EXTRACTION OF TANTALUM FROM TANTALITE ORE SOLUTION USING SOLVENT-IMPREGNATED SORBENT CARBONIZED AT 350°C AND ACTIVATED WITH 0.5 M HYDROFLUORIC ACID

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FACULTY OF LIFE SCIENCES

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PROJECT WORK SUBMITTED TO THE DEPARTMENT OF SCIENCE LABORATORY TECHNOLOGY, IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE AWARD OF BACHELOR DEGREE (B.Sc.) IN SCIENCE LABORATORY TECHNOLOGYDEPARTMENT OF SCIENCE LABORATORY TECHNOLOGY, FACULTY OF LIFE SCIENCES

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DEDICATION

I dedicate this work to Almighty God who has been my ultimate source of strength and for His divine provision to me throughout my academic journey. I also dedicate this work to my ever supportive parents, Mr and Mrs Wilson Okpiabhele, for their relentless support and compassion towards my pursuit for B.Sc. Degree in Science Laboratory Technology in the University of Benin.

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ABSTRACT

Solvent- impregnated sorbent extraction of tantalum is reported. Tantalum was effectively extracted with 350^0C carbonized and activated with 0.5 M hydrofluoric acid and impregnated with methyl isobutyl ketone from hydrofluoric acid leached tantalite ore. The transport study of tantalum ions through a supported solvent-impregnated based sorbent (coconut shell) has been carried out using a design expert (RSM). Factors affecting extraction of tantalum was studied via pH, temperature and contact time is discussed. The final extracts of tantalum complex were directly inserted after colour development in the spectrophotometer for absorbance measurement which enhances the sensitivity. It was observed that at a pH of 1, a temperature of 28^oC and at a contact time of 5 minutes, the percentage (%) tantalum extraction was decreased to 53.37% and it was observed that at a pH of 1, an increased temperature of 68^oC and at a contact time of 30 minutes, the percentage (%) tantalum extraction was increased to 91.55%. the sorbent under study can therefore be used as alternative in order to reduce the high cost of conventional adsorbent used in extraction management.

CHAPTER ONE

1.0 Introduction

The process of treating crude ores and mineral products in order to separate the valuable minerals from the waste rock is known as mineral processing. It involves the physical and chemical methods used to separate valuable minerals from their ores. It encompasses various processes such as crushing, grinding, sizing, gravity separation, magnetic separation, flotation, and leaching, among others, to extract and concentrate valuable minerals from the raw ore. Mineral processing chemistry involves the study of the chemical principles and reactions used or involved in mineral processing operations. It focuses on understanding how different chemical agents, such as reagents and solvents, interact with minerals to facilitate their separation and extraction. This includes understanding the surface chemistry of minerals, the role of pH, the selection of appropriate reagents, and the optimization of chemical processes to maximize mineral recovery and purity.

Mineral processing chemistry involves a series of physical and chemical processes aimed at extracting valuable minerals from ores. First, the size of the ore particles is reduced and this is typically done through crushing and grinding, which increases the surface area of the ore, making it easier to extract valuable minerals. Once the ore is crushed and ground, various techniques are used to separate the valuable minerals from the gangue (unwanted material). This can include techniques like gravity separation, flotation, magnetic separation, and electrostatic separation. Each technique exploits differences in physical and chemical properties between the minerals and the gangue. Leaching is a process used to extract minerals from ore by dissolving them in a liquid solvent. Commonly used solvents include acids, cyanide, and alkalis. The choice of solvent usually depends on the mineral being extracted and environmental considerations. After the minerals are separated from the ore, they are often further processed through smelting and refining. Smelting involves heating the minerals to high temperatures to separate the metal from the impurities. Refining involves purifying the metal to remove any remaining impurities, resulting in a high-purity final product. Throughout the mineral processing process, various reagents are added to facilitate the separation and extraction of minerals. These reagents can include frothers, depressants, pH modifiers, and dispersants, among others. The selection and dosage of these reagents are critical for achieving optimal separation efficiency.

The mineral group tantalite with general formula, (Fe, Mn)Ta_2O_6 is the primary source of the chemical element tantalum, which is a corrosion (heat and acid) resistant metal. Iron-rich tantalite is the mineral tantalite-(Fe) or ferrotantalite and manganese-rich is tantalite-(Mn) or manganotantalite. Tantalum was discovered in the year 1802 by Anders Ekeberg, a Swedish chemist. He was investigating the mineral columbite, which is a source of both tantalum and niobium. Ekeberg was able to isolate a new element from columbite, which he named tantalum after Tantalus, a figure in Greek mythology who was punished by being condemned to stand in water with food just out of reach (Agulyansky, 2004). Tantalite is black to brown in both color and streak. Manganese-rich tantalites can be brown and translucent.

Tantalite occurs in granitic pegmatities that are rich in rare-earth elements, and in placer deposits derived from such rocks. It has been found in Australia, Brazil, Canada, Colombia (Guainia and Vichada), Egypt, northern Europe, Madagascar, Namibia, Nigeria, Rwanda, The Democratic Republic of Congo, the United States (California, Colorado, Maine, and Virginia), and Zimbabwe. Brazil has the world's largest reserve of tantalite (52.1%).

Fig 1.1. Tantalite

Tantalum is a rare metal, and it is not found in its pure form in nature. It is a very ductile and malleable metal. In recent years, there has been growing interest in the use of tantalum for a variety of new applications, such as in the production of fuel cells and batteries. Tantalum is mainly extracted from the ore bearing concentrates through extraction and refining techniques. The rock

ore hosting the minerals are first mined and processed to increase the grade of valuable minerals as columbite-tantalite concentrates.

1.1. Background of the Study

The economic importance of mineral processing chemistry is profound due to its role in several key aspects. Mineral processing chemistry enables the extraction of valuable metals and minerals from ore deposits. These metals and minerals serve as essential raw materials for various industries such as construction, manufacturing, electronics, and energy production. By efficiently extracting and processing these resources, mineral processing chemistry facilitates the utilization of natural resources, driving economic growth and development. Processing raw ore into refined metals and minerals adds significant value to the extracted materials. For example, processing iron ore into steel increases its value and expands its range of applications in construction, automotive manufacturing, and infrastructure development. Similarly, processing rare earth elements into high-purity forms enhances their value for use in advanced technologies such as electronics, renewable energy systems, and medical devices. This value addition through mineral processing chemistry contributes to increased economic output and competitiveness.

Mineral processing chemistry involves a range of activities, including exploration, extraction, processing, and refining, all of which require skilled labor. As a result, the mineral processing industry creates job opportunities across various sectors, from mining operations to chemical engineering, metallurgy, and environmental management. The employment generated by mineral processing chemistry contributes to local and regional economic development by providing livelihoods and supporting communities. It also drives innovation and technological advancement in the mining and materials processing industries. Researchers and engineers continuously develop new extraction, separation, and refining techniques to improve efficiency, reduce environmental impact, and unlock the economic potential of mineral resources.

Granite rocks such as pegmatite often contain an acceptable composition of tantalite. Other minerals such as Microcline, Quartz, Spodumene, Beryl, Lepidolite, Tourmaline, muscovite, Cassiterite are found in association with pegmatite. The decomposition and erosion of these rocks may lead to the concentration of tantalum in alluvial deposits (Baba et al., 2007; Küster, 2009;

Melcher et al., 2015). At present, economically exploitable tantalite ores are found only in few countries of the world such as Australia, Brazil, Canada, Ethiopia, Mozambique, Nigeria, Portugal, Thailand and Democratic Republic of the Congo and about 40% of the deposit are found in Africa. Emerging economics in Africa today are at the forefront of the re-investment of natural resources for the sustainable growth of the overall economy (Hayes and Burge, 2003). Africa has immense natural resource endowments, especially in minerals used for technological development and manufacturing. As concerns about global climate change continue to fuel transition to renewable energy. Because of a lack of systematic geological mapping and exploration, the full extent of the region's mineral base remains unknown (Hayes and Burge, 2003), even though the race for Africa's mineral wealth has been raging for over two centuries. Mineral exploration and production have been at the forefront of redefining geostrategic relationships between Africa and the rest of the world, as the major emerging markets – namely, China, Brazil, and India – are increasingly investing in Africa's resources (Hayes and Burge, 2003). It can be found in some states in Nigeria in traces, small and huge deposits such as; Nasarawa, Kogi, Osun, Ekiti, Kwara, and Cross Rivers. Nigeria has world-class tantalite deposits in the Central African Tin belt (CATB) and West African Craton (WAC).

Different methods have been employed over the years for the extraction of tantalum, but the latest method of separation performed by solvent extraction from acidic solution to alcohol or ketone proved to be more economical (Theron *et al*., 2011). Solvent extraction is also known as liquid-liquid extraction, it's a method of separating compounds based on their relative solubilities in two different immiscible liquids. It is an extraction of a substance from one liquid phase into another liquid phase. Solvent extraction is a basic technique in chemical laboratories, were it is performed using a separating funnel (Theron *et al*., 2011). It can be explained in simple term: if a compound (solute) is dissolved in a liquid (water, acid, base, etc.) and the solution is brought into contact with second immiscible liquid (solvent, may be a hydrocarbon etc.) then a part of the solute is transferred to the second liquid phase by a force called the chemical potential. This process is a physical chemical process of transferring the solute between the bulks of the two immiscible phases (Zhu and Cheng, 2011). During intensive mixing of the liquid in the cause of time the mass transfer of the solute between the two liquid phases diminishes and finally at a very long contact time vanishes: an equilibrium is encountered (Zhu and Cheng, 2011). Solvent impregnated sorbents are the most recent method of extraction and purification of metals from their compound.

To avoid generation of large toxic waste due to the use of liquid-liquid extraction and losses resulting from volatility or partial blending the application of impregnated sorptive materials containing the relevant organic solvent with the use of reverse chromatography which involves stripping of the metals, is the solution to the said problems in liquid-liquid method. The method uses sorbent as the organic solvent carriers and the system has limited losses of organic solvent by immobilization (Turkowska *et al.*, 2022).

Solvent-impregnated sorbents (SIS) can be tailored to selectively extract specific metals from complex mixtures. In the case of rare earth metals or precious metals, which often occur in low concentrations within ores or industrial process streams, selective extraction is crucial for maximizing metal recovery and minimizing the co-extraction of unwanted impurities. SIS can be regenerated relatively easily, allowing for the recovery of the extracted metals and the reuse of the sorbent material. This makes the process more cost-effective and environmentally sustainable compared to traditional extraction methods that may involve complex chemical treatments or disposal of spent sorbents. By selectively extracting target metals, SIS minimize the generation of waste streams and the release of harmful pollutants into the environment. This is especially important for rare earth metals or precious metals extraction, as these metals may be associated with environmental concerns due to their toxicity or ecological impact. All these benefits make SIS an attractive option for metal extraction processes in various industries, including mining, metallurgy, recycling, and environmental remediation.

1.2. Statement of the Problem

The use of highly concentrated hydrofluoric and sulphuric acid mixture have been adopted as a method for the dissolution of the tantalite ore after roasting and then it is subjected to direct solvent extraction which has environmental challenges due to its high volatility and corrosivity. The separation and getting sharp interface is a problem due to solid material accumulation. The solid materials are humates and inorganic colloidal salts present in the ore. Due to the presence of the solid materials, there is interference in the efficiency of the liquid-liquid extraction process (Ungerer *et al*., 2014) and large volume of waste materials are generated (Schulz *et al.*, 2017). In the 2022 list of 50 strategic and critical minerals to developed nations, tantalum mineral has shown to be one with so much use value. Though it's abundance in Africa have still not reflected on the

GDP. Separation and purification of tantalum requires high tech machinery cost and intensive stage processing. These and many others factors drove the attention of industrial and academic researchers to advance a cost-effective and eco-friendly alternative process that can help to overcome the supply risks and replace the machinery operations with process development, recovery, optimization and purification of tantalum from both low-grade and high-grade ores (Deblonde *et al.*, 2016).

The solvent impregnated sorbents extraction system will be an alternative method, suitable for the purpose of extraction, separation and purification of tantalum ion. It will exhibit the combined technology; crushing and grinding unit, the magnetic separation unit and solvent impregnated sorbents separation and purification units since carbonized and activated biomass is chemically inert and can serve as a collector of ions and selectively under specific acid concentration desorb metals. This study is set out to investigate this possibility and to determine the suitability of the solvent impregnated sorbents complexes as a basis for a spectrophotometric method of analysis, thereby reducing the amount of solvents employed in solvent extraction methods and also establishing a newer path in alkanol impregnation of sorbents for extraction of tantalum ion, using response surface method in optimizing the process.

1.3. Justification of the Study

The use of solvent impregnated sorbents media in the extraction and purification of tantalum ion from tantalite or columbite ore is an alternative to alkaline /oxalate or direct solvent extraction from fluoride media. This separation process will serve as a novel route to improve the chemistry as well as the process of extraction of tantalum ion and to devise a more efficient method for industrial purpose. According to Magdalena *et al*. (2022), the use of solvent-impregnated sorbents media in the extraction and purification of tantalum ion from its ore after hydrofluoric/ sulphuric acid leaching offer several advantages over other methods. Hence, emphasis is on developing a method for extracting of tantalum (v) ion having a favorable distribution of the ion in the solvent impregnated sorbents from hydroxyl group and carbonyl in the extractants so that when the acidic solution comes in contact with the solvent impregnated sorbents phases transfer of ion is monitored via desorption and spectrophotometric determinations.

1.4. Aim and Objectives of the Study:

Aim of the research:

The research is aimed at extraction and purification of tantalum from its ore via solvent-impregnated sorbent using response surface matrics design for optimization.

The objectives of the study are to**:**

- characterize the tantalite ore
- characterize the carbonized and activated sorbents after sorbent impregnation
- optimize the conditions using response surface methodology
- determine spectrophometrically the level of tantalum extracted using response surface design matrix.

1.5. Scope of the Study:

The focus of this research is on process development for the extraction of tantalum from its ore which can serve as an alternative for the alkali roasting decomposition and liquid-liquid extraction of tantalum. The process development requires the use of solvent impregnated sorbents which are chemically inert, highly sensitive and can easily desorp metals in a low acidic solution for improved distribution and purification of tantalum ion from solution via surface response method design for variables like concentration, contact time, temperature and pH.

CHAPTER 2

2.0 LITERATURE REVIEW

2.1.0. Metals and their ores

A mineral is a naturally occurring substance having a definite chemical composition, constant physical properties, and a characteristic crystalline form. Ores are a mixture of minerals: they are processed to yield an industrial mineral or treated chemically to yield a single or several metals. Minerals may be classified into two groups: metallic and non-metallic. Metallic minerals are the chief raw materials for the manufacture of metals. Non-metallic minerals which constitute about 75% of all the minerals, are so-called because they are not used to manufacture metals and also because they lack metallic lustre. Of these about 300 are used industrially in the chemical industry, in building materials, in fertilizers, as fuels, etc., and they are also known as the industrial minerals (Habashi, 2017).

Metals are substances that form naturally below the surface of the Earth. Most metals are lustrous or shiny. They are typically found in nature as ores, which are compounds containing the metal along with other elements. The extraction of metals from their ores involves various processes depending on factors like the type of ore and the desired metal. Metal ores are first mined from the earth's crust. This can be done through surface mining or underground mining, depending on the depth and location of the ore deposit.

Once mined, ores undergo processing to extract the metal. This often involves crushing and grinding the ore to a fine powder to increase surface area for chemical reactions. Many ores contain only a small percentage of the desired metal. Concentration processes, such as flotation, magnetic separation, or gravity separation, are used to increase the metal content of the ore by removing unwanted materials. Smelting is a high-temperature process where the concentrated ore is heated in a furnace with a reducing agent such as carbon (in the form of coke) to extract the metal. During smelting, the metal oxides in the ore are reduced to metal, while impurities are removed as slag. The extracted metal often needs further purification to remove impurities and improve its properties. Refining processes vary depending on the metal but may involve electrolysis, chemical precipitation, or other methods.

Examples of some metals and their ores include: Iron Ore: Hematite (Fe_2O_3), Magnetite (Fe_3O_4), Siderite ($FeCO_3$), Silver Ore: Argentite (Ag_2S), Tin Ore: Cassiterite (SnO_2).

2.1.1 Tantalum Production

The global production of tantalum in 2022 was estimated to be 2,000 metric tons. The Democratic Republic of the Congo (DRC) was the leading producer, accounting for 43% of global production. Other major producers included Brazil, Nigeria, China, and Rwanda. Democratic Republic of Congo produced about 860 metric tons followed by Brazil at about 350 metric tons then Nigeria with about 200 metric tons; followed by China and then Rwanda with 150 and 100 metric tons respectively. It is important to note that the global tantalum market is relatively volatile, and production levels can fluctuate significantly from year to year due to limited supply of tantalum, political instability and environmental concern. It is important to note that these figures only represent the mined production of tantalum. There is also a significant amount of tantalum that is produced as a by-product of other mining operations, such as tin and tungsten mining. The global production of tantalum is relatively small, and the market is dominated by a few countries for these reasons the market is susceptible to supply shocks and price volatility.

2.1.2. Occurrence and mineralogy of tantalum metal

Relative to other metallic elements on our planet, tantalum is amongst the least abundant metals (Jeangrand, 2005), the overall estimated abundance of tantalum 0.9 ppm in the upper continental earth's crust and 0.7 ppm in the bulk continental crust (Linen *et al.*, 2013). In nature tantalum co-occur extensively as oxide with niobium in series of complex oxide minerals, often in solid-solution that contains also iron, manganese, tin, titanium, thorium and uranium (Allain *et al*., 2019). Tantalum has a very strong geochemical properties and a high ionic radius-to-charge ratio, which results in formation of strong bond with oxygen (Linen *et al*., 2013). Tantalum is a mineral that is found in a variety of geological settings, but it is most commonly found in granitic pegmatites. Pegmatites are igneous rocks that are rich in rare earth elements and other minerals. Tantalite is also found in placer deposits, which are formed when minerals are eroded from their

original source and transported by water or wind. There are two main types of tantalite deposits, Pegmatite and Placer deposits. The pegmatites deposits are the most common type of tantalite deposit. They are found in association with other rare-earth element minerals, such as niobium, beryl, and tourmaline. The placer deposit is typically found in streambeds, riverbeds, and beaches. They can be very small, but they can also be very rich in tantalum. The world's largest known tantalite deposits are the Mbalam deposit in the Democratic Republic of the Congo. This deposit is estimated to contain over 100 million tons of tantalite. Other major tantalite deposits are found in Brazil, Australia, Rwanda and Nigeria. Tantalum is found mostly in columbite-tantalite series in which columbite ($FeNb_2O_6$), and tantalite ($FeTa_2O_6$) occur in highly variable ratio. In addition to columbite- tantalite series, the important mineral for production of tantalum is pyrochlore-microlite series and wodginite (Mackay and Simandi, 2014).

2.1.4. The Chemistry of Tantalum

Tantalum mainly occur in nature exclusively as oxide in a series of complex oxide minerals, often the solid-solution also contain iron, manganese, tin, titanium, thorium and uranium (Allain *et al*., 2019). These oxide minerals mainly occur in variety of tectonic and metamorphic settings particularly in minerals of the columbite-tantalite series hosted in granitic pegmatites, peraluminous granites, and in their weathered placer deposits associated with carbonatite complexes and alkaline igneous rocks (Mackay and Simandi, 2014). Its chemistry is a complex and fascinating one. Tantalum is a transition metal with the atomic number 73 and the chemical symbol Ta. It is a very hard, ductile, and lustrous metal that is highly corrosion-resistant. Tantalum is also a relatively rare element, with only about 1 part per million in the earth's crust. The chemistry of tantalum is similar to that of niobium, another transition metal with the atomic number 41 and the chemical symbol Nb. Both tantalum and niobium are found in the same minerals, and they have similar chemical properties. However, tantalum is more stable in the +5-oxidation state than niobium, which means that tantalum compounds are more likely to have a +5-oxidation state than niobium compounds (Burnham *et al.*, 2012). Tantalum forms a variety compounds, including oxides, halides, sulfides, carbides, and nitrides. The most common oxide of tantalum is tantalum pentoxide (Ta_2O_5), which is a white powder that is used as a refractory material and a pigment.

Tantalum halides are also known, and they are typically colourless or white solids. Tantalum sulfides are black or brown solids, and tantalum carbides are hard, high-melting-point materials that are used in cutting tools and other applications.

Tantalum exist in a series of complex oxide minerals. Often the solid-solution contains iron, manganese, tin, titanium, thorium and uranium (Allain *et al*., 2019). These oxide minerals mainly occur in variety of tectonic and metamorphic settings particularly in minerals of the columbite-tantalite series hosted in granitic Pegmatites, peraluminous granites, and in their weathered placer deposits associated with carbonatite complexes and alkaline igneous rock (Mackay and Simandi, 2014). Columbite-tantalite, pyrochlore, wodginite, and microlite are economic minerals for production of tantalum (Mackay and Simandi, 2014). Tantalum- rich mineral is called 'tantalite' and contains 42-84% of Ta_20_5 (Purcell *et al.*, 2014). Columbite-tantalite minerals have similar chemical composition with tapiolite series but different crystal structure, tetragonal for tapiolite. The tapiolite group includes tapiolite-Fe and tapiolite-Mn minerals. The chemical specialty of these oxides is their reactivities with halogens (fluoride and chloride) which has significantly influenced to their separation and purification processes (Agulyonsky, 2004; Nete, M., Purcell, *et al.*, 2014a). In the presence of fluoride ion, tantalum form highly soluble and distinct fluoride complexes depending on the acidity level and metals concentration. Fluorination of tantalum containing minerals enabled the development of the Marignac process in 1866, the first industrial process for separation of tantalum (Agulyonsky, 2004; Deblonde *et al.*, 2016; El-Hussaini, 2009).

2.1.4. Properties of tantalum

Tantalum is a transition metal located in the same group (VB) of the periodic table (Richard and Kathryn, 2011). Tantalum is a rare, silvery-gray, ductile transition metal with a high melting point. It is chemically similar to niobium, and the two elements are often found together in nature. Tantalum is a good conductor of electricity, with a conductivity of about 6.5×10^7 S/m and has a high electrical resistivity, with a resistivity of about 170 μΩcm. The dielectric constant of tantalum metal is 27.9 at 1kHz. By this tantalum can store more electrical charge than other materials with a lower dielectric constant. This makes tantalum useful for making capacitors, which are devices that store electrical charge. The dielectrical constant of a material is a measure of how easily it can

be polarized by an electric field. Polarization occurs when the positive and negative charges in a material are aligned in the direction of the electric field. This alignment of charges creates an electric field of its own, which opposes the external electric field. The dielectric constant is a measure of the strength of this opposing electric field. Tantalum has a high dielectric constant because it has a strong ionic bond. Ionic bonds are formed when one atom donates electrons to another atom. In the case of tantalum, the tantalum atom donates electrons to oxygen atoms. This creates a positive charge on the tantalum atom and a negative charge on the oxygen atoms. The alignment of these charges creates a strong electric field, which makes tantalum a good dielectric material. The dielectric constant of tantalum can be affected by temperature and frequency. The dielectric constant decreases with increasing temperature and increasing frequency. This is because the thermal energy of the atoms increases with temperature, which makes it more difficult to align the charges. The increasing frequency also makes it more difficult to align the charges, because the electric field is changing more rapidly. Tantalum has a high melting point of 2996^0C (5376^0F), boiling point of 5425^0C (9797^0F) and high density of 16.69 g/cm^3. Tantalum has excellent high-temperature and corrosion-resistance due to their strong metallic bonds and the impervious oxide layer formed on the metal surface when exposed to air and moisture. The layers act as protective barrier which prevents occurrence of further reaction and chemical attacks (Rodriguez-Contreras *et al*., 2021). Tantalum is an important refractory metal with high resistance to extreme heat and wear, and with is, the metal and its alloys are ideal candidates for high-pressure and high-temperature applications. Tantalum metal has both rectifying and insulating properties. These properties make it useful in a variety of applications, including electronics, medical devices, and chemical processing equipment. Tantalum metal is a semiconductor. When voltage is applied to a tantalum metal surface, the surface will conduct electricity in one direction but not the other.

2.1.5. Mineral Processing of tantalum from its ore.

To be able to separate essential metals from their deposited mineral, the ore mineral particle size must be reduced to fine sizes to allow for partition. This procedure is called “comminution” or size diminishment. There are two noteworthy targets for comminution in mineral beneficiation: The primary goal of comminution is ‘liberation’. Liberation is the procedure to open composite

minerals in the deposit ore into more autonomous particles, and without it one can't gather the needed segments as it were. The second target of comminution in mineral preparing is to alter the measure of mineral particles to adjust to the ideal size for the progressive division forms (Beyecha, 2016). Mineral beneficiation is the initial phase in the initial phase in the extraction of metal from common assets. With the exhaustion of high review metal minerals, it is essential to building the metal review by physical techniques; which are named mineral beneficiation. The destinations of mineral beneficiation are: to increase the metal review of minerals; to lessen the measure of gangue minerals with the goal that lower volume of slag shapes in the pyrometallurgical extraction of metals and slag contains gangue minerals; to diminish the warm vitality required to isolate the fluid metal from gangue minerals; and to reduce the liquid arrangement necessity in the hydrometallurgical extraction of metals (Beyecha, 2016).

Tantalite has specific gravity of 8.0 $g.cm^{-3}$ in contrast to columbite with 5.2 $g.cm^{-3}$. This distinction can be utilized to separate tantalite from columbite using shaking table and gravity separation after washing the mined ore. Tantalite has its very own progression structure of Fe and Mn bases shift significantly without much impact on properties to have a kind name of ferrotantalite (colombite) and manganotantalite (tantalite), individually. Mn-rich tantalites can be dark, coloured, translucent, fragile, feeble magnetic, insoluble in concentrated acids such as, hydrochloric, nitric and sulphuric, dissolvable in hydrofluoric and phosphoric acids (Mitchell, 2015; Koko, *et al.*, Pienaar, 2015; Ungerer, 2012).

Mineral processing of tantalite ore from the pegmatite ore depends on the presence of radioactive oxides, response to the magnetic field, tantalum content and grade of tantalite. The present treatment techniques for tantalite concentrates, among the most part: flotation, buoyancy, planktonic electric detachment and chemical treatment, incorporate gravity and magnetic separation are the most well-known and financial technique to advance a high grade and quality tantalum mineral.

CHAPTER 3

3.0. MATERIALS AND METHODS

3.1.0. Materials/Apparatus

Coconut shell: This was used to prepare the carbonized adsorbent.

- Volumetric flask
- Conical flask
- Funnel
- Filter paper
- Measuring Cylinder
- Oven
- pH meter
- Centrifuge
- Test tubes
- Test tube rack
- Desiccator
- UV spectrophotometer
- Thermometer
- Syringe
- Crucible
- Stirrer
- Weighing balance

3.1.1. Reagents Used

- Distilled Water
- Ammonium oxalate
- Hydrogen chloride (HCl)
- Hydrogen fluoride (HF)

- Potassium hydroxide (KOH)
- Pyrogallol

3.1.2. Preparation of Adsorbent

The coconut shells were washed and sun dried for seven days, followed by moisture content. This was followed by grinding of the coconut shells with an electric grinding machine to reduce the size. The moisture free coconut shells were loaded into different clean silica crucibles and then into the muffle furnace and allowed to carbonize at a temperature of 350^{o}C for a period of 25 minutes. Then, the coconut shell was allowed to cool overnight thereby yielding the base carbon material (Gimba *et al.,* 2004). The carbonized coconut shell was sieved using a 250 um sieve to obtain coconut shell carbon of uniform size. The filtrates were then washed thoroughly to remove the unwanted matter and impurities introduced during the crushing process. The Carbonization helped create a porous structure within the sorbent, which enhanced its surface area and adsorption capacity.

3.1.3. Activation with Hydrofluoric Acid

After carbonization, the sorbent was activated with 0.5M hydrofluoric acid (HF). Activation with HF serves to increase the surface reactivity and enhance the sorbent's affinity for tantalum ions in the aqueous solution. HF treatment introduces functional groups onto the surface of the sorbent which can chemically interact with tantalum ions through coordination or ion exchange mechanisms.

3.1.4. Preparation of solvent-impregnated sorbents

1kg of each sorbent was weighed into a 2L conical flask containing 500ml of Methyl isobutyl ketone (MIBK). The mixture was stirred and allowed to stand for 24 hours. The MIBK immersed sorbents were done using the vacuum method and the impregnated sorbents were left to achieve air-dried condition, sieved with a 25µm sieve size and placed in a sealed container.

3.2. Solution Preparation

3.2.1. Ammonium Oxalate Solution

1.5g of ammonium oxalate was weighed. 10ml of distilled water was added and the mixture was stirred until the ammonium oxalate crystals dissolved completely. Then 76ml of hydrochloric acid(HCl) was added and the mixture was stirred again. The mixture was then made up to 100ml in a standard volumetric flask.

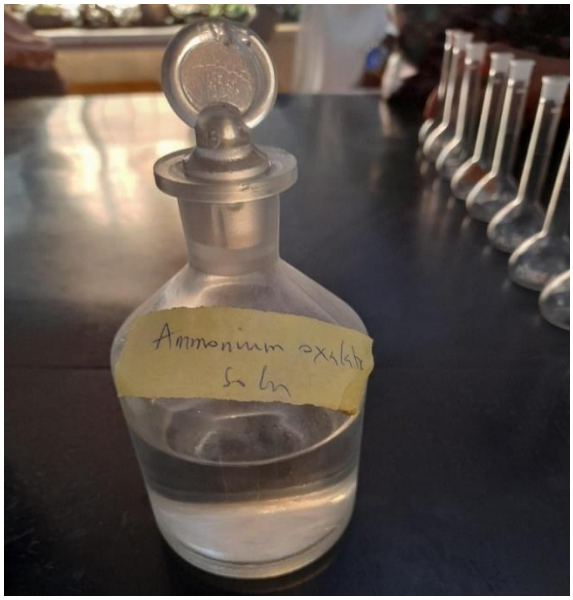

Plate 1: Prepared Ammonium Oxalate Solution

Source: Okpiabhele, 2024.

3.2.2. Modified Pyrogallol Solution

8g of potassium hydroxide(KOH) was weighed using a weighing balance and placed in a beaker. 6.5ml of distilled water was then added. 0.5g of pyrogallol was weighed and added to the mixture and stirred.

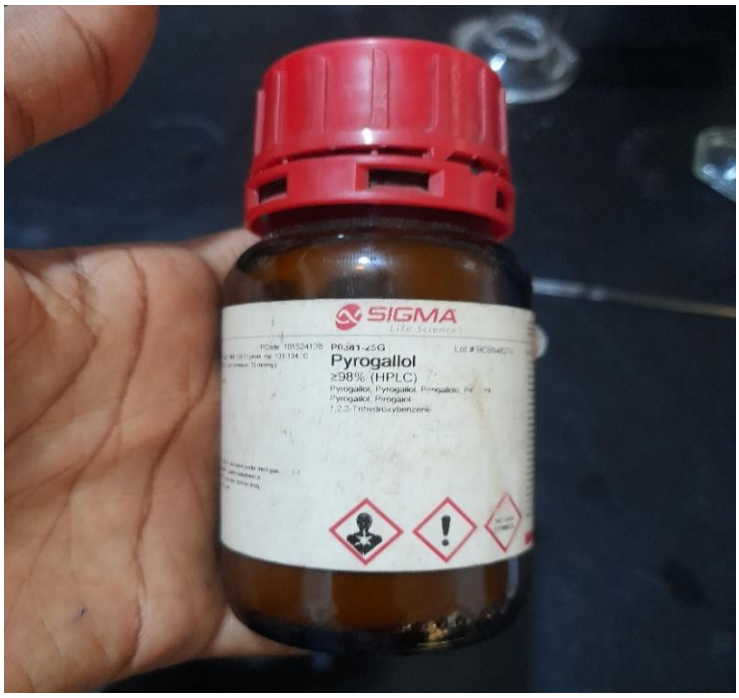

Plate 2: Pyrogallol

Source: Okpiabhele, 2024.

3.2.3. Preparation of HF Dissolved Tantalite Ore

A 25.012g crucible was weighed using a weighing balance. The tantalite ore was then placed into the crucible and the crucible was placed in an oven for about 30 minutes. The crucible was removed and placed in a desiccator and then weighed. 0.346g of tantalite ore was then dissolved in concentrated Hydrogen Fluoride(HF) acid solution.

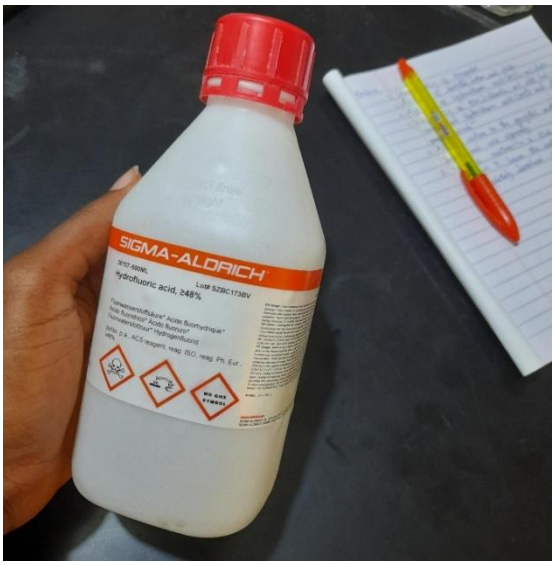

Plate 3: Hydrofluoric acid

Source: Okpiabhele, 2024.

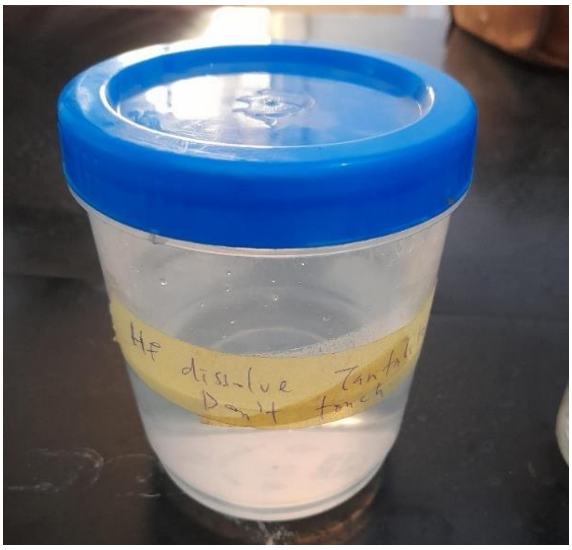

Plate 4: HF Dissolved tantalite ore

Source: Okpiabhele, 2024.

3.2.4. Preparation of Stock Solution

1ml of stock solution (the HF dissolved tantalite ore) was taken by a syringe and put in a 25ml volumetric flask. 0.5ml of the colour developer (modified pyrogallol solution) was added to the solution. Distilled water was then added to make it up to the 25ml mark. The mixture was then placed in a UV spectrophotometer for analysis.

Plate 5: Centrifuge
Source: Okpiabhele, 2024.

Plate 6: pH meter
Source: Okpiabhele, 2024.

3.3. Contact with Aqueous Solution

The solvent-impregnated sorbent was brought into contact with the aqueous solution containing tantalum ions. The solution was obtained from the leaching of tantalum-bearing

minerals or from a process stream containing dissolved tantalum compounds. Upon contact, tantalum ions in the solution interact with the functional groups on the sorbent surface, leading to their adsorption onto the sorbent.

3.4. Extraction of Tantalum

Tantalum ions in the aqueous solution undergo extraction onto the surface of the solvent-impregnated sorbent through various mechanisms, including electrostatic attraction, coordination, and ion exchange. The porous structure and high surface area of the sorbent facilitate the adsorption process, allowing for efficient capture of tantalum ions from the solution.

3.5. Separation and Recovery

Once extraction was completed, the sorbent was separated from the aqueous solution using filtration. The sorbent-bound tantalum ions were then desorbed from the sorbent surface using an appropriate eluent or desorption agent. This step released the captured tantalum ions, allowing for their recovery in concentrated form.

These processes occurred in the following steps:

0.2g of the adsorbent (carbonized 350oC coconut shell was weighed 20 places. The weighed adsorbent was placed in 20 different test tubes and 10ml of the prepared stock solution of the tantalite ore was added. The pH and temperature parameters for each stock solution was also regulated. Each of the resulting mixtures, were then run into a centrifuge for the contact time analysis. After each completed contact time runs, the mixture-containing test tubes were removed from the centrifuge and filtered into a 25ml conical flask. In a 25ml volumetric flask, 1ml of the filtered stock solution was added. Then, 0.5ml of the color developer was added to the mixture in the volumetric flask. Distilled water was then added to make it up to the 25ml mark, after which they were then placed in a UV spectrophotometer for analysis.

CHAPTER 4

4.0. RESULTS AND DISCUSSION

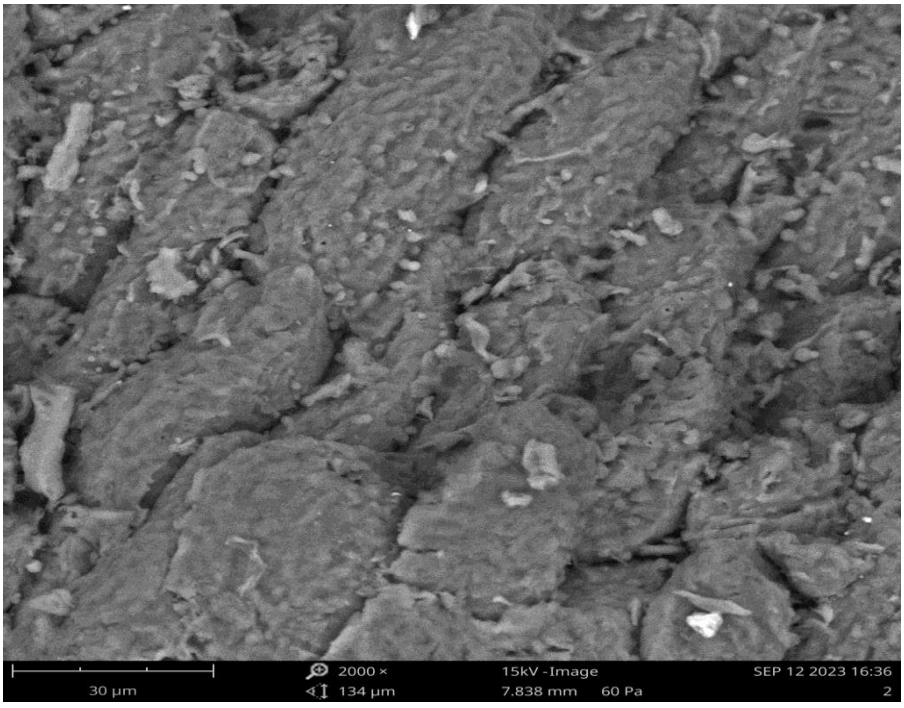

Figure 4.1. 350^{0}C Carbonization /0.5 M HF activation of Cocos nucifera (coconut) shell.

Table 4.1: Absorbance reading for optimal wavelength for tantalum.

Wavelength	Absorbance
320nm	1.462
330nm	1.441
340nm	1.468
350nm	1.403
360nm	1.332
370nm	1.270

Table 4.2: 350^{0}C Carbonization /0.5 M HF activation of Cocos nucifera (coconut) shell after impregnation with methyl isobutyl ketone [MIBK]

Frequency	Appearance	Bonds	Compounds
2929.52	Medium to strong absorption band	C-H stretch	Aldehydes, RCHO
1693.21	Strong to sharp absorption band	C=C stretch	Amides $R\text{-}CONH_2$
1588.98	Medium to strong absorption band	C=C stretch N-H stretch	Arenes, N-H Secondary amine
1156.75	Strong and sharp absorption band	C-O stretch C-N stretch	Acids, esters, anhydrides. Tertiary amine C-N
747.36	Strong to sharp absorption band	C-H bend	Mono-substituted benzene

Table 4.3: Box-bekhen design matrix for experimental design tantalum extraction using solvent-impregnated sorbent carbonized @ 350°c and extracted with 0.5 M hydrofluoric acid solution

	Factor 1	Factor 2	Factor 3	Response 1
Run	A:pH	B:Contact time	C:Temperature	TANTALUM EXTRACTION (350^0C)
		mins	^{0}C	%
10	1	5	28	53.37
12	3	5	68	53.85
5	3	5	28	53.46
11	2	17.5	28	57.88
15	1	5	68	57.42
2	2	10	48	54.92
18	3	30	28	63.89
1	2	17.5	48	64.20
6	3	30	68	69.94
7	1	30	28	64.98
9	2	20	38	65.04
20	1	17.5	48	69.83
3	1	17.5	38	65.34
19	2	10	58	71.40
13	1	17.5	58	76.93
17	2	15	38	73.22
14	2	25	48	77.64
16	2	25	58	82.33
4	2	15	58	88.71
8	1	30	68	91.55

Table 4.4: Fit Summary

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	
Linear	**0.0039**		**0.4736**	**0.2739**	**Suggested**
2FI	0.3858		0.4829	-0.3101	
Quadratic	0.3106		0.5224	-0.5169	
Cubic	0.1974		0.9651		**Aliased**

Table 4.5: Sequential Model Sum of Squares

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Mean vs Total	91923.24	1	91923.24			
Linear vs Mean	**1377.24**	**3**	**459.08**	**6.70**	**0.0039**	**Suggested**
2FI vs Linear	221.38	3	73.79	1.10	0.3858	
Quadratic vs 2FI	253.56	3	84.52	1.36	0.3106	
Cubic vs Quadratic	617.29	9	68.59	15.09	0.1974	Aliased
Residual	4.55	1	4.55			
Total	94397.25	20	4719.86			

Table 4.6: ANOVA for quadratic model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1852.17	9	205.80	3.31	0.0380	significant
A-pH	83.95	1	83.95	1.35	0.2723	
B-Contact time	788.79	1	788.79	12.68	0.0052	
C-Temperature	448.05	1	448.05	7.21	0.0229	
AB	46.18	1	46.18	0.7426	0.4090	
AC	82.87	1	82.87	1.33	0.2752	
BC	94.11	1	94.11	1.51	0.2468	
A^2	41.81	1	41.81	0.6724	0.4313	
B^2	12.80	1	12.80	0.2058	0.6598	
C^2	0.8471	1	0.8471	0.0136	0.9094	
Residual	621.83	10	62.18			

The analysis of ANOVA for the quadratic model was performed as it was required to test the significance and adequacy of the model. From Table 6, it shows that the model and linear (B and C) were significant, with small p-value less than 0.05.

Table 4.7: Fit statistics

Std. Dev.	8.28		**R^2**	0.5567
Mean	67.80		**Adjusted R^2**	0.4736
C.V. %	12.21		**Predicted R^2**	0.2739
			Adequate Precision	10.2827

The coefficient of determination R^2 is close to 1, which indicates a better correlation between the experimental data and predicted (Okolo *et al.*, 2018). Also, the adjusted R^2 and predicted R^2 are in reasonable agreement which shows the model is significant. The Adequate Precision(AP) ratio should be higher than 4 for the predicted model to be used to navigate the space. For this study, AP for the model is 10.2827, which is an adequate signal for the model. The coefficient of variation (CV) and standard deviation (SD) indicates the degree of precision. The low values of CV and SD shows the adequacy with which the experiment was conducted. In this study, the CV value was 12.21 while the SD value was 8.28.

Table 4.8: Coefficients in terms of coded factors

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	67.57	1	1.89	63.56	71.58	
A-pH	-3.23	1	2.55	-8.64	2.17	1.00
B-Contact time	9.13	1	2.68	3.45	14.80	1.00
C-Temperature	6.68	1	2.53	1.32	12.03	1.00

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-colinearity, the higher the VIF the more severe the correlation of factors. From the results, the VIFs are all equal to 1 and hence are orthogonal.

Table 4.9: Final equation in terms of actual factors

TANTALUM EXTRACTION (350^0C)	=
+45.23750	
-3.23417	pH
+0.730152	Contact time
+0.333775	Temperature

The equation in terms of actual factors was used to make predictions about the response for given levels of each factor.

Test for Significance of Regression Model for Tantalum Extraction

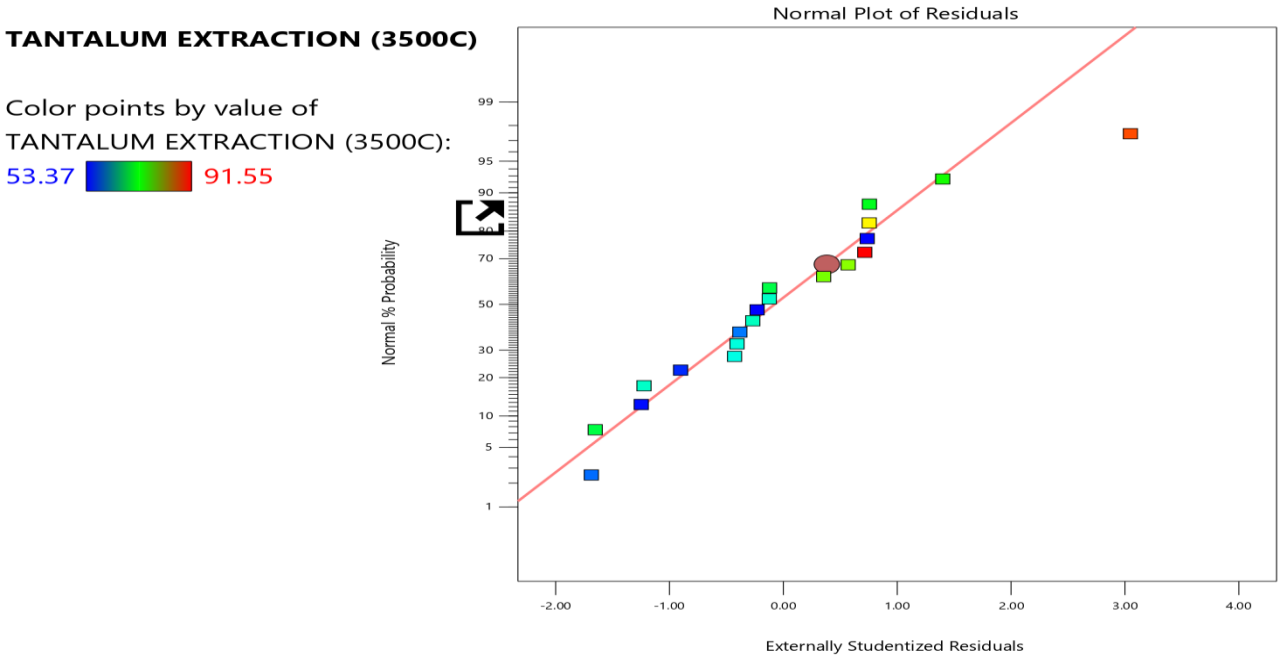

Fig 4.2. Normal plot of residual

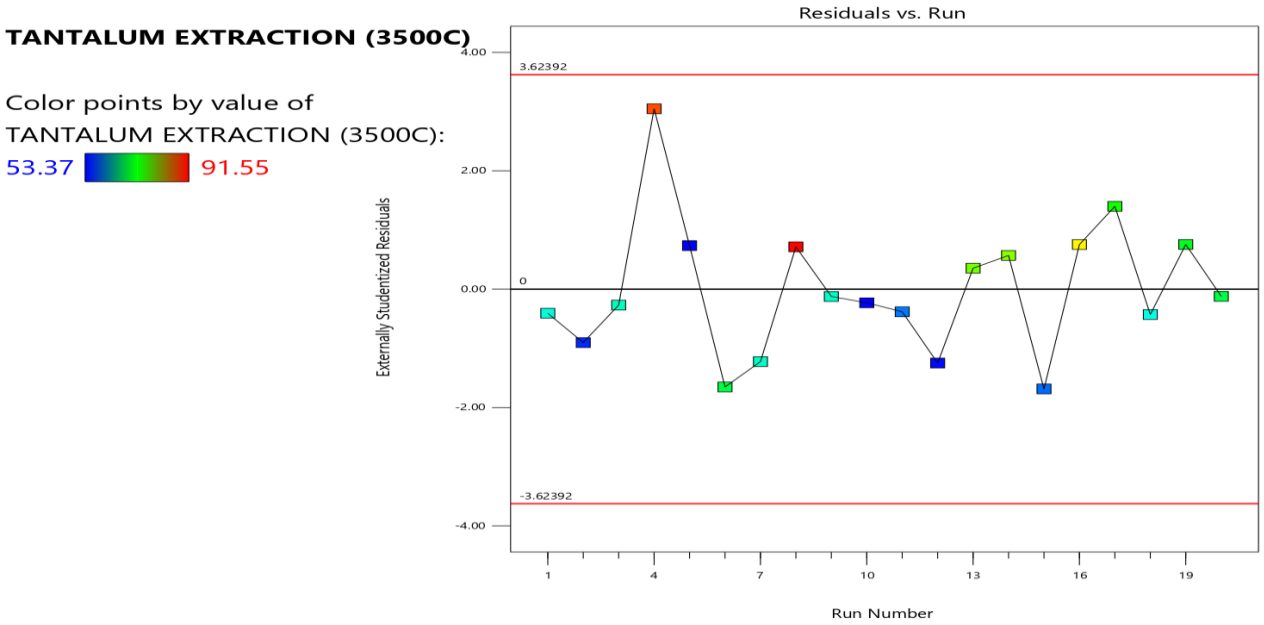

Fig 4.3. Residual vs runs

A good estimated regression model explains the variation of the dependent variable in the sample, if the points of the residual plot approximate a straight line, then the normality assumption is satisfied. Normality indicates whether or not a set of data is normally distributed by plotting the data against the theoretical normal distribution in order to form an approximate straight line (Okolo *et al.,* 2018).

The normalization plots indicated in Fig. 4.2 and Fig 4.3, help in judging if the model is satisfactory. The first plot, normal probability is shown in Figure 4.2. The data were plotted against a theoretical normal distribution in such a way that the points should form an approximate straight line and a departure from this line would indicate a departure from a normal distribution. From the result, the data points are slightly deviating from the normal distribution given, but not very critical (Okolo *et al.*, 2018).

Also, the second plot of residuals versus the fitted value in Figure 4.3 shows that the data points are scattered randomly and does not form a trend. However, all the data points in the plot are within the boundaries marked by the red lines. Therefore, there are no outlier data.

Table 5.0: Box-cox power transformation report for tantalum extraction

Run Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residuals	Externally Studentized Residuals	Cook's Distance	Influence on Fitted Value DFFITS	Standard Order
1	64.20	67.57	-3.37	0.052	-0.418	-0.407	0.002	-0.095	18
2	54.92	62.09	-7.17	0.089	-0.907	-0.902	0.020	-0.281	17
3	65.34	67.46	-2.12	0.140	-0.277	-0.269	0.003	-0.108	10
4	88.71	69.08	19.63	0.081	2.474	3.048	0.136	0.908	12
5	53.46	48.53	4.93	0.366	0.748	0.737	0.081	0.560	2
6	69.94	80.14	-10.20	0.384	-1.570	-1.652	0.385	-1.306	8
7	64.98	73.25	-8.27	0.312	-1.205	-1.224	0.165	-0.825	3
8	91.55	86.60	4.95	0.324	0.726	0.715	0.063	0.495	7
9	65.04	66.06	-1.02	0.077	-0.128	-0.124	0.000	-0.036	16
10	53.37	55.00	-1.63	0.313	-0.238	-0.230	0.006	-0.156	1
11	57.88	60.89	-3.01	0.140	-0.392	-0.382	0.006	-0.154	14
12	53.85	61.88	-8.03	0.375	-1.227	-1.248	0.225	-0.966	6
13	76.93	74.14	2.79	0.144	0.364	0.354	0.006	0.145	9
14	77.64	73.04	4.60	0.091	0.582	0.570	0.009	0.181	11
15	57.42	68.35	-10.93	0.316	-1.597	-1.686	0.295	-1.146	5
16	82.33	76.38	5.95	0.118	0.765	0.754	0.019	0.275	15
17	73.22	62.40	10.82	0.077	1.360	1.400	0.039	0.404	13
18	63.89	66.79	-2.90	0.368	-0.440	-0.428	0.028	-0.327	4
19	71.40	65.43	5.97	0.114	0.766	0.756	0.019	0.271	20
20	69.83	70.80	-0.9722	0.119	-0.125	-0.121	0.001	-0.044	19

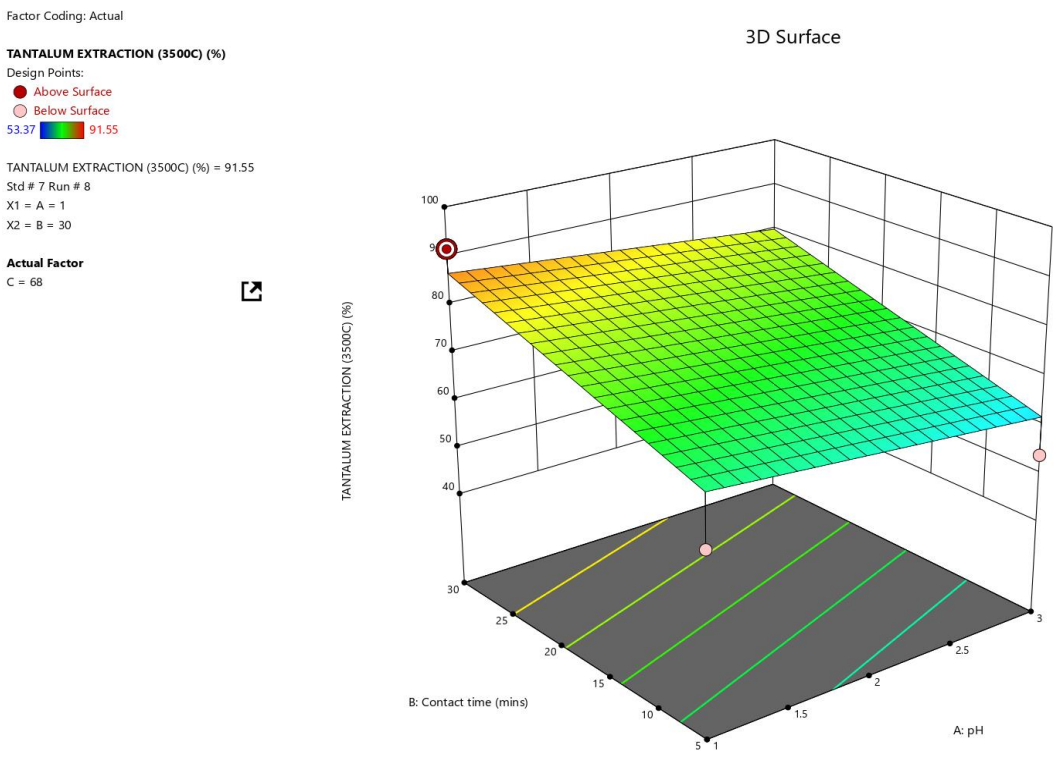

Figure 4.4.1. Response surface for the effect of contact time vs pH for Run 8

The 3D surface plots from the effect of contact time and pH on the extraction of tantalum from tantalite ore solution in Fig 4.4.1. The pH for this experiment was kept at 1 and it is evident that the % tantalum extraction increased as the contact time was increased to 30 minutes. At a contact time of 30 minutes and a pH of 1, the % tantalum extraction was 91.55%.

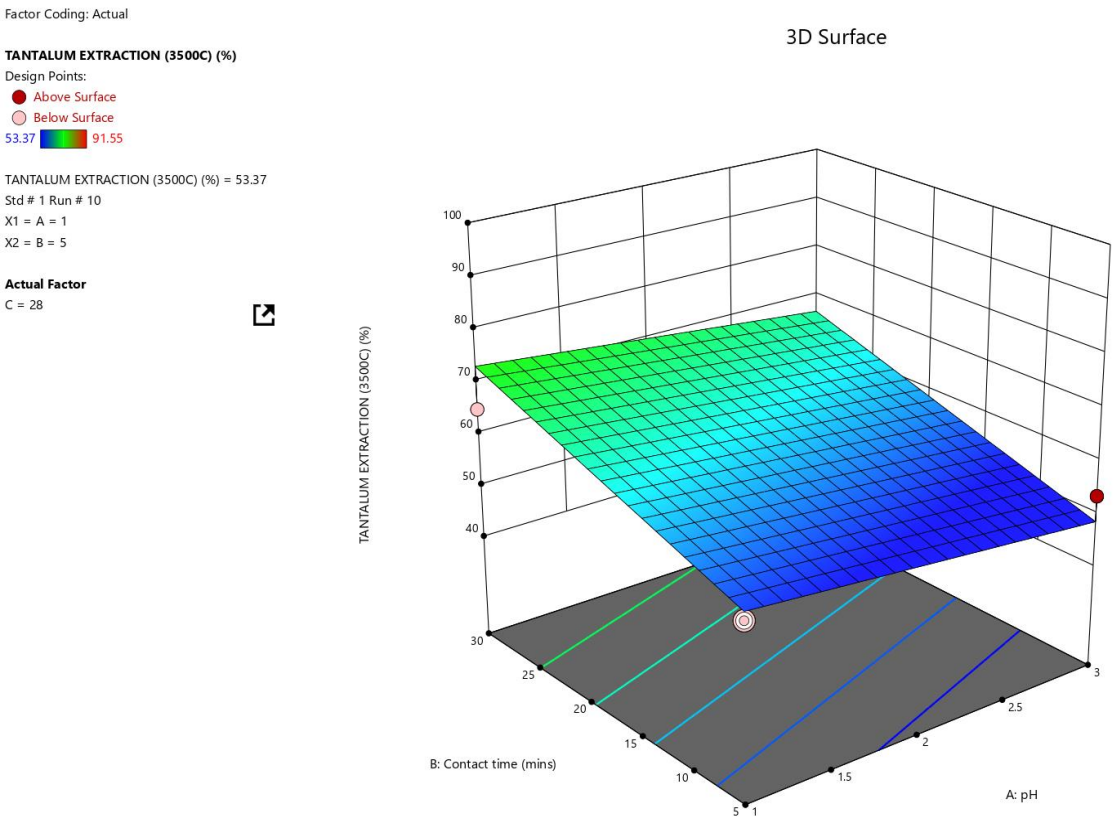

Figure 4.4.2. Response surface for the effect of contact time vs pH for Run 10

The 3D surface plots from the effect of contact time and pH on the extraction of tantalum from tantalite ore solution in Fig 4.4.2. The pH for this experiment was kept at 1 and it is evident that the % tantalum extraction decreased as the contact time was decreased to 5 minutes. At a contact time of 5 minutes and a pH of 1, the % tantalum extraction was 53.37%.

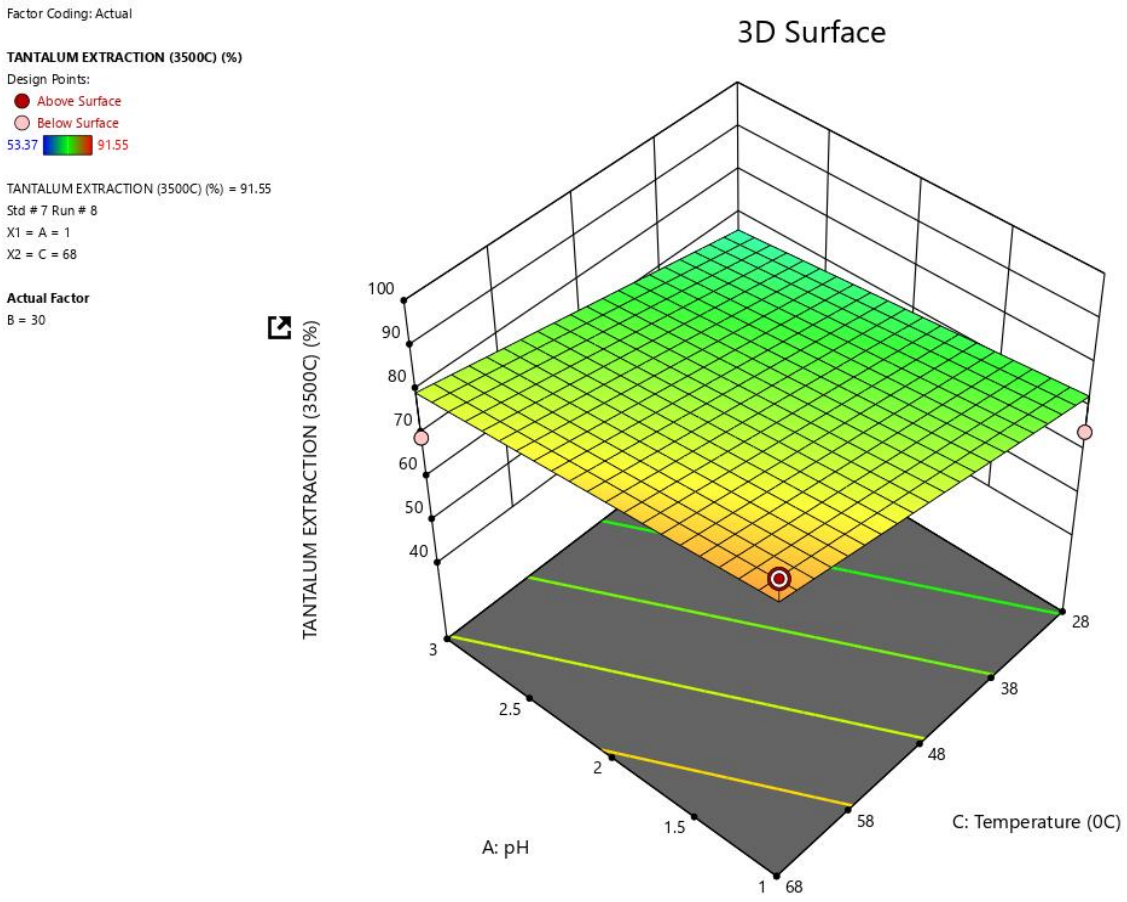

Figure 4.4.3. Response surface for the effect of pH vs temperature for Run 8

The 3D surface plots from the effect of temperature and pH on the extraction of tantalum from tantalite ore solution in Fig 4.4.3. The pH for this experiment was kept at 1 and it is evident that the % tantalum extraction increased as the temperature was increased to 68^{o}C. At a temperature of 68^{o}C and a pH of 1, the % tantalum extraction was 91.55%.

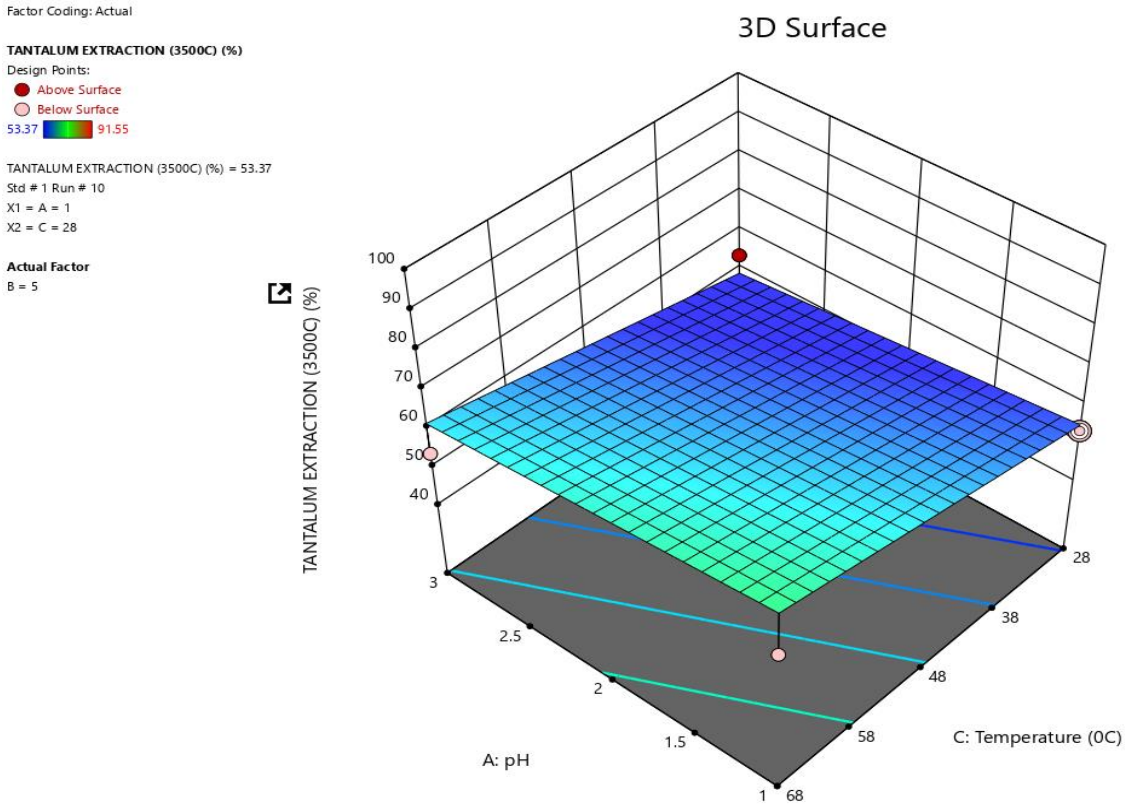

Figure 4.4.4. Response surface for the effect of pH vs temperature for Run 10

The 3D surface plots from the effect of temperature and pH on the extraction of tantalum from tantalite ore solution in Fig 4.4.4. The pH for this experiment was kept at 1 and it is evident that the % tantalum extraction decreased as the temperature was decreased to 28^{o}C. At a temperature of 28^{o}C and a pH of 1, the % tantalum extraction was 53.37%.

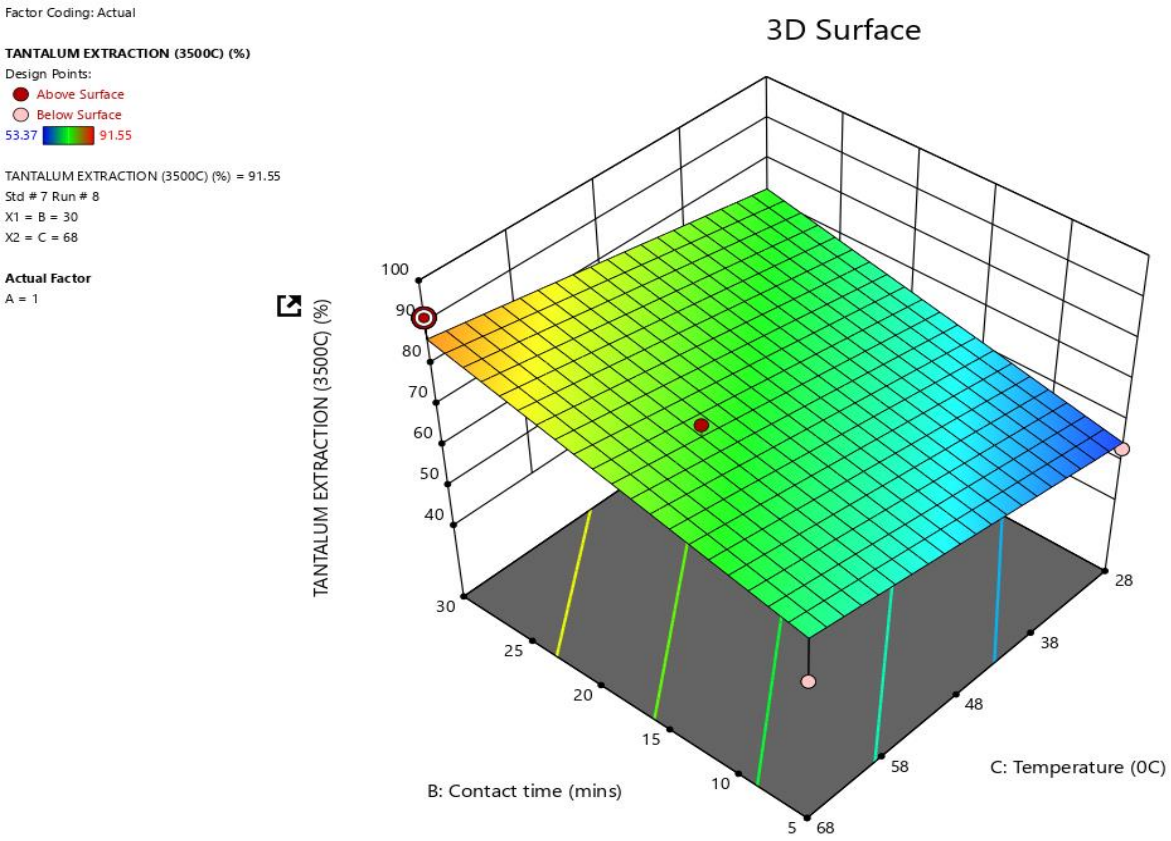

Figure 4.4.5. Response surface for the effect of contact time vs temperature for Run 8

The 3D surface plots from the effect of contact time and pH on the extraction of tantalum from tantalite ore solution in Fig 4.4.5. The temperature for this experiment was kept at 68^{o}C and it is evident that the % tantalum extraction increased as the contact time was increased to 30 minutes. At a contact time of 30 minutes and a temperature of 68^{o}C, the % tantalum extraction was 91.55%.

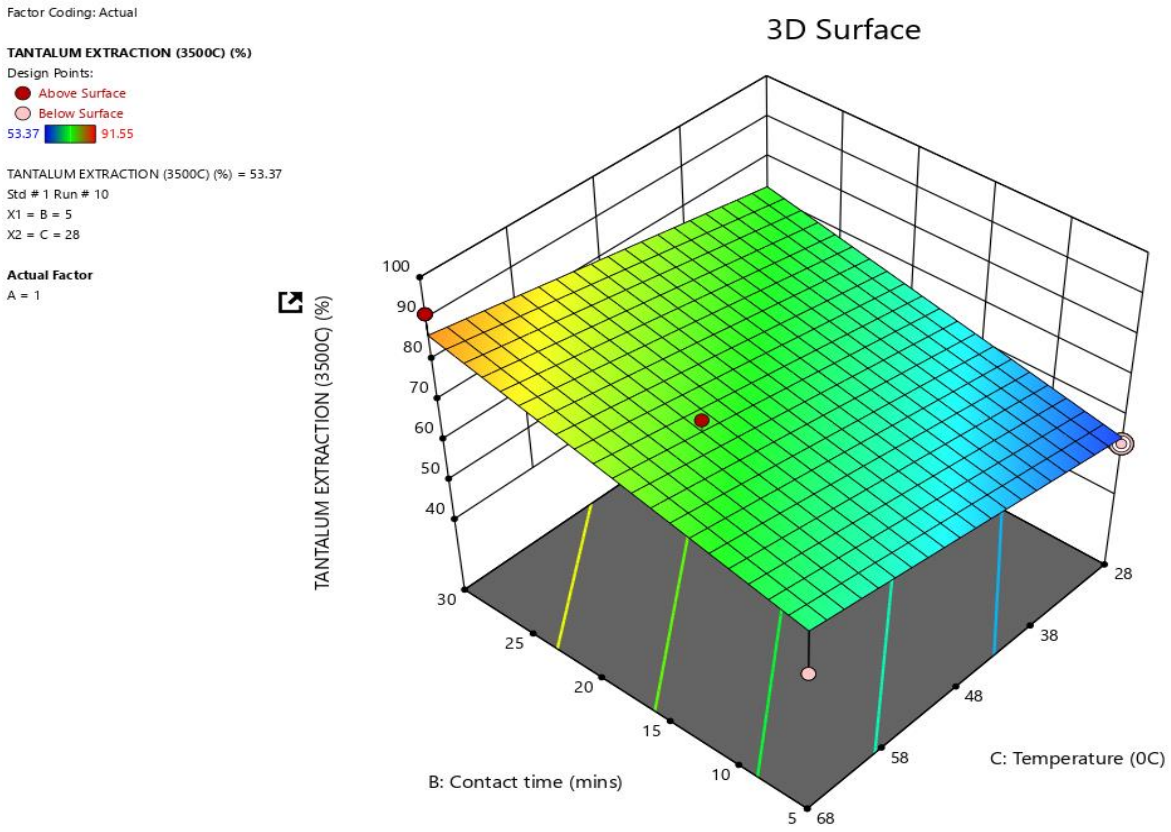

Figure 4.4.6. Response surface for the effect of contact time vs temperature for Run 10

The 3D surface plots from the effect of contact time and temperature on the extraction of tantalum from tantalite ore solution in Fig 4.4.6. The temperature for this experiment was kept at 28^{o}C and it is evident that the % tantalum extraction decreased as the contact time was decreased to 5 minutes. At a contact time of 5 minutes and a temperature of 28^{o}C, the % tantalum extraction was 53.37%.

CHAPTER 5

5.0. CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

This study reports on an optimised extraction of tantalum from tantalite ore solution. In addition, the study also provided an in-depth understanding of how the optimal extraction parameters and impregnated sorbent affected tantalum extraction.

The key findings revealed that the percentage tantalum extracted improved as the pH increased from pH 3 to pH 1, extraction temperature from 28^0C to 68^oC and 30 minutes contact time. The statistical analysis showed that the variables are significant and were noted to regulate the percentage extraction of tantalum Response Surface Matrix [RSM] model.

5.2 RECOMMENDATIONS

The data presented in this work showed that the solvent-impregnated sorbent carbonized at 350 ^{0}C and activated with 0.5 M hydrofluoric acid strongly influence the extraction of tantalum from tantalite ore solution using design expert (RSM). The activation of the carbonized sorbent improve the adsorption capacity of the material hence, this material can be sourced locally and are cheap to process, I will employ the relevant authorities to look into its usage which will help our local content and does improve our research database. It is suggested that research should be carried out on the stripping efficiency of different strippants in the removal or extraction of tantalum from solvent impregnated sorbent surface. It is also recommended that further research be carried out on the extraction of tantalum using other agricultural materials to study extraction efficiency of different biomass.

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