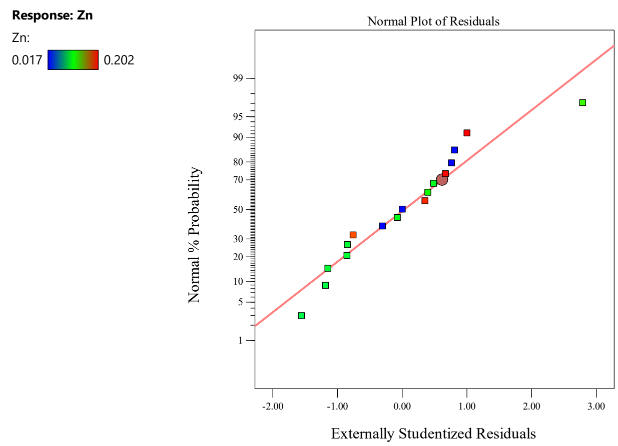
(e)



(f)



(g)



(h)

Figure 4.13 (a) – (h): Normal plot of residuals showing the distribution of the response variables (a) Ca, (b) Cu, (c) Fe, (d) K, (e) Mg, (f) Na, (g) Mn and (h) Zn.

For the Ca residuals, they show good normality (Figure 4.13 (a)), with responses ranging from 98.78 to 999.43 mg/kg. This indicates effective model prediction across experimental conditions. The wide response range suggests that both experimental factors significantly influence calcium content (Sai et al., 2023). The Cu residuals show acceptable plots, as shown in Figure 4.13 (b), with a response range of 0.28 – 6.74 mg/kg. This finding indicates that the model effectively predicts Cu variations across experimental conditions (Montgomery, 2013). The Fe content residuals show a normal distribution (Figure 4.13 (c)), between 1.63 and 90.13 mg/kg, with the studentized residuals being evenly distributed around the theoretical line. This broad range indicates a strong effect of both the mineral solution and groundnut oil processing waste on the Fe content (Myers et al., 2004). The K residuals show good normality (Figure 4.13 (d)), with responses between 2961.89 and 5340 mg/kg. This indicates strong model performance for this macronutrient (Danbaba et al., 2015; Javanbakht and Ghoreishi, 2017). The Mg normal probability plot shows some slight deviation from good normality (Figure 4.13 (e)), with residuals between -6.00 and 4.00. However, these values are still acceptable for RSM analysis; according to Khuri (2017), minor deviations from normality in residual plots may not significantly affect the validity of the model, unless other diagnostic plots indicate serious problems (Khuri, 2017). The overall pattern supports the model's adequacy for optimizing magnesium in feed formulation. The Na residuals show good normality (Figure 4.13 (f)), with responses between 1008.18 and 1189.84 mg/kg. This indicates consistent model performance under various experimental conditions. The narrow response range suggests stable sodium levels across different formulation combinations (Sai et al., 2023). The Mn residuals show good normality, as shown in Figure 4.13 (g), with a response range of 0.79 – 8.997 mg/kg. This validates the model's effectiveness in predicting manganese variations (Danbaba et al., 2015). The Zn residuals show good normality (Figure 4.13 (h)), ranging from 0.017 to 0.202 mg/kg. The normal distribution of residuals confirms the RSM model's reliability for zinc optimization decisions (Myers et al., 2009). The statistical validation ($R^2 > 0.99$ for most parameters) confirms the achievement of the primary objectives; successful extraction of essential elements from platinum tailings, and successful predictable formulation: RSM models confirm precise optimization.

4.5.2.4 Optimization of nutrients

The eleven (11) response variables (carbohydrate, protein, lipid, Ca, Cu, Fe, K, Mg, Na, Mn and Zn) were correlated with the independent variables using the second-order polynomial as represented by equation (4.1):

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_{12} AB + \beta_{13} AC + \beta_{23} BC + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 Y \quad (4.1)$$

where A = mineral solution (ml), B = groundnut oil processing waste (g) and C = moisture content (%).

4.5.2.4.1 Carbohydrate content

The carbohydrate content in the experimental feed formulations ranged from 24.8% to 57.8% (Table 4.11), with an average of 40.54% (Table 4.12). A linear regression model was produced:

$$Carbohydrate = 139.88278 + 0.92593A - 1.87303B - 12.36117C - 0.01331AB - 0.04716AC - 0.03360BC - 0.00208A^2 + 0.04776B^2 + 0.50800C^2$$

Mineral solution (A) has a positive coefficient of 0.926, and (B) groundnut waste has a negative coefficient of -1.873, which indicates that mineral supplementation improves carbohydrate availability and groundnut processing waste decreases carbohydrate availability. This decrease can be an advantage to the carnivorous fish that has a poor carbohydrate digestive ability (Kamalam et al., 2017). This slight decrease may be due to the possibility that the binding interactions of carbohydrates with minerals can result in a lower apparent carbohydrate content according to proximate analysis. This happens because the bound carbohydrates may not be completely accessible for digestion and absorption (Prodić et al., 2023). In general, the carbohydrate content aligns with research by Agbo and Boateng (2011), who reported similar carbohydrate compositions in groundnut byproducts used in aquafeeds (N. W. Agbo et al., 2011). Similarly, Kari et al. (2023) reported that groundnut waste serves as an excellent carbohydrate source in fish feed formulations (Kari et al., 2023).

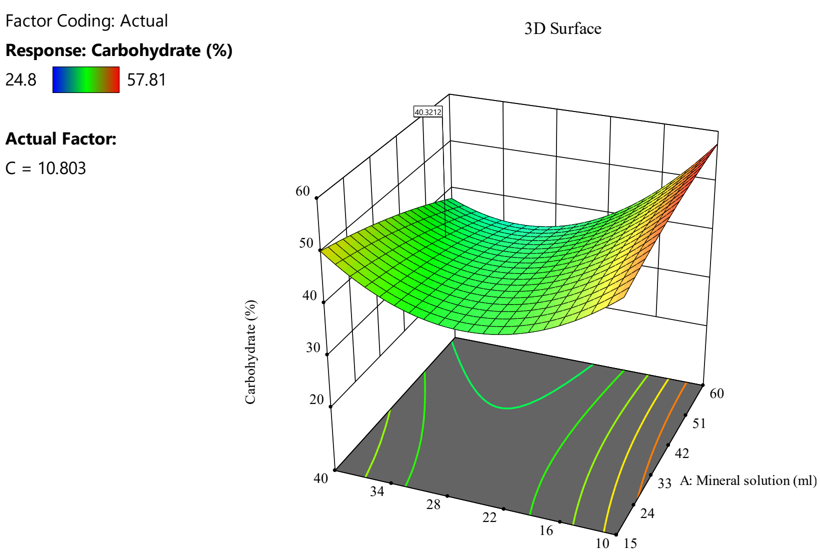


Figure 4.14: 3D response surface plot showing how two variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect carbohydrate content.

The 3D surface response surface plot (Figure 4.14) illustrates that the maximum carbohydrate content in the pellets was obtained at relatively higher levels of groundnut oil processing waste and relatively lower levels of mineral solution. This observation is consistent with the findings of Monday et al., 2024, who reported that the carbohydrate content in fish feed is relatively directly proportional to the proportion of plant-based ingredients (Monday et al., 2024). $R^2 = 0.9898$) and C.V = 2.97% (Table 4.12) demonstrated the reliability of the model in determining the carbohydrate content in the fish feeds. Carbohydrates are important in fish diets, are energy sources, that are important for the formation of glycoproteins and glycolipids, and their carbon structure acts as a building block for creating nonessential amino acids, nucleic acids, and other essential biomolecules (Adéyèmi et al., 2020). The general optimal range for carbohydrate is usually between 20% and 40% dry weight for most fish species (National Research Council, 2011a).

4.5.2.4.2 Protein content

The protein content varies across the formulations, ranging from 1.39% to 11.20% (Table 4.11) with an average of 6.09% (Table 4.12). The linear regression equation was

$$\text{Protein} = 35.63294 + 0.27659A - 0.55578B - 3.68167C - 0.00397AB - 0.01404AC - 0.01027BC - 0.00063A^2 + 0.01425B^2 + 0.15152C^2$$

Positive results of coefficient (A) mineral solution (0.277) demonstrate that mineral supplementation enhances protein utilization, which complies with the body of evidence indicating that trace minerals such as zinc, iron, and copper are necessary to synthesize proteins and metabolize amino acids (Lall and Kaushik, 2021a). Similarly to carbohydrate, the protein content was strongly positively correlated with the percentage of groundnut waste (r = 0.994). This suggests that groundnut oil processing waste can contribute to the protein fraction of the feed, making it a potential ingredient for meeting the protein needs of aquaculture species (Agbo et al., 2011; Zhao et al., 2023).

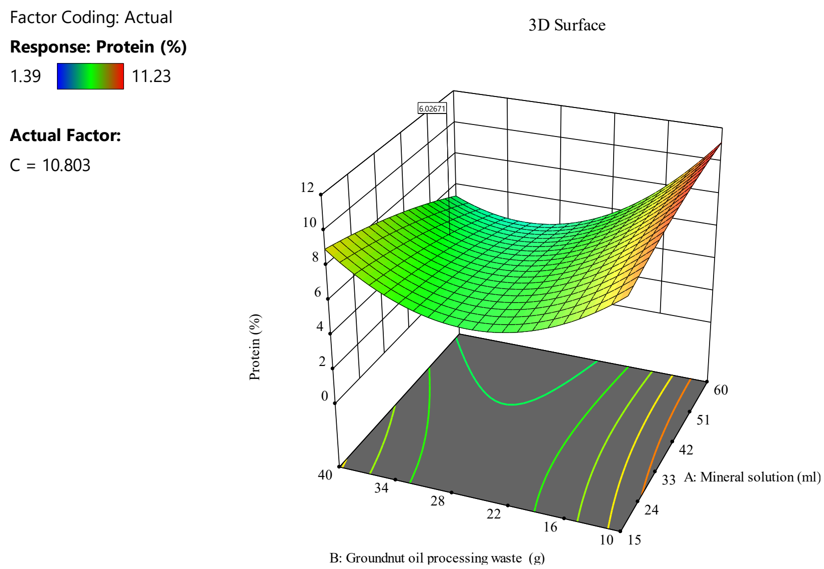


Figure 4.15: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect protein content.

The 3D surface plot (Figure 4.15), revealed that the protein content increased in a manner directly proportional to the increase in groundnut oil waste. These findings show that groundnut oil waste contributes to the protein content, which matches existing research on oilseed by-products as protein sources in aquafeeds (Agbo et al., 2011). The high R^2 value of 0.9897 and CV value of 5.96% in Table 4.12 support the reliability of determining the protein content in the formulated fish feed pellets (Myers et al., 2009). In terms of protein content, the positive effect of groundnut waste was consistent across all mineral solution concentrations, though it slightly decreased at higher mineral solution concentrations. This suggests that while groundnut waste is the main contributor to protein content, high mineral solution levels affect protein availability, and high mineral contents can form complexes with proteins, affecting the availability of protein (Balasubramanian et al., 2023; Mokolopi, 2022). The protein content is low considering that fish feed protein requirements range from approximately 25% to 70%, depending on the species and growth stage (Teles et al., 2020).

4.5.2.4.3 Lipid content

The lipid content in the experimental formulations ranged from 2.95% to 15.50% (Table 4.11) with an average of 8.93% as shown in Table 4.12. The linear regression equation for determining the lipid content for given levels of each factor was as follows:

$$\text{Lipid} = 46.69867 + 0.35189A - 0.71633B - 4.69033C - 0.00507AB - 0.01796AC - 0.01253BC - 0.00078A^2 + 0.01817B^2 + 0.19264C^2$$

The positive correlation between lipid content and the percentage of groundnut waste (r = 0.994) shows that groundnut oil processing waste is a great lipid source in fish feed formulations. This finding aligns with the known composition of groundnut oil processing waste, which retains a significant amount of oil content even after extraction (Aluyor et al., 2009; Mahfoud et al., 2023). The lipid levels in high-groundnut waste formulations are suitable for many fish species, potentially reducing the need for extra lipid supplementation in practical feed formulations (Siddiqua and Khan, 2022).

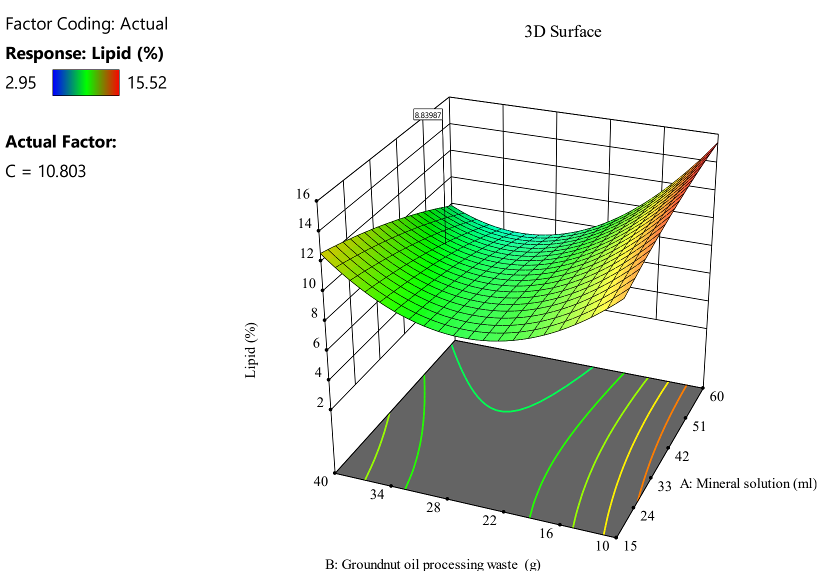


Figure 4.16: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect lipid content.

The 3D response surface plot (Figure 4.16) illustrates that there is a similar trend to that of protein. Lipid content increased with increase of groundnut oil waste, indicating that groundnut oil waste contributes significantly to the lipid fraction, which is consistent with the literature highlighting the effects of ingredient proportions on lipid content in formulated fish products (Siddiqua and Khan, 2022). The high R^2 value of 0.9899 and coefficient of variation (C.V.) of 5.11% as shown in Table 4.12, indicate reliability of the determination of the lipid content in the formulated fish feed (Myers et al., 2009). The lipid requirements generally range from 10% and 20% dry weight for the formulation of fish feeds (Huerta-Ortiz et al., 2018). Lipids are important, especially the highly unsaturated fatty acids, for fish growth and reproduction (Carr et al., 2023). They also act as a vital energy source during the embryonic and larval phases, in conjunction with proteins and carbohydrates (Lall and Kaushik, 2021a).

4.5.2.4.4 Moisture content

The moisture content had minimal direct effects on macronutrient and mineral composition, with correlation coefficients near zero for most response variables (Gracious and Nandakumar, 2024). The limited influence of moisture content on nutritional composition suggests that this factor mainly affects the physical pellet characteristics rather than the nutritional profile, although it is still an important factor for pellet manufacturing and stability (Ruscoe et al., 2005; Auwal et al., 2017; Erinle et al., 2020). The optimum moisture content for the fish feed formulation was determined to be approximately 10%, balancing nutrient availability with pellet stability (Zhang et al., 2021).

4.5.2.5 Elemental composition analysis

The formulated pellets were analyzed to determine the content of essential elements: Ca, Cu, Fe, K, Mg, Na, Mn and Zn.

4.5.2.5.1 Ca content

The Ca content varied greatly across formulations, ranging from 98.78 to 999.43 mg/kg (Table 4.11), with a very high positive correlation with the mineral solution concentration (R = 0.999) (Table 4.12). The linear regression equation for Ca:

$$Ca = 3879.28 + 33.7235A + 18.2162B - 699.534C + 0.66709AB - 4.00031AC - 0.10980BC - 0.00536A^2 - 0.98665B^2 + 35.8572C^2$$

The mineral solution (A) shows a positive coefficient (33.72), which indicates how the platinum mineral tailing extract directly contributes to calcium supplementation. The Ca content indicated that the mineral solution concentration was the key factor regardless of the percentage of groundnut waste. In similar studies reported, the results of the studies by Goodrich et.al (2022) indicate that Ca was supplemented through the addition of the mineral solution to the feed formulation (Goodrich et al., 2022).

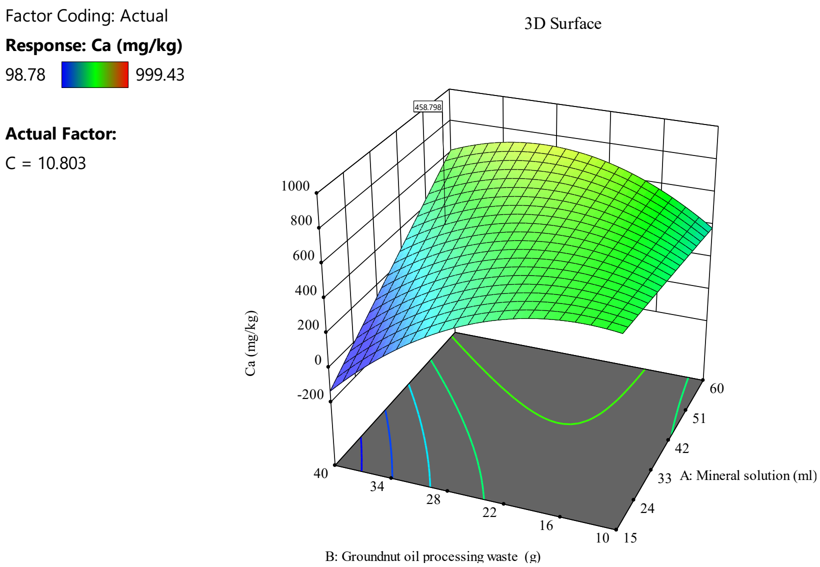


Figure 4.17: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect Ca content.

The 3D response surface plot (Figure 4.17) illustrates this result, showing that maximum calcium content is achieved at higher levels of mineral solution. This finding highlights the importance of mineral supplementation in meeting the calcium needs of fish, especially plant-based ingredients that generally have lower calcium levels than animal-derived ingredients (Lall and Kaushik, 2021a; Muleya et al., 2024). Compared with the other elements, the model has a very high R^2 value = 0.9999 and a C.V. = 0.69 % (Table 4.12), confirming its reliability for determining the Ca content in the fish feed formulations. The dietary requirements for Ca are in the range 5 – 7 g/kg. Calcium is vital for bone development and metabolic functions, so its optimization is key for fish health and growth performance (Lall and Dumas, 2015).

4.5.2.5.2 Cu content

The Cu content ranged from 0.28 to 6.74 mg/kg, as shown in Table 4.11, and was positively correlated with the mineral solution concentration (R = 0.996). The determination of Cu content produced a quadratic regression equation:

$$Cu = 25.9653 + 0.25239A + 0.14478B - 4.81017C + 0.00441AB - 0.02560AC + 0.00087BC - 0.00050A^2 - 0.00738B^2 + 0.24120C^2$$

The copper model revealed a complex interaction of factors includes the terms (AB, AC, BC) and quadratic terms (A^2, B^2, C^2), The positive coefficient for mineral solution (A) and groundnut oil processing waste (B) indicates that increasing the amount of the solution and the groundnut processing oil waste increases the Cu content, while the negative coefficients for moisture content (C) suggest that increasing it decreases copper content in the formulated pellets.

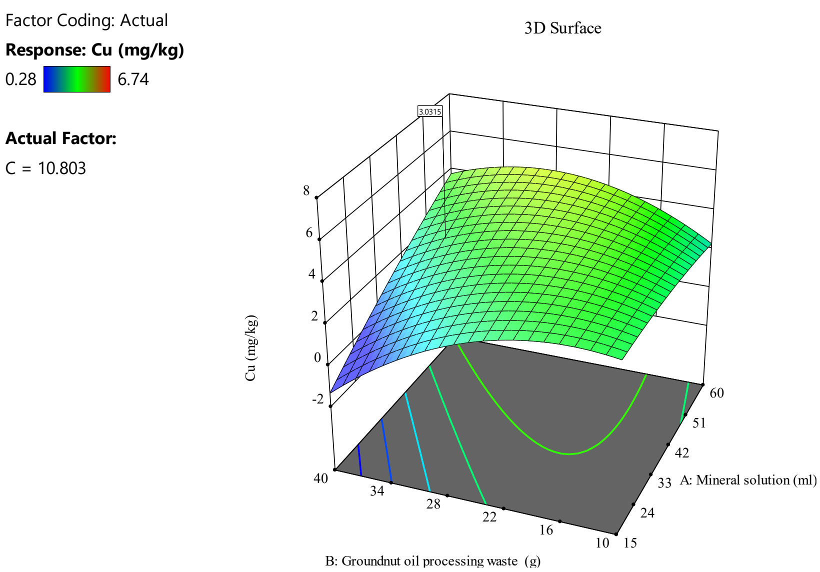


Figure 4.18: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect Cu content.

Then the 3D response surface plot (Figure 4.18) clearly illustrates this relationship, showing that maximum Cu content is achieved at specific combinations of the variables rather than at their extremes. This result is similar to previous findings that Cu supplementation in fish feed requires careful optimization due to potential interactions with other minerals and its role as an essential micronutrient (El-Sayed et al., 2023). Cu is crucial for fish growth and functions as an enzyme cofactor, with typical requirements between 3 and 5 mg/kg in complete feed (Kokkali et al., 2023; Lall and Dumas, 2022).

4.5.2.5.3 Fe content

The Fe content varied from 0.72 to 90.13 mg/kg, with a moderate positive correlation with the mineral solution percentage (R = 0.785) (Table 4.12). The iron content analysis yielded a quadratic regression equation:

$$\mathrm{Fe} = 372.504 - 4.03539A + 2.33408B - 54.1870C + 0.05374AB - 0.23804AC - 0.05253BC + 0.07578A^2 - 0.08571B^2 + 2.74780C^2$$

That describes the relationship between the iron content and formulation variables. Like the Cu, the Fe content showed interaction of the terms (AB, AC, BC) and quadratic terms (A^2, B^2, C^2). The negative coefficients for the main effects suggest that increasing the concentration of mineral solution (A), at high moisture content (C) it decreases iron levels in the formulated pellets. While the trace mineral concentrations were positively related to the mineral solution percentage, the increase in the Fe concentration was due to the groundnut oil processing waste itself, also considering the C.V. was 18.69% (Table 4.12). This finding was supported by the proximate and elemental analyses of the groundnut waste before mixing (Table 4.10), which revealed relatively high concentrations of Fe compared with the elemental analysis of the mineral solution (Table 4.9). The observed phenomena highlight that the Fe content was derived mainly from groundnut oil processing waste rather than from the addition of the mineral solution. Phytic acid, which is a significant form of phosphorus in plant-based feed ingredients, can attach to important minerals such as iron, zinc, and calcium, forming insoluble complexes that lower their availability for animals (Boccio et al., 2002).

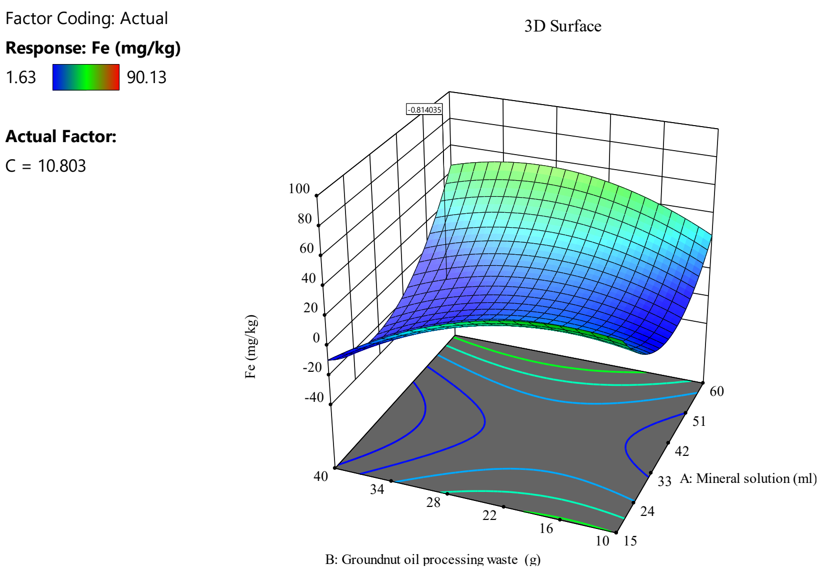


Figure 4.19: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect Fe content.

The 3D response surface plot (Figure 4.19) shows that the maximum iron content is achieved at specific combinations of the variables. This finding aligns with research by Prabhu et al. (2019), who reported that Fe supplementation in fish feed requires careful optimization due to potential interactions with other minerals (Prabhu et al., 2019). The model's high R^2 value of 0.9918 (Table 4.12) confirms its reliability in determining the Fe content in fish feed formulations. Generally, the Fe requirements in fish feeds are between 30 and 100 mg Fe/kg of feed. Fe is required for different metabolic processes, such as oxygen transport and enzyme function (Lall and Dumas, 2022).

4.5.2.5.4 K content

The K content ranged from 2961.89 to 5340.00 mg/kg (Table 4.11) and was positively correlated with mineral solution concentration (R = 0.982) (Table 4.12). The following is the linear regression equation for the K content for given levels of each factor:

$$K = 13197.18 + 98.6073A + 35.1538B - 1885.61C + 1.5402AB - 7.9471AC + 0.5855BC - 0.47461A^2 - 2.37665B^2 + 91.6432C^2$$

The positive coefficients for mineral solution (A) and groundnut oil processing waste (B), with lower moisture content (C), increased the potassium levels in the formulated pellets, with the mineral solution having the most significant effect. The consistent relationship between the mineral solution concentration and micromineral content highlights the contribution of the mineral solution, which is supported by Kokkali et. al (2023) who reported that the level of macro-elements can be supplemented in fish feed formulations, especially when plant-based ingredients that may have unbalanced mineral profiles are used (Kokkali et al., 2023). The 3D response surface plot, in Figure 4.20, displays the relationship, revealing that the maximum K content is achieved at higher levels of mineral solution.

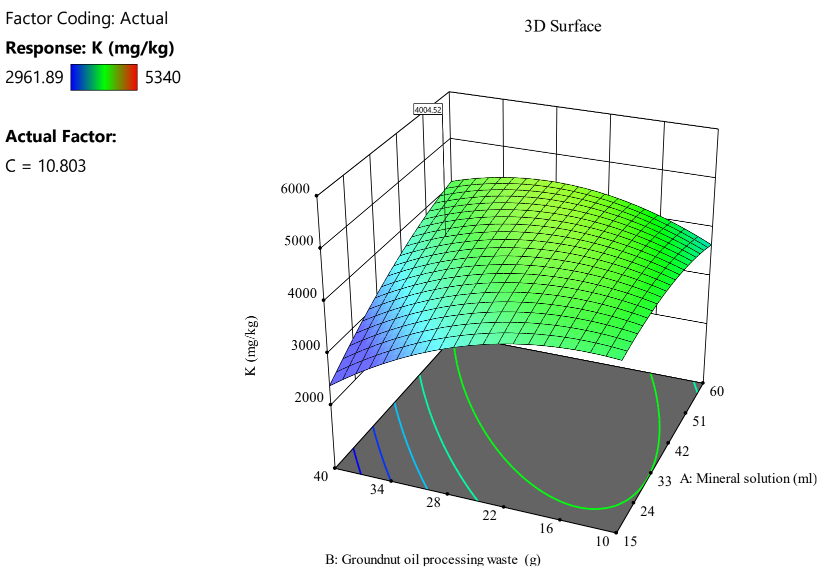


Figure 4.20: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect the K content.

Similar findings were reported by Kousoulaki et al. (2023), who emphasised the importance of mineral supplementation in fish feed to ensure adequate K levels (Kokkali et al., 2023). The model's high R^2 value (0.9961) and C.V. (1.76%) in Table 4.12 validate the reliability of the determination of K content in the fish feed formulations (Arfini et al., 2025). High K levels contribute to groundnut oil processing waste, which is naturally rich in this essential mineral (Booth and Fielder, 2016). In general, the requirement in feed is 1-3 g/kg; however some species like the *salmonella* require up to 8g/kg in their diets. K regulates osmotic pressure, maintains acid–

base balance and improves nerve and muscle function (Booth and Stewart Fielder, 2016).

4.5.2.5.5 Mg content

The Mg content ranged from 321.04-1144.11 mg/kg (Table 4.11) and was positively correlated with the mineral solution percentage (R = 0.996). The following is the linear regression equation for Mg content:

$$Mg = 3423.18 + 29.6686A + 25.1866B - 592.531C + 0.58375AB - 3.27484AC - 0.56687BC - 0.03592A^2 - 0.92516B^2 + 30.6014C^2$$

The positive coefficients for mineral solution (A) and groundnut oil processing waste (B) indicate increased Mg levels, whereas the negative coefficient for moisture content (C) suggests that increasing this component slightly decreases the Mg content in the formulated pellets. This inverse relationship between moisture content and Mg content may be due to interactions between Mg and other components in the groundnut waste, as reported in the literature (Lall and Kaushik, 2021a; Muntean et al., 2024).

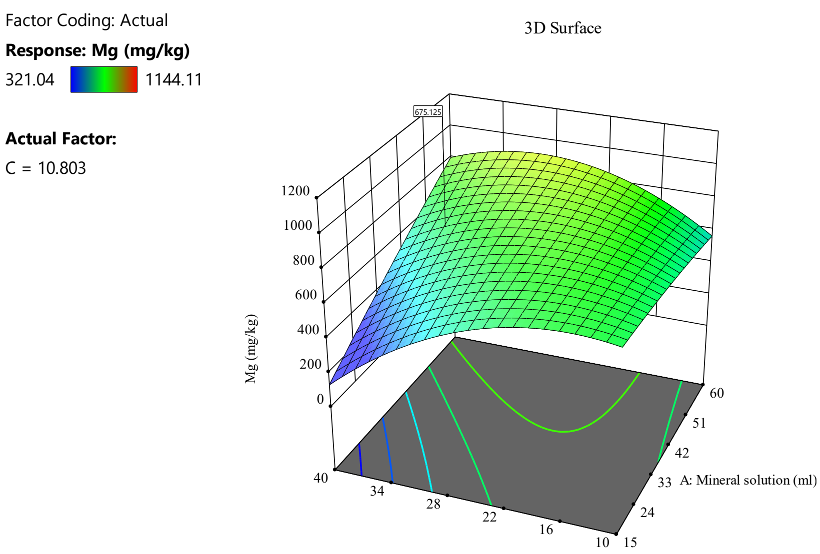


Figure 4.21: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect Mg content.

The 3D response surface plot (Figure 4.21) displays the relationship, confirming that the maximum Mg content occurs at relatively high levels of mineral solution and slightly low levels of groundnut oil processing waste. The model's high R^2 value (0.9931) and C.V. (3.5%) (Table 4.12) confirm its reliability for determining the Mg content in fish feed formulations (Arfini et al., 2025). The range of dietary requirements for Mg in fish feeds is 400 - 700 mg/kg. Mg contributes to enzyme activation, osmoregulation, and ATP metabolism (Lall and Dumas, 2015).

4.5.2.5.6 Na content

The Na content ranged from 1008.18 - 1189.84 mg/kg (Table 4.11), Similarly to K was positively correlated with the mineral solution percentage (R = 0.995). The following equation is used for the Na content for given levels of each factor:

$$\mathrm{Na} = 1748.58 + 7.47676A + 2.37656B - 137.251C + 0.12579AB - 0.77458AC + 0.07967BC - 0.01353A^2 - 0.19147B^2 + 6.95936C^2$$

The positive coefficients for mineral solution (A) and groundnut oil processing waste (B) indicate that increasing their amounts improves Na levels, whereas the negative coefficient for moisture content (C) suggests that at high moisture content the Na level slightly decreases in the formulated pellets (Booth and Stewart Fielder, 2016). This is further displayed by the 3D response surface plot, in Figure 4.22, showing that maximum sodium content is achieved at higher levels of mineral solution and slightly lower moisture content.

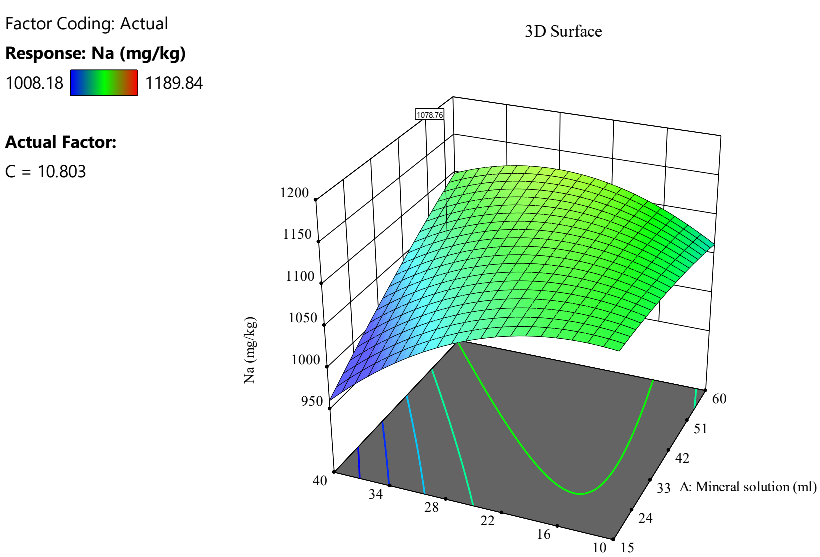


Figure 4.22: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect the Na content.

The high R^2 value (0.9907) and C.V. (0.5957 %) in Table 4.12 confirm the reliability for the determination of the Na content in the fish feed formulations via this model (Domingo et al., 2019).

The Na dietary requirements range from 1 –3 g/kg. Na is important for osmoregulation and nerve function (Booth and Friedel, 2016; Lall and Kaushik, 2021).

4.5.2.5.8 Mn content

The Mn content ranged from 0.79-8.997 mg/kg, and it was positively correlated (R = 0.997) (Table 4.12). The following equation is the linear regression equation for determining the Mn content for the given levels of each factor:

$$Mn = 31.2334 + 0.26287A + 0.22875B - 5.78535C + 0.00523AB - 0.03129AC - 0.00097BC + 0.00005A^2 - 0.00943B^2 + 0.29550C^2$$

Where, at a high amount of groundnut oil processing waste and with increasing mineral solution (A), high concentrations of Mn are obtained. The 3D response surface plot (Figure 4.23) displays this relationship, showing that the maximum Mn content was achieved at higher levels of mineral solution and slightly lower levels of moisture content.

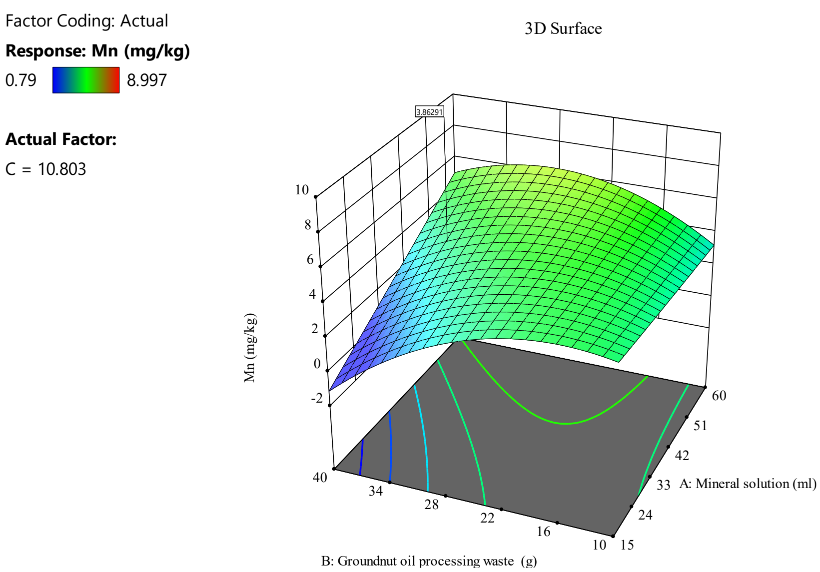


Figure 4.23: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect the Mn content.

The model's high R^2 value (0.9956) and C.V. (4.25 %) in Table 4.12 suggest good reliability for the determination of Mn content in the fish feed formulations (Arfini et al., 2025; Domingo et al., 2019). The results of the elemental analysis of raw groundnut processing waste (Table 4.9), confirm the improvement in micronutrients in the formulated fish feed pellets. The dietary requirements for Mn are in the range 2 – 15 mg/kg (National Research Council, 2011b). The observed range falls within typical dietary needs. Manganese is important for enzyme function and bone development in fish (Lall and Kaushik, 2021a).

4.5.2.5.9 Zn content

The Zn content ranged from 0.017-0.202 mg/kg, indicating a positive correlation with the amount of mineral solution (R = 0.995) (Table 4.12). The linear regression equation for the determination of the Zn content for given levels of each factor was as follows:

$$Zn = 0.84043 + 0.00586A + 0.00157B - 0.14595C + 0.00014AB - 0.00079AC + 0.00008BC + 6.02\times10^{-6}A^2 - 1.79\times10^{-4}B^2 + 0.00733C^2$$

The positive coefficients for mineral solution (A) and groundnut oil processing waste (B) indicate that with lower moisture content (C) increased the Zn levels. The 3D response surface plot in Figure 4.24 displays this relationship, with the maximum zinc content obtained at high levels of mineral solution.

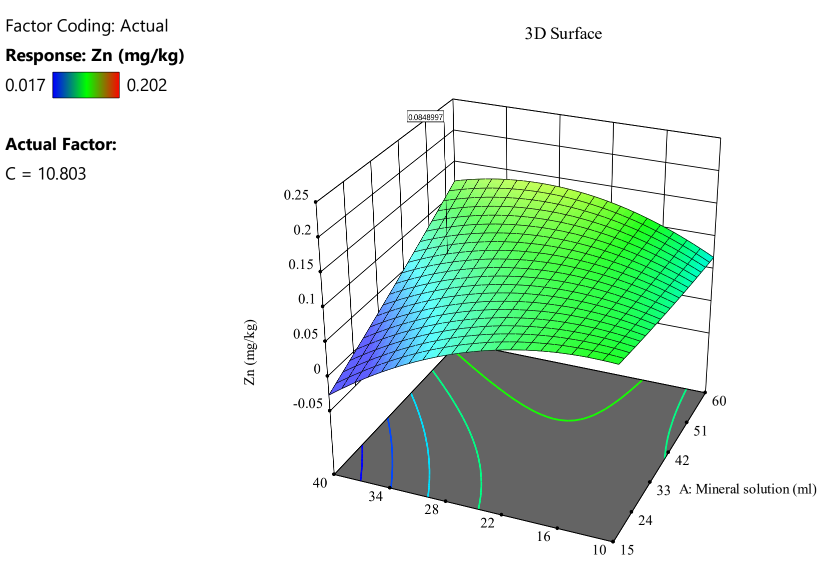


Figure 4.24: 3D response surface plot showing how two key variables, mineral solution (% of feed) and groundnut oil processing waste (% of feed), affect Zn content.

The model's high R^2 value (0.9912) and C.V. (6.27 %) in Table 4.12 confirm the reliability of the determination of the Zn content in the fish feed formulations (Zakaria et al., 2012; Domingo et al., 2019). Zinc is essential for growth, immune function, and enzyme activity in fish. The dietary requirements for Zn are in the range 20 – 200 mg/kg (Gatlin, 2003; Lima et al., 2024). The optimization process, which uses the desirability function approach, identified the following optimal formulation: 5% mineral solution, 65% groundnut oil processing waste, and 10% moisture content. This formulation provides fair level of macronutrients (carbohydrates, proteins, and lipids) and minerals (Ca, Cu, Fe, K, Mg, Na, Mn, and Zn) to meet the nutritional requirements of fish (Zhang et al., 2021; Lima et al., 2024).

4.5.2.6 Optimum formulation for fish feed pellets

Table 4.13 shows the optimum formulation selected to maximize both the macronutrient (protein, lipid, carbohydrate) and mineral (Ca, Fe, K, Mg, Na, Cu, Mn, Zn) contents. The highest values for protein, lipid, and minerals were observed at the upper levels of the coded factors for both the mineral solution (% of feed) and the groundnut oil processing waste (% of feed), with varying moisture contents of approximately 10%.

Table 4.13: The optimum formulation for fish feed pellets

Parameter	**Optimal Level**
Mineral Solution Volume	25 ± 0.218 ml
Groundnut oil processing waste	37.5 ± 0.55g
Moisture content of groundnut oil processing waste	11 ± 0.09%

Reported fish feed formulations including plant-based raw materials with various ingredients, some researchers use mineral premixes for essential elements (Prabhu et al., 2018; Monje et al., 1996; Yimer et al., 2023). A premix is a mix of vitamins or minerals, blended in the right amounts with suitable carriers or diluents (Philar et al., 2005; Silva De, 1991). Some reports in the literature in which various fish diets have been formulated include; Idah et. al (2019) who formulated diets with fish meal (40% - 60%), soybean meal (15% - 45%), wheat (7% - 21%), and fish oil (10% – 12%) for carnivorous marine species. They produced proximate analysis ranges of moisture content (71.8% – 74.05%), crude protein content (17.37% – 18.6%), crude lipid content: 6.5% – 7.64% and ash content (2.45% – 3.12%) (Idah et al., 2019). Ovie and Ovie (2006) formulated diets with 40% groundnut cake combined with (14.39% to 38.15%) fish meal,(19% to 43%)

Guinea corn, and (2%) premix (S. O. Ovie and Ovie, 2006) and performed proximate analysis: moisture (4% - 11%), protein (26% - 50%), Lipid (5% - 7%), ash (5% - 9%), fiber (1% - 4%), nitrogen free extracts (18- 45%) (Ovie, Ovie, 2008). Tiamiyu et. al (2013) formulated diets with fish meal 29.12% (constant in all diets), groundnut cake: 0% – 29.12%, cotton seed meal: 0% – 29.12%, yellow corn: 20.14% (constant in all diets), rice bran: 20.14% (constant in all diets), vitamin and mineral premix: 1% (constant in all diets), salt: 0.5% (constant in all diets) and they obtained proximate analysis of moisture: 5.23% – 6.4%, crude protein: 34.6% – 34.9%, crude fiber: 6.11% – 8.34%, ether extract: 7.56% – 7.8%, ash: 3.1% – 4.06% and nitrogen-free extract : 40.3% – 42.8% (Tiamiyu et al., 2013).

4.6 Proximate analysis of the formulated fish feed pellets

Table 4.14: Proximate analysis of the formulated fish feed pellets compared with other fish feed products in the local market

Parameter	**Reported dietary requirements/limits**	**Formulated fish feed**	**Local Market Feed A**	**Local Market Feed B**	**Local market Feed C**
Carbohydrates	20 – 40%	56.62 %	–	–	–
Protein	34 – 50%	10.79 %	32 %	44.5 %	59%
Lipids	5 – 15%	15.91 %	5 %	8 %	14.5%
Ash	7 – 12%	4.26 %	6 %	8.8 %	14 %
Fiber	3 – 10%	2.18 %	–	2.5 %	–
Moisture Content	< 14 %	9.07%	10 %	12 %	8.5 %

The formulated feed containing 10% crude protein from groundnut oil processing waste does not fully meet the nutritional requirements of most cultured fish species, which range from 25 to 50% protein (NRC, 2011). To address this, supplementary agro-based protein sources such as soybean meal, and insect meal are recommended to enrich the formulation. Soybean meal, widely regarded for its balanced amino acid profile and digestibility, could sustainably increase protein levels while maintaining cost efficiency (FAO, 2015). Insect meal provides functional benefits such as enhanced immunostimulation and may reduce environmental footprint (Henry et al., 2015)

Table 4.15: Elemental analysis of the formulated fish feeds compared with other fish feed products in the local market

Element	**Reported dietary requirements/limits**	**Formulated fish feed (**mg/kg)	**Local Market Feed A**	**Local Market Feed B**	**Local Market Feed C**
As	< 1 mg/kg	ND	–	–	–
Ca	3 – 5 g/kg	961.49 ± 0.80	3%	1.3 %	–
Cr	1 – 5 mg/kg	ND	–	–	–
Cd	< 0.050 mg/kg	ND	–	–	–
Cu	3–12 mg/kg	6.54 ± 0.12	–	–	–
Fe	30 – 170 mg/kg	79.71 ± 0.50	–	–	–
K	1 – 3 g/kg	5118.06 ± 2.5	–	–	–
Mg	0.4 – 0.6 g/kg	1090.10 ± 0.85	–	–	–
Mn	2 – 15 mg/kg	6.46 ± 0.11	–	–	–
Na	1 – 3 g/kg	1183.07 ± 1.4	–	–	–
P	5 – 8 g/kg	–	0.7 %	0.88 %	–
Pb	< 0.3 mg/kg	ND	–	–	–
Zn	20 – 115 mg/kg	0.296 ± 0.0050	–	–	–

A comparison of the proximate and elemental analyses of the groundnut cake (Table 4.10) before mixing with the mineral solution and the resulting formulated fish feed pellet (Table 4.9) revealed that while the mineral solutions was a major source of the elements, the groundnut cake had significant contents of K, Zn and Cu. The notable differences in the elemental and proximate contents of the pellets and the groundnut processing oil waste show that the mineral solution contributed to the elemental nutrient levels without affecting the composition of other nutrients, carbohydrates, proteins and lipids. A comparison of the formulated fish feeds with samples from the market (Feed A, B and C), (Table 4.15), revealed that the feeds on the local market lack most of the essential elements as highlighted by the dashes.The formulated fish feed pellet has carbohydrates (Table 4.15) and has potential as a multielement nutrient feed considering that those from the market either have none of the elements or just only Ca and P. In general, fish do not have a particular dietary requirement for carbohydrates; therefore, their availability in a diet provides a relatively inexpensive source of energy, especially for the non-carnivorous fish species (National Research Council, 1993). The carbohydrate utilization by fish varies among different species; Herbivorous and omnivorous fish produce enzymes that help digest carbohydrates, whereas carnivorous fish do not have these enzymes (Arenas et al., 2021). Considering the cost of protein ingredients, getting fish to use carbohydrates and lipids for energy, while reserving proteins for growth, would greatly benefit fish farmers (Daniel, 2016). Carbohydrates, which include starches and sugars, are the cheapest sources of energy for fish diets. While they are not essential, carbohydrates are added to aquaculture diets to lower feed costs and help bind ingredients during feed production (Craig et al., 2017). These findings support the potential of mineral solution enrichment in aquafeed formulation for improving the nutritional quality of plant-based ingredients.

4.7 X-ray diffraction analysis to determine phase and elemental changes

XRD was performed to confirm any key elemental or phase interactions. Figure 4.22 shows the dictograms for the raw platinum mineral tailings, which are presented as RAW-PT (Raw platinum minerals tailings), the fish feed pellet and the residue after extraction of elements.

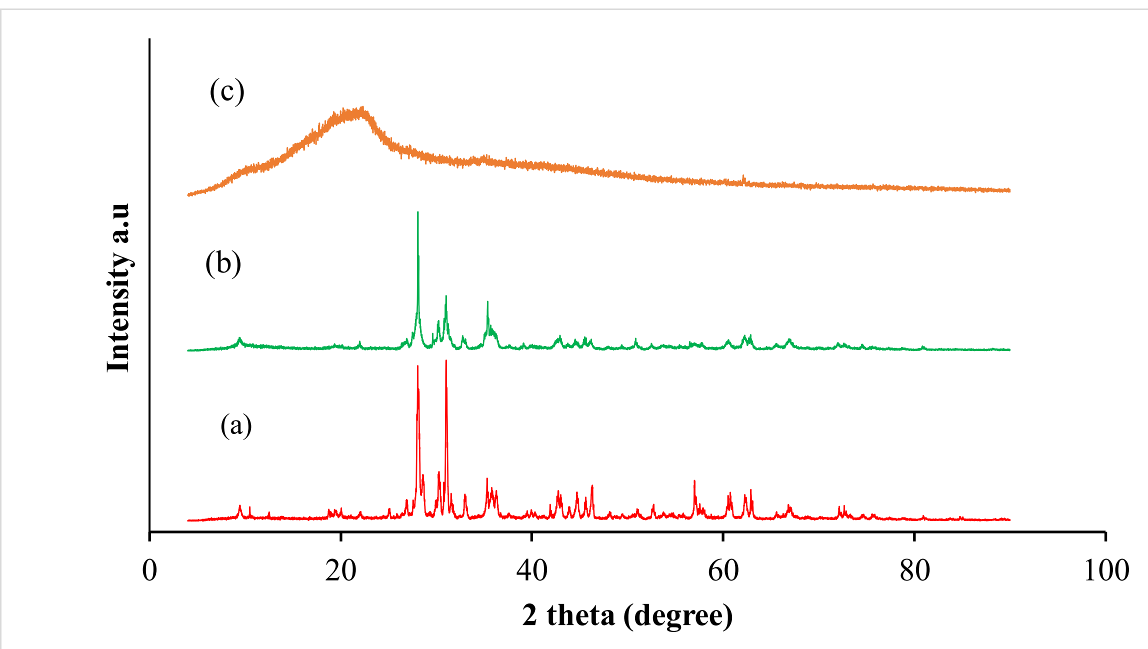


Figure 4.25: XRD diffractograms of (a) raw platinum mineral tailings, (b) platinum residue and (c) fish feed pellets

The X-ray diffractogram (Figure 4.25a) of the raw platinum tailings reveals prominent diffraction peaks within the 30 to 35° 2θ range, with intensities ranging from approximately 1400 to 1500 counts. These peaks are primarily associated with forsterite $(Mg,Fe)_2SiO_4$). The secondary peaks found between 27 and 28° are attributed to plagioclase feldspar ($CaAl_2Si_2O_8$), indicating its notable abundance in the mineral matrix. The peaks detected in the 35– 37 °C range are linked to chromite ($FeCr_2O_4$), which is recognized for its spinel crystal structure. Several other low-intensity peaks points are revealing the presence of secondary minerals such as diopside ($CaMgSi_2O_6$), orthopyroxene, kaersutite, actinolite, and enstatite. The distinct and broad characteristics of the primary peaks suggest a high degree of crystallinity in these major mineral phases. The observed peaksalign with reported ultramafic characteristics of other platinum group tailing findings (Motha et al., 2023; Rabatho et al., 2010).

Considering the residue (Figure 4.25b), the selective organic acid extraction process mainly dissolved secondary silicates while keeping the forsterite matrix intact. This is shown by the increased intensity of the forsterite peaks at 2θ = 18.5° and 21.3° in the residue tailings, compared to the reduced intensity of chromite peaks at 2θ = 31.8° and 36.9° (Borhade et al., 2021; X. Zhang et al., 2020). The final fish feed pellet XRD pattern shows successful mixing of the mineral solution with groundnut oil processing waste. It displays typical calcite precipitation at 2θ = 29.4° from the organic matrix, likely introduced during the palletization process. The continued presence of forsterite and plagioclase diffraction peaks in the final feed confirms the effect of important magnesium and calcium-bearing mineral phases needed for aquaculture nutrition.

The X-ray diffractogram (Figure 4.25c) of the fish feed pellets revealed the absence of distinct crystalline peaks within the examined 2θ range. This indicates the minimal presence of crystalline mineral phases in the material. Rather, the diffraction pattern displays a continuous background between 40° and 80°, which is characteristic of amorphous scattering. The overall amorphous pattern results from the thermal processing conditions experienced during extrusion. These conditions disrupt the organized crystalline structures present in the raw materials, resulting in a predominantly amorphous pellet matrix. This alteration in structure is consistent with previous studies demonstrating that extrusion leads to molecular rearrangement. These processes increase pellet uniformity and digestibility (Liu et al., 2021; Tumuluru, 2023).

4.8 Physical properties of the formulated fish feed pellets

The physical properties of fish pellets are the structural and mechanical characteristics of aquaculture feed pellets, and include measurements such as diameter, length, surface area, and volume (Khater et al., 2014). Density-related properties include bulk density and true density (Moses and Augustina, 2017). Mechanical traits involve durability, crushing load, and expansion ratio (Khater et al., 2014). Aquatic behaviour features are floatability, water stability, and sinking velocity (Vassallo et al., 2005). Water stability is particularly important because it shows how well the pellet keeps its shape when submerged until fish consume it. Poor water stability causes nutrients to leak out quickly, leading to wasted feed and environmental issues from water pollution (Zaabwe et al., 2020). Two key physical properties were investigated: leaching and floatability tests. For leaching (Table 4.16), the ICP-OES analysis after an hour of immersion of the pellets in distilled water revealed leaching rates that consistently ranged between 2.14% and 2.73% across all the tested elements.

Table 4.16: Element pellet leaching analysis

Element	Initial content (mg/kg)	Leached amount (mg/L)	Leaching rate (%)	Detection limit (mg/L)	RSD (%)
Ca	949.29	21.54	2.27	0.01	2.8
Cu	6.81	0.15	2.28	0.001	4.2
Fe	77.91	1.69	2.18	0.01	3.6
K	5098.56	109.37	2.14	0.05	2.1
Mg	1032.06	26.52	2.57	0.01	3.4
Na	1098.15	24.67	2.25	0.02	2.9
Mn	5.66	0.14	2.58	0.001	5.1
Zn	0.11	0.003	2.73	0.001	6.8

The leaching characteristics (Table 4.16) of the formulated fish feed were consistent and had low leaching rates for all the measured minerals, ranging from 2.14% to 2.73%. This consistency indicates good water stability and pellet integrity, which is crucial for reducing nutrient loss and preventing water quality issues in aquaculture systems (Zaabwe et al., 2020). The observed leaching rates of approximately 2.1-2.7% are very low, suggesting potentially better pellet formulation and manufacturing processes (Haetami et al., 2017).

Table 4.17: Floatability test results

	No. of floating Pellets						**% Floating**					
Exp.	5 min	10 min	15 min	20 min	25 min	30 min	5 min	10 min	15 min	20 min	25 min	30 min
1	9	7	4	3	2	1	36	28	16	12	8	4
2	8	7	2	1	1	0	32	28	8	4	4	0
3	7	5	4	3	2	1	28	20	16	12	8	4
4	8	5	5	4	3	1	32	20	20	16	12	4
5	10	6	5	4	3	1	40	24	20	16	12	4

Table 4.18: Statistical analysis of the floatability test

Time	**Mean floating (%)**	**Std dev (%)**	**Coefficient of variation (CV%)**	**Standard error (%)**
5 min	33.6	4.32	12.86	1.93
10 min	24	3.16	13.17	1.41
15 min	16	1.58	9.88	0.71
20 min	11	1.58	14.36	0.71
25 min	9	1.58	17.58	0.71
30 min	2.6	1.14	43.85	0.51

Tables 4.17 and 4.18; the statistical analysis indicates that the fish feed pellets formulated from groundnut oil processing waste mixed with mineral tailings solution have poor floatability characteristics. For example, the high fluctuations in the coefficient of variation (CV) indicate increasing variability in pellet floatability performance over time (Fashina et al., 2019). Research suggests that high-quality floating feeds can achieve over 90% floatability after 50 minutes of water exposure (Wan Nooraida et al., 2023). These low-floating properties may suggest variations in pellet density, binding agents, or processing conditions that impact buoyancy (Felix and Oscar, 2018). To produce floating feeds, these results suggest the need to modify the formulation to enhance floating, as demonstrated in successful formulations of floating pellets, for example, by optimising pellet extrusion parameters. Fish types such as catfish consume sinking pellets better

than floating pellets do (Emam and Mohamed, 2019). In contrast, tilapia feed at all depths and eat both floating and sinking pellets (Milstein and Hernández, 2017). Sinking pellets have several advantages; they offer better water stability, eliminate feed competition from surface-feeding species, and improve access for species with downturned mouths (Haetami et al., 2017; Zaabwe et al., 2020). The production of high-quality floating feeds is achieved by optimizing factors such as temperature, die diameter, and feed preconditioning time during extrusion. This optimization will improve properties such as the expansion ratio, bulk density, floatability, durability, and water absorption (Irungu et al., 2018).

4.9 Comparison with commercial pellets

The elemental analysis of the prototype pellets shows that calcium, 0.96 g/kg, (Table 4.15) and Zn ,0.30 mg/kg are significantly less than the 3 – 5 g/kg, Ca and 20 – 115 mg/kg, Zn range of the recommended dietary ranges as shown in Table 4.15. Conversely, K (5.1 g/kg) and Mg (1.09 g/kg) are optimal compared to the recommended 1 – 3 g/kg and 0.4 – 0.6 g/kg respectively , and this level might disrupt the ionic balance in freshwater depending with the freshwater natural elemental composition (Starling, n.d.). The levels of As, Cr, Cd and Pb that are below the levels of detection aligning with FAO/WHO feed safety limits; <1, 5, 0.05 and 0.3 mg/kg respectively (Saha, Mottalib, et al., 2020). Considering physical properties; leaching of elements it was within 2 – 3 % which is well below the 5% technological threshold that prevents loss of nutrients and eutrophication (Kaddour, 2018). However, physical performance was quite poor: after 5 min floats, only 34% of the pellets could float but 30 min later this was about to 3% (Table 4.18), in comparison, modern extruded feeds are now regularly maintained at floats of >90% after 50 or 60 min at optimal extrusion parameters of barrel temperatures (>120° C), 2 mm dies, and with ≤25% moisture in the feed (Irungu et al., 2018). All data consistently indicate toxicological safety, however, show potential Ca and Zn deficiencies and poor buoyancy; In future this will need specific mineral fortification and extrusion optimization to match production to better quality the manufacturing standards.

4.10 Costing of the Fish Feed Formulation

The economic feasibility of this fish feed formulation shows strong potential for cost-effective production using locally available waste materials. The main raw materials, platinum mineral tailings and groundnut oil processing waste, come with low acquisition costs. Tailings are often underused byproducts stored in containment facilities and sometimes used for road surfacing, while groundnut processing waste is easily sourced from local crop processing operations.

However, the research was quite expensive in terms of analysis. ICP-OES elemental analysis for solutions cost \$5.66 per sample, and for the pellets, it was \$17.34 per sample. The optimization process involved 29 experimental runs based on RSM, which added up to \$492.42 for the runs and replicate analysis. There were also costs for elemental extraction optimization and 15 runs for feed formulation optimization, followed by triplicate verification of optimal conditions, leading to total analytical costs of \$780.30.

While the research was costly due to extensive optimization and validation, the established optimal parameters will allow for future production at significantly lower costs. Raw material acquisition will account for less than 5% of total feed production costs. The demonstrated potential nutrient recovery efficiency and potential detoxification confirm the economic viability of scaling up this process, where analytical costs would be spread out over larger production volumes. This cost structure makes the formulated feed competitive with traditional aquaculture feeds and offers the added environmental advantage of waste valorization, promoting both economic sustainability and circular economy principles in the aquaculture industry.

CHAPTER 5

SUMMARY OF FINDINGS, CONCLUSIONS AND RECOMMENDATIONS

5.1 Summary of findings

This chapter gives the main key highlights of the findings and future recommendations.

5.1.1 Extraction of essential elements

A Box–Behnken design optimization study successfully demonstrated the effectiveness of acetic and citric acid mixtures (0.1–0.5 M) for recovering essential elements from platinum mineral tailings. The 3D surface plots revealed complex interactions between pH (2 – 8), contact time (1 – 5 hours), temperature (65 – 75 °C), and acid concentration that significantly influenced the element recovery rates. Calcium showed the highest recovery potential, reaching 132 mg/L under optimal conditions, while sodium recovery reached the broadest range (240 – 445 mg/L). The statistical analysis demonstrated excellent model performance, with R^2 values exceeding 0.85 for most elements, particularly calcium ($R^2 = 0.9858$), iron ($R^2 = 0.9760$), and zinc ($R^2 = 0.9678$). Low pH conditions (2--3) combined with high acid concentrations (0.3--0.5 M) and moderate temperatures (70 °C) generally favour maximum element extraction, whereas long contact times (3--5 hours) increase the recovery efficiency for most target elements. The predicted versus actual value plots confirmed the reliability of the response surface models, with adequate precision values ranging from 10.80--32.86, indicating sufficient signal–to–noise ratios for practical optimization applications.

5.1.2 Formulation of fish feed pellets

The response surface plots show that the optimization of fish feed pellets using renewable food resources in the form of mineral solutions obtained through the processing of wastes of platinum tailings and groundnut oil is effective. This research highlights the possibility of mineral fortification with a high potential for enhancement: 10-fold Ca (98.78 – 999.43 mg/kg), 55-fold

Fe (1.63–90.13 mg/kg), and 24-fold Cu (0.28 – 6.74 mg/kg). The macronutrient optimization revealed carbohydrate contents ranging from 24.8 – 57.81%, protein contents ranging from 1.39 – 11.23%, and fat contents ranging from 2.95 – 15.52%. The parameters optimized were the volume of the mineral solutions (which ranged from 10 – 40 ml) and the groundnut oil waste (which ranged from 15 – 60 g), with a moisture content of 10.803. The mechanism illustrates the concept of the circular economy in which industrial waste material is always appreciated and massive nutritional enrichments are made, particularly at the level of trace minerals, which was found to be most sensitive to experimental formulation changes. The experiment confirms the formulation of sustainable solutions in the sector of aquaculture feed and is defined by the integrated valorisation of waste substances as well as nutritional enrichment through the application of response surface methodology.

5.2 Conclusions

This research offers a potential sustainable fish feed formulation in Zimbabwe, using two key raw materials: elements extracted from platinum minerals tailings and combined with groundnut oil processing waste. The optimized extraction process using organic acids successfully recovered bioavailable macro- and microelements crucial for fish nutrition; 42% Ca, 45.61% K, 35.48% Mg, 64.42% Na, and 16.62% Zn. The formulated feed exhibited favorable physical properties and mineral composition supportive of aquaculture species requirements. Importantly, this work departs from traditional approaches by valorizing industrial mineral waste in sustainable aquafeed, contributing a novel circular economy model to address both resource scarcity and environmental impact issues facing fish feed production. However, the crude protein content of the current formulation (approximately 10%) falls below recommended levels for most cultured fish species. To bridge this gap, incorporation of complementary agro-based protein sources such as soybean meal or insect meal is proposed to ensure nutritional adequacy while preserving economic and environmental sustainability. Overall, this research provides a basis of potential for mineral-enriched, sustainable fish feed pellets derived from unconventional raw materials. Future work should focus on in vivo feeding trials to evaluate growth performance and health outcomes, exploration of multi-source protein supplementation, and comprehensive life cycle assessment to validate environmental benefits. These efforts will be crucial to translating experimental findings into practical aquaculture solutions and enabling industry adoption.

5.3 Recommendations

This study shows that there is high potential to produce feasible sustainable aquaculture feeds in the use of essential elements extracted from platinum mineral tailings and groundnut oil processing waste as a cheap and extensive alternative to fish meal and the use of vitamin and mineral premixes. Nonetheless, a number of crucial constraints should be mentioned, and further research will be necessary to confirm commercial feasibility and safety. This study has assessed the extraction of elements, formulation parameters and nutritional composition, but there have been no in vivo feeding trials, which remains essential for determining the growth performance, feed conversion efficiency and overall biological value of formulated feeds. Moreover, aflatoxin contamination is one of the key safety issues because, under conditions of tropical storage, groundnut products are highly vulnerable to contamination. Phytic acid, trypsin inhibitors, and oxalates, which are antinutritional factors in groundnut cake, further restrict the bioavailability of protein and mineral absorption. Fermentation was carried out however its effects on mineral bioavailability or antinutrient reduction was not quantified. Future studies should include: before/after fermentation mineral analysis, phytate and antinutrient quantification, digestibility trials and bioavailability assessments. There is need for protein enrichment strategies, as the existing protein content (1.39 – 11.23%) might not satisfy the carnivorous fish species that are commonly required to obtain 30 – 55% dietary protein. It is vital to conduct safety analyses of the platinum mineral extracts in which bioavailability tests and full heavy metal contamination testing should be undertaken to assess the food safety of the feed. There is need for a species-specific optimization of different fish groups (carnivorous vs. omnivorous), life-stage requirements and palatability research among different aquaculture species. Scale-up feasibility studies must be geared toward the development of cost-effective extraction processes and quality control standardization procedures that are economically viable for scaling to commercial levels. Further areas of research could include environmental impact assessment on the basis of life cycle analysis, optimization technology with respect to processing, such as extrusion parameter refinement, and exploration of microencapsulation as a means of nutrient protection.

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LIST OF APPENDICES

APPENDIX A: ICP-OES calibration

Calibration for analysing the elements As, Cr, Pb, Cd, Ca, Cu, Fe, K, Mg, Na, Mn and Zn was performed via the ICP Multi-Element Standard Solution IV provided by Merck (Product No. 1.11355), which contains 23 elements, namely, Ag, Al, B, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, In, K, Li, Mg, Mn, Na, Ni, Pb, Sr, Tl, and Zn, all at a concentration of 1000 mg/L in 6.5% nitric acid. The calibration standards of the ICP Multi-Element Standard Solution IV were analysed on an iCAP 7000 ICP–OES (Thermo Scientific, USA). Serial dilution was performed in 2% HNO_3 (v/v) to make working calibration standards at concentrations of 0.5, 2.0, 10.0 and 50.0 mg/L and a calibration blank of 2% HNO_3. After 30 minutes of instrument warming and plasma stabilization (RF power of 1400 W, nebulizer flow of 0.7 L/minute, and auxiliary flow of 0.5 L/minute), calibration curves were developed, and the correlation coefficient (r^2) was ≥ 0.995 for all the elements. Yttrium was used as an internal standard to perform internal standardization at a concentration of 5 mg/L, and a method detection limit between 0.01 and 0.5 mg/L was reached. The leachates were diluted 1:10 in 2% HNO_3.

APPENDIX B: Optimisation of extraction of elements

Std	Run	F1	F2	F3	F4	R1	R2	R3	R4	R5	R6	R7	R8
		H+	hr.	T°C	M	Ca	Cu	Fe	K	Mg	Mn	Na	Zn
15	6	5	1	75	0.3	121	0.03	0.62	69	238	24	348	0.64
14	7	5	5	65	0.3	121	0.03	0.62	69.8	239	24.9	349	0.64
19	8	2	3	75	0.3	130	0.04	0.68	75	265	29	375	0.68
12	9	8	3	70	0.5	110	0.01	0.62	70	210	22	320	0.55
21	10	5	1	70	0.1	103	0.02	0.53	63	189	19	250	0.53
27	11	5	3	70	0.3	123	0.03	0.63	70.5	240	25.2	351	0.65
10	12	8	3	70	0.1	90	0.01	0.5	58	180	16	240	0.48
7	13	5	3	65	0.5	128	0.02	0.68	88	288	39	438	0.7
16	14	5	5	75	0.3	124	0.03	0.64	71	243	26	353	0.66
4	15	8	5	70	0.3	99	0.01	0.57	62	203	18	308	0.52
23	16	5	1	70	0.5	125	0.02	0.68	87	285	38	435	0.69
5	17	5	3	65	0.1	102	0.02	0.53	62	188	18	249	0.52
24	18	5	5	70	0.5	129	0.02	0.69	95	295	41	445	0.72
2	19	8	1	70	0.3	98	0.01	0.56	61	202	18	305	0.51
18	20	8	3	65	0.3	97	0.01	0.55	60	201	17	302	0.5
29	21	5	3	70	0.3	123	0.03	0.63	70.6	240	25.2	351	0.65
26	22	5	3	70	0.3	123	0.03	0.63	70.6	240	25.2	351	0.65
20	23	8	3	75	0.3	101	0.01	0.58	64	205	19	310	0.53
25	24	5	3	70	0.3	123	0.03	0.63	70.6	240	25.2	351	0.65
1	25	2	1	70	0.3	124	0.04	0.68	72	258	28	365	0.69
9	26	2	3	70	0.1	110	0.03	0.6	66	210	22	300	0.6
22	27	5	5	70	0.1	105	0.02	0.58	70	200	22	265	0.57
17	28	2	3	65	0.3	126	0.04	0.67	73	260	27	368	0.68

13	29	5	1	65	0.3	119	0.03	0.62	68	237	24	347	0.63

APPENDIX C: ICP-OES detection limits

Common mineral tailings elements	Symbol	Wavelength (nm)	LOD Axial (µg/L)	LOD Radial (µg/L)	LOQ Axial (µg/L)	LOQ Radial (µg/L)
Al	Al	167.079	0.07	0.4	0.23	1.33
As	As	189.042	3.2	7.8	10.7	26
Ca	Ca	393.37	0.02	0.10	0.07	0.33
Cd	Cd	214.44	0.25	0.70	0.83	2.33
Co	Co	228.62	0.50	1.50	1.67	5.00
Cr	Cr	267.72	0.50	1.50	1.67	5.00
Cu	Cu	324.75	1.00	2.00	3.33	6.67
Fe	Fe	259.94	0.40	1.00	1.33	3.33
K	K	766.49	3.00	10.00	10.00	33.00
Mg	Mg	279.55	1.00	2.00	3.33	6.67
Mn	Mn	257.61	0.10	0.25	0.33	0.83
Na	Na	589.59	26.00	77.00	87.00	257.00
Ni	Ni	231.60	0.10	0.10	0.33	0.33
Pb	Pb	220.35	2.50	6.50	8.30	22.00
Zn	Zn	213.86	0.35	1.00	1.17	3.33

Note: LOD values are based on 3σ criterion with 99% confidence level. LOQ values are calculated as 10σ for reliable quantification.

Comment:

Statement of Conformity to a Specification or Standard: SAZS 560:1997
Decision Rule Based on: Result implies noncompliance with an Upper limit if the measured value plus the uncertainty exceed the limit
Parameter marked * implies non-compliance
Any Additions to,deviations,or exclusions from the method done? No

Parameters/ Elements	Method Code	Summary of Method (s)
**(all)Mg,Ca,K,Na,Fe,Cu,Zn,Mn,Li,Cr	**CHW101	Acid digestion and AAS finish

Elements	Symbol	Wavelength (nm)	Best LOD Axial (µ	Typical LOD Axial (	Best LOQ Axial (µg	Typical LOQ Axial (µg/L)
Al	Al	167.079	0.027	0.07	0.09	0.23
As	As	189.042	0.676	3.2	2.25	**10.7**
Ca	Ca	393.37	0.01	0.02	0.02	0.07
Cd	Cd	214.44	0.08	0.25	0.27	0.83
Co	Co	228.62	0.25	0.50	0.83	1.67
Cr	Cr	267.72	0.25	0.50	0.83	1.67
Cu	Cu	324.75	0.50	1.00	1.67	3.33
Fe	Fe	259.94	0.20	0.40	0.67	1.33
K	K	766.49	1.50	3.00	5.00	10.00
Mg	Mg	279.55	0.50	1.00	1.67	3.33
Mn	Mn	257.61	0.05	0.10	0.17	0.33
Na	Na	589.59	13.00	26.00	43.00	87.00
Ni	Ni	231.60	0.05	0.10	0.17	0.33
Pb	Pb	220.35	1.20	2.50	4.00	8.30
Zn	Zn	213.86	0.18	0.35	0.60	1.17

Note: LOD values are based on 3σ criterion with 99% confidence level. LOQ values are calculated as 10σ for reliable quantification.

Comment:

Statement of Conformity to a Specification or Standard: SAZS 560:1997
Decision Rule Based on: Result implies noncompliance with an Upper limit if the measured value plus the uncertainty exceed the limit
Parameter marked * implies non-compliance
Any Additions to,deviations,or exclusions from the method done? No

Various Elements	LOD Axial (µg/L)	LOD Radial (µg/L)	LOQ Axial (µg/L)	LOQ Radial (µg/L)
Ag	1.5	2	5	6.7
Al	0.07	0.4	0.23	1.33
As	3.2	7.80	10.70	26.00
Au	10	20.00	33.00	67.00
B	0.75	0.80	2.50	2.67
Ba	0.03	0.14	0.10	0.47
Be	0.02	0.04	0.07	0.13
Ca	0.02	0.10	0.07	0.33
Cd	0.25	0.70	0.83	2.33
Co	0.5	1.50	1.67	5.00
Cr	0.5	1.50	1.67	5.00
Cu	1	2.00	3.33	6.67
Fe	0.4	1.00	1.33	3.33
K	3	10.00	10.00	33.00
Li	0.1	0.30	0.33	1.00
Mg	1	2.00	3.33	6.67
Mn	0.1	0.25	0.33	0.83
Na	26	77.00	87.00	257.00
Ni	0.1	0.10	0.33	0.33
Pb	2.5	6.50	8.30	22.00
Pd	5	10.00	17.00	33.00
Pt	10	20.00	33.00	67.00
Rb	0.01	0.05	0.03	0.17
Zn	0.35	1.00	1.17	3.33

Note: LOD values are based on 3σ criterion with 99% confidence level. LOQ values are calculated as 10σ for reliable quantification. .

Comment:

Statement of Conformity to a Specification or Standard: SAZS 560:1997
Decision Rule Based on: Result implies noncompliance with an Upper limit if the measured value plus the uncertainty exceed the limit
Parameter marked * implies non-compliance
Any Additions to,deviations,or exclusions from the method done? No

Parameters/ Elements	**Method Code**	**Summary of Method (s)**
**(all)Mg,Ca,K,Na,Fe,Cu,Zn,Mn,Li,Cr	**CHW101	Acid digestion and AAS finish

Element	Symbol	Wavelength (nm)	LOD Axial (µg/L)	LOD Radial (µg/L)	LOQ Axial (µg/L)	LOQ Radial (µg/L)	Comments
Au	Au	267.595	10	20	33	67	preconcentration recommended
Pt	Pt	214.423	10	20	33	67	Fire assay preconcentration recommended
Pd	Pd	340.46	5.00	10.00	17.00	33.00	preconcentration recommended
Rh	Rh	343.49	8.00	15.00	27.00	50.00	preconcentration recommended
Ru	Ru	349.89	12.00	25.00	40.00	83.00	preconcentration recommended
Ir	Ir	224.27	15.00	30.00	50.00	100.00	preconcentration recommended
Os	Os	228.23	20.00	40.00	67.00	133.00	preconcentration recommended

Note: LOD values are based on 3σ criterion with 99% confidence level. LOQ values are calculated as 10σ for reliable quantification.

Comment:

Statement of Conformity to a Specification or Standard: SAZS 560:1997
Decision Rule Based on: Result implies noncompliance with an Upper limit if the measured value plus the uncertainty exceed the limit
Parameter marked * implies non-compliance
Any Additions to,deviations,or exclusions from the method done? No

APPENDIX D: Optimisation of leaching of essential elements

Experiment 1 – 15

ELEMENT	EXP. 1	EXP. 2	EXP. 3	EXP. 4	EXP. 5	EXP. 6	EXP. 7	EXP. 8	EXP. 9	EXP. 10	EXP. 11	EXP. 12	EXP. 13	EXP. 14	EXP. 15
Pb (ppm)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cr (ppm)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Ca (ppm)	122.7	104.5	132.2	127.8	129.2	121	120.5	130	110	103.4	122.6	90	128	124	99
Al (ppm)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cu (ppm)	0.03	0.02	0.04	0.04	0.02	0.03	0.03	0.04	0.01	0.02	0.03	0.01	0.02	0.03	0.01
Fe (ppm)	0.63	0.54	0.69	0.68	0.69	0.62	0.62	0.68	0.62	0.53	0.63	0.5	0.68	0.64	0.57
K (ppm)	70.6	63.8	75.9	74.2	90	69	69.8	75	70	63	70.5	58	88	71	62
Mg (ppm)	240.2	189.5	289.5	260	290	238	239	265	210	189	240.1	180	288	243	203
As (ppm)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Mn (ppm)	25.2	19.2	39.5	28	40	24	24.9	29	22	19	25.2	16	39	26	18
Cd (ppm)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Na (ppm)	350.9	255.5	442.4	370	440	348	349	375	320	250	350.8	240	438	353	308
Zn(ppm)	0.65	0.54	0.71	0.7	0.71	0.64	0.64	0.68	0.55	0.53	0.65	0.48	0.7	0.66	0.52

Experiment 16 – 29

ELEMENT	EXP. 16	EXP. 17	EXP. 18	EXP. 19	EXP. 20	EXP. 21	EXP. 22	EXP. 23	EXP. 24	EXP. 25	EXP. 26	EXP. 27	EXP. 28	EXP. 29
Pb (ppm)	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cr (ppm)	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Ca (ppm)	125	102	129	98	97	122.7	122.7	101	122.7	124	110	105	126	119
Al (ppm)	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cu (ppm)	0.02	0.02	0.02	0.01	0.01	0.03	0.03	0.01	0.03	0.04	0.03	0.02	0.04	0.03
Fe (ppm)	0.68	0.53	0.69	0.56	0.55	0.63	0.63	0.58	0.63	0.68	0.6	0.58	0.67	0.62
K (ppm)	87	62	95	61	60	70.6	70.6	64	70.6	72	66	70	73	68
Mg (ppm)	285	188	295	202	201	240.2	240.2	205	240.2	258	210	200	260	237
As (ppm)	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Mn (ppm)	38	18	41	18	17	25.2	25.2	19	25.2	28	22	22	27	24
Cd (ppm)	-	-	-	-	-	-	-	-	-	-	0	0	0	0
Na (ppm)	435	249	445	305	302	350.9	350.9	310	350.9	365	300	265	368	347
Zn(ppm)	0.69	0.52	0.72	0.51	0.5	0.65	0.65	0.53	0.65	0.69	0.6	0.57	0.68	0.63

APPENDIX E: Optimisation of formulation of fish feed pellets

	EXP. 1	EXP. 2	EXP. 3	EXP. 4	EXP. 5	EXP. 6	EXP. 7	EXP. 8	EXP. 9	EXP. 10	EXP. 11	EXP. 12	EXP. 13	EXP. 14	EXP. 15	EXP. 16	EXP. 17
Pb (mg/kg)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cr (mg/kg)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Ca (mg/kg)	100.58	991.81	100.66	557.93	999.43	547.41	998.16	548.32	544.76	100.09	552.22	553.7	553.67	547.72	993.81	98.78	548.86
Al (mg/kg)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cu (mg/kg)	0.34	6.74	0.32	3.68	6.21	3.49	6.55	3.58	3.77	0.29	3.62	3.66	3.35	3.67	6.24	0.28	3.87
Fe (mg/kg)	8.52	90.13	8.25	8.42	79.73	1.63	71.38	1.72	5.12	7.98	4.03	11.84	5.12	4.03	80.29	7.45	11.84
K (mg/kg)	2961.89	5340	2982.63	4209.85	5071.18	4353.92	5113.34	4333.73	4344.81	2974.42	4248.76	4192.92	4365.76	4309.52	4989.18	2971.18	4238.47
Mg (mg/kg)	323.17	1144.11	325.34	767.64	1111.27	711.85	1144	758.69	743.98	326.72	739.06	741.24	734.17	704.93	1063.67	321.04	749.78
As (mg/kg)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Na (mg/kg)	1008.95	1180.33	1009.12	1095.48	1178.45	1103.72	1172.95	1092.84	1096.13	1008.18	1106.44	1104.56	1101.73	1105.44	1189.84	1008.47	1105.92
Mn (mg/kg)	0.863	8.997	0.919	4.579	8.04	4.511	8.475	4.956	4.988	0.79	4.724	4.654	4.506	4.429	8.352	0.918	4.583
Cd (mg/kg)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Zn (mg/kg)	0.019	0.195	0.018	0.112	0.202	0.12	0.188	0.1	0.1	0.017	0.105	0.104	0.096	0.1	0.201	0.018	0.098

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