**TAILINGS DEWATERING: THICKENING FOLLOWED BY FILTRATION**

Lappeenranta–Lahti University of Technology LUT

Master's Degree in Chemical Engineering for Water Treatment, Master's thesis

2022

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ABSTRACT

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Every year, the mineral processing industry produces a significant amount of tailings that are difficult to effectively manage. Tailings dewatering is a very effective method of tailings management, which helps recover process water and safely store the tailings. Dewatering the tailings reduces the volume of tailings which in turn reduces the risks associated with dam failure and environmental degradation. Thickeners can process large volume of tailings, produce dense underflow and clear overflow liquor. Different kinds of flocculants are added in thickening operation to enhance dewatering of tailings. Flocculant addition increases the settling rate of solids, promotes consolidation, and clarifies the overflow liquor. Underflow produced from thickener can either be disposed directly to the tailings storage facility or filtered to create cakes with less residual moisture for dry stacking. Pressure filters can produce dry cakes, which can be transported by trucks or conveyor belts for 'dry-stacking'. Dewatering tailings reduces the capital and operating costs, as well as the demand for natural water consumption.

The main aim of this thesis was to study the dewatering of two different tailings. First, the tailings were thickened with addition of flocculant, and the resulting underflow was filtered. Additionally, the effects of various flocculant doses on underflow density, overflow clarity, filtration capacity and cake moisture were studied.

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Lappeenranta, 12 December 2022

Sanchit Bista

# SYMBOLS AND ABBREVIATIONS

Roman characters

| | | |
|---|---|---|
| *D* | Diameter | [m] |
| *d10* | Particle diameter, 10% of the sample's solid particles are smaller than this size | [µm] |
| *d50* | Particle diameter, 50% of the sample's solid particles are smaller than this size | [µm] |
| *d80* | Particle diameter, 80% of the sample's solid particles are smaller than this size | [µm] |
| *dp/dx* | Pressure difference across thickness | [Pa/m] |
| *k* | Permeability | [$m^2$] |
| *q* | Liquid superficial velocity | [m/s] |
| *v* | Stokes Velocity | [m/s] |
| *V* | Volume | [$m^3$] |
| *w* | Dry cake mass | [g] |

Greek characters

| | | |
|---|---|---|
| $\mu$ | Viscosity of liquid | [kg/ms] |
| $\alpha$ | Specific cake resistance | [m/kg] |
| $\varepsilon$ | Cake porosity | |

| | | |
|---|---|---|
| $\rho$ | Density | [g/L], [kg/m$^3$] |
| Constants | | |
| g | Acceleration due to gravity | [m/s$^2$] |
| Subscripts | | |
| f | fluid/liquid | |
| s | solids | |
| w | water | |
| Abbreviations | | |
| HCT | High-compression thickener | |
| HRT | High-rate thickener | |
| M10 | Magnafloc 10 | |
| M155 | Magnafloc 155 | |
| M5250 | Magnafloc 5250 | |
| PF | Pressure Filter | |
| PP | Polypropylene | |
| PSD | Particle Size Distribution | |
| PT | Paste thickener | |
| SG | Specific Gravity | |
| TSF | Tailings Storage Facility | |
| YS | Yield Stress Measurement | |

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# 1 Introduction

Today's mining operations are focused on extracting metals from rocks, like gold and silver, or non-metallic minerals like gemstones and fossils. Mining techniques used today include drilling into the rock to extract ore bodies and then processing that ore through separation techniques such as crushing and grinding before shipping it out for further treatment or sale. However, there are certain drawbacks of mining operations: pollution, biodiversity loss, and contamination of surface water, groundwater, and soil (Balasubramanian, 2017). The significant portion of ore that is still there after the valuable products have been taken from it is referred to as tailings.

Tailings are the wastes produced from mechanical and chemical processes in mineral processing and might make up more than 95 % of the total ore. As ore degrades and large amounts are processed, tailings management becomes more challenging. They are typically uneconomical fraction; however, disposal of tailings results in large capital and operating expenses, as well as delays and losses in water recovery (Du, McLoughlin and Smart, 2014). Tailings management comes with higher costs, difficulties in water management, and challenges in mine closure and rehabilitation (Liu *et al.*, 2020). After the ore has been treated, residual solids and process liquid are disposed of in a tailings storage facility (TSF) (Dunne, Kawatra and Young, 2019). Tailings have major consequences if not handled properly. Both surface and groundwater resources may become contaminated. A tailings dam failure, such as the one that occurred in 2015 at the Somarco mine in Brazil, could occur if there is too much water in the tailings pond (Escobar, 2015). Tailings dams have been classified industrially as productivity limiting and hazardous due to significant safety and environmental impacts like water contamination, soil saturation, acid mine drainage, and dam construction failure (Alam *et al.*, 2011).

Tailings can be dewatered which reduces the water content and makes disposal safer. Dewatering is an important and critical step in minerals processing. Various methods of dewatering include sedimentation/gravity settling, vacuum filtration, centrifugal filtration and pressure filtration (Usher, Hons and Sc, 2002). It has two distinct objectives: to thicken slurry and to clarify liquids. Both processes, however, are desired in specific activities and are frequently referred to as thickening and clarification, respectively (Crust, 2017). Dewatering the tailings reduces the amount of the tailings dams' input in mining sites, reducing the land area and expenses associated with disposal (Yang *et al.*, 2019). Figure 1 shows the different stages in mineral processing with dewatering process of tailings which is the primary objective of this thesis.

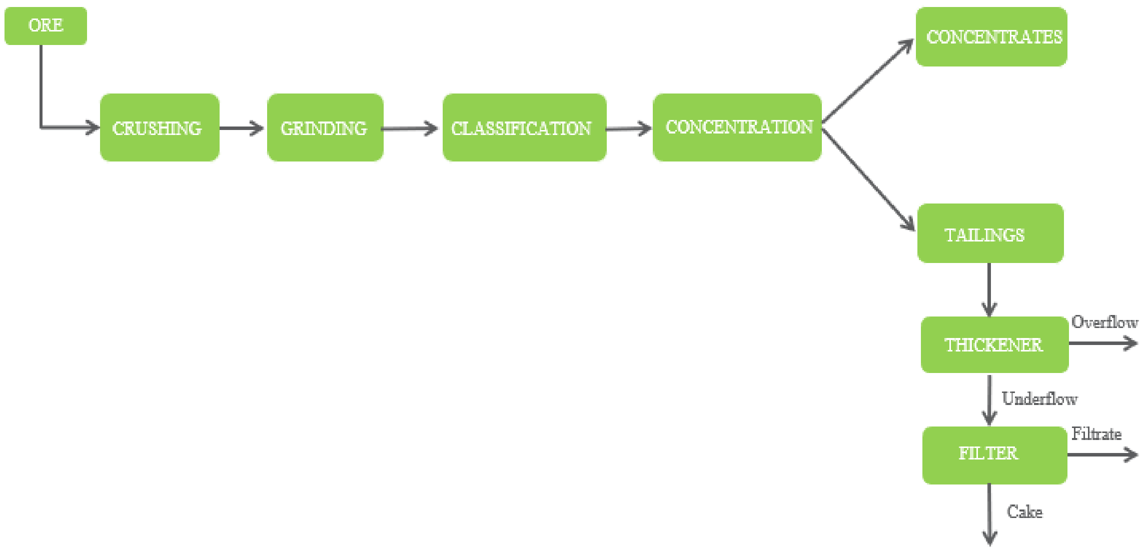



**Figure 1** Tailings dewatering in mineral processing (Metso Outotec)

The tailings produced is further processed to reduce the amount of liquid. Typically, a thickener or a clarifier is used to dewater the tailings. It follows the principle of sedimentation to separate the solids and liquid. Sedimentation works on the principle that solids settle due to difference in densities influenced by gravitational force (Buratto *et al.*, 2014). Flocculant is added to aggregate fine solids to achieve faster settling, increased solids volume fraction and clean overflow liquor. Polymer flocculants are commonly used for dewatering tailings because of their ability to significantly increase the separation of solids and liquids by flocculating small particles (Hogg, 1999). Thickeners are used to make suspensions more concentrated through sedimentation and the release of a clear liquid called overflow. Overflow is recycled back to the process, reducing freshwater consumption (Tan, Bao and Bickert, 2017).

After the thickening process, filters can be used to dewater the tailings underflow. This is an alternative way to tailings dam disposal as it instantly produces dry solids and clean liquor for process reuse which saves significant amount of money (Alam *et al.*, 2011). Filtration works on the idea that as the liquid flows through the filter, a filter media separates the liquid from the solid particles (Buratto *et al.*, 2014). Cake produced by an effective filtration contains less residual moisture thus requiring less storage area. This in turn reduces the construction and operational costs of tailings management facility.

Dewatering the tailings facilitates and improves tailings management. When tailings are efficiently dewatered, the solids concentration increases, and clear filtrate may be returned to the plant, lowering the need for water from natural sources. Dewatered tailings are lower in volume which naturally leads to smaller tailings storage facilities, lowering the costs of both construction and operation.

This thesis focuses on the dewatering of two different tailings. The tailings are first thickened with addition of flocculant. The resulting underflows from thickening tests are then filtered using pressure filter (PF). In the literature part of this thesis, Chapter 1 provides an overview of mining, minerals processing and tailings management. Chapter 2 presents the dewatering techniques that are used to dewater tailings. Additionally, coagulation and flocculation are discussed in this chapter. After the literature review, experimental part of the thesis is described and discussed in Chapter 3, 4 and 5. Chapter 3 describes the objective of this study. In chapter 4, the samples, tools, and the techniques used to dewater the tailings are described. The results are presented and discussed in Chapter 5. The conclusion of this study and future studies are presented in Chapter 6.

# 2 Mining

Mining is the process of extracting minerals and other valuable metals from the earth. The recovery of rock and valuable minerals from surface and subterranean deposits begins at the mine and quarry fronts. Mining techniques can vary depending on the type of ore being extracted (Metso, 2015). The two main techniques of mining i.e., surface mining and underground mining are briefly discussed below:

I. Surface mining

Surface mining involves removing large amounts of surface material overlying the mineral deposit. There are three primary types of surface mining: strip mining, open pit mining, and mountain top removal mining.

Strip mining is the method of extracting minerals that are in shallow deposits by first removing a broad strip of the surrounding rock and soil. Strip mining is best suited for horizontally located deposits.

In open pit mining, the earth's rocks and minerals are removed using an open pit or borrow. It is best suited for minerals that are close to the earth’s surface but accumulated vertically. Open pits can be accessed in different levels and are worked in benches. Open pit mines require a large amount of space and can take several years to complete before production begins. An open pit must be extended on the sides more as it becomes deeper, which requires extracting more waste rock.

Mountaintop removal is the process of removing a mountain's top and mining the resulting area. For the material to be hauled away quickly, it is typically done on steep slopes (Balasubramanian, 2017).

Ore is carried from an open pit by trucks, bucket elevators, or pipes carrying water. Surface mines are generally less expensive than underground mines, but they can cause environmental damage when they are not properly managed. Currently, surface mining techniques are used to extract most metallic minerals (more than 90%), practically all non-metallic minerals (more than 95%), and a significant portion of coal (more than 60%). Nearly 25 billion tons of the more than 30 billion tons of ore extracted annually are produced by surface mining. Surface mining involves removing rock or soil by chiseling or drilling holes into it (Ramani, 2012).

II. Underground mining

Underground mines are located under the ground, making them much quieter than open pit mines and more effective at extracting resources from their environment. Gold, silver, and lead are common minerals that are mined underground. In an underground mining operation, the mineral is accessed and transported to the surface through a network of tunnels and shafts (Balasubramanian, 2017). Underground mines are more expensive than surface mines, but they can produce higher quality products with fewer emissions and less extensive land disturbance than open pit mines. In comparison to surface mining, underground mining requires less waste rock removal. Underground mines require specialized equipment and trained workers for safe operation (Harraz, 2016).

## 2.1. Mineral processing

Minerals are naturally occurring metallic and non-metallic substances. Mineral processing is science of extracting valuable minerals from unusable gangue. Ores are extracted out of the ground either at the surface or below during mining. The valuable minerals are contained inside the large, mined ores. Therefore, the ore must be crushed to reduce the size and release the valuables (Haldar, 2018). Generally, the ores go through following steps in minerals processing as shown in Figure 2.

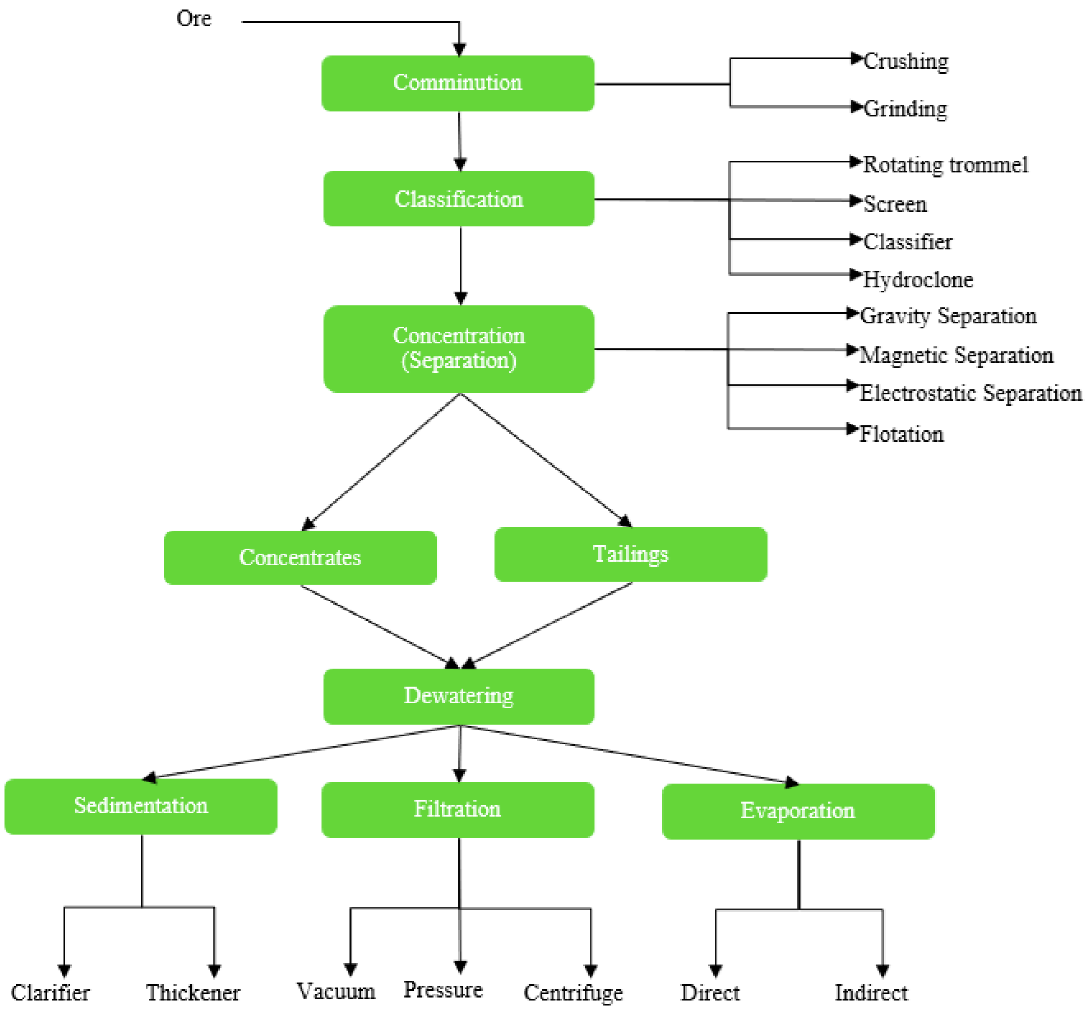



**Figure 2** Different stages in mineral processing (Metso Outotec)

The term "comminution" refers to the process of size reduction that occurs after the ore is removed from the ground. The primary objective of comminution is to release valuable minerals from the ore by crushing and grinding.

Following extraction, the ore first passes through crushers where valuable ores are crushed into smaller pieces, up to 5 mm. The primary purpose of crushing is to liberate valuable minerals from the ore and obtain solids of a specific size range. Crushing ore increases surface area for high reactivity. Crushing ores into smaller size also helps in transportation to different processing units. Crushers like jaw crushers and cone crushers are used for crushing (Metso Outotec, 2022).

Grinding reduces the ore size by turning it into fine powder. Grinding mills further reduce the size of ore to increase mineral liberation. After grinding, ore has narrower and smoother particle size distribution. Particle size affects mineral processing techniques. Analyzing the particle size distribution (PSD) is essential to determine the quality of grind. Ores can be ground by tumbling, stirring and vibrating grinders. Following grinding, a variety of screens and classifiers are used to control the ore's size (Haldar, 2018).

Classification is the process of sorting mineral ore particles according to their sizes and densities. While classifiers and hydrocyclones utilize a liquid medium to sort the particles by weight, trommels and screens sieve the particles using meshes of different sizes. Hydrocyclones use centrifugal force to separate light particles from heavy particles.

Concentration is the process of extracting valuable minerals from the ore. Valuable minerals are released from the ore after it has been crushed and ground. Then, the desired minerals are separated from the undesired ones. The unwanted portion are called wastes or tailings. Concentration is also referred as separation. There are several methods for separating valuable minerals, including gravity separation, magnetic separation, electrostatic separation, and flotation. Separation occurs due to the density of the particles in gravity separation. The magnetic characteristics of the particles are the basis for magnetic separation. Electrostatic separation is based on conductivity of particles. Flotation is based on hydrophobic properties of particles. Hydrophobic particles stick to air bubbles, rise to the top, and are subsequently skimmed off.

Dewatering is the process of removing solids from suspension. Both the valuable minerals ‘concentrate’ and the unwanted fraction ‘tailing’ can be upgraded. While upgrading concentrates, the goal is to increase product value; when dewatering tailings, the goal is to manage waste to reduce environmental effect, recover process water, and turn some fractions into valuables. Some of the upgrading methods are sedimentation/thickening/clarifying, filtration, and evaporation  (Metso Outotec, 2022).

## 2.2. Tailings Management

Tailings are the wastes generated from mechanical and chemical procedures used to extract a desired product from ore. Minerals, metals, chemicals, organics, and process water that are unprofitable and undesired are released, typically as slurry, to a tailings storage facility or a disposal site (Dunne, Kawatra and Young, 2019).

Large tailings dams and tailings storage facilities have historically been used to store a significant volume of tailings material. Direct disposal of tailings into rivers, oceans and lakes have also been practiced which has significant social and environmental challenges (Franks *et al.*, 2011). A dam failure could have disastrous effects. The Boliden dam disaster in 1998 in Spain, which was holding lead-zinc waste, is one dam failure worth mentioning. The dam that was storing five million cubic meters of process water with high amounts of heavy metal-containing particles burst, poisoning two rivers and flooding farmland. At that time, no lives were lost, but there was a significant financial loss (Boger, 2009). In 2012, Talvivaara mine in Sotkamo Finland, hundreds of thousands of cubic meters of contaminated wastewater leaked from a gypsum pond through a 'funnel shaped' hole. Uranium levels in stream water close to Talvivaara mine's leaky gypsum pond were found to be more than 50 times above average. The uranium concentrations measured by Finnish Environment Institute (SYKE) reached 350 mg/l. The levels of nickel and zinc were higher than what is considered to be detrimental to organisms (WISE, 1998).

Every year, mines produce huge amounts of tailings. Five to fifteen tons of tailings are produced for every ton of concentrate produced (Haldar, 2018). With the expansion of mineral exploration and the mining of lower grade deposits, tailings disposal is a significant environmental concern. To reduce negative effects on the environment and society, these tailings must be managed with extreme attention. There are several methods for managing tailings, including:

- Tailings impoundment on land
- Use as back fill for both open pit and underground mines
- Tailings disposal in deep lake beds
- Offshore disposal

- Recovering metals/valuables from further processing  and disposal (Ritcey, 2005)

Traditionally, mines around the world have been using tailings storage facilities (TSF) to store the tailings, which contains a significant amount of water. One of the main reasons for a dam failure is the lack of control of water balance. Heavy metals in tailings contaminate groundwater from seepage. Instead of dumping tailings straight to the TSF, tailings can be dewatered and then disposed in TSF as paste, back fill the underground mines and dry stacked (Edraki *et al.*, 2014).

Thickened tailings can flow a sufficient distance from the discharge point for surface deposition. Depending on the measures of their yield stress, thickened tailings can be further categorized into low, medium, and high-density tailings. Conventional and high-rate thickeners (HRT) can thicken tailings up to 65 % solids or more depending upon the slurries. High compression thickeners (HCT) and paste thickeners (PT) can produce tailings with solids percentage higher than that of conventional and high-rate thickeners, up to 85 % w/w. High-density tailings usually referred as ‘paste’ are mainly used for back fill purposes (IIED, 2002).

Tailings can be dewatered using filters, which produce cake with less residual moisture. These are commonly known as "dry" tailings as they contain very low amount of residual moisture. The cakes are transported by conveyor belt and/or truck after being dewatered to a moisture level where they can no longer be pumped. They are then deposited and compacted in a dry stack that does not necessarily need a retention dam because it is dense, stable, and unsaturated (Davies, 2011). After dewatering tailings, they can be managed based on the following methods:

- **Tailings storage facility (TSF)**

Tailings in slurry form are stored in large quantities in tailings storage facilities. A tailings storage facility's main goal is to safely store tailings for an extended period with less impact to the environment and society. Each TSF's design is unique and takes site conditions and the mining operations into account. TSF is designed and built to hold tailings waste during the life of the mine, as well as after closure. The terrain of the site, the type of tailings, process and environmental limits, and mining rules are few of the variables that affect a TSF (IIED, 2002).

- **Paste back fill**

Paste tailings are dewatered to such extent where they do not have a critical flow velocity when pumped. When deposited, they do not segregate and barely release any bleed water. Paste thickeners can dewater slurries to 70-85 % solids which can be disposed in underground mines as backfill. Paste backfill provides ground and wall support in underground mines. To strengthen the back fill mixture, binders may occasionally be added in tiny amounts to the paste tailings. Dewatering of tailings to produce paste results in lower space requirements for final disposal and reduced tailings dam failure risks. Recovered water can be recycled back in the process (Napier-Munn and Wills, 2005).

- **Dry stacking**

Tailings are dewatered into almost dry stage, higher solids concentration than paste. The concentration of solids in the cakes can be greatly increased by thickening and filtering the tailings. Typically, filters dewater cakes to more than 85 % of solids, which can be transported to a TSF and dry stacked. These dewatered tailings are usually transported by conveyor belt and/or trucks, deposited and compacted. The clean overflow and filtrate can be recycled back to the process. Dry stacking has less environmental impacts because of its efficiency and operation (Napier-Munn and Wills, 2005).

Tailings disposal after dewatering makes it easy to handle as the volume of tailings decrease. The pressure on the demand for freshwater is reduced by the ability to recycle the water. Tailings management done correctly lowers the dangers of dam failure, contaminated water and soil, and loss of biodiversity.

# 3 Solid-Liquid Separation

Solid-liquid separation is a separation technology that is used in many industries like minerals and chemical processing, pharmaceuticals, biotechnology, pulp, and paper industries. It separates solid particles existing in a dispersed or colloidal state in suspension. Solid-liquid separation is applied primarily to recover and dewater the solids from the slurry. If required, the solids can further be washed. Also, the liquid fraction i.e., overflow and filtrate can be recycled with or without further treatment (Svarovsky, 2001).

Dewatering, a technique used in solid-liquid separation, aims to lower the moisture content of filter cakes and sediments. It is widely achieved by gravitational sedimentation (thickeners) and compression of cakes followed by air displacement (filtration). With the addition of dewatering aids, dewatering performances can be improved (Svarovsky, 2001).

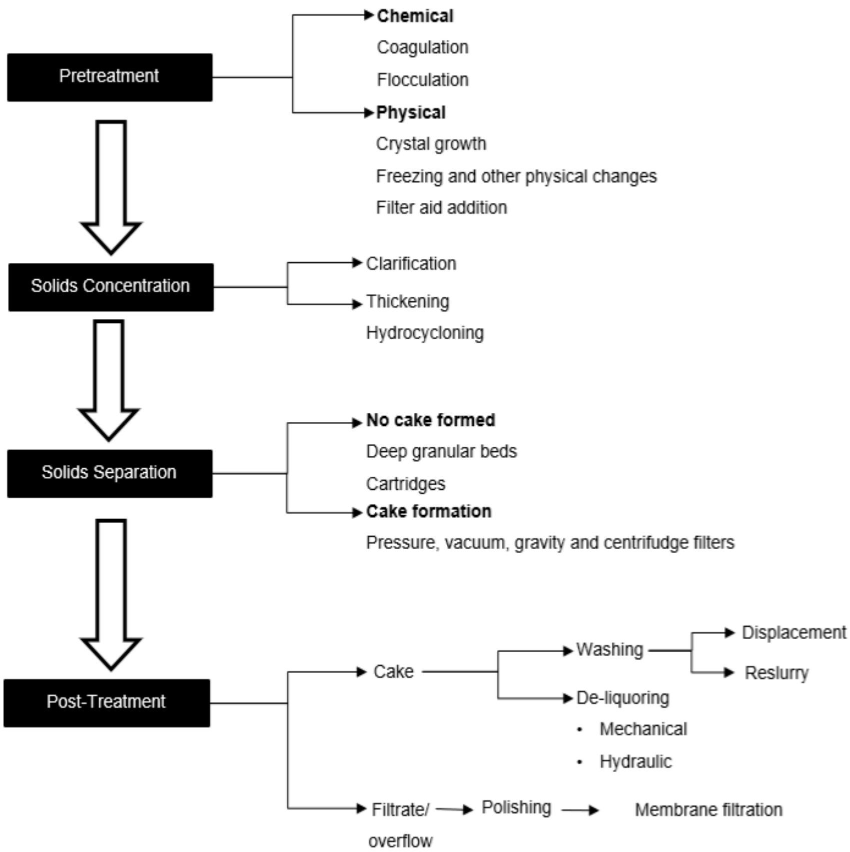



**Figure 3** Different stages in solid-liquid separation (Tiller, Alciatore and Shirato, 1987).

Figure 3 illustrates the different stages in solid-liquid separation

1. Pretreatment: Slurry properties are changed, for instance through chemical treatment, flocculation, or coagulation, to enhance its particle size and filterability. Filter aids are the substances that are used to keep the filter medium from getting clogged and to create an open, porous cake, which lowers the resistance to the filtrate flowing through the filter (Cheremisinoff, 1998).
2. Solids concentration: Thickeners or hydrocyclones can be used to extract liquid from slurries and concentrate the solids. Addition of filter aids can result in increased permeability.
3. Solids separation: Concentrated solids can further be filtered.
4. Post treatment: Any residual liquid can be washed away from the cake. Cakes can be dried to reduce the residual moisture (Svarovsky, 2001).

Tailings dewatering requires effective solid-liquid separation. Thickeners can thicken the tailings to produce dense underflow, usually supported by flocculant addition and clear overflow. The tailings can also be filtered using centrifugal, vacuum, or pressure filters.

## 3.1. Coagulation and Flocculation

Coagulation and flocculation are often mistaken for one single process. They are different processes though the difference can sometimes be very small. Coagulation is the destabilization of solids through the addition of chemicals that neutralize the charges. It brings solid particles together and creates micro flocs. These micro flocs of destabilized solid particles are agglomerated together through flocculation to produce large size flocs, which settle quickly in a sedimentation unit. Figure 4 demonstrates how coagulation destabilizes and gathers solid particles, and how flocculation connects these small flocs to form larger flocs.

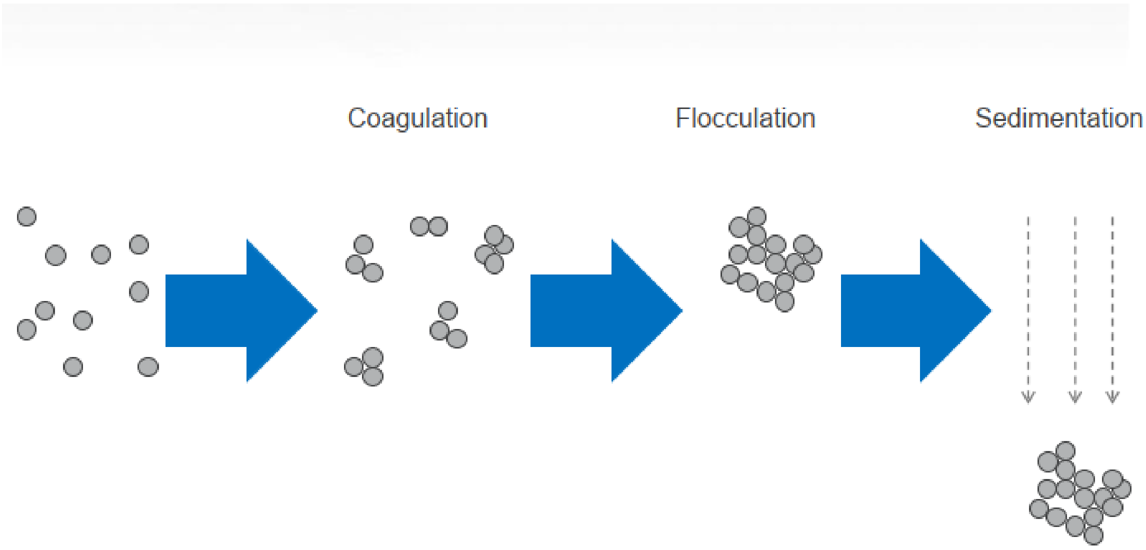



**Figure 4** Coagulation and flocculation process (Adapted from Ahmad, Hashlamon and Hong, 2015)

Coagulation eliminates the repulsion between solid particles, brings the solids closer and aids in their consolidation. Fine colloidal particles come into contact during coagulation, resulting in the formation of micro agglomerates. The consolidated clumps can reach a size of 1 mm, settle, and separate from liquid. Typically these are higher valence cationic salts. (Svarovsky, 2001).

To achieve good coagulation, a high energy rapid mixing is necessary which ensures proper dispersion of coagulant and collision of solid particles. A coagulated aggregate can reform after breaking for example agitation and pumping (Metso, 2015). There are two different types of coagulants: organic and inorganic coagulants.

- Organic coagulants

Organic coagulants, generally referred as polyelectrolytes are a wide variety of water-soluble macromolecular substances, either natural or synthetic, with the potential to destabilize the charges of colloidal particles in a solution. They have very high cationic charge density, so they can neutralize negative charges of the colloidal or suspended solids. Typically, organic coagulants have low molecular weight which allows good dispersion of the cationic charge around each colloidal or suspended solids and low viscosity so they would easily distribute to the effluent (Svarovsky, 2001). Polyamines and PolyDADMACs are some polyelectrolytes that are commonly used (Bae *et al.*, 2007).

- Inorganic coagulants

Aluminum and iron salts are most used inorganic coagulants. Inorganic coagulants when added to a solution, neutralize the suspended particles. Aluminum sulphate, aluminum chloride, poly aluminum chloride, and sodium aluminate are examples of aluminum coagulants. Ferric sulfate, ferrous sulfate, and ferric chloride are iron coagulants. Inorganic coagulants are widely available, cheaper and when applied properly remove most suspended solids (Bratby, 2006).

Flocculants are long-chained polymers that agglomerate destabilized particles into flocs enabling for faster solid liquid separation (Svarovsky, 2001). Flocculation is a time dependent process. Flocculation, in general consists of three main steps:

- Destabilization: Destabilizing the suspended fine particles is the initial step in the flocculation process. It is usually controlled by pH modifications.
- Formation of floc and growth: When particles collide and adhere to each other, bigger aggregates called flocs are created. Flocculated solids settle faster.
- Degradation of floc: Flocs break and degrade under shear and tension from slurry agitation and turbulence (Hogg, 1999).

Figure 5 illustrates the flocculation process. Two solid particles in suspension can be seen to repel one another. Once the flocculant is introduced in the suspension, the flocculant adsorbs to the solids surface, creates bridges and brings them together (Metso Outotec, 2022).

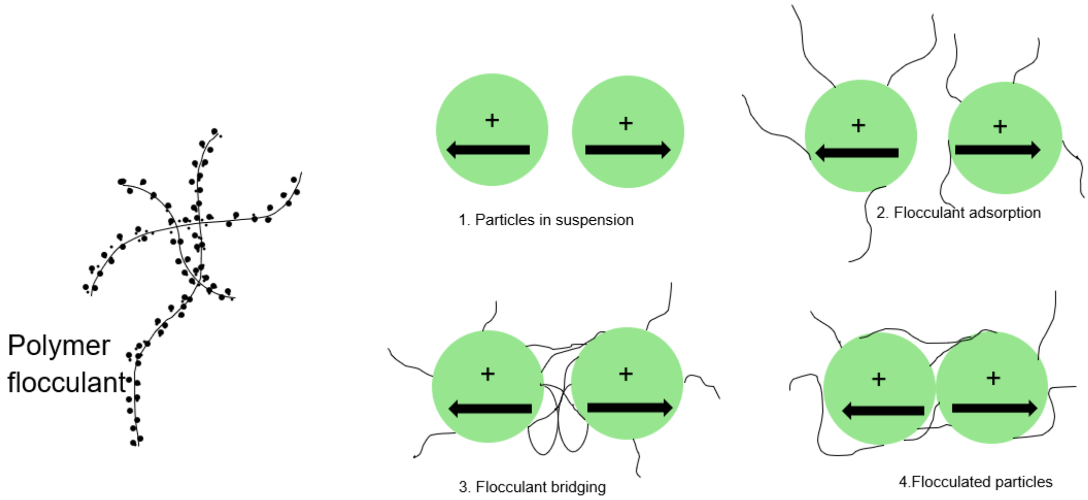



**Figure 5** Flocculation process (Metso Outotec)

Organic and inorganic flocculants can be used for flocculation. Organic flocculants can be natural and/or synthetic polymers. Guar gum and water-soluble starch are organic flocculants (Chukwudi, Eng and Uche, 2008). Inorganic flocculants are the inorganic salts of multivalent metals. The most commonly used inorganic flocculants are aluminum and iron salts because they are readily available, effective, and relatively inexpensive (Chatsungnoen and Chisti, 2019). Flocculants can have different charges and molecular weights.

- Anionic flocculants

Anionic flocculants are widely utilized to flocculate positively charged fine solids in slurry as they produce big agglomerates that settle faster. With anionic flocculants bridging mechanism is prime over charge neutralization. They are used for thickening of concentrates and tailings such as copper, zinc and lead concentrates and tailings, phosphate slimes and bauxite red mud (Cytec, 2010).

- Cationic flocculants

Cationic polymers effectively flocculate low solids slurry, generally by coagulation. These are extensively used in coal tailings dewatering. Cationic polymers usually yield clear overflow but struggle in settling of solids resulting in slow settling rate and decreased efficiency. Cationic flocculants adhere to negatively charged tiny particles in slurry by a charge neutralization mechanism (Edraki *et al.*, 2014).

- Non-ionic

Generally, non-ionic flocculants are applied to slurries with solids that have multivalent cations or high ionicity for both thickening and filtration processes. They work especially well in media that are acidic, like pregnant uranium leach liquors (Cytec, 2010).

Utilization of flocculants has resulted in high-rate and high-capacity designs that are best suited to their application. They have lowered the size requirements for sedimentation equipment (Dunne, Kawatra and Young, 2019). Flocculants are added to accelerate the settling rate of the solid particles in the tailings slurry. Addition of flocculant increases adhesion and consequently optimize the solid liquid separation in aqueous solution (B.C. Chukwudi, 2008).

There are thresholds beyond which flocculants lose their effectiveness. Excessive addition of flocculant can have adverse effects on the process. Some of the effects are:

- Excessive particle cohesion: solid particles will adhere to the structural surfaces such as thickener wall.
- Over-flocculation results in fluffy underflow bed.
- Over-flocculation results in channeling as the polymeric chains form relatively robust structures within the bed.
- Over-flocculation sometimes causes larger flocs to float.

Therefore, using excessive flocculant is not effective. The primary goal of adding flocculant to a thickening feed is to speed up the settling of solid particles. With fast settling solids, a larger volume of slurry can be processed in a small area in relatively shorter period (Probst, 2001). Flocculants have expensive operating costs. There can be a trade-off between equipment size and required dose, with higher throughput per unit area requiring a higher flocculant dose to a certain point (Robert C. Dunne, 2019).

## 3.2. Sedimentation

Sedimentation is the process where gravitational force causes suspended solid particles to separate from suspension. The solid particle settles when its density exceeds that of suspending liquid medium. After sedimentation, huge volume of water can be removed, producing 55-60 percentage of solids. It usually is done before filtration as it puts less strain on the filters increasing the filtration capacity (Ruthven, 1997). It aids in minimizing the filter's size and the amount of room required for its installation.

Clarification and thickening are two categories of gravitational sedimentation. The size of the particle, the concentration of the solids in the slurry, and the flocculation of the particles all have an impact on how solid particles settle under gravity. Settling solids act as individual particles in a dilute suspension, known as *free settling*. Most clarifying processes involve free settling. In a concentrated suspension, suspended particles are close to each other and form aggregates. These aggregates settle together as a mass and is referred as *hindered settling*. In hindered settling, the concentration rather than the particle size has a greater impact on the settling behavior of the solids. As the solids continue to settle, solid bed is formed. The sediment on top of the settled solid bed compresses it, releasing liquid trapped in between the particles. The zone is called *compression zone*. Flocculants can be added to create flocs and speed up the settling of suspended solids. Most of the thickening operations fall under hindered settling and compression zone (Dorf, 2004). The different zones in a sedimentation unit are shown in the Figure 6.

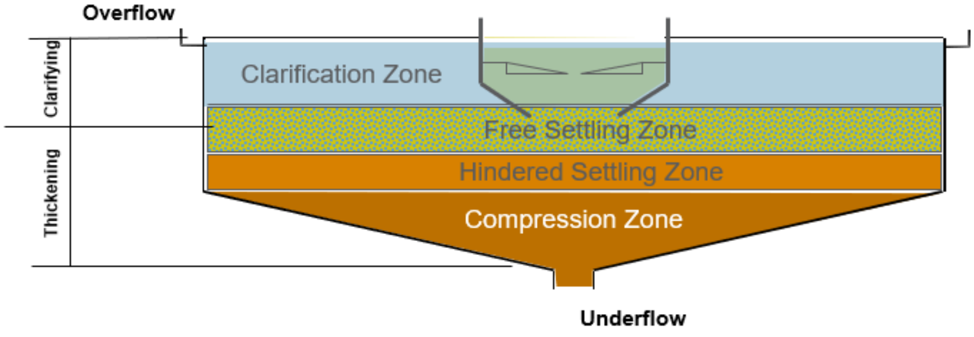



**Figure 6** Different zones in a sedimentation unit (Metso Outotec)

### *3.2.1. Clarifier*

A clarifier works under the same principle as of thickening, but clarifiers focus on the clear overflow liquid. Clarifiers, typically, treat low density slurries with less than 5 % solids w/w. Clarification process uses gravity for settling of solids. Coagulants and flocculant are added to achieve high level of clarity in the overflow. Usually, the clarified liquid is recycled back to the plant. Clarifiers are commonly used when overflow clarity is of high importance. The settled solids/underflow can be recycled to the feed to enhance flocculation of solids (Metso Outotec, 2022). Figure 7 shows how a typical clarifier operates.

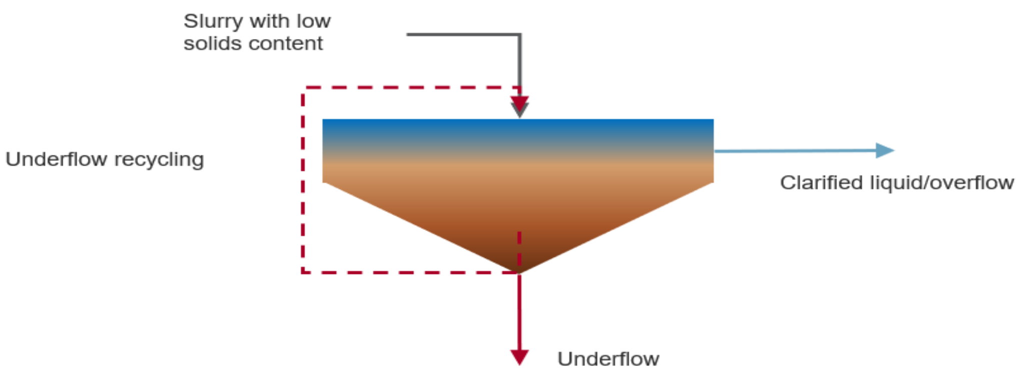



**Figure 7** A typical clarifier (Metso Outotec)

### 3.2.2. *Thickener*

Thickeners produce concentrated underflow and clear overflow. The settling velocity of solid particles affects thickener performance, which is influenced by the difference in solid and liquid densities as well as particle size.

For a single spherical solid particle of diameter $D$ and density $\rho_s$ suspended in liquid with density $\rho_f$ and viscosity $\mu_f$, the settling velocity is given by the stokes velocity,

$$\mathrm{v} = \frac{D^2(\rho_s - \rho_f)G}{18\mu_f} \tag{1}$$

where, $g$ is the acceleration of gravity (Betancourt *et al.*, 2014).

Sedimentation equipment is commonly known as thickener and clarifier in the minerals industry. Thickeners usually produce denser underflow while clarifiers usually clarify the liquid. Minerals processing industries generate a large amount of wastewater with suspended solids in it. Thickeners are widely utilized in minerals processing because of its simple establishment, operation, and cost (Garmsiri and Unesi, 2018). Thickeners are basically used to dewater the feed slurry utilizing the principle of sedimentation. Thickening technology is a cost-effective method of solid liquid separation where an incoming feed stream is used to produce two phases:

- Overflow - Clarified water with minimal solids
- Underflow - Concentrated solids with minimal liquid

A well designed and operated thickener can produce both clarified overflow and concentrated underflow. In a functional thickener, successful thickening involves several means and processes:

- Settling under gravitational force
- Flocculation - Addition of flocculant to incoming feed to enhance gravity settling by agglomeration of solid particles

- Underflow bed compression due to self-weight of solid materials and raking which releases water trapped in between the solids

The particle sizes in a mineral suspension can vary greatly. The rate of sedimentation of bigger particles is faster than that of small particles. When the density difference between solid particles and liquid suspension is small and the size of the particles is fine, it can result in a larger diameter tank and a longer retention time. Flocculant addition, agglomerates solid particles to form flocs, which enhances the settling rate. The separation process becomes more challenging as particle size gets finer, however, the concentration of solids in the slurry is crucial (Dunne, Kawatra and Young, 2019).

Feed dilution enhances the dewatering performance of a thickener. It ensures maximum number of solid particles get exposure to the flocculant added. In a diluted feed, flocculated solids act as independent unit and settle freely and quickly. The effect of feed dilution on floc size and density increases flocculation efficiency (Hogg, 1999). Feed dilution reduces flocculant consumption, increases settling flux and enhances the underflow bed compressibility (M.D. Green, 1997). Overflow can be recycled to dilute the feed to reduce the fresh water consumption.

Yield stress (YS) measurement provides valuable information on handling of underflow from thickeners as a function of solids concentration. It indicates the upper limit of solids concentration in underflow that can be handled (Usher, Hons and Sc, 2002).

Generally, thickeners are cylindrical in shape as shown below in Figure 8. The feed slurry enters the thickener via feedwell, where it is diluted and flocculated. The flocculated feed gets dispersed, forms larger agglomerates, and rapidly settles on the thickener bed. Settled solids then start to compact in the compression zone, creating dense underflow. Clarified water rises to the top and overflows through launders into collection launder. Overflow launders are the U or V shaped notches located on the periphery of the thickener for the disposal of overflow liquid. Thickeners have rake mechanism, slowly turning around the center column located close to the bottom and spanning the full diameter. Rakes have attached angled plates called scraper blades, which scrapes and aids solids discharge through the bottom central underflow outlets (Rudman *et al.*, 2008).

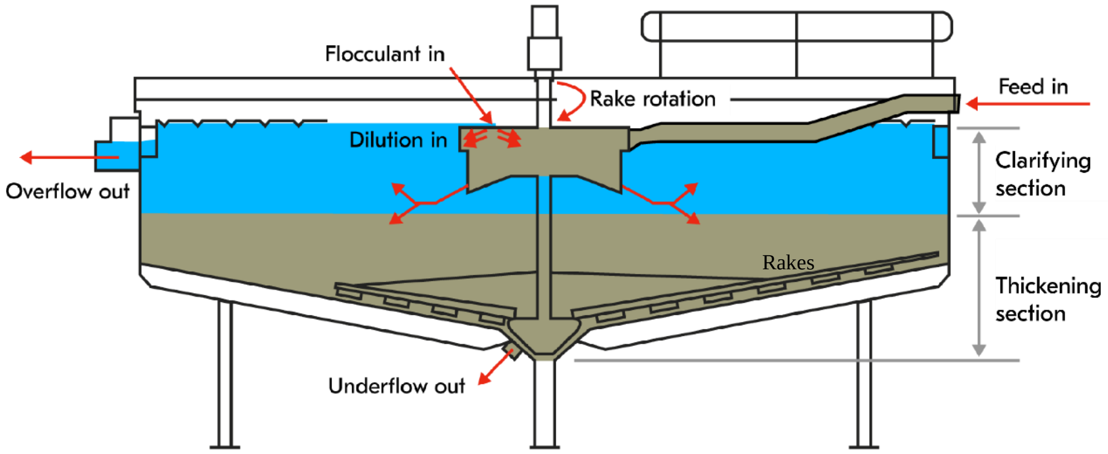



**Figure 8** A typical thickener (Metso Outotec)

Thickeners can be installed on the ground or in elevated steel tanks. Generally, large diameter thickeners are built on the ground with concrete while small thickeners are elevated steel tanks. Also, the construction material of thickeners depends on the slurry being processed.

A poorly operated thickener results in inconsistent operation with either less than optimal underflow densities or an undesirable level of solids in overflow. Valuable products can be lost and downstream operations like filtration can be affected. Flocculants are commonly used to enhance the settlement, increase the underflow density, and acquire cleaner overflow. It is imperative that optimal amount of flocculant is utilized to ensure maximum benefits.

There are different types of thickeners in operation depending upon the application.

- **Conventional thickener**

In a conventional thickener, flocculant is either not required or could impact the downstream process adversely. However, the addition of flocculant may enhance the overflow clarity, increase the underflow density, and increase the settling rate of solids resulting in higher tonnage of slurry handling (Dunne, Kawatra and Young, 2019). Conventional thickeners are generally larger in size and occasionally utilized as storage when there are issues within the plant facility. A feedwell, raking mechanism, and a bridge to support the feedpipe and permit center access are typical features of traditional thickeners. Conventional thickeners are suitable for highly variable flow and solids loading. They have large footprint and underflow density varies (Cacciuttolo Vargas and Marinovic Pulido, 2022). Figure 9 shows a conventional thickener where flocculant is not utilized.

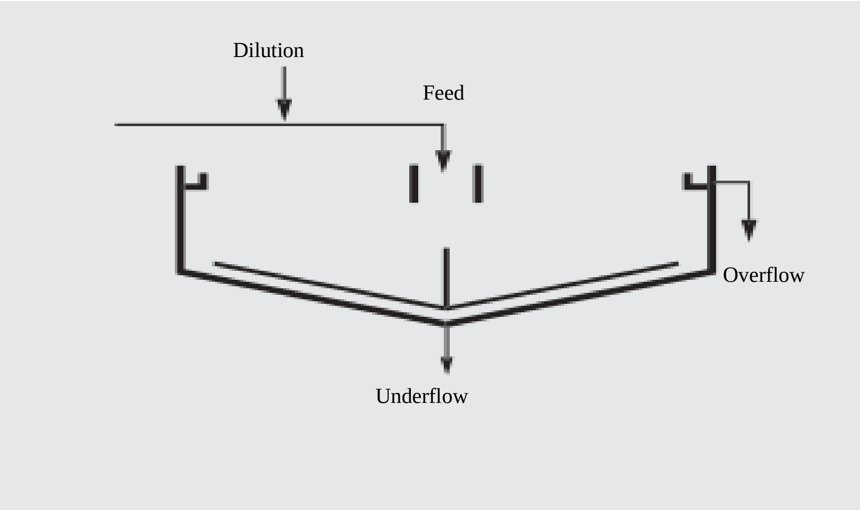



**Figure 9** A conventional thickener (Outotec, 2011)

- **High-rate thickener**

Since the throughput rates for flocculated slurries were significantly higher than those for non-flocculated slurries, the introduction of synthetic flocculant is largely responsible for the creation of high-rate thickeners (HRT) (Schoenbrunn *et al.*, 1983). In terms of size, feedwell design, and control, HRT are different from traditional thickeners. The HRT feedwell is made to distribute flocculants into the feed end uniformly so that flocculated slurry can enter the settling zone without harming newly created flocs (Bergh, Ojeda and Torres, 2015). Figure 10 illustrates the operation of a high-rate thickener, where flocculant is fed to the feed, and underflow is recycled back to the feed to improve flocculation.

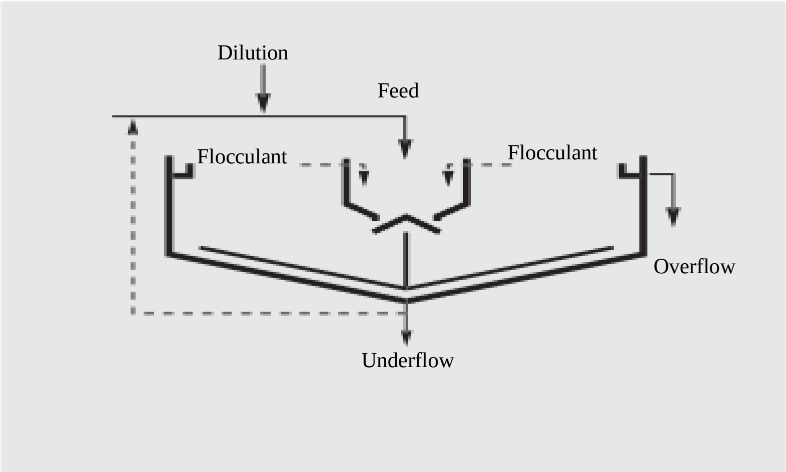



**Figure 10** A high-rate thickener (Outotec, 2011)

- **High compression thickener**

In comparison to HRT, it achieves consistent higher underflow density, usually 2-6 % solids w/w because of the deeper mud bed that increases the bed's available compression forces. High compression thickeners (HCT) typically add 1-3 meters in sidewall to an HRT (Dunne, Kawatra and Young, 2019). HCT have high torque but similar footprint to HRT. Common HCT applications are tailings dewatering, pre leach and post leach duties and clay and mineral sand slimes. The operation of a high compression thickener can be seen in Figure 11, where the increase in sidewall height is apparent.

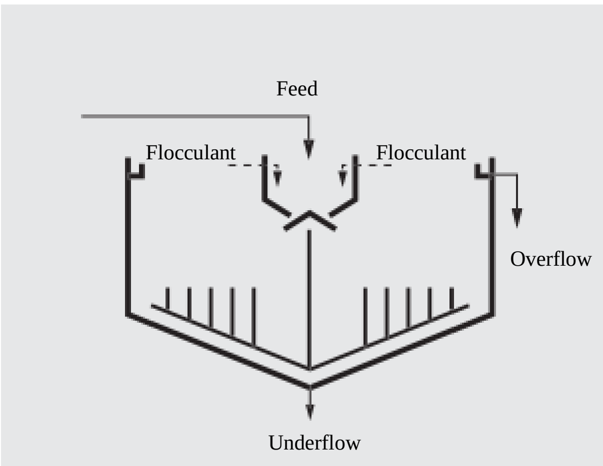



**Figure 11** A high compression thickener (Outotec, 2011)

- **Paste thickener**

Paste thickener (PT) also known as deep cone thickeners are used when high density underflow is the goal. PT uses very deep mud bed to take advantage from the bed compression force and provide sufficient time to dewater. The tank diameter to height ration is 1:1 or higher (Dunne, Kawatra and Young, 2019). PT produces underflows with yield stress (YS) measuring more than 200 Pa. Common PT applications are tailings dewatering, pre leach and post leach duties and alumina washing (Metso Outotec, 2022). In Figure 12, a paste thickener operation is shown, and its sidewall height is much higher than that of both high-rate and high compression thickeners.

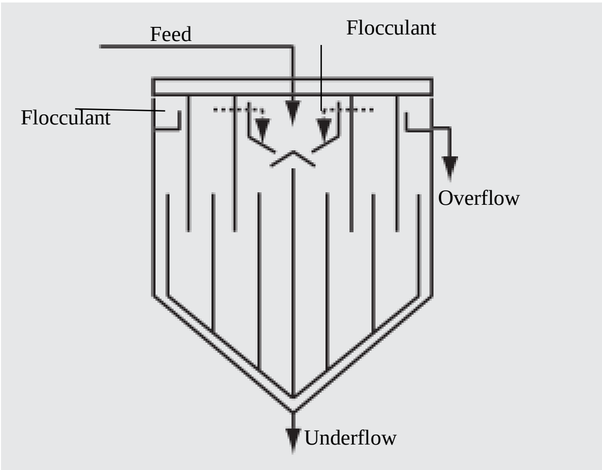



**Figure 12** A paste thickener (Outotec, 2011)

## 3.3. Filtration

The removal of suspended solid particles from a liquid by depositing them on a filter medium with pore sizes too tiny for the solid particles to pass through is known as filtration. Solids are deposited either on the surface or within its depth. Liquid permeates through the filter medium by a pressure difference across the medium. Solids deposited on the filter medium are called ‘cake’ and liquid that has been displaces is called ‘filtrate’ (Ripperger, Gösele and Alt, 2009).

Filtration is typically done for one of the following reasons:

- clarification of liquid and/or
- recovery of solids
- separation aiming to facilitate and enhance other plant operations (Cheremisinoff, 1998).

Different process variables, such as particle size distribution (PSD), solids concentration in feed, composition of particles, including density, shape, and surface characteristics, feed temperature, liquid viscosity, pH, and filter aid, all have an impact on filtration. Larger solid particles in feed make filtration easier and increase filtration capacity. A wide particle distribution results in delayed filtration process because of the formed cake is too dense and resists liquid and air to pass through. Higher solid content in feed increases the filtration capacity. Solids with high density filter easily, increasing filtration capacity. High temperature of feed and low viscosity of liquid also increases filtration capacity.

Basically, filters can be classified into following two types: Depth filters and cake filters (Mullin, Lowrison and Svarovsky, 1977).

- **Depth filters**

Depth filters are used for deep bed filtration where solids are deposited inside the filter medium. The filter medium consists of a deep bed with pores that are much bigger than the particles that the filter is meant to trap. Gravity, diffusion, and inertial forces cause solids to penetrate the filter medium and separate there. Porous media, sand, filter cartridges are commonly used as filter medium in deep bed filters (Svarovsky, 2001). Figure 13 illustrates the mechanism for deep bed filtration.

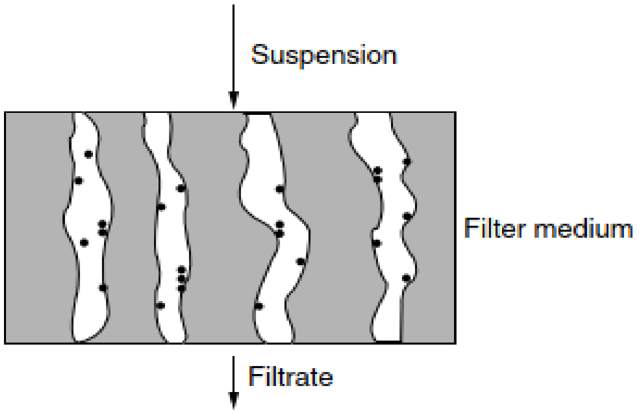



**Figure 13** Mechanism of deep bed filtration (McCabe, Smith and Harriot, 1993)

In deep bed filtration, the solid particles are deposited inside and within the filter medium after the slurry has passed through it. It is undesirable for solid particles to accumulate on the filter medium's surface. Deep bed filtration is preferred when the suspension is very dilute. Sand and synthetic fibers bed are used to trap fine solids from the suspension. Solids in the suspension will penetrate deep into the bed before being trapped (McCabe, Smith and Harriot, 1993).

- **Cake filters**

The solid particles in suspension are retained on the surface of the filter medium in cake filters (Svarovsky, 2001). The retained solids on the filter cloth gradually accumulate to create a cake, which then serves as the filter media. Cake filters can separate large quantities of slurries. For cake filters to function, either vacuum is applied from downstream or above-atmospheric pressure is applied upstream of the filter medium (McCabe, Smith and Harriot, 1993). Figure 14 illustrates the mechanism for cake filtration.

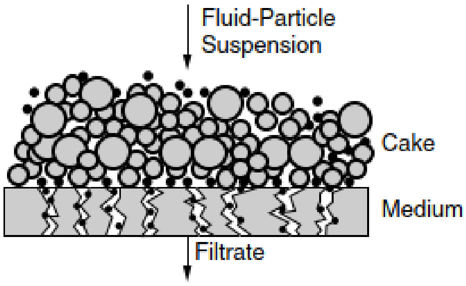



**Figure 14** Mechanism of cake filtration (McCabe, Smith and Harriot, 1993)

It is necessary to look at fundamental aspects of flow of fluid through porous medium to understand cake filtration. Darcy's law states that there is a basic connection between the pressure difference and the rate at which liquid flows through porous medium. The equation for steady laminar flow through homogenous and incompressible porous media is given by

$$q = \frac{k}{\mu}\frac{dp}{dx} \tag{2}$$

where, *dp/dx* is the dynamic pressure difference across thickness *dx* of porous medium with permeability *k*, *q* is the liquid superficial velocity i.e., volume flow rate/cross sectional area, and *μ* is the liquid viscosity(Gray and Miller, 2004).

Darcy's law is modified in filtration. The permeability *k* is replaced by specific flow resistance α and the pressure gradient *dp/dx* is replaced by pressure loss per unit mass of solid deposited by *dp/dw*:

$$q = \frac{1}{\mu\alpha}\frac{dp}{dw} \tag{3}$$

where *dw* is the dry cake mass per unit filter area deposited and is normally used in filtration rather than the distance from media, *dx*. Relation between mass *dw* and *dx* is given by:

$$dw = \rho_s\,(1-\varepsilon)dx \tag{4}$$

where, $\varepsilon$ is the porosity of the cake.

Substituting Equation 4 into Equation 3 gives:

$$q = \frac{1}{\mu\alpha\rho_S(1-\varepsilon)}\frac{dp}{dx} \tag{5}$$

Now comparing Equations 5 and 2, the relation between permeability $k$ and specific flow resistance $\alpha$ is given as:

$$k = \frac{1}{\alpha\rho_S(1-\varepsilon)} \tag{6}$$

Darcy's conventional equation assumed solid velocity to be negligible for flow through a fixed and incompressible bed (Tiller and Green, 1973).

Application of pressure difference impacts filtration. Pressure differences can be generated by gravitational force, by vacuum, by pressurized air/fluid, or by centrifugal force (Ripperger, Gösele and Alt, 2009). Filters run by gravity are simple and cost effective but are bulky and dewatered solids still content significant percentage of residual moisture. In gravity filters, atmospheric pressure forces the solids to pass through the filter medium. Filter medium receives slurry feed from the top of the filter. A cake builds up on the filter medium surface while clear filtrate flows from the bottom. Some of the examples of gravity filters are rotary drum gravity filters, belt filters and sand filters (Svarovsky, 2001). Filters driven by vacuum, pressure and centrifugal forces are briefly discussed below.

- **Vacuum filters**

In vacuum filters, pressure difference is created by application of vacuum. Vacuum can be created using a gas displacement device or the suction of a liquid pump. Generally, the driving force of 0.5 – 0.9 bar is sufficient to provide good filtration rate in many applications.

Slurry is forced through the filter medium by the atmospheric pressure in front of it due to vacuum created behind the filter medium, which filters the suspended solids in the suspension. Some of the examples of vacuum filters are vacuum belt filters, disc filters and rotary drum filters (Svarovsky, 2001).

- **Centrifugal filters**

In centrifugal filters, feed is driven through the filter medium by centrifugal force. During filtration, liquid passes through the interstices of solid particles deposited on the filter medium. Cyclones and centrifuges are centrifugal filters (Svarovsky, 2001).

- **Pressure filters**

Pressure filters use pressurized air or water to force feed slurry through the filter medium as it enters the filter. Smaller filter units can fit more easily in a process circuit since pressure filters produce more output per unit area (Svarovsky, 2001). Also, pressure filters provide better washing and drying results. Two basic types of pressure filtration exist: plate and frame press, and chamber press.

Plates and frame are arranged in an alternating pattern on the plate and frame press. Filter cloth separates the hollow frame from the plate. Hydraulic piston closes the filter press and compresses the filter cloth between plates and frames preventing leakage, creating a tight chamber between plates. Slurry is pumped to the empty frames from the holes in the corners of the plates and frames. As cake builds inside the frame, filtrate flows through the filter cloth, down the groove, exits the filter. The cakes are discharged by opening the plates and frames. The number of plates depend on the required filtration capacity and cake thickness (Napier-Munn and Wills, 2005). Plate and frame press filters can produce cakes that are up to 50mm thick. These filters have benefits such easier filter cloth change, more consistent cakes, and longer filter cloth life (Tarleton and Wakeman, 2008).

The chamber press filters are similar to plate and frame filters, with the exception that it only consists of the recessed filter plates. The individual filter chambers are formed between successive plates. A hole in the middle of each plate connects all the chambers. The plate is covered by a filter cloth with a central hole, and slurry is fed through the inlet. Filtrate exits the filter after passing through the filter cloth and tiny holes in the plate as cake accumulates on the chamber(Napier-Munn and Wills, 2005). Cakes up to 32mm in thickness can be produced with chamber press filters. These filters have lower initial costs, less leakage and can process higher concentration feeds (Tarleton and Wakeman, 2008).

Some of the examples of pressure filters are fast filter press, horizontal filter press and tube press (Napier-Munn and Wills, 2005). Figure 15 shows the operation principle of Metso Outotec Larox® PF filter.

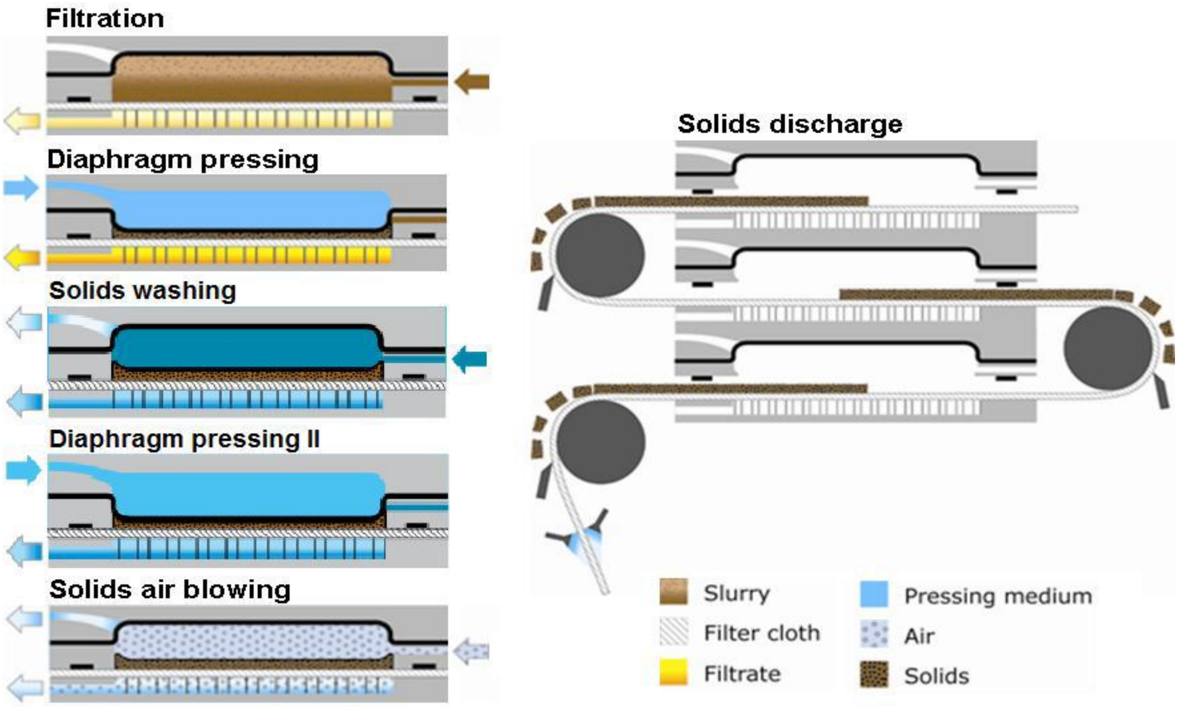



**Figure 15** Operation principle of Metso Outotec Larox® PF filter (Metso Outotec)

*Filtration*: Slurry is simultaneously pumped into each filter chamber. As more slurry is fed to the chamber, it displaces the filtrate and solids (cake) start to form on the filter cloth surface. The pumping pressure rises as the solids accumulate, forcing the filtrate through the cloth until the desired solids thickness is reached.

*Pressing*: High-pressure air or water automatically inflates a diaphragm at the top of each chamber. By doing so, the volume of the chamber is decreased, and the solids are compressed to extract more filtrate. Clear filtrate is produced by the solid's filtration process and tightly woven filter cloth. The high pressure increases the efficiency of the filtration process. Dewatered solids of uniform thickness with minimal excess liquid generated by diaphragm pressing facilitate the air blowing operation.

*Air Drying*: After pressing the solids', compressed air is blown through the solids for final dewatering. The moisture content of the cake can be lowered and controlled by adjusting the pressure and duration of the air blow.

*Cake discharge and cloth wash*: The dewatered solids are transported out of each chamber on the moving filter cloth once the plate pack opens. To reduce cloth blindness, the integrated wash unit uses high-pressure water to spray both sides of the cloth.

Pressure filters allow high pressure differences, withstands higher pressures, and can produce dry cakes. Tailings can be filtered using pressure filters, resulting in dry cakes and a clear filtrate. It reduces the environmental impact by removing the need for large dams to store tailings with a high-water content.

# 4 Objectives

The primary object of this thesis was to dewater two different tailings sample by thickening first and then filtering the underflow produced from thickening using pressure filter. Additionally, the impact of flocculant dose on the underflow densities and overflow clarities during thickening and filtration capacities and cake moistures during filtration were studied.

The goal of the thickening process was to obtain dense underflow and clear overflow. Flocculant is a key component in thickening. The best performing flocculant type and optimum dose was determined by flocculant screening tests. Each thickener underflow was filtered using pressure filter to achieve maximum filtration capacity. The following chapter describes the tools, techniques and details of the experiment and results.

# 5 Materials and Methods

## 5.1. Materials

Sample from two different tailings were used in this study i.e., Graphite tailings and Composite tailings. Graphite tailings used for this experiment was extracted from flotation tailings in a pilot study, which consisted of silicate gangue, graphite, and process water. Composite tailings sample was obtained from a Spodumene mine, which comprised of flotation tails, slime, and process water. The tailings sample had crystal quartz and arsenic in it.

## 5.2. Analysis Methods

For analysis of the samples, following methods, tools and formulas were used.

- pH: pH for the tailings samples were measured with pH papers. For pH adjustment, calcium hydroxide was added to the slurry.
- Temperature (T): Temperature of tailings samples and dilution waters were measured with thermometer.
- Density (ρ): Density is the weight per unit volume of a substance, i.e.

$$\rho = \frac{m}{\mathrm{V}} \tag{7}$$

Where, $\rho$ is density

$m$ is the mass in grams (g) and

$V$ is the volume in liter (L).

- Specific Gravity (SG): Specific Gravity is the density ratio of the substance to water, i.e.

$$SG = \frac{\rho_s}{\rho_w} \quad (8)$$

Where, SG is the specific gravity, $\rho_s$ is the density of substance and $\rho_w$ is the density of water.

- Solids content (% w/w): Solids content is the amount of dry solids present in slurry and usually represented in percentage. It is calculated by

$$Solids\ \% = \frac{Net\ weight\ of\ dry\ sample[g]}{Net\ weight\ of\ original\ sample[g]} \times 100\% \quad (9)$$

A known volume of slurry was weighed and let dry overnight in an oven at 105°C. The next day dry solids were weighed and solids content in slurry was calculated.

- Particle size distribution (PSD): Particle size distribution for tailings samples were analyzed with Malvern Mastersizer 3000. Slurry samples were continuously mixing and recirculating via peristaltic pumps. Representative sample was taken from feed stock slurry and continuously mixed before pouring it in the analyzer beaker. The samples were then added with a pipette to a 1000 mL water-filled analyzer beaker. After that, the sample was stirred using a motorized stirrer while the volumetric PSD was assessed by laser diffraction 3-5 times at intervals of 15 seconds. Fraunhofer method was used to measure the PSD. The average value was determined after each experiment was carried out at least 3 times.
- Suspended solids in overflows and filtrates (mg/L): A vacuum filtration device was used to filter 100 mL of sample liquid through a pre-weighed Whatman GF/C Filter (1.2 µm) to measure the suspended particles in the overflow and overflow samples. Before being dried in an oven at 105°C, the collected overflow solids were thoroughly cleaned with water. To determine the suspended particles content, the filter was reweighed.

For determining the moisture content, cakes and underflows were dried in an oven overnight at 105°C. The following formula is used to determine the moisture content:

$$cake\ moisture\ [\%\ w/w] = \frac{wet\ cake\ weight\ [g] - dry\ cake\ weight\ [g]}{wet\ cake\ weight\ [g]} \times 100\% \quad (10)$$

- Underflow density (% w/w): Underflow samples were taken when the underflow bed height in testing rig reached 240 mm. Underflow samples were pumped at low speed of 100 mL/min with a peristaltic pump. 148 mL of underflow was collected in a vial and weighed, that was suitable for measuring yield stress. The underflow was dumped onto a tray and let to dry overnight at 105°C after the yield stress had been measured. Dry underflow solids were weighed the following day, and the underflow's solids content was calculated.
- Rheology, Yield stress measurement (Pa): A Thermo Haake VT550 rheometer was used for all rheological measurements. Underflow samples were collected and weighed in a 148 mL phial; and the solids content was then calculated. The technique plots shear stress against time, with the yield stress represented as the apex of the curve. The shear rate was held constant at 0.1 $sec^{-1}$. A simple un-sheared yield stress was measured for each underflow sample.

## 5.3. Sample preparation and characterization

Feed stock slurry was received in slurry form which was mixed in a well agitated and baffled container. Once properly mixed, different slurry properties were determined.

Sample characterization is the description of the primary characteristics of the particles, such as their size distribution, shape, density, along with primary properties of the liquid. The secondary properties, such as the settling velocity of the particles and the specific resistance of a filter cake, are influenced by primary characteristics of the particles (Svarovsky, 2001). The characteristics of both tailings that were dewatered are given in Table 1.

**Table 1** Tailings characterization

| | Graphite Tailings | Composite Tailings |
|---|---|---|
| **Feed** | | |
| Solids content (% w/w) | 2.4 | 11 |
| Solids SG | 2.72 | 2.57 |
| Slurry density (g/L) | 1015 | 1073 |
| Liquid density (g/L) | 1000 | 1000 |
| pH | 10 | 6.5 |
| Temperature (°C) | 20 | 20 |
| PSD d10 (μm) | 2.32 | 4.84 |
| PSD d50 (μm) | 8.61 | 70.3 |
| PSD d80 (μm) | 42.2 | 168 |
| PSD <10 μm (%) | 53.69 | 16.61 |

*For graphite tailings, pH was adjusted to 10 from 6 by adding calcium hydroxide as flocculation was extremely slow at pH 6.

## 5.4. Equipment

### *5.4.1. Metso Outotec Supaflo 99mm HRT*

99mm HRT was used to thicken the tailings samples which imitates a larger thickener. The apparatus has an underflow exit, rakes powered by a motor on top, a feed pipe, and a feed defector plate. A calibrated peristaltic pump is used to pump slurry into the test unit, where flocculant and dilution water (fluid) are added, mixed in the feedwell, and then allowed to settle to the bottom of the test unit by gravity. The 99mm Supaflo high-rate thickener used to thicken the tailings for this thesis is shown in Figure 16.

**Figure 16** 99mm diameter high-rate thickener (Metso Outotec)

Typically, 15 kg of dry solids and 100 litres of process water are needed for 99mm HRT testing. The solids and process water must be combined to form the feed stock slurry in a well agitated and baffled container. Dynamic thickening testing with 99mm HRT has the following benefits:

- Determining the exact correlations between feed rate per unit area, flocculant rate, underflow density, and overflow clarity.
- Overflow clarity is evaluated with greater accuracy than in static test work.
- Accurately calculating the necessary amount of feed dilution.
- It is possible to assess the impact of recirculating underflow in some low feed solids applications.
- Eliminating the need to use a safety factor whether the feed is normal or "worst case."

### 5.4.2. *Metso Outotec Larox® Labox 25*

Labox 25 is a bench scale pressure filtration unit, with filtration area of 25 $cm^2$ which works on the principles of Metso Outotec Larox® PF technology. Maximum 16 bar of pressing pressure can be applied to press and dewater the cake. The Metso Outotec Larox® Labox 25 bench scale unit is depicted in Figure 17.


**Figure 17** Metso Outotec Larox® Labox 25 bench scale unit (Metso Outotec)

## 5.5. Method

### 5.5.1. *Dynamic Thickening*

Flocculants used for the test works were in powder form, which were made into solution before using it to flocculate the slurries. Flocculants used were anionic polyacrylamide polymer flocculants with high molecular weights. A stock flocculant solution was prepared and diluted before thickening tests were carried out. Lappeenranta tap water was used to hydrolyze the stock flocculant solution to 0.25 % w/v (2.5 g/L), and it was then diluted as required. To prepare stock solution, 2.5 g of dry flocculant was gradually added to water, making sure that no "fisheyes" formed (granules did not adhere to each other). For flocculant screening, the stock flocculant solution was further diluted to 0.025% w/v.

Flocculant screening tests were conducted in identical 500 mL graduated cylinder. Slurry with desired solids content was poured in the cylinder and diluted with process water till 500 mL line (Farrow and Swift, 1996). Feed was diluted sufficiently to avoid hindered settling. Flocculant screening was conducted by adding flocculant to the diluted feed and gently mixing by inverting three times. Settling rates achieved by different flocculant types were measured by timing the mud bed line as it dropped over a given distance. The physical and chemical properties of Magnafloc flocculants used for screening tests are presented in Table 2.

**Table 2** Physical and chemical properties of flocculants used for flocculant screening.

| **Flocculant type** | **Polymer type** | **Charge** | **Molecular weight** | **Water solubility** |
|---|---|---|---|---|
| Magnafloc 10 (M10) | Polyacrylamide | Slightly Anionic | Very High | Yes |
| Magnafloc 155 (M155) | Polyacrylamide | Anionic low | High | Yes |
| Magnafloc 5250 (M5250) | Polyacrylamide | Anionic medium | High | Yes |

Magnafloc is a range of synthetic flocculants made for thickening, clarifying, filtering and centrifugation, among other mineral processing applications. High molecular weight flocculants enhance the settling of solids by several order of magnitude than low molecular weight flocculants (BASF, 2022).

Polyacrylamide flocculants are commonly used in thickeners. These flocculants come in a variety of molecular weights, charge densities, uses, and physical forms (Costine *et al.*, 2018). Flocculants used for screening tests for this study were anionic and high in molecular weight. Once suitable flocculant and dose was selected, and feed dilution was decided, dynamic thickening test work begin.

Figure 18 illustrates the set-up of dynamic thickener test work where the stock slurry is already dilute, and then the flocculant is dosed in two different points in the feed pipe.

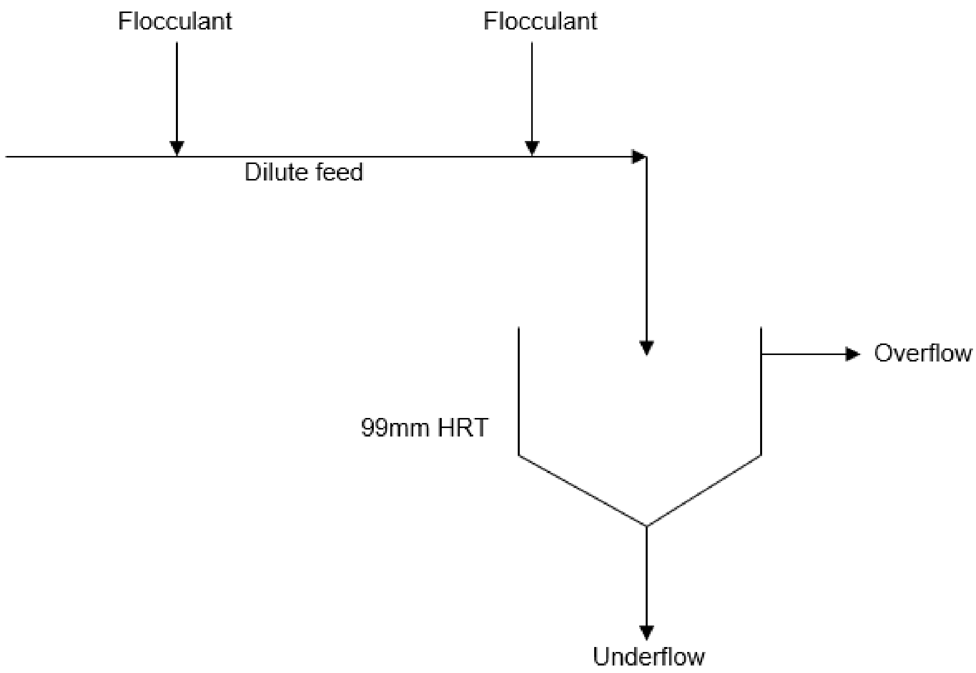



**Figure 18** 99mm dynamic thickening test work set up

### *5.5.2. Pressure Filtration*

Labox 25 is a bench scale filtration unit designed for research purposes in laboratory. Filtration tests for this study were carried out using Labox 25 test kit, with filtration area of 25 $cm^2$.

The filter medium for the filtration tests was filter cloth AINO T31. It is made from polypropylene and has air permeability of 1 $m^3/m^2$ min at 200 Pa.

Cycle times for filtration testing must be optimized first. For each tailing, thickener feed slurry was flocculated and let settle overnight. The settled solids were recovered after supernatant water was decanted. For both tailings, the solids content in the settled solids was matched with the average underflow solids produced from thickening runs. Thus, prepared slurry was agitated before filtering.

Filter cloth AINO T31 was placed on top of the grid. The cylinder that collects feed slurry was carefully tightened. 148 mL of homogenous underflow was poured into the cylinder. The slurry was pressed with a pressing pressure of 16 bar. Filtration cakes were dried with 5 bar of air-drying pressure.

# 6 Results and discussions

Thickening and filtration tests were conducted on graphite and composite tailings. The results obtained from the dewatering tests are presented and discussed in this chapter. The tailings were first thickened with a 99mm dynamic thickener, and the resulting underflows were filtered using pressure filter Labox 25.

## 6.1. Thickening

**Metso Outotec 99mm dynamic thickening testing**

The tailings were dynamically thickened in the 99mm HRT. Stock slurry was pumped from an agitated container for each test using a variable speed peristaltic pump.

For each test, the required dose of flocculant was pumped at a specified flow rate. To help with dewatering, the underflow bed was raked using two rotating pickets and two stationary pickets at a pace of two revolutions per minute. As soon as the underflow bed height reached 160 mm, overflow samples were taken. When the underflow bed height of each test reached 240 mm, the test was stopped, and underflow samples were taken.

Suspended solids in overflow were calculated using the analysis technique outlined in section 5.2.

Underflow samples were collected and weighed in a 148 mL phial; the solids concentration was then determined as described in section 5.2.

### *6.1.1. Graphite tailings HRT*

Three different Magnafloc flocculants were used for flocculant screening tests: Magnafloc 10 (M10), Magnafloc 155 (M155) and Magnafloc 5250 (M5250). Flocculant screening was conducted on the graphite tailings samples in 500 mL static cylinders. Results from the flocculant screening tests can be seen in Figure 19 and Figure 20.

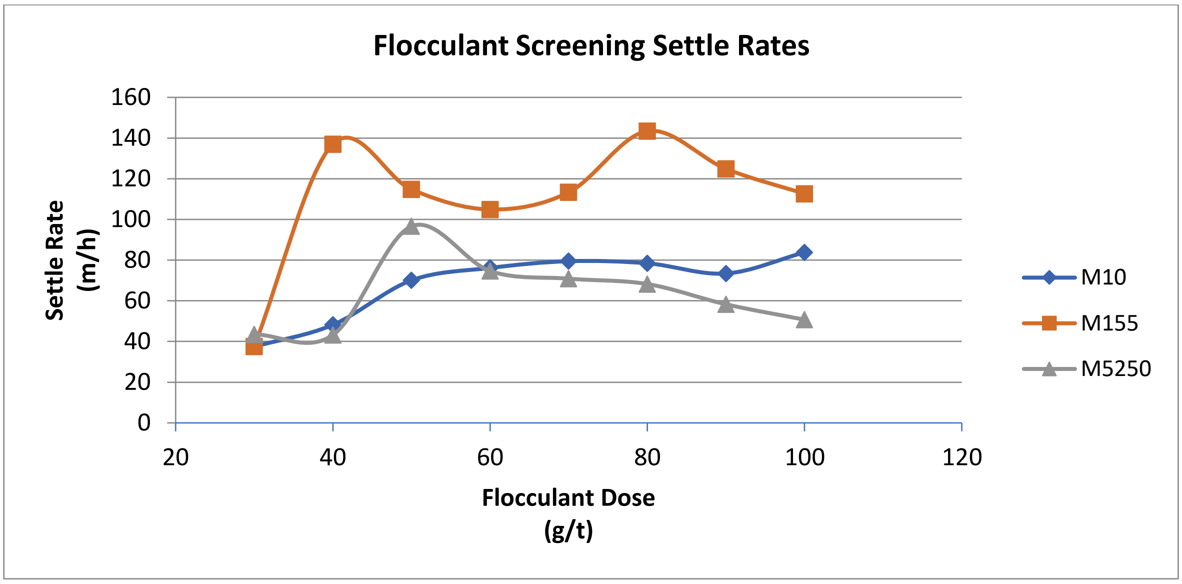



**Figure 19** Flocculant screening tests for graphite tailings at pH 6

M155 at 40 g/t provided fastest settling of solids, however overflow was cloudy. The settling rate with M10 was slower than that of other flocculants but provided clearer overflow.

**Figure 20** Flocculant screening tests for graphite tailings at pH 6. From left to right; M10, M155 and M5250 respectively

Figure 20 shows that the flocculation was not effective at pH 6. The overall overflow quality was poor with plenty of suspended solids in it. Even at higher doses of flocculant addition, overflow clarity did not improve. The slurry pH was adjusted to 10 by adding calcium hydroxide and flocculant screening was performed again. Figure 21 shows the settling rates of these flocculants.

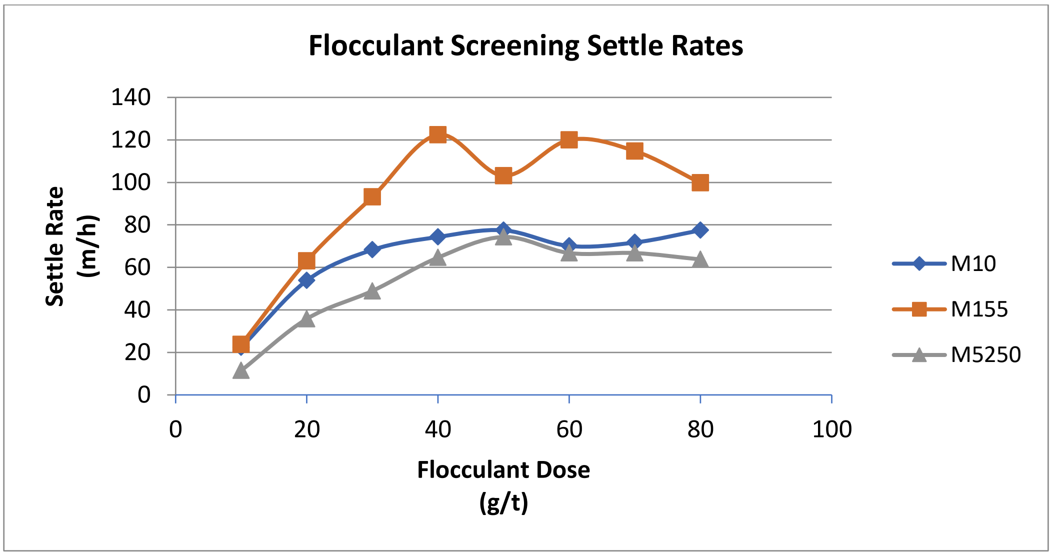



**Figure 21** Flocculant screening tests for graphite tailings at adjusted pH 10.

At slurry pH 10, settling rate of solids was slightly slower compared to settling rates at pH 6 but provided clear overflows already at lower flocculant doses. The optimal dose of flocculant was found to be 40 g/t of M155 based on the flocculant screening tests. Flocculant M155 was chosen for dynamic thickening tests as it provided fastest settling with clear overflow as seen in Figure 22.

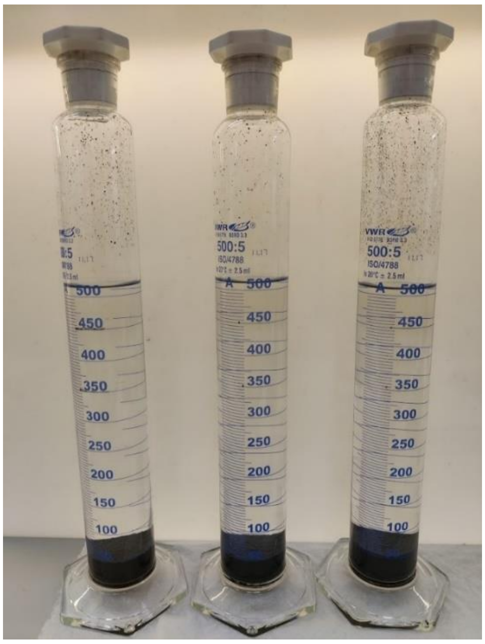


**Figure 22** Flocculant screening tests for graphite tailings at pH 10. From left to right; M10, M155 and M5250 respectively

Dynamic thickening tests for graphite tailings were carried out at a feed concentration of 2.4 % solids w/w. The results from dynamic thickening tests for graphite tailings are shown in Table 3.

**Table 3** Dynamic thickening test work results for graphite tailings

| Run No. | Feed | | Flocculant | | Underflow | | Overflow | Solids SG |
|---|---|---|---|---|---|---|---|---|
| | Flux (t/(m$^2$·h)) | Liquor RR (m/h) | Type | Dose (g/t) | Meas. Solids (% (w/w)) | YS (Pa) | Solids (mg/L) | (Measured) (t/m$^3$) |
| 1 | 0.40 | 16.80 | M155 | 40 | 49.2 | 28 | 152 | 2.75 |
| 2 | 0.40 | 16.80 | M155 | 60 | 48.7 | 29 | 275 | 2.69 |
| 3 | 0.40 | 16.80 | M155 | 20 | 50.4 | 35 | 130 | 2.71 |

The first run employed 40 g/t of flocculant M155. Underflow bed height reached 240 mm in 24 minutes, at which point the test was stopped and sample was taken. This resulted in an underflow density of 49.2% solids w/w and a yield stress of 28 Pa. Overflow sample was taken when the underflow bed height reached 160 mm in height. The overflow was clear, measuring 152 mg/L of suspended solids. Figure 23 shows that overflow clarity was almost clear at optimum flocculant dose of 40 g/t.

**Figure 23** Graphite tailings HRT Run 1

The second run was conducted at the same conditions as the first run, however the flocculant dose was increased from 40 to 60 g/t. It took 26 minutes for the bed height to reach 240 mm, when the underflow sample was taken. The underflow density decreased slightly to 48.7 % solids w/w.

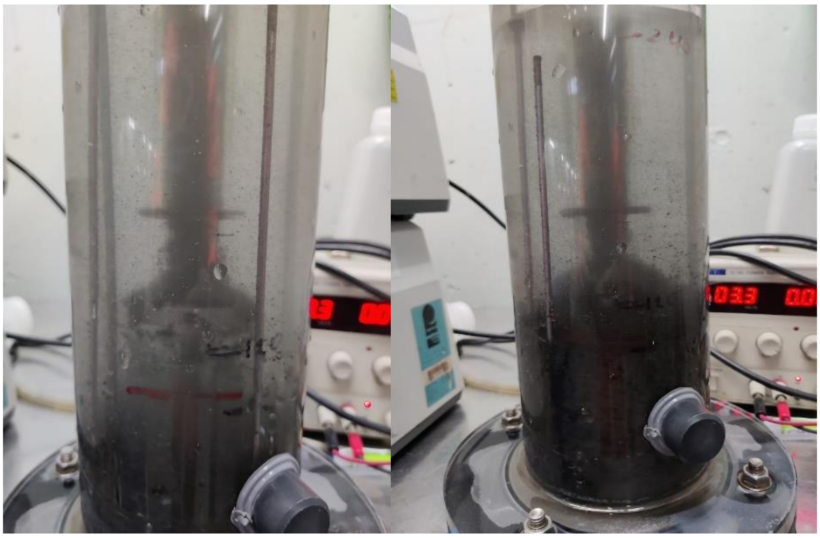


**Figure 24** Graphite tailings HRT Run 2

The overflow was cloudy, measuring 275 mg/L of suspended solids. The underflow yield stress measured 29 Pa. Figure 24 shows the second thickening test for graphite tailings which was run with 60 g/t of flocculant M155. The overflow clarity did not look as clear as the first thickening run which was run with lower flocculant dose.

In the third thickening test, flocculant dose was reduced to 20 g/t while the rest of the parameters remained the same as in the previous runs. With a yield stress of 35 Pa, the underflow density was 50.4 percent solids w/w. Overflow was clear, measuring only 130 mg/L of suspended solids.

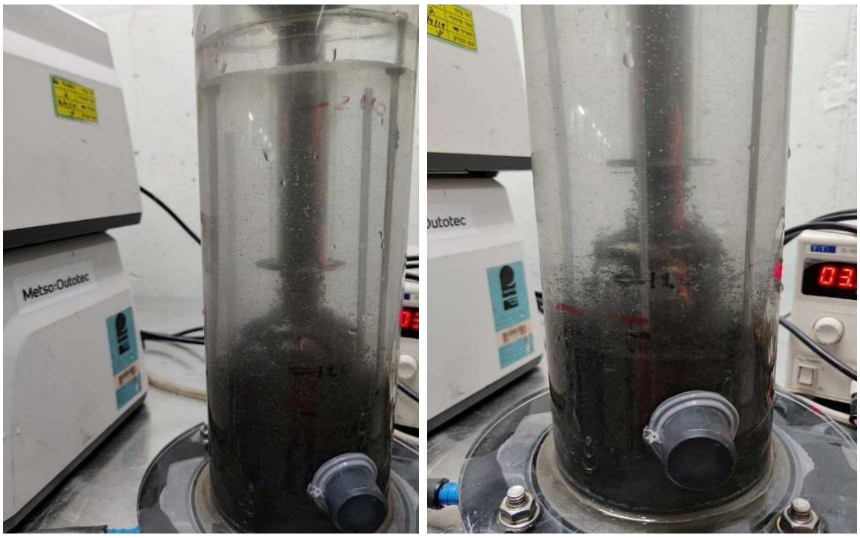



**Figure 25** Graphite tailings HRT Run 3

The overflow looks clear in Figure 25, which was run with 20 g/t of flocculant M155. From the flocculant screening tests, fastest settling rate with clear flow was achieved at 40 g/t of M155. However, in dynamic thickening testing, clear overflow was achieved already at 20 g/t of flocculant addition.

The effects of different flocculant doses on the underflow density and its yield stress, and overflow clarity of graphite tailings are presented in Figure 26 and Figure 27.

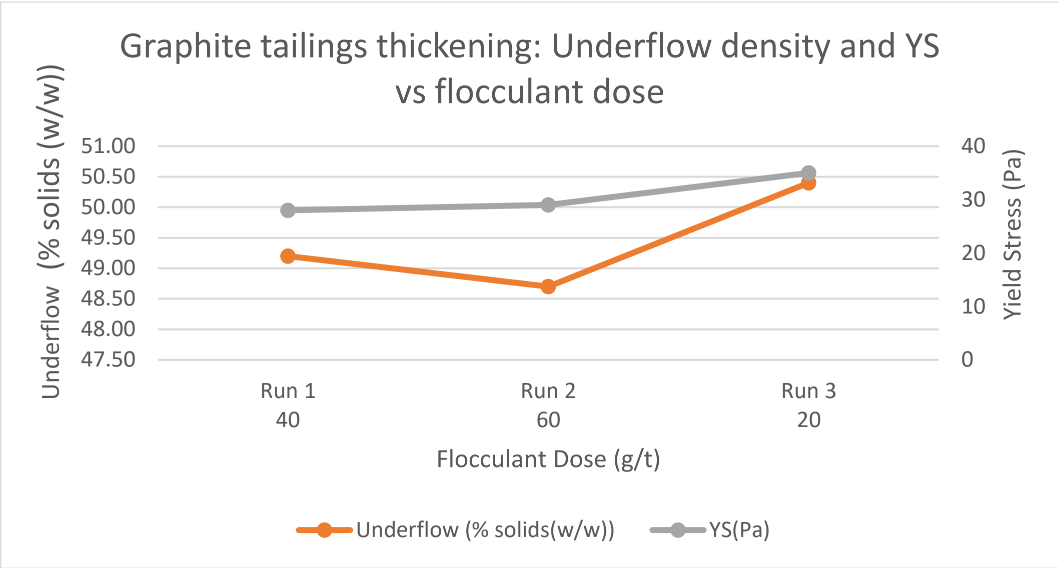



**Figure 26** Underflow density and yield stress measurement vs flocculant dose for graphite tailings thickening

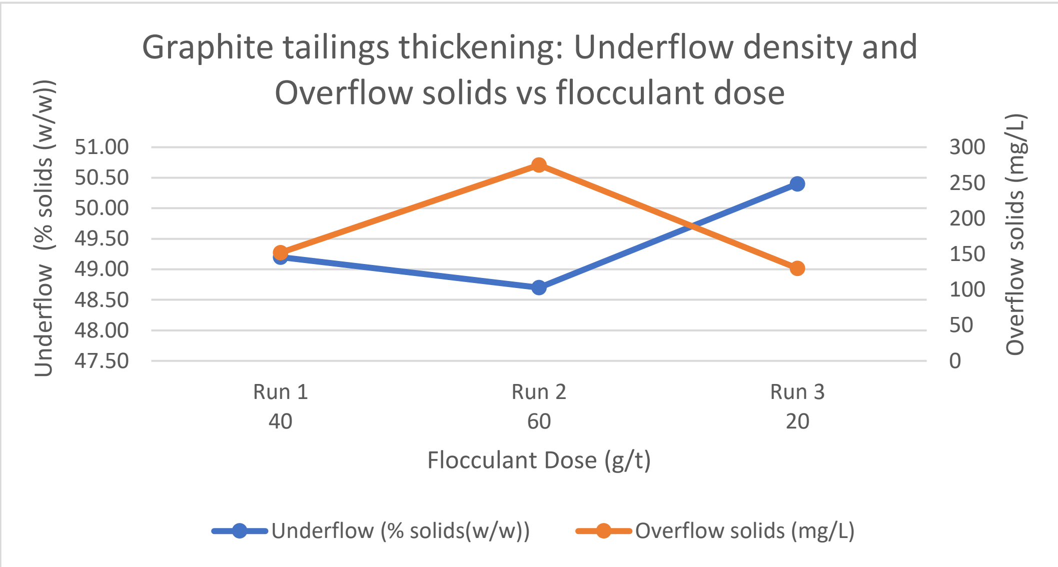



**Figure 27** Underflow density and overflow solids vs flocculant dose for graphite tailings thickening

From flocculant screening tests, flocculant type M155 provided fastest settling rates. The optimal dose of flocculant was calculated at 40 g/t from the flocculant screening tests. In thickening test run 1, 40 g/t of flocculant M155 was used.

The second thickening test was run at flocculant dose of 60 g/t to observe its effect on the underflow density and overflow clarity. The underflow density decreased slightly than in the thickening run carried out with optimal flocculant dose. Increasing the flocculant dose did not increase the underflow density. The overflow was relatively cloudier than the first thickening test, measuring 275 mg/L of suspended solids. The increased flocculant dose did not help improve overflow clarity either.

The third thickening test for graphite tailings was conducted with lowered flocculant dose of 20 g/t i.e., less than the optimal flocculant dose, again to observe its effect in underflow and overflow properties. In this instance, the underflow density climbed to 50.4% solids w/w. Flocculant dose when lowered than the optimal dose, resulted in increased underflow density and yield stress measurement. Overflow clarity was better than previous tests, measuring 130 mg/L of suspended solids. Even at lowered flocculant dose than that of optimal dose, underflow density increased, and overflow clarity got better.

### *6.1.2. Composite tailings HRT*

Magnafloc 10 (M10), Magnafloc 155 (M155) and Magnafloc 5250 (M5250) flocculants were used for flocculant screening tests for composite tailings. Flocculant screening was conducted on the samples in 500 mL static cylinders.

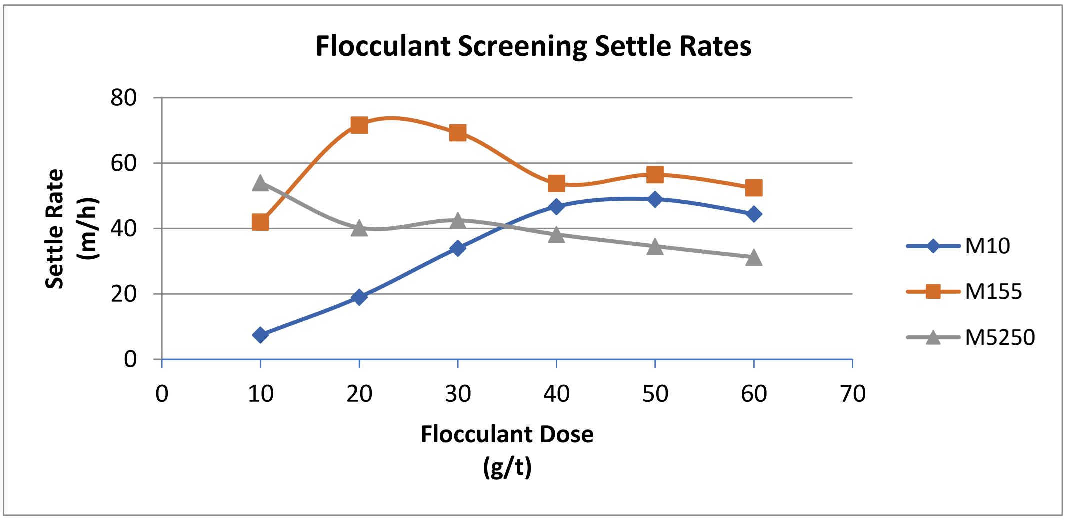



**Figure 28** Flocculant screening tests for composite tailings

Figure 28 shows the results from flocculant screening tests. Clear overflows were achieved already at lower flocculant doses as can be seen in Figure 29. Flocculant M155 was chosen for dynamic thickening tests as it provided fastest settling rate with clear overflow.

**Figure 29** Flocculant screening tests for Composite tailings. From left to right; M10, M155 and M5250 respectively

Dynamic thickening tests for composite tailings were carried out at a diluted feed concentration of 11 % solids w/w. The results from dynamic thickening tests for composite tailings are given in Table 4.

**Table 4** Dynamic thickening test work results for Composite tailings

| | **Feed** | | **Flocculant** | | **Underflow** | | **Overflow** | **Solids SG** |
|---|---|---|---|---|---|---|---|---|
| Run No. | Flux ($t/(m^2 \cdot h)$) | Liquor RR (m/h) | Type | Dose (g/t) | Meas. Solids (% (w/w)) | YS (Pa) | Solids (mg/L) | (Measured) ($t/m^3$) |
| 1 | 0.50 | 4.41 | M155 | 20 | 67.9 | 59 | 595 | 2.57 |
| 2 | 0.50 | 4.41 | M155 | 30 | 63.9 | 48 | 287 | 2.57 |
| 3 | 0.50 | 4.41 | M155 | 10 | 68.5 | 74 | 799 | 2.57 |

The first thickening run used 20 g/t of flocculant M155. This resulted in an underflow density of 67.9 % solids w/w with YS of 59 Pa. The overflow contained 595 mg/L of suspended particles and was cloudy.

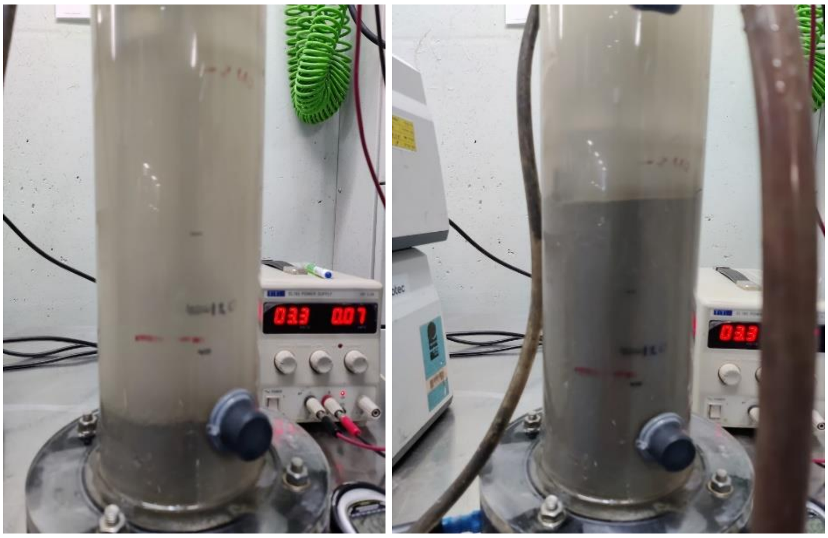


**Figure 30** Composite tailings HRT Run 1

Figure 30 depicts the thickening of composite tailings at 20 g/t of flocculant M155. The overflow clarity was already cloudy before the test began, and it did not become clearer as time went on.

The second run was conducted at the same conditions as the first run, however the flocculant dose was increased from 20 to 30 g/t. The underflow density decreased slightly to 63.9 % solids w/w. The underflow yield stress measured 48 Pa. The overflow was less cloudy, measuring 287 mg/L of suspended solids.

**Figure 31** Composite tailings HRT Run 2

The overflow clarity got better in the second thickening test for composite tailings, which was run at 30g/t of M155 as can be seen in Figure 31.

In the third thickening test, flocculant dose was reduced to 10 g/t while the rest of the parameters remained the same as in the previous run. With a yield stress of 74 Pa, the underflow density was 68.5 % solids w/w. Overflow was cloudy, measuring 799 mg/L of suspended solids.

**Figure 32** Composite tailings HRT Run 3

The overflow was cloudy again in the third thickening test for composite tailings, carried out with flocculant dose of 10 g/t of M155 as can be seen in Figure 32.

The effects of different flocculant doses on the underflow density and its yield stress, and overflow clarity of composite tailings are presented in Figure 33 and Figure 34.

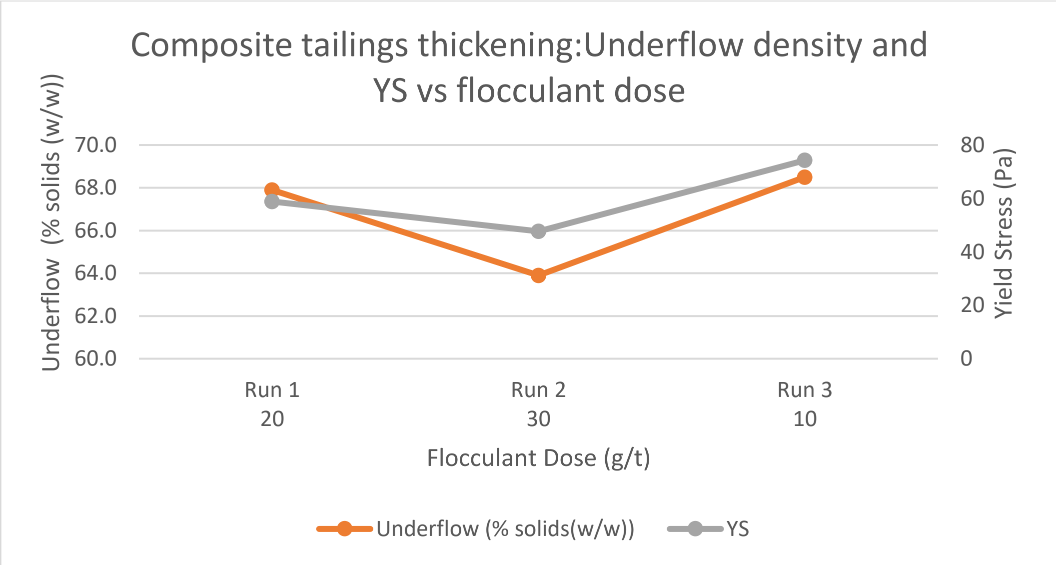



**Figure 33** Underflow density and yield stress measurement vs flocculant dose for composite tailings thickening

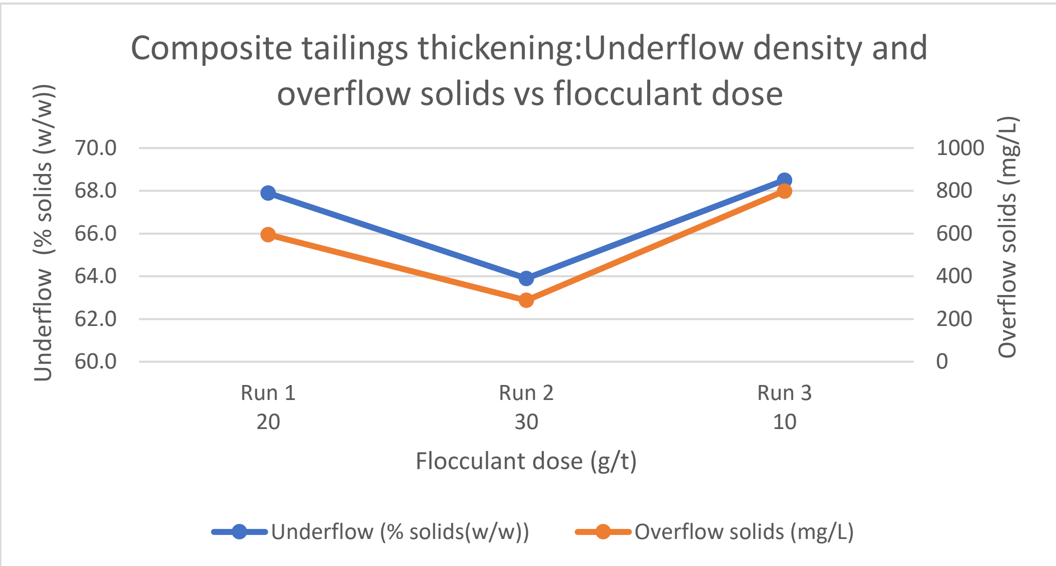



**Figure 34** Underflow density and overflow solids vs flocculant dose for composite tailings thickening

From flocculant screening tests, flocculant type M155 provided fastest settling rates. The optimal dose of flocculant was calculated at 20 g/t. In thickening test run 1, optimal dose of 20 g/t of flocculant M155 was used. The overflow contained 595 mg/L of suspended particles and was cloudy.

The second thickening test was run with increased flocculant dose; 30 g/t of flocculant M155 to observe its effect on the underflow density and yield stress measurement, and overflow clarity. Increased flocculant dose resulted in a decrease in underflow density, indicating that underflow density was not increased by increasing flocculant dose. Compared to the first thickening test, the overflow was comparatively clearer. Overflow clarity did get better as a result of the increased flocculant dose.

A lower flocculant dose of 10 g/t of M155 was used for the third thickening test on graphite tailings, which was done so that its impact on underflow and overflow properties could be studied. The underflow density increased to 50.4% solids w/w, indicating that underflow density increased when flocculant dose was below optimal dose. Overflow, however, was cloudier than in previous thickening tests, showing that a lower flocculant dose does not adequately clarify overflow.

## 6.2. Filtration

Underflow samples generated from thickening of graphite and composite tails were filtered using Metso Outotec filter technology.

AINO T31 filter cloth was used as the filter medium. Same filter cloth was used for all three filtration tests for each sample. After each test, filter cloth was rinsed and turned to be used in the next run. Figure 35 shows different components of Labox 25 put together to filter the tailings underflow.

**Figure 35** Metso Outotec Larox® Labox 25 (Metso Outotec)

### *6.2.1. Graphite tailings Labox 25 filtration*

Thickener feed slurry was decanted to 49 % solids w/w after addition of flocculant M155 to simulate the average underflow density from thickening tests. The slurry then was filtered to find the optimal parameters and cycle time.

Pumping: Since labox 25 test unit was used to filter the underflows, 148 mL of underflow was poured into the chamber. During calculation, 1 minute of pumping was used in data sheet.

Pressing: The cake was pressed with 16 bar of pressing pressure. The optimal pressing time was determined to be 2 minutes.

Drying: The cake was air dried with 5 bar of air-drying pressure. The optimal drying time was determined to be 1.5 minutes.

After the original slurry was filtered simulating underflow from thickening tests with addition of flocculant to optimize pressing and drying times, underflow generated from each thickening tests were filtered.

### Graphite tailings underflow filtration Run 1

The underflow generated from thickening of graphite tailings was filtered with Labox 25. The solids content in the underflow was 49.2 % w/w, which was produced by addition of 40 g/t of M155. The first filtration test was carried out with pressing pressure of 16 bar for 2 minutes. Filtrate started to get clear after first 10-15 seconds of pressing. The duration of air drying was 90 seconds and was found to be effective. The cake was dried with 5 bar of drying air. The resulting capacity from the first test was 286 kg DS/$m^2$h. A 27 mm thick cake was produced with residual cake moisture of 21.46 % w/w. Although the cake released smoothly from the cloth, bottom edges of the cake was sticking to the cloth, which under tap water was rinsed away easily. Figure 36 shows the cake and filtrate from filtration of graphite tailings underflow with 40 g/t of flocculant M155.

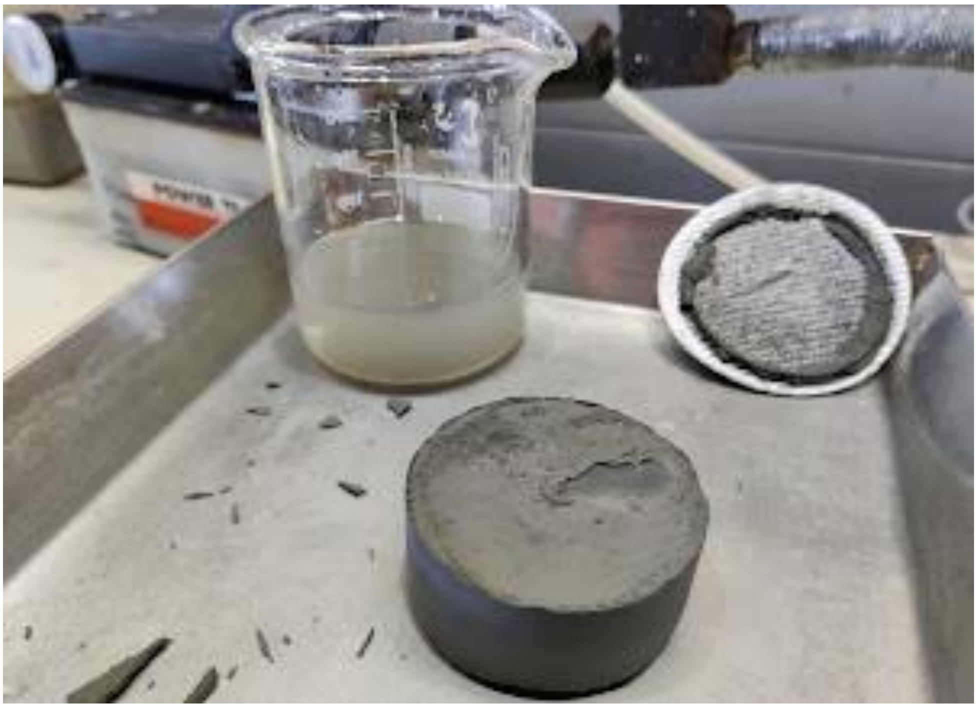


**Figure 36** Graphite filtration Run 1; 40 g/t of flocculant M155

### Graphite tailings underflow filtration Run 2

The second test was conducted using the same process times that were used in first test run. The underflow which was filtered had 48.7 % solids w/w and was produced by addition of 60 g/t of M155. The resulting filtration capacity from the second test was

282.7 kg DS/m$^2$h. Again, a 27 mm thick was produced with residual cake moisture of 22.12 % w/w. Cake released smoothly from the cloth, however some solids from the bottom edges of the cake stuck with the cloth as can be seen in Figure 37. The filter cloth was washed under tap water prior to the following test.

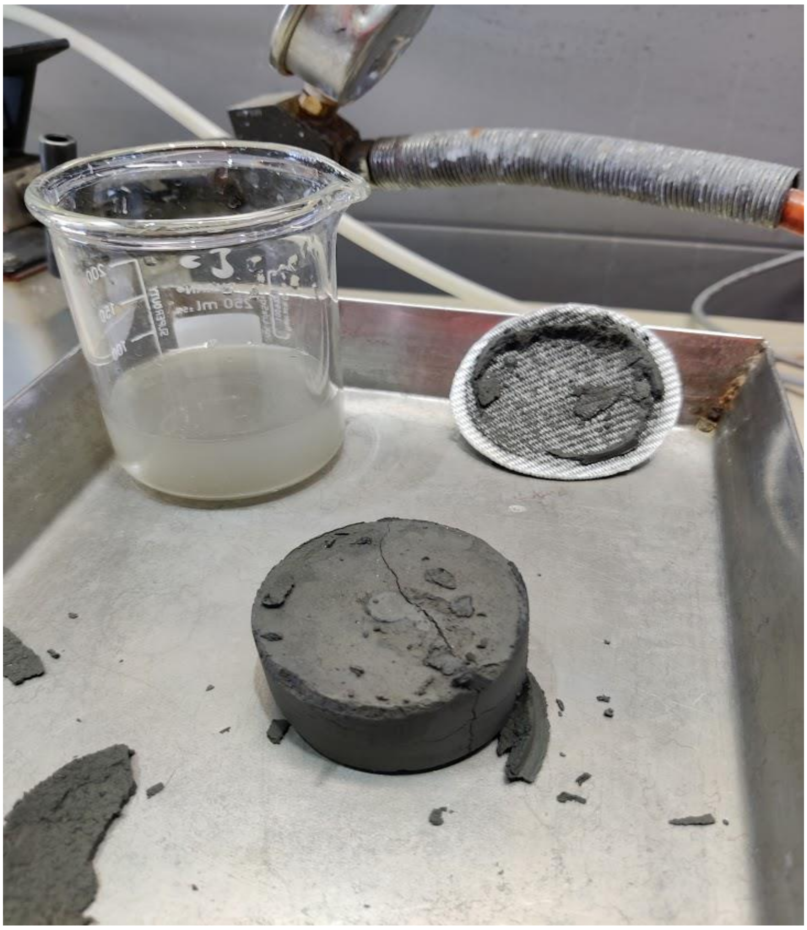


**Figure 37** Graphite tailings filtration Run 2; 60 g/t of flocculant M155

### Graphite tailings underflow filtration Run 3

The third test was also conducted using the same process times that were used in first and second test run. The underflow which was filtered had 50.4 % solids w/w and was produced by addition of 20 g/t of M155. The resulting capacity from the second test was 295.1 kg DS/m$^2$h. A 28 mm thick cake was formed with residual cake moisture of 22.52 % w/w. Cake released smoothly with small amount sticking to the cloth, which was rinsed away easily under tap water. Figure 38 shows the cake and filtrate produced from filtration of graphite tailings underflow thickened with 20 g/t of flocculant M155.

**Figure 38** Graphite tailings filtration Run 3; 20 g/t of flocculant M155

All Labox 25 tests for graphite tailings underflow filtration were performed with 16 bar pressing and 5 bar drying air pressure values. The thickness of resulting cakes from each test were consistent measuring 27-28 mm. Cake sticking to the filter cloth was minimal, which could be washed away under tap water.

The cake moistures, filtration capacities and suspended solids in filtrate for graphite tailings underflow filtration tests are tabulated in Table 5.

**Table 5** Labox 25 filtration test results for graphite tailings

| Run | Flocculant dose (g/t) | Total cycle time (min) | Cake Moisture (% w/w) | Filtration Capacity (kg DS/m²h) | Solids in filtrate (mg/L) |
|---|---|---|---|---|---|
| 1 | 40 | 8.5 | 21.46 | 286 | 777 |
| 2 | 60 | 8.5 | 22.12 | 282.7 | 1347 |
| 3 | 20 | 8.5 | 22.52 | 295.1 | 1273 |

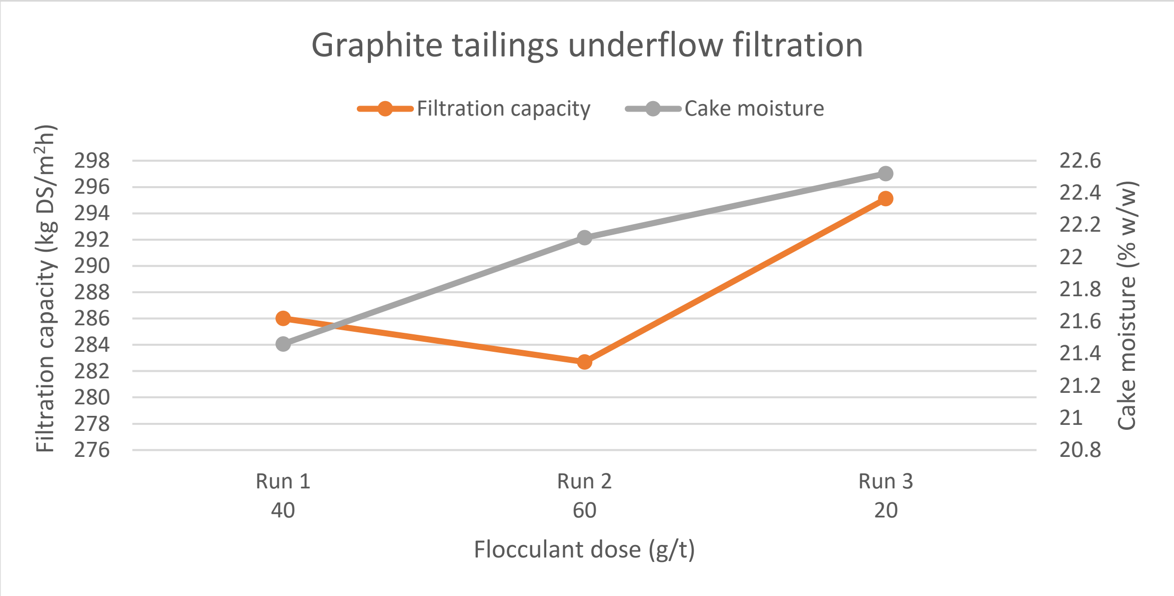



**Figure 39** Filtration capacity and cake moisture vs. flocculant dose for graphite tailings underflow filtration

The filtration capacities with relative cake moistures at different flocculant doses are presented in Figure 39. All filtration tests for graphite tailings underflows were carried out with constant cycle time of 8.5 minutes, pressing pressure of 16 bar and air-drying pressure of 5 bar.

In first filtration test, underflow generated with optimal dose of 40 g/t of flocculant M155 was filtered. The filtration capacity reached 286 kg DS/$m^2$h with residual cake moisture of 21.46 % w/w. Filtrate contained 777 mg/L of suspended solids.

The underflow produced with 60 g/t of the flocculant M155 was the subject of the second filtration test. Comparing the second filtration test to the first, the filtration capacity dropped, and cake moisture increased. The increase in cake thickness may have contributed to the increase in residual cake moisture. Filtrate also became cloudier. The filtrate was cloudy for the first 15 seconds before clearing. The sediments from the first test that were caught between the pores may be the reason of the increased amount of suspended solids in the filtrate.

The underflow produced by 20 g/t of flocculant M155 was filtered in the third filtration test. The filtration capacity increased significantly, so did the cake moisture. The rise in residual cake moisture may have been influenced by the thickness of the cake. Filtrate was slightly better than the second filtration test.

### *6.2.2. Composite tailings Labox 25 filtration*

Slurry was decanted to 67 % solids w/w after addition of flocculant M155 to simulate the average underflow density from thickening tests. The slurry then was filtered to determine the optimal parameters and cycle time.

Pumping: Since labox 25 test kit was used to filter the underflows, 148 mL of underflow was poured into the chamber. During calculation, 1 minute of pumping was used in data sheet.

Pressing: The cake was pressed with 16 bar of pressing pressure. The optimal pressing time was determined to be 30 seconds.

Drying: The cake was air dried with 5 bar of air-drying pressure. The optimal drying time was determined to be 2 minutes.

### Composite tailings underflow filtration Run 1

The underflow that was filtered, which was produced by adding 20 g/t of M155, had a solids content of 67.9 % w/w. The underflow was pressed with pressing pressure of 16 bar for 30 seconds. Filtrate started to get clear after first 10-15 seconds of pressing. The duration of air drying was 2 minutes and was found to be effective.  The cake was dried with 5 bar of drying air. The resulting capacity from the first test was 526.3 kg DS/m$^2$h. A 43.5 mm thick cake was formed with residual cake moisture of 13.97 % w/w. Small quantities of cake stuck to the filter cloth, but the cake easily released from it. The cloth could be rinsed easily under tap water. The cake, filtrate and filter cloth from composite tailings underflow filtration run 1 is shown in Figure 40.

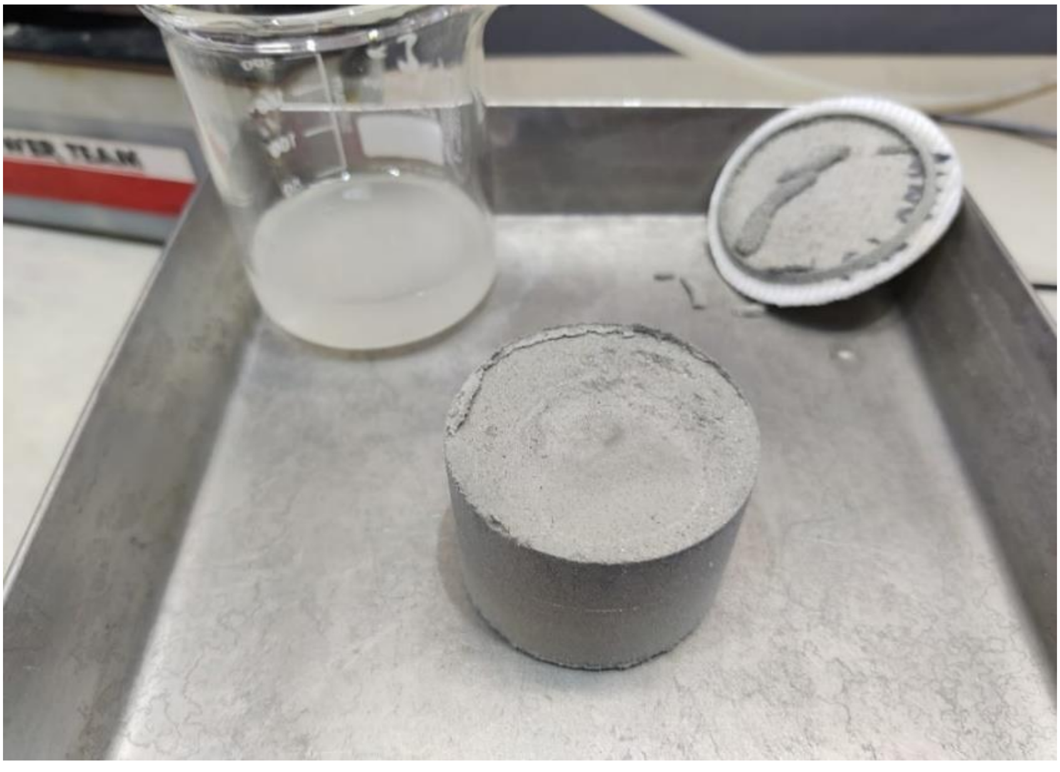


**Figure 40** Composite tailings filtration Run 1; 20 g/t of flocculant M155

**Composite tailings underflow filtration Run 2**

The second filtration test was conducted using the same process times as the initial test. The underflow that was filtered was created by adding 30 g/t of M155 and contained 63.9 % w/w solids. After the first 10-15 seconds of pressing, the filtrate began to become clear, just like in the first filtration test. The second test produced a 473.1 kg DS/$m^2$h. A 41 mm thick cake with a moisture content of 13.93 % w/w was produced. The cake released from the filter cloth easily, much like in the previous filtering test, with some cake from the bottom edges sticking to the cloth. Cloth was rinsed under tap water. The cake and filtrate produced from the second composite tailings underflow filtration can be seen in Figure 41.


**Figure 41** Composite tailings filtration Run 2; 30 g/t of flocculant M155

### Composite tailings underflow filtration Run 3

The third composite underflow filtration test used the same process times as the first and second test runs. The underflow that was filtered had 68.5 % w/w solids and was produced by adding 10 g/t of M155. Similar to earlier filtration tests, the filtrate began to become transparent after the first 10-15 seconds of pressing. The resulting filtration capacity from the second filtration test was 523.8 kg DS/$m^2$h. A 46 mm thick cake with a 15.04 % w/w cake moisture content was produced. Cake released smoothly from the filter cloth as can be seen in Figure 42.

**Figure 42** Composite tailings filtration Run 3; 10 g/t of flocculant M155

The cake moistures, filtration capacities and suspended solids in filtrate for composite tailings underflow filtration tests are tabulated in Table 6.

**Table 6** Labox 25 filtration test results for composite tailings

| Run | Flocculant dose (g/t) | Total cycle time (min) | Cake Moisture (% w/w) | Filtration Capacity (kg DS/m²h) | Solids in filtrate (mg/L) |
|---|---|---|---|---|---|
| 1 | 20 | 7.5 | 13.97 | 526.3 | 1072 |
| 2 | 30 | 7.5 | 13.93 | 473.1 | 1141 |
| 3 | 10 | 7.5 | 15.04 | 523.8 | 908 |

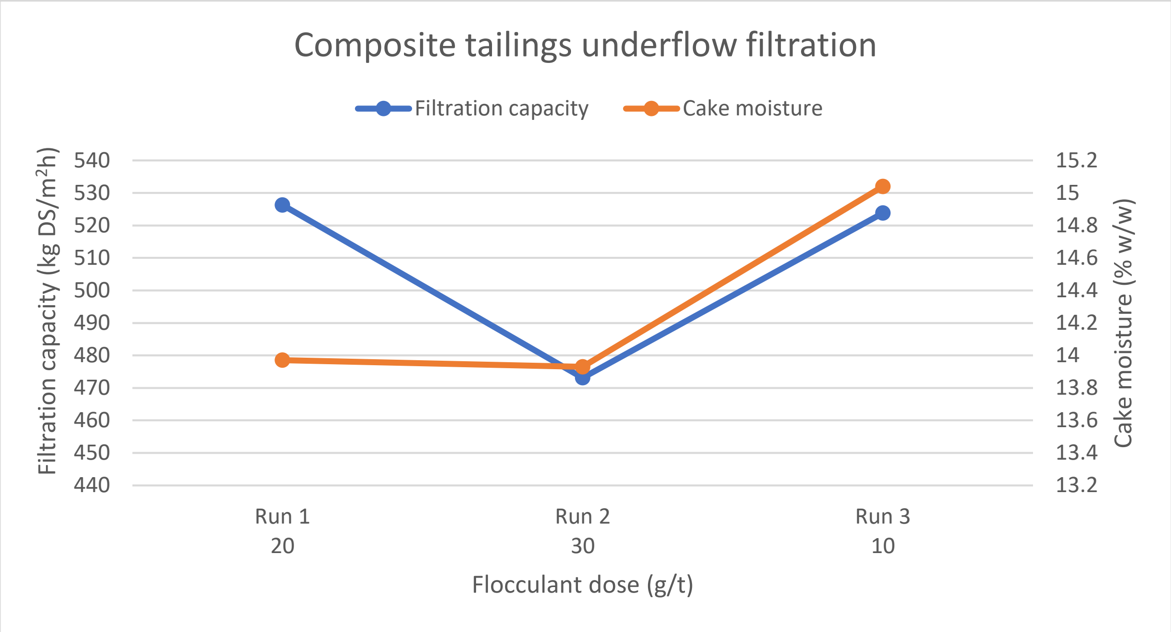



**Figure 43** Filtration capacity and cake moisture vs. flocculant dose for composite tailings underflow filtration

Figure 43 shows the filtration capacities with respect to cake moisture at various flocculant doses for composite tailings underflow. All filtration tests for composite tailings underflows were carried out with constant cycle time of 7.5 minutes, pressing pressure of 16 bar and air-drying pressure of 5 bar.

In first filtration test, underflow generated with optimal dose of 20 g/t of flocculant M155 was filtered. The filtration capacity reached 526.3 kg DS/$m^2$h with residual cake moisture of 13.97 % w/w. Filtrate contained 1072 mg/L of suspended solids.

The underflow produced by 30 g/t of the flocculant M155 was filtered in the second filtration test. The filtration capacity decreased significantly and the residual cake moisture in cake decreased slightly compared to the first composite underflow filtration test. In addition, the filtrate clarity declined from the prior filtration test. The sediments from the first filtration test that were caught between the pores may be the reason of the increased amount of suspended solids in the filtrate.

The underflow produced by 10 g/t of the flocculant M155 was subjected to the third filtration test. The filtration capacity increased significantly than the second filtration test along with the residual cake moisture. The increased residual moisture in the cake may be caused by the thickness of the cake that was formed. Filtrate was still cloudy, but with lowered number of suspended solids.

Graphite tailings with characteristics as mentioned in Table 1 provided better results when 20 g/t of M155 was added in the thickening run 3. It provided the densest underflow with least suspended solids in the overflow. Underflow density decreased and suspended solids in overflow increased when excess flocculant than the optimal flocculant dose was added to the feed. The results from graphite tailings thickening shows that, application of excess amount of flocculant than the optimal dose does not result in denser underflow and clearer overflow. The yield stress measurements for all graphite tailings underflows were less than 100 Pa, so, pumping those underflows should not be an issue. Increased slurry yield stress requires significantly more torque to aid slurry transportation and bigger motor to pump. The underflow generated from addition of 20 g/t of M155 resulted in maximum filtration capacity when filtered. However, the cake produced had highest residual moisture in it which may have been contributed by the thickness of the cake. Significant amounts of suspended solids were present in the filtrates. Compared to composite tailings, graphite tailings had finer solid particles. The presence of finer solid particles in the slurry may be the reason of the excess suspended solids in the filtrate.

Composite tailings with characteristics as mentioned in Table 1 provided densest underflow when 10 g/t of M155 was added in the thickening run 3. However, it also resulted in highest number of suspended solids in the overflow. The clearest overflow was produced with 30 g/t of flocculant M155, which is higher dose of flocculant than the optimal dose. For composite tailings, higher flocculant dose than the optimal dose provided clearer overflow but not the densest underflow. Yield stress measurements were less than 100 Pa for all thickening runs for composite tailings too. Maximum filtration capacity was achieved when the underflow with optimal flocculant dose of 20 g/t was filtered. The cake formed after filtering the densest underflow from composite tailings, obtained from flocculant dose less than that of optimal dose, had the largest residual moisture but the lowest amount of suspended particles in the filtrate. The thickness of the cake may have contributed to the increased residual moisture in the cake.

Dewatering tailings is a particularly efficient way to manage tailings because it yields a clear liquid as overflow and/or filtrate and a denser solid fraction in the form of thick underflow and/or cakes with less residual moisture. Dewatering reduces the volume of tailings, which lowers the need for pumping and associated expenses, reducing operating costs. Additionally, a smaller tailings storage facility can accommodate a reduced volume of tailings. Small TSF can be constructed quickly on small area and with low dam walls, which reduces capital expenses. Small TSF has small footprint. Dewatering tailings reduces the freshwater demand since the overflow and filtrate can be recycled back into the process, reducing the amount of water that needs to be discharged to the tailings storage facility (Fabian *et al.*, 2015). Tailings dewatering lowers the cost of following treatment operations, particularly the drying operations. The reduced moisture content in the underflow and cakes requires less fuel to dry, thus saving significant amount of drying costs.

Thickeners can dewater higher volumes of slurry, recovering higher volumes of water. They produce dense underflow which minimizes the tailings storage facility footprints. Reduced water content in the TSF reduces the potential for seepage and dam failures. These dense underflows can be used to backfill underground mine-voids.

Flocculants are added to the feed slurry during thickening to enhance the solids settling. It increases the effectiveness of solid-liquid separation while also assisting in reducing the overall cost of dewatering. Although flocculant enhances dewatering, overusing flocculant does not always result in improved thickener performance. It is very important to choose the right flocculant type and dose (Alam *et al.*, 2011). The thickening of graphite and composite tailings from this study shows that the underflow density and overflow clarity are not always increased by adding too much flocculant to the feed during thickening.

Tailings can be dewatered more efficiently by filters. It produces cake with less moisture content and can be transported with trucks or conveyor belt to be dry stacked. Additionally, groundwater contamination from seepage is also eliminated. The type of flocculant and its doses affects the filtration performance characteristics such as permeability, specific cake resistance and specific cake surface area. Filtration of tailings is significantly improved when thickeners are used prior to the filtration process. The dense underflow produced from the thickeners when used as filter feed results in higher filtration capacity (Cheremisinoff, 1998).

# 7 CONCLUSIONS AND FUTURE LINES OF RESEARCH

The primary objective of this thesis was to dewater graphite and composite tailings using a thickener and a filter. The effects of the flocculant dose on the underflow densities, overflow clarities, filtration capacities, and cake moistures during the thickening and filtration of tailings were also studied. While thickening of the tailings sample, the goal was to obtain dense underflow and clear overflow. The best performing flocculant type and optimum dose was determined by flocculant screening tests. Each thickener underflow was filtered using pressure filter to achieve maximum filtration capacity with least percentage of residual moisture in the cake.

Graphite tailings could be dewatered easily using a 99mm dynamic HRT. From the flocculant screening tests, M155 at 40 g/t provided fastest settling of solids with clear overflow. The underflow produced by the thickening graphite tailings had an average solid content of 49 % w/w, removing around 51% w/w of water that could be recycled back into the plant. The underflow solids content did not differ considerably for the thickening tests, with a variance of less than 1 %. However, there was a large disparity in overflow clarities. When 20 g/t of M155 of flocculant dose was added, the underflow produced was the densest and the overflow had the least suspended solids. Upon filtering the same underflow, the filtration capacity was at its maximum but with higher residual cake moisture. When 60 g/t of M155 of flocculant dose was added, the underflow produced was the least dense and the overflow had most suspended solids. Although the cake moisture did not significantly reduce after filtering that underflow, the filtration capacity did.

Composite tailings could be easily dewatered with a 99mm dynamic HRT, just like graphite tailings. From the flocculant screening tests, fastest solids settling was achieved with clear overflow using M155 at 20 g/t. With the optimal flocculant dose, an underflow of roughly 68% solids w/w was produced, which upon filtering yielded the maximum filtration capacity. All thickening runs produced underflows that contained more than 60% solids by weight. When 10 g/t of M155 of flocculant dose was added, the underflow produced was the densest however the overflow had the most suspended solids. Upon filtering the same underflow, cake with maximum residual moisture was formed. Underflow produced with a flocculant dose that was higher than the optimal flocculant dose resulted in the least dense underflow and filtration capacity. In comparison to the optimal dose, higher flocculant dose resulted in the clear overflow.

Flocculation enhances sedimentation process. Without flocculant addition, the overflow will retain more solids and the underflow will have less solids. On the other hand, excessive flocculant use during dewatering may not necessarily produce better outcomes. Proper screening and optimization of flocculant increases dewatering efficiency and thus helps save flocculant expenditures.

In conclusion, dewatering tailings reduces the volume of tailings. It also, allows the recycling of the clear liquid back into the process in the form of overflow and filtrate, reducing the need for fresh water. Pumping and associated expenses are reduced when the volume of tailings is lowered. Conveyor belts or trucks can be used to move dry cakes that cannot be pumped. Additionally, it requires less area for disposal, which reduces the capital and operational costs of a tailings storage facility. Effective dewatering of tailings significantly reduces the risks related pollution, biodiversity loss, and contamination of surface water, groundwater, and soil. Risks relating the loss of life and property caused by tailings dam failure is also considerably reduced.

For further research, more flocculant screening tests could be done with additional flocculant types at narrower dose gaps; this might provide more accurate results. Another research area could be the study of coagulation together with flocculation to understand the overflow clarity. Dewatering with bigger test units will provide more accurate results. Dewatering performances can be greatly affected by particle shapes, so particle shape and its effect could be studied in future. For better filtrate clarity, tighter filter cloth could be utilized.

# BIBLIOGRAPHY


Ahmad, A., Hashlamon, A. and Hong, L. (2015) 'Pre-treatment methods for seawater desalination and industrial wastewater treatment: A brief review -', *International Journal of Scientific Research in Science, Engineering and Technology*, 1(April), pp. 422–428.

Alam, N. *et al.* (2011) 'Dewatering of coal plant tailings: Flocculation followed by filtration', *Fuel*, 90(1), pp. 26–35. Available at: https://doi.org/10.1016/j.fuel.2010.08.006.

Bae, Y.-H. *et al.* (2007) 'Potable Water Treatment By Polyacrylamide Base Flocculants, Coupled With an Inorganic Coagulant', *Environmental Engineering Research*, pp. 21–29. Available at: https://doi.org/10.4491/eer.2007.12.1.021.

Balasubramanian, P.A. (2017) 'By University of Mysore , Mysore', (February), pp. 1–8. Available at: https://doi.org/10.13140/RG.2.2.15761.63845.

Bergh, L., Ojeda, P. and Torres, L. (2015) 'Expert control tuning of an industrial thickener', *IFAC-PapersOnLine*, 28(17), pp. 86–91. Available at: https://doi.org/10.1016/j.ifacol.2015.10.083.

Betancourt, F. *et al.* (2014) 'Modeling and controlling clarifier-thickeners fed by suspensions with time-dependent properties', *Minerals Engineering*, 62, pp. 91–101. Available at: https://doi.org/10.1016/j.mineng.2013.12.011.

Boger, D. V. (2009) 'Rheology and the resource industries', *Chemical Engineering Science*, 64(22), pp. 4525–4536. Available at: https://doi.org/10.1016/j.ces.2009.03.007.

Bratby, J. (2006) *Coagulation and flocculation in water and wastewater treatment*, *Water 21*. Available at: https://doi.org/10.2166/9781780407500.

Buratto, B. *et al.* (2014) 'Wall effects during settling in cylinders', *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 449(1), pp. 157–169. Available at: https://doi.org/10.1016/j.colsurfa.2014.02.045.

Cacciuttolo Vargas, C. and Marinovic Pulido, A. (2022) 'Sustainable Management of

Thickened Tailings in Chile and Peru: A Review of Practical Experience and Socio-Environmental Acceptance', *Sustainability (Switzerland)*, 14(17). Available at: https://doi.org/10.3390/su141710901.

Chatsungnoen, T. and Chisti, Y. (2019) *Flocculation and electroflocculation for algal biomass recovery*. Second Edi, *Biofuels from Algae*. Second Edi. Elsevier B.V. Available at: https://doi.org/10.1016/b978-0-444-64192-2.00011-1.

Cheremisinoff, N.P. (1998) 'Environmental Policy and Technology Project United States Agency for International Development'. Available at: http://www.bh.com.

Chukwudi, B., Eng, M. and Uche, R. (2008) 'Flocculation of Kaolinite Clay using Natural Polymer.', *The Pacific Journal of Science and Technology*, 9(2), pp. 495–501. Available at: http://www.akamaiuniversity.us/PJST9_2_495.pdf.

Costine, A. *et al.* (2018) 'Variations in the molecular weight response of anionic polyacrylamides under different flocculation conditions', *Chemical Engineering Science*, 176, pp. 127–138. Available at: https://doi.org/10.1016/j.ces.2017.10.031.

Crust, A.A.H. (2017) 'Optimisation of Thickener Performance: Incorporation of Shear Effects', p. 288.

Cytec (2010) 'Mining Chemicals', pp. 1–434.

Davies, M. (2011) 'Filtered dry stacked tailings: the fundamentals', *Proceedings Tailings and Mine Waste 2011, Vancouver B.C.*, p. 9 p.

Dorf, R.C. (2004) 'The engineering handbook, second edition', *The Engineering Handbook, Second Edition*, pp. 1–3047.

Du, J., McLoughlin, R. and Smart, R.S.C. (2014) 'Improving thickener bed density by ultrasonic treatment', *International Journal of Mineral Processing*, 133, pp. 91–96. Available at: https://doi.org/10.1016/j.minpro.2014.10.003.

Dunne, R.C., Kawatra, S.K. and Young, C.A. (2019) *SME Mineral Processing &Extractive Metallurgy Handbook*. Society for Mining, Metallurgy & Exploration.

Edraki, M. *et al.* (2014) ‘Designing mine tailings for better environmental, social and economic outcomes: A review of alternative approaches’, *Journal of Cleaner Production*, 84(1), pp. 411–420. Available at: https://doi.org/10.1016/j.jclepro.2014.04.079.

Escobar, H. (2015) ‘Mud tsunami wreaks ecological havoc in Brazil’, *Science*, 350(6265), pp. 1138–1139. Available at: https://doi.org/10.1126/science.350.6265.1138.

Fabian, K. *et al.* (2015) ‘The impacts of using thickened tailings on water management and CAPEX of tailings storage facilities’, *Proceedings of the 18th International Seminar on Paste and Thickened Tailings*, pp. 535–546. Available at: https://doi.org/10.36487/acg_rep/1504_41_fabian.

Farrow, J.B. and Swift, J.D. (1996) ‘A new procedure for assessing the performance of flocculants’, *International Journal of Mineral Processing*, 46(3–4), pp. 263–275. Available at: https://doi.org/10.1016/0301-7516(95)00084-4.

Franks, D.M. *et al.* (2011) ‘Sustainable development principles for the disposal of mining and mineral processing wastes’, *Resources Policy*, 36(2), pp. 114–122. Available at: https://doi.org/10.1016/j.resourpol.2010.12.001.

Garmsiri, M.R. and Unesi, M. (2018) ‘Challenges and opportunities of hydrocyclone-thickener dewatering circuit: A pilot scale study’, *Minerals Engineering*, 122(November 2017), pp. 206–210. Available at: https://doi.org/10.1016/j.mineng.2018.04.001.

Gray, W.G. and Miller, C.T. (2004) ‘Examination of Darcy’s law for flow in porous media with variable porosity’, *Environmental Science and Technology*, 38(22), pp. 5895–5901. Available at: https://doi.org/10.1021/es049728w.

Haldar, S.K. (2018) *Mineral Processing*, *Mineral Exploration*. Available at: https://doi.org/10.1016/b978-0-12-814022-2.00013-7.

Harraz, H.Z. (2016) ‘Topic 7 : Mining Methods Part III : Surface mining- Placer Mining Hassan Z . Harraz’, (February 2010). Available at: https://doi.org/10.13140/RG.2.1.4027.9924.

Hogg, R. (1999) ‘The role of polymer adsorption kinetics in flocculation’, *Colloids and*

*Surfaces A: Physicochemical and Engineering Aspects*, 146(1–3), pp. 253–263. Available at: https://doi.org/10.1016/S0927-7757(98)00723-7.

IIED, I.I. for E.D. (2002) ‘Mining for the Future Appendix A: Large Volume Waste Working Paper’, *Mining, Minerals and Sustainable Development*, (31), pp. 1–55.

Liu, D. *et al.* (2020) ‘Improved water recovery: A review of clay-rich tailings and saline water interactions’, *Powder Technology*, 364, pp. 604–621. Available at: https://doi.org/10.1016/j.powtec.2020.01.039.

McCabe, W.L., Smith, J.C. and Harriot, P. (1993) ‘Unit operations of chemical engineering.’, *Chemical Engineering Science*, p. 1154. Available at: https://doi.org/10.1016/0009-2509(57)85034-9.

Metso (2015) ‘Basics in minerals processing’, p. 354.

Metso Outotec, M.O. (2022) ‘Thickening technologies’, *Africa*. Available at: https://www.mogroup.com/portfolio/?q=&page=1.

Mullin, J.W., Lowrison, G.C. and Svarovsky, L. (1977) *Solid-Liquid Separation*. London: The Butterworth Group.

Napier-Munn, T. and Wills, B.A. (2005) *Preface to 7th Edition*. 7th edn, *Wills’ Mineral Processing Technology*. 7th edn. Available at: https://doi.org/10.1016/b978-075064450-1/50000-x.

Outotec (2011) *Outotec ® Thickening technologies Outotec ® Thickeners and Clarifiers*, *Outotec Oyj*. Available at: https://d3pcsg2wjq9izr.cloudfront.net/files/40011/download/332960/low_res_Outotec_thickening_18022011.pdf.

Probst, A. (2001) *for Thickener Control*. Mcgill university, Canada.

Ramani, R. V. (2012) ‘Surface mining technology: Progress and prospects’, *Procedia Engineering*, 46, pp. 9–21. Available at: https://doi.org/10.1016/j.proeng.2012.09.440.

Ripperger, S., Gösele, W. and Alt, C. (2009) ‘Filtration, 1. Fundamentals’, *Ullmann’s*

*Encyclopedia of Industrial Chemistry*, pp. 1–38. Available at: https://doi.org/10.1002/14356007.b02_10.pub2.

Ritcey, G.M. (2005) ‘Tailings management in gold plants’, *Hydrometallurgy*, 78(1-2 SPEC. ISS.), pp. 3–20. Available at: https://doi.org/10.1016/j.hydromet.2005.01.001.

Rudman, M. *et al.* (2008) ‘Raking in gravity thickeners’, *International Journal of Mineral Processing*, 86(1–4), pp. 114–130. Available at: https://doi.org/10.1016/j.minpro.2007.12.002.

Schoenbrunn, F. *et al.* (1983) ‘Solid-Liquid Separation.’, *Engineering and Mining Journal*, 184(7), pp. 1067–1112.

Svarovsky, L. (2001) *Solid-Liquid Separation*. 4th edn. Butterworth-Heinemann.

Tan, C.K., Bao, J. and Bickert, G. (2017) ‘A study on model predictive control in paste thickeners with rake torque constraint’, *Minerals Engineering*, 105, pp. 52–62. Available at: https://doi.org/10.1016/j.mineng.2017.01.011.

Tarleton, S. and Wakeman, R. (2008) *Solid Liquid Separation Equipment Selection and Process Design*. Oxford: Butterworth-Heinemann.

Tiller, F.M., Alciatore, A. and Shirato, M. (1987) *Filtration in the Chemical Process Industry. In Filtration: Principles and Practices*. 2nd edn. Edited by M.M. J. and O. C. New York.

Tiller, F.M. and Green, T.C. (1973) ‘Role of porosity in filtration IX skin effect with highly compressible materials’, *AIChE Journal*, 19(6), pp. 1266–1269. Available at: https://doi.org/10.1002/aic.690190633.

Usher, S.P., Hons, B.E.C. and Sc, B. (2002) ‘SUSPENSION DEWATERING : by’, (May).

WISE (1998) *Chronology of major tailings dam failures, 2022*. Available at: http://www.wise-uranium.org/mdaf.html.

Yang, Y. *et al.* (2019) ‘Effect of primary flocculant type on a two-step flocculation process on iron ore fine tailings under alkaline environment', *Minerals Engineering*, 132(November 2018), pp. 14–21. Available at: https://doi.org/10.1016/j.mineng.2018.11.053.

APPENDIX

PSDs’

Particle size distribution for graphite tailings sample

# MO PSD Analysis

**Metso:Outotec**

**Measurement Details**

**Sample Name** Graphite Tailings
**Operator** SanBis

**Measurement Details**

**Analysis Date Time** 9.2.2022 10.37.55
**Result Source** Averaged
**Instrument Type** Mastersizer3000
**Accessory Name** Hydro EV
**Software Version** 3.72.1810.240

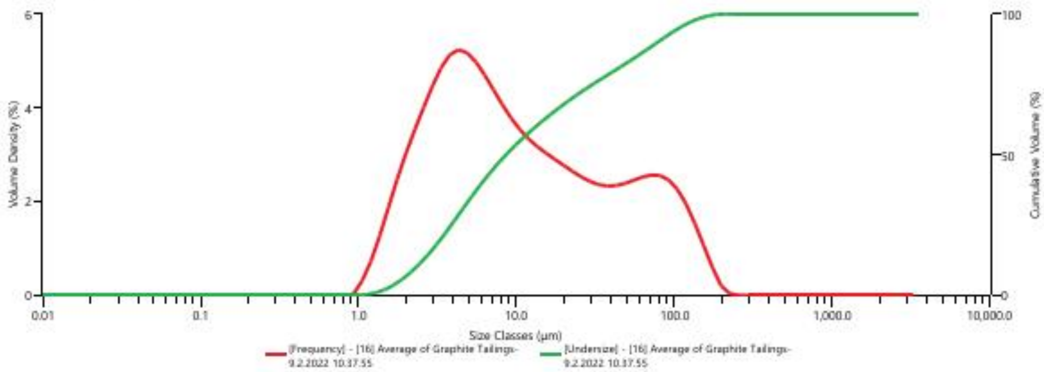
**Analysis**

**Scattering Model** Fraunhofer
**Analysis Model** General Purpose
**Weighted Residual** 0.88 %
**Residual** 0.80 %
**Laser Power** 73.40 %
**Laser Obscuration** 17.47 %
**Ultrasound Achieved** 0 %
**Stirrer Speed Achieved** 2400 rpm

**Result**

**Concentration** 0.0116 %
**Span** 8.855
**Uniformity** 2.453
**Specific Surface Area** 999.0 $m^2$/kg
**D [3;2]** 5.72 μm
**D [4;3]** 25.3 μm
**Dv (10)** 2.32 μm
**Dv (20)** 3.32 μm
**Dv (50)** 8.61 μm
**Dv (80)** 42.2 μm
**Dv (90)** 78.5 μm
**Volume Below (10) μm** 53.69 %

**Frequency (compatible) and Undersize**




**Result**

| Size (μm) | % Volume In | Size (μm) | % Volume In | Size (μm) | % Volume In | Size (μm) | % Volume In | Size (μm) | % Volume In | Size (μm) | % Volume In | Size (μm) | % Volume In | Size (μm) | % Volume In |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.0100 | 0.00 | 0.0463 | 0.00 | 0.214 | 0.00 | 0.991 | 0.25 | 4.58 | 4.32 | 21.2 | 2.21 | 98.1 | 1.89 | 454 | 0.00 | 2100 | 0.0 |
| 0.0114 | 0.00 | 0.0526 | 0.00 | 0.243 | 0.00 | 1.13 | 0.60 | 5.21 | 4.15 | 24.1 | 2.11 | 111 | 1.63 | 516 | 0.00 | 2390 | 0.0 |
| 0.0129 | 0.00 | 0.0597 | 0.00 | 0.276 | 0.00 | 1.28 | 1.05 | 5.92 | 3.92 | 27.4 | 2.02 | 127 | 1.28 | 586 | 0.00 | 2710 | 0.0 |
| 0.0147 | 0.00 | 0.0678 | 0.00 | 0.314 | 0.00 | 1.45 | 1.57 | 6.72 | 3.65 | 31.1 | 1.97 | 144 | 0.89 | 666 | 0.00 | 3080 | 0.0 |
| 0.0167 | 0.00 | 0.0771 | 0.00 | 0.357 | 0.00 | 1.65 | 2.07 | 7.64 | 3.39 | 35.3 | 1.94 | 163 | 0.49 | 756 | 0.00 | 3500 | |
| 0.0189 | 0.00 | 0.0876 | 0.00 | 0.405 | 0.00 | 1.88 | 2.53 | 8.68 | 3.15 | 40.1 | 1.95 | 186 | 0.15 | 859 | 0.00 | | |
| 0.0215 | 0.00 | 0.0995 | 0.00 | 0.460 | 0.00 | 2.13 | 2.94 | 9.86 | 2.95 | 45.6 | 1.99 | 211 | 0.02 | 976 | 0.00 | | |
| 0.0244 | 0.00 | 0.113 | 0.00 | 0.523 | 0.00 | 2.42 | 3.34 | 11.2 | 2.78 | 51.8 | 2.04 | 240 | 0.00 | 1110 | 0.00 | | |
| 0.0278 | 0.00 | 0.128 | 0.00 | 0.594 | 0.00 | 2.75 | 3.72 | 12.7 | 2.64 | 58.9 | 2.10 | 272 | 0.00 | 1260 | 0.00 | | |
| 0.0315 | 0.00 | 0.146 | 0.00 | 0.675 | 0.00 | 3.12 | 4.05 | 14.5 | 2.53 | 66.9 | 2.14 | 310 | 0.00 | 1430 | 0.00 | | |
| 0.0358 | 0.00 | 0.166 | 0.00 | 0.767 | 0.00 | 3.55 | 4.29 | 16.4 | 2.42 | 76.0 | 2.14 | 352 | 0.00 | 1630 | 0.00 | | |
| 0.0407 | 0.00 | 0.188 | 0.00 | 0.872 | 0.00 | 4.03 | 4.38 | 18.7 | 2.31 | 86.4 | 2.06 | 400 | 0.00 | 1850 | 0.00 | | |

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Mastersizer - v3.72
Page 1 of 1

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Printed: 9.2.2022 10.55

Particle size distribution for composite tailings sample

# MO PSD Analysis

Metso:Outotec

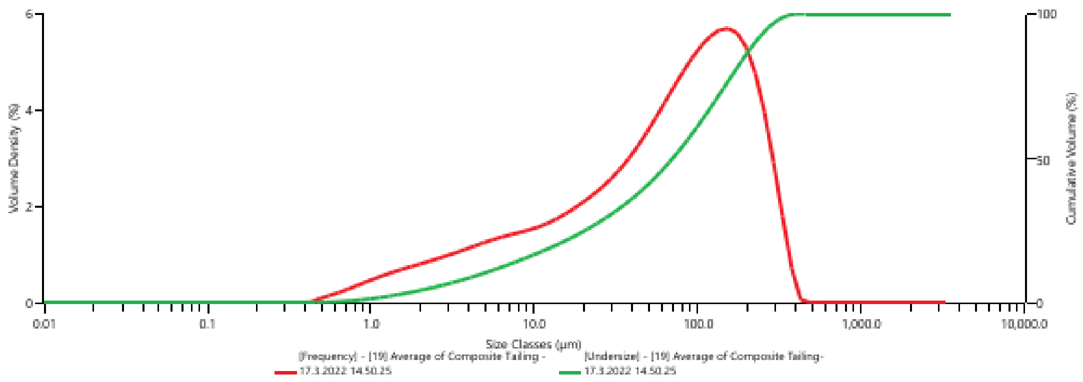
| Measurement Details | |
|---|---|
| **Sample Name** | Composite Tailings |
| **Operator** | SanBis |

| Measurement Details | |
|---|---|
| **Analysis Date Time** | 17.3.2022 14.50.25 |
| **Result Source** | Averaged |
| **Instrument Type** | Mastersizer3000 |
| **Accessory Name** | Hydro EV |
| **Software Version** | 3.72.1810.240 |

| Analysis | |
|---|---|
| **Scattering Model** | Fraunhofer |
| **Analysis Model** | General Purpose |
| **Weighted Residual** | 1.11 % |
| **Residual** | 1.11 % |
| **Laser Power** | 75.30 % |
| **Laser Obscuration** | 18.59 % |
| **Ultrasound Achieved** | 0 % |
| **Stirrer Speed Achieved** | 2830 rpm |

| Result | |
|---|---|
| **Concentration** | 0.0334 % |
| **Span** | 3.112 |
| **Uniformity** | 0.971 |
| **Specific Surface Area** | 467.9 $m^2/kg$ |
| **D [3;2]** | 12.2 μm |
| **D [4;3]** | 94.0 μm |
| **Dv (10)** | 4.84 μm |
| **Dv (20)** | 13.7 μm |
| **Dv (50)** | 70.3 μm |
| **Dv (80)** | 168 μm |
| **Dv (90)** | 224 μm |
| **Volume Below (10) μm** | 16.61 % |

Frequency (compatible) and Undersize




Result

| Size (μm) | % Volume In | Size (μm) | % Volume In | Size (μm) | % Volume In |
|---|---|---|---|---|---|
| 0.500 | 1.22 | 38.0 | 3.49 | 212 | 9.00 |
| 1.00 | 2.91 | 45.0 | 3.79 | 300 | 2.66 |
| 2.00 | 7.63 | 53.0 | 4.47 | 400 | 0.00 |
| 6.00 | 4.80 | 63.0 | 2.96 | 500 | 0.00 |
| 10.0 | 8.07 | 70.0 | 2.02 | 850 | 0.00 |
| 20.0 | 3.23 | 75.0 | 11.32 | 1400 | |
| 25.0 | 4.05 | 106 | 12.65 | | |
| 32.0 | 3.20 | 150 | 12.47 | | |

Composite tailings(tailings+slime) After decantation

Malvern

Malvern Instruments Ltd.
www.malvern.com

Mastersizer - v3.72
Page 1 of 1

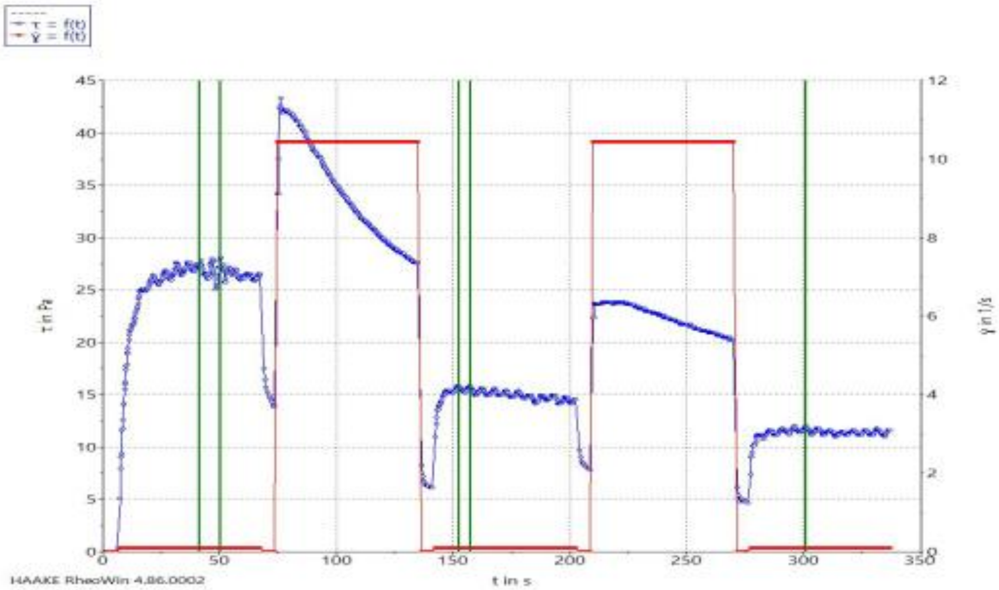
Created: 26.1.2022
Printed: 17.3.2022 15.02

YS measurement for graphite tailings HRT Run 1

**HAAKE RheoWin 4.86.00** Page 1

| | | | | | |
|---|---|---|---|---|---|
| **Company** | Outotec (Finland) Oy | **Measuring device** | VT550 | | |
| **Operator** | Outotec (Finland) Oy | **Temperature device** | | | |
| **Date/Time** | 17.02.2022 / 13.49.42 | **Measuring geometry** | | **Gap** | 1,000 mm |
| **Substance** | Graphite Tailings | **A-factor** | 2,110e+04 Pa/Nm | | |
| **Sample no** | Beaker | **M-factor** | 1,998 (1/s)/(rad/s) | | |
| **Description** | Run 1 40 gpt M155 | | | | |

**Comment**




**Filename:** ----

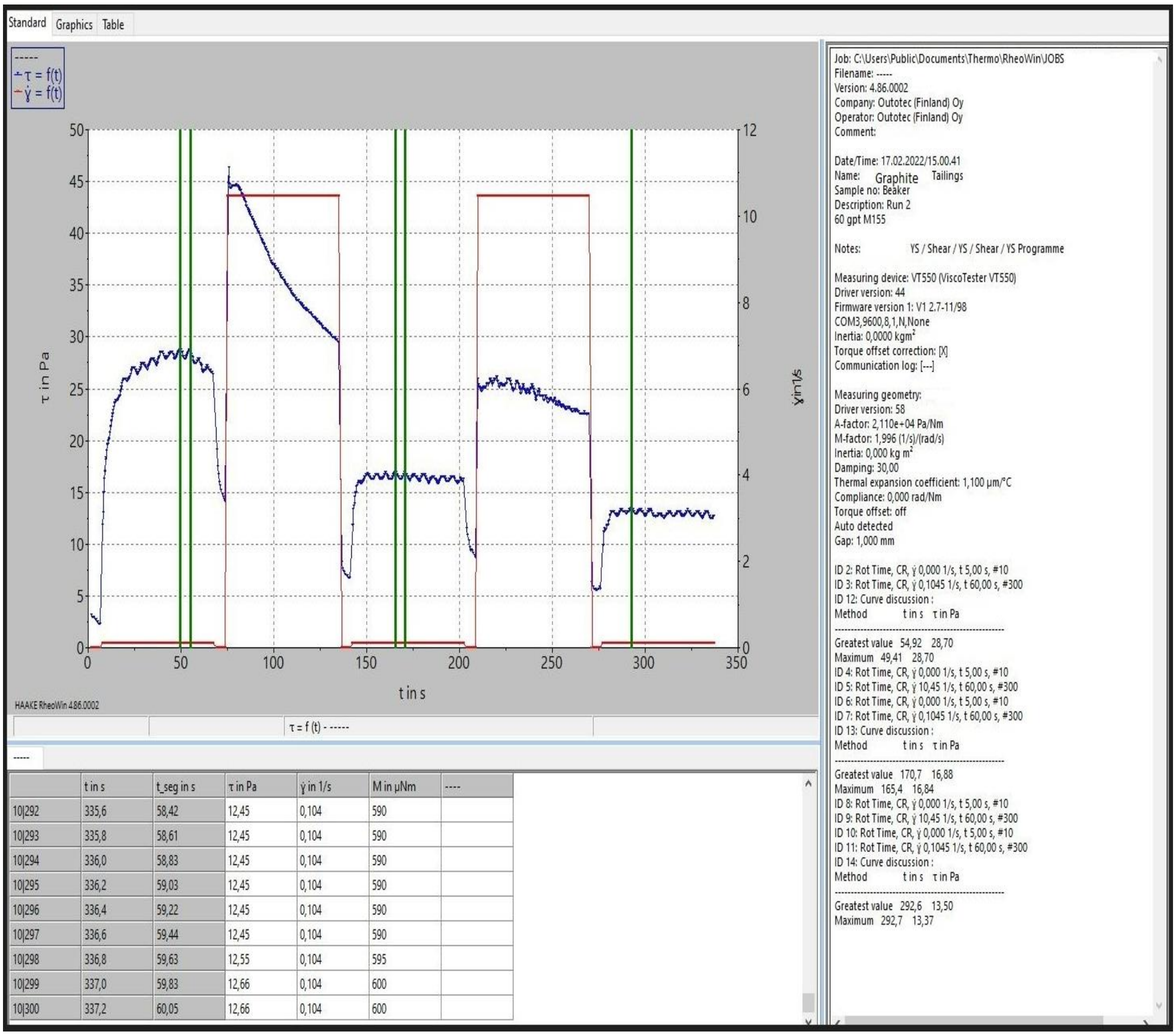
**Job:** **Element definition / Notes**

ID 2: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 3: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 4: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 5: Rot Time; CR; □□10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 6: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 7: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 8: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 9: Rot Time; CR; □□10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 10: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 11: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
Notes

**Evaluation**

ID 12-2: Curve discussion :
Method t in s τ in Pa
---------------------------------------------
Greatest value 50,56 28,13
Maximum 41,51 27,54
ID 13-2: Curve discussion :
Method t in s τ in Pa
---------------------------------------------
Greatest value 157,6 15,82
Maximum 152,4 15,77
ID 14-2: Curve discussion :
Method t in s τ in Pa
---------------------------------------------
Greatest value 300,9 12,03
Maximum 300,8 11,90

YS measurement for graphite tailings HRT Run 2

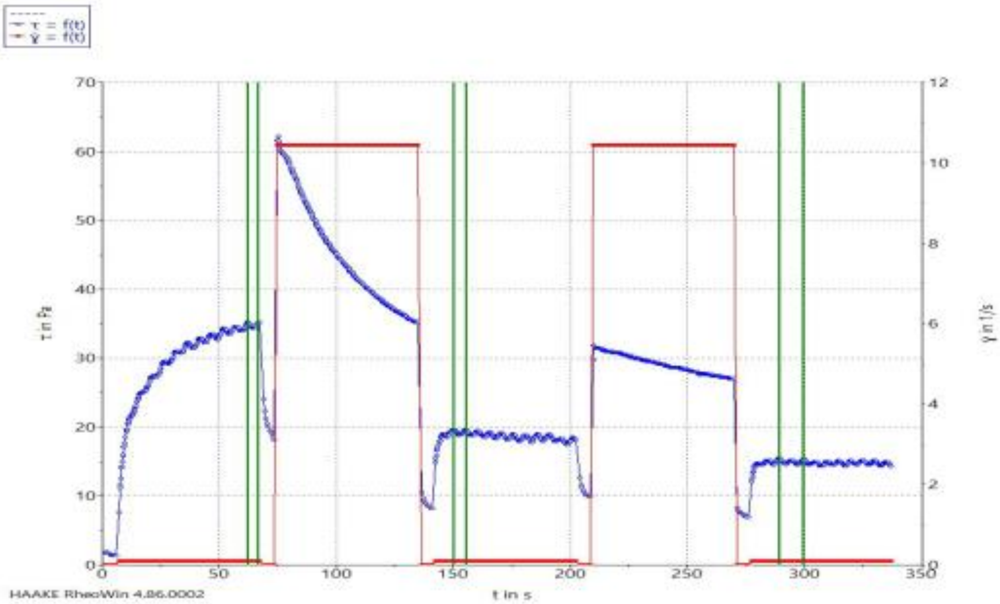


YS measurement for graphite tailings HRT Run 3

**HAAKE RheoWin 4.86.00** Page 1

| | | | | | |
|---|---|---|---|---|---|
| **Company** | Outotec (Finland) Oy | **Measuring device** | VT550 | | |
| **Operator** | Outotec (Finland) Oy | **Temperature device** | | | |
| **Date/Time** | 17.02.2022 / 16.25.50 | **Measuring geometry** | | **Gap** | 1,000 mm |
| **Substance** | GraphiteTailings | **A-factor** | 2,110e+04 Pa/Nm | | |
| **Sample no** | Beaker | **M-factor** | 1,996 (1/s)/(rad/s) | | |
| **Description** | Run 3 20 gpt M155 | | | | |

**Comment**




**Filename:** -----

**Job:** **Element definition / Notes**

ID 2: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 3: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 4: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 5: Rot Time; CR; □□10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 6: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 7: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 8: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 9: Rot Time; CR; □□10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 10: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 11: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
Notes

**Evaluation**

ID 12-2: Curve discussion :

| Method | t in s | τ in Pa |
|---|---|---|
| Greatest value | 66,99 | 35,24 |
| Maximum | 62,48 | 35,07 |

ID 13-2: Curve discussion :

| Method | t in s | τ in Pa |
|---|---|---|
| Greatest value | 155,6 | 19,62 |
| Maximum | 150,2 | 19,63 |

ID 14-2: Curve discussion :

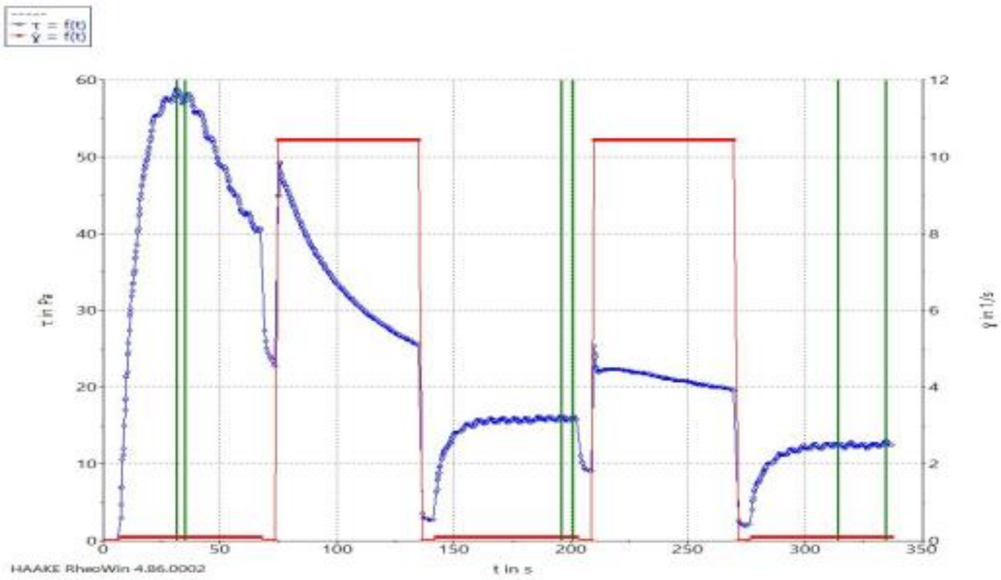
| Method | t in s | τ in Pa |
|---|---|---|
| Greatest value | 299,7 | 15,40 |
| Maximum | 289,4 | 15,36 |

YS measurement for composite tailings HRT Run 1

**HAAKE RheoWin 4.86.00** Page 1

| | | | | | |
|---|---|---|---|---|---|
| **Company** | Outotec (Finland) Oy | **Measuring device** | VT550 | | |
| **Operator** | Outotec (Finland) Oy | **Temperature device** | | | |
| **Date/Time** | 16.03.2022 / 10.31.36 | **Measuring geometry** | | **Gap** | 1,000 mm |
| **Substance** | Composite tailings | **A-factor** | 2,110e+04 Pa/Nm | | |
| **Sample no** | Beaker | **M-factor** | 1,996 (1/s)/(rad/s) | | |
| **Description** | Run 1 20 gpt M155 | | | | |

**Comment**




**Filename:** ------

**Job:** **Element definition / Notes**

ID 2: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 3: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 4: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 5: Rot Time; CR; □□10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 6: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 7: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 8: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 9: Rot Time; CR; □□10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 10: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 11: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
Notes

**Evaluation**

ID 12-2: Curve discussion :

| Method | t in s | τ in Pa |
|---|---|---|
| Greatest value | 31,77 | 58,87 |
| Maximum | 35,24 | 58,00 |

ID 13-2: Curve discussion :

| Method | t in s | τ in Pa |
|---|---|---|
| Greatest value | 201,0 | 16,25 |
| Maximum | 196,1 | 16,25 |

ID 14-2: Curve discussion :

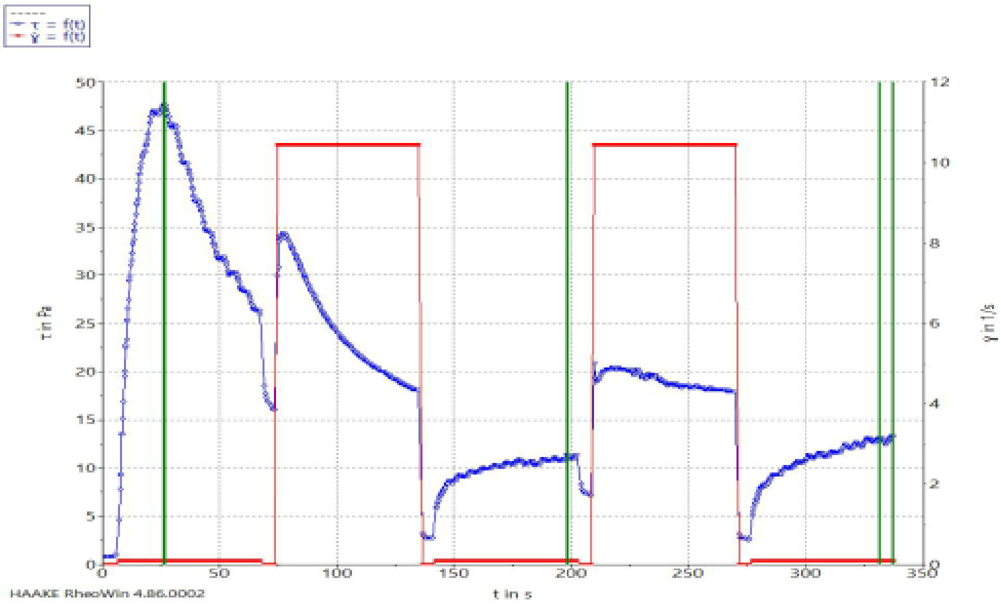
| Method | t in s | τ in Pa |
|---|---|---|
| Greatest value | 334,7 | 13,08 |
| Maximum | 314,2 | 12,69 |

YS measurement for composite tailings HRT Run 2

**HAAKE RheoWin 4.86.00** Page 1

| | | | |
|---|---|---|---|
| **Company** | Outotec (Finland) Oy | **Measuring device** | VT550 |
| **Operator** | Outotec (Finland) Oy | **Temperature device** | |
| **Date/Time** | 16.03.2022 / 13.20.20 | **Measuring geometry** | **Gap** 1,000 mm |
| **Substance** | Composite tailings | **A-factor** | 2,110e+04 Pa/Nm |
| **Sample no** | Beaker | **M-factor** | 1,996 (1/s)/(rad/s) |
| **Description** | Run 2 30 gpt M155 | | |

**Comment**




**Filename:** -----

**Job: Element definition / Notes**

ID 2: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 3: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 4: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 5: Rot Time; CR; □□10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 6: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 7: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 8: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 9: Rot Time; CR; □□10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 10: Rot Time; CR; □□0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 11: Rot Time; CR; □□0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
Notes

**Evaluation**

ID 12-2: Curve discussion :

| Method | t in s | τ in Pa |
|---|---|---|
| Greatest value | 26,92 | 47,69 |
| Maximum | 26,36 | 47,61 |

ID 13-2: Curve discussion :

| Method | t in s | τ in Pa |
|---|---|---|
| Greatest value | 198,7 | 11,39 |
| Maximum | 198,5 | 11,37 |

ID 14-2: Curve discussion :

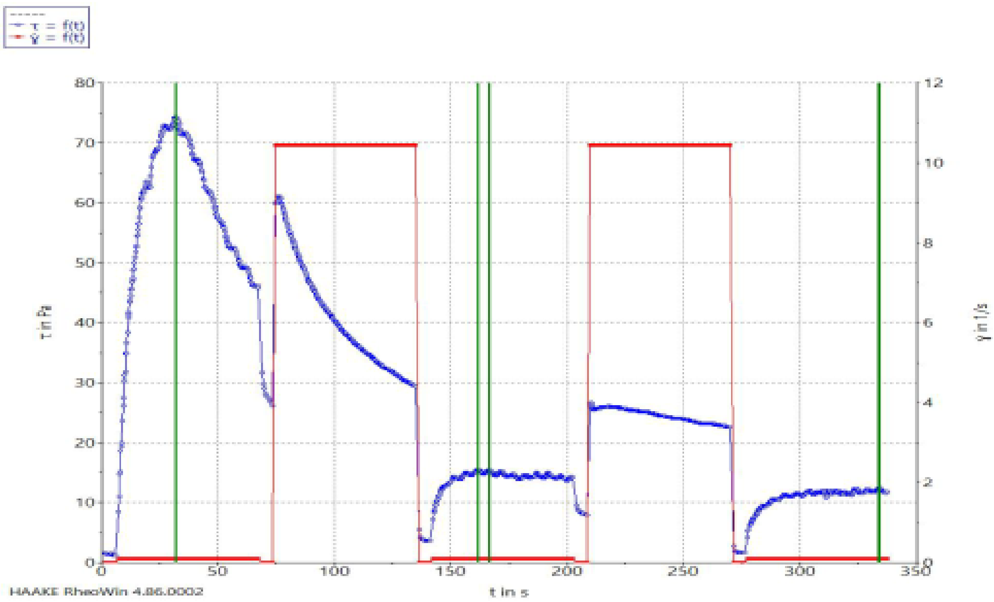
| Method | t in s | τ in Pa |
|---|---|---|
| Greatest value | 337,2 | 13,29 |
| Maximum | 331,7 | 13,11 |

YS measurement for composite tailings HRT Run 3

| **HAAKE RheoWin 4.86.00** | Page 1 |
|---|---|

| | | | | | |
|---|---|---|---|---|---|
| **Company** | Outotec (Finland) Oy | **Measuring device** | VT550 | | |
| **Operator** | Outotec (Finland) Oy | **Temperature device** | | | |
| **Date/Time** | 16.03.2022 / 14.35.21 | **Measuring geometry** | | **Gap** | 1,000 mm |
| **Substance** | Composite tailings | **A-factor** | 2,110e+04 Pa/Nm | | |
| **Sample no** | Beaker | **M-factor** | 1,996 (1/s)/(rad/s) | | |
| **Description** | Run 3 10 gpt M155 | | | | |

**Comment**




**Filename:** -----

**Job:** **Element definition / Notes**

ID 2: Rot Time; CR; γ̇ 0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 3: Rot Time; CR; γ̇ 0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 4: Rot Time; CR; γ̇ 0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 5: Rot Time; CR; γ̇ 10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 6: Rot Time; CR; γ̇ 0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 7: Rot Time; CR; γ̇ 0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
ID 8: Rot Time; CR; γ̇ 0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 9: Rot Time; CR; γ̇ 10,45 1/s; t 60,00 s; #300; T 23,00 °C;
ID 10: Rot Time; CR; γ̇ 0,000 1/s; t 5,00 s; #10; T 23,00 °C;
ID 11: Rot Time; CR; γ̇ 0,1045 1/s; t 60,00 s; #300; T 23,00 °C;
Notes

**Evaluation**

ID 12-2: Curve discussion :
Method t in s τ in Pa
---------------------------------------------------
Greatest value 32,34 74,27

ID 13-2: Curve discussion :
Method t in s τ in Pa
---------------------------------------------------
Greatest value 166,6 15,40
Maximum 161,7 15,40

ID 14-2: Curve discussion :
Method t in s τ in Pa
---------------------------------------------------
Greatest value 334,4 12,45
Maximum 334,0 12,33