

**APPLICATION OF HALLIMOND TUBE FOR THE
FLOTABILITY STUDY OF PURE SCHEELITE**

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UNIVERSITI SAINS MALAYSIA

2022

**SCHOOL OF MATERIALS AND MINERAL
RESOURCES ENGINEERING**

UNIVERSITI SAINS MALAYSIA

**APPLICATION OF HALLIMOND TUBE FOR THE
FLOTABILITY STUDY OF PURE SCHEELITE**

by

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**A thesis submitted fulfillment of the requirements
for the degree of
Bachelor of Engineering with Honours
(Mineral Resources Engineering)**

August 2022

ACKNOWLEDGEMENT

First of all, I would like to express our gratitude and praises to Allah opportunity and help us endlessly finishing the thesis successfully.

I also own a deep sense of gratitude to my precious lecturer who acts as my research supervisor, Assoc. Prof. Dr. Hashim bin Hussin through the preparation of this thesis. He is always guiding and providing invaluable guidance on whatever I ask and always correcting my understanding on the froth flotation principle and mechanism. His expertise, dynamism, vision, sincerity, and motivation have greatly motivated me to continue my quest to advance my knowledge at this point in my academic career. He also taught me how to conduct research and how to convey the results as simply as possible.

Next, I would love to sent my biggest appreciation to the involved technician such as Encik Zul, Encik Meor, Encik Asrul and Puan Haslina from the first stage (characterization mineral) in the flow work until the end of final stage (flotation test) in the project. I am incredibly grateful to Cik Nordalilah Mohd Sobri from my senior course for helping me do my study throughout the entire approach. Lastly, I will always cherish my family and friends because they were one of my pillars of support during the good times and the bad.

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APLIKASI TIUB HALLIMOND UNTUK MENGENAL KEBOLEHAPUNGAN SCHEELITE TULEN

ABSTRAK

Scheelite adalah salah satu sumber untuk pembentukan tungsten mineral yang biasanya wujud bersama dengan mineral asing yang seperti kalsium yang mempunyai Ca-atom aktif yang sama pada permukaan mineral. Kajian kebolehapungan menggunakan tiub Hallimond dijalankan bagi memisahkan scheelite daripada kalsit. Projek ini dilaksanakan untuk mengkaji kebolehapungan scheelite tulen melalui penggunaan tiub Hallimond dan menentukan keberkesanan jenis pengumpul yang berbeza ke atas scheelite mineral dan parameter-parameter yang lain untuk pemulihan scheelite. Proses mengkaji ciri-ciri mineral melibatkan teknik-teknik seperti mikroskop terpolarisasi, XRF dan XRD. Jenis pengumpul yang digunakan ialah pengumpul campuran (acid oleik + asid naphthenik) dan asid oleik sahaja. Seterusnya, perbandingan peratusan pemulihan scheelite tulen antara dua jenis pengumpul tersebut. Berdasarkan keputusan hasil eksperimen, peratusan pemulihan scheelite menunjukkan nilai yang lebih tinggi daripada 45% untuk kedua-dua pengumpul pada tiga minit waktu penyesuaian. Pada pH nilai 9, peratusan pemulihan scheelite untuk asid oleik pengumpul dan pengumpul campuran mempunyai nilai masing-masing yang tertinggi iaitu 50.72% dan 54.20%. Peratusan pemulihan scheelite ketika menggunakan pengumpul campuran ialah 44.98% dan 37.46% untuk penggunaan asid oleik sahaja. Kesimpulannya, parameter yang optimum adalah tiga minit waktu penyesuaian, dua minit masa kebolehapungan dan Sembilan untuk nilai pH. Peratusan pemulihan scheelite tulen lebih tinggi ketika menggunakan pengumpul campuran berbanding penggunaan asid oleik sahaja.

**APPLICATION OF HALLIMOND TUBE FOR THE FLOTABILITY
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ABSTRACT

Scheelite one of the sources for occurrence of tungsten, commonly coexists best in ore deposits with other gangue minerals such as calcite that have the same active Ca-atoms. The study of flotation was carried out via Hallimond Tube to separate scheelite from calcite. This project was implemented to investigate the floatability of pure scheelite via application of Hallimond Tube and to determine the effectiveness different types of collectors on mineral scheelite and other parameters to recover the scheelite. The mineral characterization process involved techniques such as polarised microscope, XRF and XRD. Type of collected that had been used were a mixture collector (oleic acid and naphthenic acid) and oleic acid collector. Next, the percentage recovery of scheelite were compared between both collectors. Based on the experiment's result, percentage recovery of pure scheelite was discovered higher than 45%. in the conditioning time of 3 minutes for both collectors. At pH value of 9, the percentage recovery for oleic acid collector and mixture collector was the highest which were 50.72% and 52.40%. The percentage of recovery when using mixture collector as 44.98% while the percentage recovery of scheelite when using oleic acid only was 37.46% at two minutes of flotation. As the conclusion, the optimum parameter were three minutes for conditioning time, 2 minutes for flotation time and has pH value of 9. The recovery of pure scheelite was higher when using mixture collector compared to oleic acid collector only.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Tungsten was discovered by Juan and Fausto Elhuyar in 1783. Peter Woulfe investigated a mineral from Sweden in 1779 and discovered a new metal within it, but he did not extract it. Wilhelm Scheele examined it in 1781 and was successful in isolating an acidic white oxide, which he correctly deduced was the oxide of a new metal. The brothers Juan and Fausto Elhuyar, who were interested in mineralogy and were based at the Seminary in Vergara, in Spain, are credited with discovering tungsten. In 1783, they generated the same acidic metal oxide and even converted it to tungsten metal by heating it with carbon (Caswell & Daley, 1999).

The calcium tungstate mineral, well known as scheelite, $CaWO_4$ is an important ore of tungsten. It acquired commercial value in the 20th century when tungsten became used in alloy steels and electric-light filaments. Tungsten occurs under two main mineral forms, scheelite ($CaWO_4$) and wolframite ($(Fe, Mn)WO_4$), with scheelite's importance increasing as wolframite resources are progressively depleting. Scheelite is one of the major ore minerals in various types of tungsten deposits, including skarn-, porphyry-, vein-, and metamorphic-type deposits (Allen and Folinsbee, 1944, Noble et al., 1984, Liu and Ma, 1993). Scheelite commonly occurs as compact or granular masses in contact metasomatic deposits, high-temperature veins, and granite pegmatites.

Scheelite, a major tungsten-bearing mineral, is now mostly used in flotation. Scheelite always coexists with other calcium-containing minerals like calcite and

fluorite in ore deposits. Because they all have the same Ca active sites on their cleavage planes, fatty acid anionic collectors like sodium oleate may firmly collect them all with little selectivity (Rai et al., 2002). To improve scheelite flotation, cationic collectors such as amine and quarternary ammonium, as well as collector mixtures, have been utilised. In mineral froth flotation, the collector adsorption phenomenon is crucial for bubble–particle interaction in terms of particle surface hydrophobicity.

Hydroxamate collectors, such as benzohydroxamic acid, naphthenic hydroxamates, and amide hydroxamates, have also been utilised in recent years for scheelite flotation. Alkyl hydroxamates have been shown to be good collectors for a variety of minerals, including cassiterite, kaolin clay, bastnaesite, mixed copper oxide, and sulphide minerals (Lee et al., 2009, Lee et al., 1998). The –CONHOH group in hydroxamates has a considerable chelating activity with metal ions exposed on mineral surfaces, according to those reports. However, until date, little research has been done on alkyl hydroxamate as a scheelite flotation collector.

Furthermore, it is distinctly coloured crystals are associated with brilliant fluorescence. There are various types of tungsten minerals. Among them, scheelite (CaWO_4) is considered an economically important mineral. To be specific, scheelite is easily ground to fine particles during crushing and grinding processes due to its brittleness. Generally, scheelite is partially composed of particles as fine as these particles, which is different from other types of minerals. This characteristic can be one of the reasons why many researchers have researched froth flotation for the recovery of scheelite from tungsten ore. Flotation is an extensively used separation

technique in the minerals industry that is based on differences in the surface properties of particles.

1.2 Objectives of Research

1. To study wolframite and scheelite minerals as a source of tungsten.
2. To investigate the floatability of pure scheelite via application of Hallimond Tube.
3. To determine the effectiveness different types of collectors on mineral scheelite and other parameters to recover the scheelite.

1.3 Problem Statement

Tungsten is a significant metal for manufacture of special alloys since it has high melting point and high physical properties such as electrical conductor. Scheelite and wolframite are most important sources of tungsten. If the scheelite is in the coarse size, gravity separation can be used to concentrate it. It slimes easily during comminution because it is a brittle mineral (hardness of 4.5–5 on the Mohs scale). The most common method for processing finely grained scheelite is froth flotation, which has only been used since the 1970s.

The problem in this flotation of scheelite occurs when the scheelite coexist with another calcium containing mineral such as calcite minerals have similar surface characteristics which have the same Ca active sites (hydrophilic). The primary goal was and still is to separate scheelite from other calcium-bearing minerals such as calcite, fluorite, and apatite by improving the selective separation performance of scheelite. An efficient flotation scheme was studied through divergent collectors since it works differently depending on properties study.

Based on a previous study, the flotation test was conducted by using one collector only, oleic acid. This flotation was conducted differently when a mixture of collector with the mix ratio of 1:1 was using to improve the recovery of scheelite compared to the use of only one collector at one time. There will be different parameters that were used to compare between the recovery of scheelite when using only one collector and the recovery of scheelite when using the mixture collector. The parameters that involve were conditioning time, flotation time and pH value.

1.4 Scope of work

The goal of this study is to investigate the recovery of scheelite minerals using a Hallimond tube flotation test with the optimal parameters for scheelite separation, such as collector type, conditioning time, pH value, and flotation time. The proportion of scheelite recovery that can fluctuate in the given parameter will be determined via data analysis.

In a Hallimond tube, the experiment was conducted out. The Hallimond tube flotation test requires a single mineral sample. The sample provided must be separated in order to produce pure scheelite minerals. Because scheelite is a non-magnetic material, magnetic separation is the initial step in the separation process. Mineral characterisation analysis is also required to show that the sample includes scheelite mineral and to assess the sample's chemical composition using XRF, XRD, and polarised microscopy images.

The scheelite may be easily separated from the sample after completing this project, and a flotation experiment can be performed to assess the percentage of recovery using the parameters that have been determined.

1.5 The Reseached Gap Analysis

Based on previous study, the flotation test via Hallmond Tube were implemented by using one collector only (type of fatty acid). This was because the fatty acid such as oleic acid can effectively alter the mineral surface of pure scheelite. This alteration of mineral surface had changed the mineral behaviour from hydrophobic into hydrophilic mineral. For this study, we used napthenic acid as compliment to fatty acid in enhancing the involved collector.

The previous flotation test was conducted in alkaline solution while in this study, the flotability test was implement in both condition (acidic and alkaline) in pH value parameter. The sodium carbonate and sulphric acid were used as pH modifier to gain desired pH value. The flotation time which range from 2 to 4 because the less time of flotation for having optimum recovery, the decreasing the cost of flotation process (industrial stage).

1.6 Thesis Outline

There are five main chapters covered in this thesis which are the introduction, literature reviews, methodology, results and discussions, conclusion, and recommendations. The chapter 1 (introduction) was used to provide the general outline of this thesis such as stating the objectives of this project. The literature review in the chapter 2 focuses on the fundamental of flotation especially implementation by using Hallimond tube.

The procedures of the research activity, including sample preparation for magnetic separation, were addressed in Chapter 3 (Methodology) because the presented sample is not strictly scheelite mineral. The findings of a flotation test utilising a Hallimond tube with various parameters were also discussed in this chapter. Images from X-ray Fluorescence (XRF), X-ray Diffraction (XRD), and polarised microscopes were mentioned.

In Chapter 4, the experimental results and discussion were discussed, and in Chapter 5, the conclusion and recommendation were presented.

CHAPTER 2

LITERATURE REVIEW

2.1 Geology and Mineralogy of Scheelite

On In the periodic table, tungsten is found just beneath molybdenum. It has a higher melting point and density than molybdenum. It also has a ductile to brittle transition temperature much above room temperature, making it even more brittle. The filament material in incandescent light bulbs and as an alloying element in tool steels are two of its most common applications. Ferberite (FeWO_4), hubnerite (MnO_4), wolframite (FeMnWO_4), and scheelite (CaWO_4) are four common tungsten bearing minerals (Briant, 2001).

Scheelite (CaWO_4) is a hydrothermal mineral that occurs in numerous deposit types, including skarn, greisen, veins, porphyry, and orogenic gold. Scheelite crystal lattice hosts several trace elements including REE, Mo, Sr, Y, Nb, and Na, which provide valuable information about physicochemical conditions, pathways, and sources of the hydrothermal fluids. Pure minerals were used in adsorption experiments and analysis. They are as follows which are scheelite, calcite, and fluorite from China (Solongo *et al.*, 2020) .

Calcium tungstate, sometimes known as scheelite, is a mineral. Colorless, white, grey, pale yellow, light purple, or light brown, with green, orange, or red occasionally. Scheelite that contains little or no molybdenum is usually white or nearly white. $\text{Ca}(\text{WO}_4)$ is the chemical formula, and it is mostly made up of CaO and WO_3 . CaO and WO_3 have theoretical percentages of 19.4 percent and 80.6 percent, respectively. Under UV irradiation, the fluorescence changes from light blue to yellow (Wen *et al.*, 2021).

Scheelite has extremely bright fluorescence under shortwave (SW) ultraviolet (UV) light and makes spectacular specimens as **Figure 2.1** in below.



Figure 2.1:Scheelite shows strong whitish-blue fluorescence under SW UV light

Scheelite belongs to the tetragonal crystal system, space group $C4H6-I41/a$. $a_0 = b = 5.25$, $c_0 = 11.40$, $Z=4$. Scheelite has a simple crystal structure with tetrahedron $[WO_4]^{2-}$ group and Ca^{2+} arranged in phase along the c -axis (**Figure 2.2**). Atomic spacing: $W-O(4) = 1.78$, $Ca-O(8) = 2.46\text{\AA}$. Scheelite, like apatite, bornite, fluorite, and carbonates including calcite, magnesite, and dolomite, is classified as a polar salt mineral (Bulatovic, 2007). It is hydrophilic because it has relatively strong ionic surface bonding and high free energy values at its surface as a polar mineral. This allows it to react with water molecules.

As a result, it's also semi-soluble(Kupka & Rudolph, 2018). The solubility of scheelite rises dramatically below pH 6. Above pH 6, the molar concentration of WO_4^{2-} ions is substantially higher than that of Ca^{2+} , resulting in a differentiated dissolution that is theoretically incompatible with the lattice ions' structure (Marinakos & Kelsall, 1987).

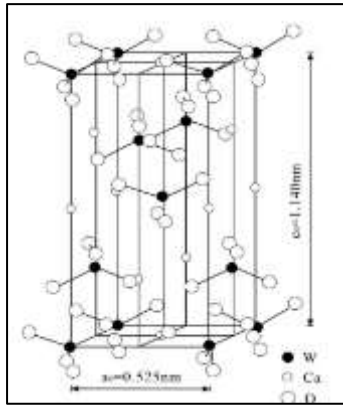


Figure 2.2: The crystal structure of scheelite (Wen *et al.*, no date b)

2.2 Mineral Characterization

2.2.1 Sampling

A geological sample is a sample of soil, rock, mineral, fluid, or gas taken from a greater mass or volume to serve as an example of that larger body or to reflect some specific trait or variation within it (Clarence, 2006).

As this for project, John Riffle Splitter was used since the sample that was gained in a small amount. A mechanical device featuring a sequence of alternating chutes that deposit one half of the sample into one discharge bin and the other half into a second bin is called a riffle splitter (sometimes referred to as a Jones splitter). (Any riffle splitter with an odd number of chutes should be avoided.) The technique is just for freely flowing samples. More so than techniques like coning and quartering, riffle splitters use numerous fractions (chutes), increasing the number of increments in each cycle.

In contrast to sectorial splitters, riffle splitters feature substantially bigger increments. Riffle splitters have been produced in a variety of forms. They come in a variety of sizes, and some allow for the simultaneous splitting of a sample into other

fractions besides halves, such as fourths or eighths. Depending on the size and model, riffle splitters may only contain 10 to 30 chutes, but with repeated passes, each chute picks up particles from different parts of the initial sample. Because of this, there are significantly more increments for the portion of the sample being processed than there are chutes.

The grouping and segregation error associated with the first pass may dominate the uncertainty associated with the entire splitting process, though, as one discards one-half of the sample in the first stage of a multiple-step mass reduction approach. This indicates that while the remainder of the sample splitting operation is carried out, there may not be much gain in reducing the grouping and segregation error after the first run (Epa, 2003).

Riffle splitters can work effectively, but the outcomes depend on the operator's competence and training. There should be no bias while providing the sample to the chutes, and it must be supplied to the riffle splitter so that each chute receives an equal amount (Pitard 1993, cited by, Epa 2003). The **Figure 2.3** shows one of type for John Riffle Splitter.



Figure 2.3: Illustration of John Riffle Splitter

2.2.2 Ore Visual Assessment and Ore Morphology Studies

The polarising microscope is both an optical measurement equipment and a microscope for examining specimens in detail. The visual evaluation and morphology of the ore is completed by looking at the polished component of the ore in the polarised microscope with a separated scale fraction. The polarised microscope's goal is to discriminate between the existence, colour, and shape of minerals. Due to the sample's optically anisotropic property, the polarised microscope only records physical details and photographs specimens that are visible.

2.2.3 X-Ray Fluorescence (XRF)

The non-destructive analytical technique XRF (X-ray fluorescence) is used to determine the elemental makeup of materials. By detecting the fluorescence (or secondary) X-ray released by a sample when it is excited by a main X-ray source, XRF

analyzers may determine the chemistry of a sample. XRF spectroscopy is an ideal technology for qualitative and quantitative investigation of material composition because each element present in a sample creates a set of characteristic fluorescent X-rays ("a fingerprint") that is unique to that element.

Determine the spectrum distribution of the X-rays emitted from the sample and measure the strength of the chosen spectral component are the two main goals of XRF spectrometers. By Bragg diffraction at several crystals, the spectrum is divided into various wavelengths in wavelength-dispersive XRF. Electronic detectors are used to measure intensities. Electronic detectors used in energy-dispersive spectrometers monitor both energy dispersion and intensity; as a result, exceptionally good energy resolutions are needed for these detectors (Kramar, 1999). The **Figure 2.4** shows the principle of XRF.

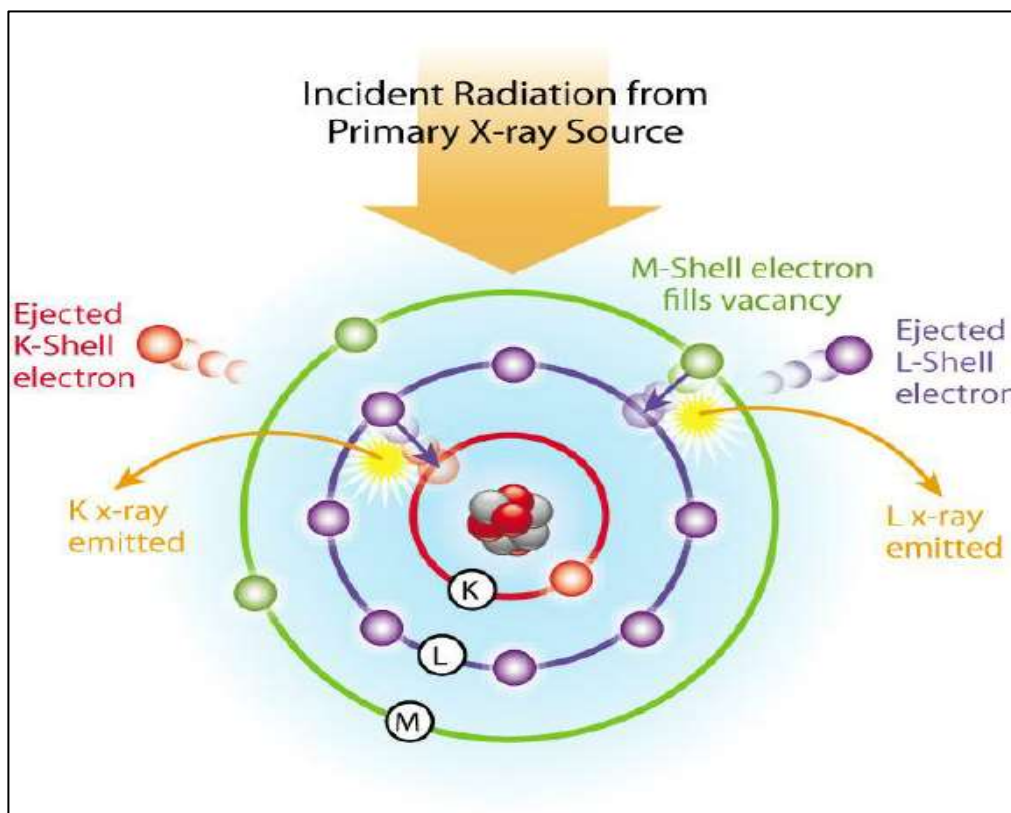


Figure 2.4: X-Ray Fluorescence principle (ThermoFisher Scientific)

2.2.4 X-Ray Diffraction (XRD)

X-ray diffraction (XRD) is a versatile technique that can be used to provide chemical information for both elemental and phase research. XRD is highly useful for stress measurements and texture analysis, in addition to chemical characterisation. Although crystalline samples are required for XRD analysis, the technique can also be used to determine the degree of crystallinity in polymers.

XRD has typically been used for bulk sample analysis, but with the introduction of new optical equipment, it can now also be utilised for thin film research. The phase detection in the sample is demonstrated using a Cu-anode, θ , and K radiation X-ray diffractometer (Nasrazadani & Hassani, 2016). During the remaining period, only about 1-3g of material with diameters of ~ 75 micrometres is normally required. If the preparation is done correctly, the process can be reproduced. The XRD findings were analysed using the X-Pert High Score tool and the PANanalytical ICSD index.

2.3 Physical Processing

Mineral processing is defined as "any of a variety of procedures for crushing and separating ore into valuable substances or waste". The physical technique for separating wanted minerals from gangue minerals such as cassiterite, wolframite, and scheelite in the tin and tungsten ore. Separation can be accomplished using separator equipment that takes advantage of magnetic property variations (Clarence, 2006).

2.3.1 Magnetic Separator

A magnetic separator is a device that removes impurities and other magnetic elements from metal with the use of a magnet. Magnetic separators can be applied before, during, and after a material's manufacturing, and they can be altered to attract

different types of magnetic materials at varied intensities. Magnetic separators can also be cylinders that allow items to pass through them. A magnetic separator's purifying material can be in the form of pieces, a finished product, or even a liquid metal. According to the samples received, scheelite is a non-magnetic target mineral that falls on the tailings. The roller and feed rates are set to the right speeds before feeding the sample into the magnetic separator.

Non-magnetic material is discharged in a natural trajectory forward of the roll. The magnetic force of the roll affects any magnetic particles, which are then discharged on the underside of the roll into a separate chute. By regulating the splitter chutes and the conveyor speed with an inverter control, separation trajectories can be set. There are three collection places or chutes where the material can be captured which are non-magnetic, middling (paramagnetic minerals), and magnetic (ferromagnetic minerals) as in the **Figure 2.5**.

Magnetic rolls come in diameters of 75mm, 150mm, and 200mm and up to 1 metre in width. There are numerous roll configurations that can be used to provide the non-magnetic fraction one more pass for increased product purity or recovery. The 75 microns to 15 mm range of material sizes can be processed by the Rare Earth Roll Magnetic Separator. Although a strict control of the particle size range promotes separation efficiency, as with any physical separation procedures.

Neodymium Iron Boron permanent magnets, the strongest permanent magnets currently on the market, are used in the Rare Earth Roll Magnetic Separator and are integrated into a composite high-intensity magnetic head pulley(Fears, 2018). High separation efficiencies are generated by:

- magnetic roll assembly's design is using premium neodymium magnets.
- the number of poles that must be spaced apart in order to produce magnetic fields with strong gradients.
- The ideal setup maximises the magnetic force that a paramagnetic particle experiences as it passes over the roll.

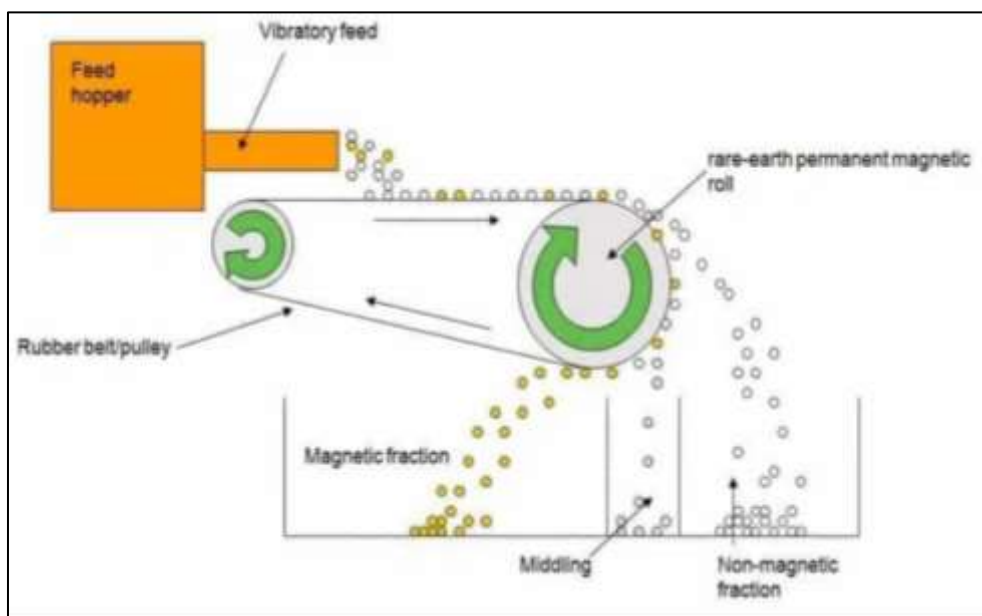


Figure 2.5: Principle of operation of Rare Earth Roll Magnetic Separator (Rowson, 2019)

The **Figure 2.6** in the below shows a magnetic separator that commonly be use in the lab. This is a small scale of magnetic separator.



Figure 2.6: Laboratory-scale Rare Earth Roll Separator.

2.4 Flotation

Flotation is a technique for separating and concentrating the valuable components of ore to generate a grade of mineral concentrate that can be fed to efficient pyrometallurgical or hydrometallurgical operations. Froth flotation was commonly start with crushing and grinding the ore to liberate distinct grains of precious minerals and gangue components, pulping the ore particles with water, and then selectively turning hydrophobic the surface of the mineral. The pulp is next pushed through a stream of air bubbles, which attach to and lift the hydrophobic particles, which gather in a froth layer that flows over the flotation cell's weir.

The mechanics behind the various flotation processes span a wide range of disciplines. The electrochemist oversees conditioning the interface between the mineral and the aqueous solution to make the particles hydrophobic. Thermodynamic and kinematic features of particle-bubble adhesion are involved. The method for meeting the requirements for adhesion in flotation systems can be demonstrated using two

material types which are oxides and sulphides. These materials' flotation demonstrates two distinct mechanisms that are ion adsorption for oxides and oxidation and chemisorption for sulphides (Woods, 1981).

Mineral and chemical engineers have employed flotation to separate and concentrate aqueous suspensions or solutions of a wide range of minerals, coal, precipitates, inorganic waste components, effluents, and even microbes and proteins. Flotation is anticipated to handle more than two billion tonnes of diverse ores and coal each year around the world. With the depletion of high-grade ore sources, this figure, which represents around 85 percent of ores extracted annually, is projected to rise in the future. Because of the growing mechanisation of mining operations, which creates enormous amounts of fine coal particles, coal flotation has increased dramatically.

Flotation technology has been applied to a variety of additional applications, including deinking waste paper for recycling, water treatment, and plastic separation. Flotation deinking now contributes roughly 130 million tonnes of recovered paper to the global paper industry each year. This quantity roughly equates to half of the yearly papermaking capability (Nguyen, 2013).

2.5 Principle of Flotation

Froth flotation is a versatile process for physically separating particles in a mineral/water slurry based on differences in air bubbles' propensity to preferentially attach to specific mineral surfaces. The particles with associated air bubbles are transported to the surface and eliminated, while those that are entirely wet remain in the liquid phase. Froth flotation can be applied to a wide range of mineral separations

because chemical treatments can be used to selectively change mineral surfaces to give them the qualities needed for separation. Separating sulphide minerals from silica gangue (and from other sulphide minerals), separating potassium chloride (sylvite) from sodium chloride (halite), separating coal from ash-forming minerals, removing silicate minerals from iron ores, separating phosphate minerals from silicates, and even non-mineral applications such as de-inking recycled newsprint are just a few examples.

It's especially helpful for treating fine-grained ores that don't respond well to gravity concentration. The **Figure 2.7** shows the fundamental flotation systems.

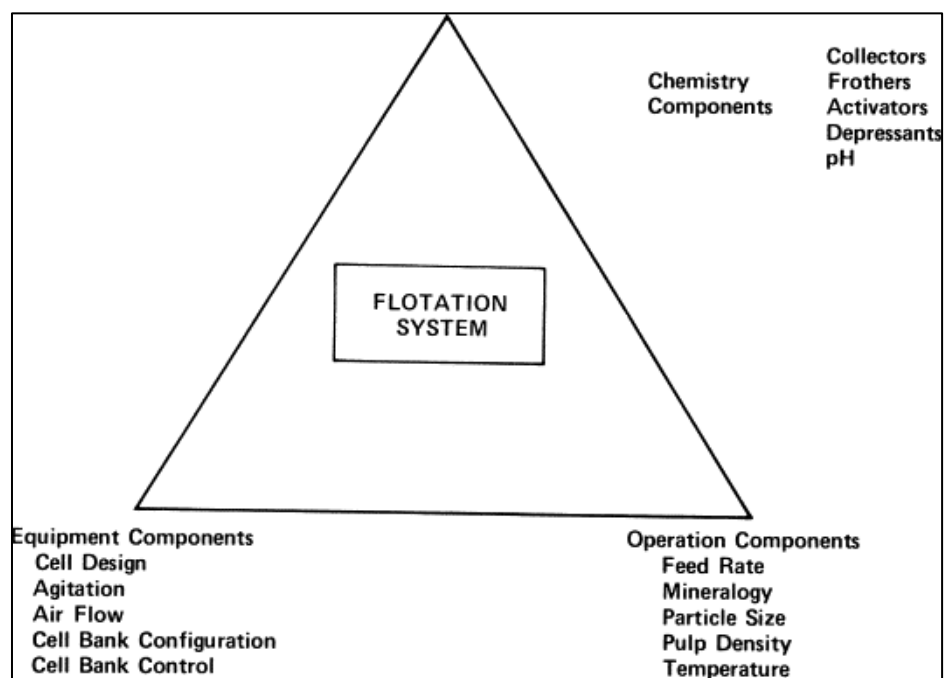


Figure 2.7: Flotation system (Klimpel, 2013)

The selective separation of solid particles, liquid droplets, chemicals, ions, or biological organisms from a bulk liquid based on their surface qualities is referred to as flotation. In this process, rising bubbles collide with matter suspended in the liquid, which is followed by probable particle adherence to the bubble surface and separation

of the resulting bubble–particle aggregate from the liquid. Colloidal suspensions of solid or liquid products, as well as the separation of certain dissolved chemicals and ions, are examples of separations based on adsorption onto a bubble from a solution. As make a separation, the removed substance must have a decrease in free energy when it binds to the bubble. This decrease must be much greater than that of competing ions or colloids for selective separations. When the substances to be removed are hydrophobic (if they are a separate solid or liquid phase) or surface-active (if they are dissolved), and the substances to remain with the carrying liquid are hydrophilic, this occurs (Burstein, 1995).

Many aspects of the flotation must be considered in archiving a better understanding of this system. The most important aspect can be grouped into three main different types which are physical aspects (particle hydrophobicity and floatability, bubble-particle interactions, froth drainage, and flotation kinetics), chemical aspects (surface chemistry of mineral and gangue particles, the chemistry of flotation reagents, and mineral reagents interactions) and engineering aspect (bubble generation, particle dispersion, and cell design and circuits).

The interplay between physical, chemical, and mechanical engineering components also plays a role in successful flotation separations. To highlight the mutual connection of the three parts of flotation science and technology, a triangular image is frequently employed as shown in **Figure 2.8**. There are numerous additional important topics for inquiry, including mineralogy, economics, and the environment.

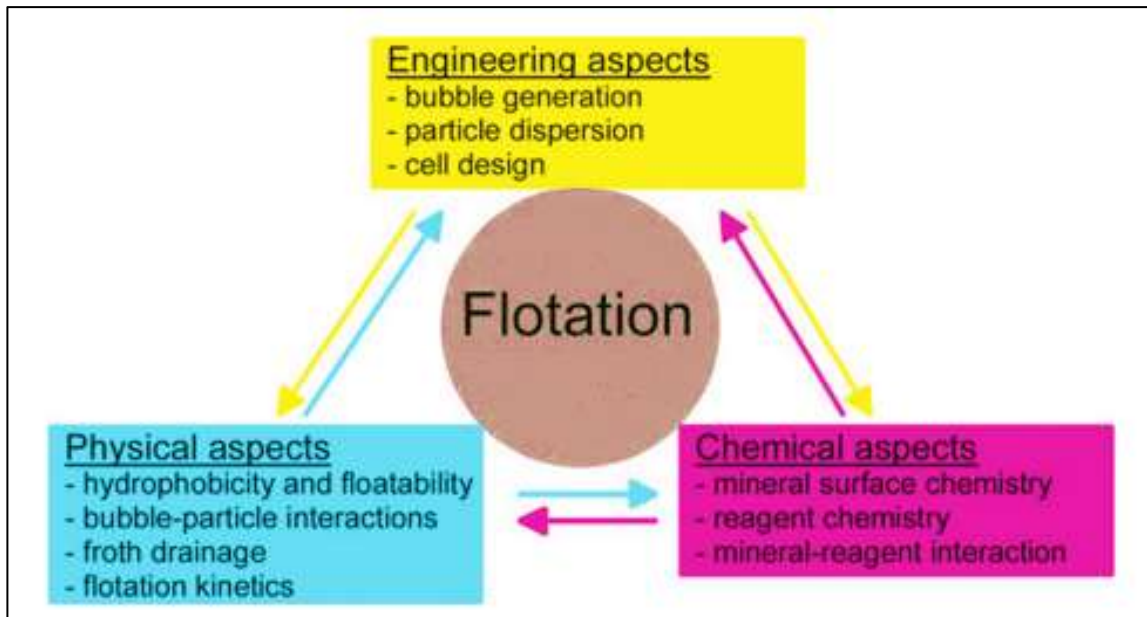


Figure 2.8: Connection of three parts (physical, chemical, engineering) in flotation.

The flotation process' core principles are well understood, but it has proven difficult to develop quantitative prediction models that can be used to simulate the functioning of flotation cells in typical industrial circuits. The complexity of the multiple micro-processes that combine to generate the overall result, which is the separation of distinct mineral species by virtue of the differential surface conditions that can be induced on the various minerals, is the source of the difficulty.

An agitated slurry is aerated in the flotation cell by introducing a cloud of air 'bubbles' that are typically around a millimeter in size. The slurry's agitation is adequate to keep the solid particles in suspension, however, the suspension is not always uniform, with the larger, heavier particles tending to stay in the cell's lower sections. The suspended solid particles can and do collide with the rising bubbles, and those particles with sufficient surface characteristics can adhere to the rising bubbles and be transported upward, eventually reaching the slurry's surface. During its ascent through

the slurry, each bubble will collide with numerous particles, and a single bubble can carry several particles to the top of the slurry (Michaud, 2016).

2.6 Hallimond Tube

For small-scale flotation research, the original Hallimond tube (Hallimond, 1944) and its several variants have been widely employed (Sutherland and Work, 1955; Fuerstenau et al., 1957; Nagy and Van Cleave, 1962; Siwek et al., 1981; Dobias, 1983, cited by Drzymala & Swatek, 2007). Other processes, such as particle aggregation and mechanical carryover of tiny particles, are generally present during flotation in the Hallimond tube. Because of the interaction between these processes, exact flotation demarcation is difficult, if not impossible. If the influence of these activities on genuine flotation is taken into consideration, an inaccuracy in the evaluation of flotation due to flotation-related processes can be eliminated or greatly reduced.

Recently, tests were carried out to see if hydrophilic particles might be entrapped in Hallimond tubes (Drzymala and Lekki, 1989a, b; Drzymala et al., 1992). The equipment needed for the flotation test is shown in **Figure 2.9**. As a result of these studies, an empirical equation was produced that emphasises the importance of particle size and density in the entrainment process. The entrainment of particles in the monobubble Hallimond tube is studied in depth in this research, which also includes an experimental verification of the entrainment equations.

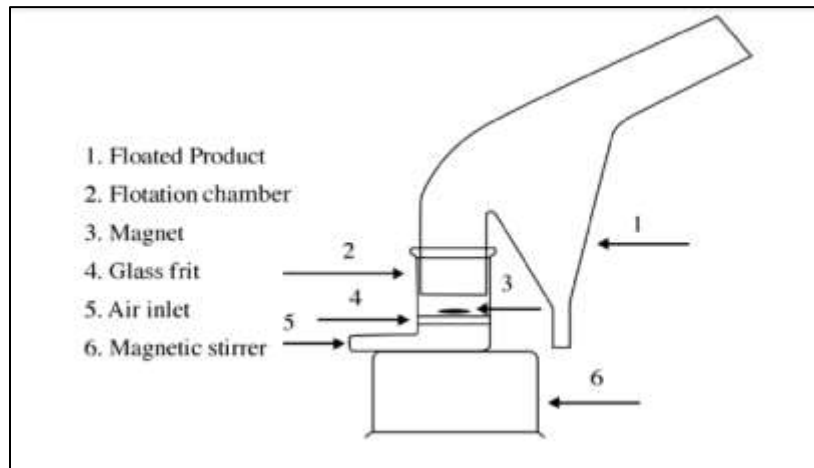


Figure 2.9: Equipment needed for flotation test (Bulut, 2013)

The **Figure 2.10** as showing the mechanisme of flotation (Hallimond Tube) on how the selected mineral drop in the collection stem.

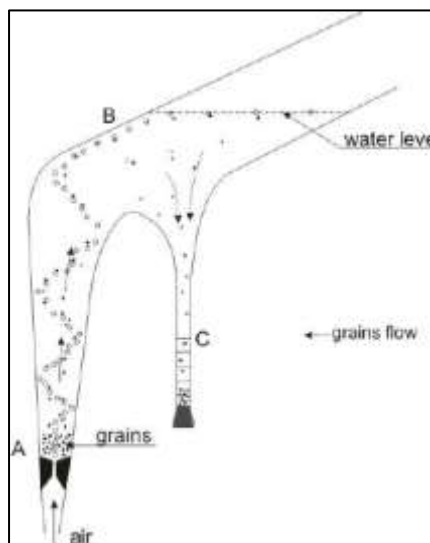


Figure 2.10: Mechanisme of flotation for Hallimond Tube (Drzymala, 2008)

Despite several major flaws, the Hallimond tube is a widely used method of floatability testing. The test sample is deposited as a loose bed of particles on top of a porous septum in the original form. The particles are contacted by air that is pushed through the bed. Because the particles can't move as freely as they do in an industrial flotation cell, it's usually easier for the bubbles to attach themselves to the mineral

particles. Klassen and Makrousov once state that particles that would not float under industrial conditions may float in a Hallimond tube as the result. The addition of a stirrer in a later version of the tube seems to solve the problem, but there are still some flaws as shown the below (Chudacek & Fichera, 1991).

- (i) incomplete flotation chemistry
- (ii) inability to generate real grade vs. recovery data, and
- (iii) unrealistic flow conditions

The capacity of a mineral surface to adhere to a gas bubble in water or another fluid is referred to as mineral flotability. The degree of occupation of the mineral-solution and solution-gas phase interfaces by molecules of the collector (surface-active substances) before their mutual collision, as well as the induction time during the collision, determine the mineral particle's attachment to the bubble (Dobias, 1981 as cited in (Drzymala, 1994a). Bubbles are produced in the capillary of the Hallimond tube.. When the bubbles are formed slowly, the diameter of the bubbles (d_b) is determined by the surface tension of the liquid (γ), the density of the liquid (ρ_l), density of the gas (ρ_g), and size of the capillary (d_c) (Tarjan, 1986).

$$d_b = \left(\frac{6d_c\gamma}{g(\rho_l - \rho_g)} \right)^{\frac{1}{3}} \quad \text{Equation 2.1}$$

In Eq. 2.1, g stands for gravitational acceleration. It is hard to compute the size of the bubbles as they slowly form in the monobubble Hallimond tube because the size of the capillary at the point where the bubbles originate is not well defined and the capillary tip is not sharp. The capillary is <125 μ m in diameter at its narrowest point, but it gradually expands in diameter and blends with the Hallimond tube's walls. The particles in the feed compartment of the tube change the form and size of the capillary

during entrainment experiments. The size of the bubbles does not change significantly as the gas flow increases, but the number of bubbles per time unit increases, making the spacing between the bubbles smaller. The space between bubbles is near zero at a certain critical throughput of the gas, and a coalescent string of bubbles forms. The size of the bubbles does not depend on the capillary size or the surface tension of the bubbles in this scenario (Koch & Norworyta, 1992).

The capillary of the flotation cell creates a bubble that travels across a bed of particles in the Hallimond tube, dragging some particles along with the water next to the bubble. The vertical velocity of the water layer and particles behind the bubbles is equal to the bubble's vertical speed, which is about in all areas of our tube.

A hydrophobic surface coating must be produced on the tiny particles of the component to be floated in order for the desired component to cling to gas bubbles. The small particles of the other components, on the other hand, must be kept hydrophilic. When considering the use of flotation as a separation technique, it is important that some, if not all, of the components are initially hydrophilic in nature when immersed in water. This condition thus allows for the possibility of selective hydrophobicity development. The selective development of hydrophobicity is achieved in practically all flotation processes by the application of particular hydrophobing chemical reagents (Michaud, 2015).

The differential in wettability of different minerals is the basis of froth flotation. Water-wettable particles range from those that are water-repellent to those that are water-repellent (hydrophobic). When a suspension of hydrophobic and hydrophilic