




Engerbø Rutile and Garnet: Froth Flotation Reagent Dosage and Consumption

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Introduction

Background

Engebø rutile and garnet ore deposit is situated along the Førde fjord in Sunnfjord Municipality, Vestland, Norway. Nordic Rutile ASA, a wholly owned subsidiary of Nordic Mining ASA, has held the rights to Engebø deposit since 2006. The Engebø deposit contains two minerals of economic interest, namely rutile and garnet. In order to separate the minerals of economic interest from the other minerals in the deposit (gangue minerals):

- The ore will be blasted (mined) from the deposit and either selectively stockpiled or processed based on the mineral content
- The ore that has substantial mineral content that can be recovered will be crushed and milled down to decrease the particle size in order to liberate the minerals in the comminution plant
- The comminuted ore will be separated in the mineral beneficiation plant using physical and physiochemical separation processes.
- The gangue minerals along with the unrecovered minerals will constitute the tailings that will be disposed at the bottom of the Førde fjord

Figure 1 shows a simplified process flow diagram of the mineral beneficiation plant. The range of typical mineralogical makeup of the ore is shown in Table 1.

Table 1: Mineralogical composition of the feed ore

Mineral	Approximate Composition (Wt.%)	Chemical Formula
Garnet	46.0%	$(\text{Fe,Ca,Mg})_3\text{Al}_2(\text{SiO}_4)_3$
Omphacite	28.0%	$(\text{Ca,Na})(\text{Mg,Fe,Al})_2\text{Si}_2\text{O}_6$
Amphibole	9.1%	$(\text{Fe,Mg,Ca})_x\text{SiO}_3$
Quartz	4.2%	SiO_2
Rutile	3.7%	TiO_2
Epidote Group	1.9%	$\text{Ca}_2(\text{Al, Fe})_3\text{Si}_3\text{O}_{12}(\text{OH})$
Chlorite	1.5%	$(\text{Mg,Fe})_3(\text{Si,Al})_4\text{O}_{10}(\text{OH})_2 \cdot (\text{Mg,Fe})_3(\text{OH})_6$
Phengite	1.4%	$\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$
Ilmenite	0.6%	FeTiO_3
Pyrite	0.5%	FeS_2
Others	3.1%	

Simplified Flow Diagram

The simplified process flow sheet for the Engebø rutile and garnet project is shown in Figure 1.

Flowsheet

The process has been designed to produce a coarse garnet product, a fine garnet product, and a rutile product. Any mass not reporting to these products will report to the tailings stream.

The mineral beneficiation plant mainly consists of physical separation of minerals which exploits the difference in specific gravity, magnetic susceptibility, or the triboelectric properties of the individual minerals in the deposit. However, the ore contains the mineral pyrite in small amounts. Pyrite exhibits similar physical, magnetic, and electrostatic characteristics as rutile. In order to separate pyrite, which is a gangue mineral, from rutile, a physiochemical process called froth flotation is employed in the processing circuit. Although only $\approx 12\%$ of the ore that is fed into the processing plant is fed into the froth flotation circuit under nominal operation, the process employs the use of chemicals, a portion of which will eventually report along with the tailings. Table 3 shows the relative percentages of mass that is expected to report from different streams into the tailings. Work has been conducted to estimate the amount and nature of the reagents that will report along with the tailings.

Froth Flotation

Froth flotation is a process which exploits the difference in surface properties between components of a system for selectively separating a set of components from the rest of the system. This is often accomplished by using reagents and wetting agents to selectively make a component of the system that needs to be separated either hydrophobic or hydrophilic. The hydrophobic component is carried along to the surface along with the air-bubbles that is introduced in the flotation cell where it is skimmed and separated. Froth flotation has been used successfully in the mineral processing, paper recycling and waste-water treatment industries.


Reagents used in flotation:

The flotation reagents can broadly be classified as below according to their function.

1. **Collectors:** These are a group of chemicals that attach to the mineral surface to make the mineral hydrophilic, thereby improving the affinity of mineral particle to the air bubbles.
2. **Frothers:** These are chemicals that improve the stability of the air bubbles and are usually surfactants that modify the surface tension of the liquid.
3. **Depressants and Activators:** Depressants are chemicals that usually suppress the affinity of mineral particles to attach to the air bubbles. Activators are chemicals that aid in increasing the affinity of mineral particles to the collectors and thereby increasing the ability of mineral particle to float to the surface along with the air bubbles
4. **Modifiers:** These are chemicals that are used to modify the characteristics of the slurry to achieve the desired objective of the flotation process. The modifiers include pH regulators (acids and bases), viscosity regulators, reducing agents, oxidising agents, etc.

Engerbø Froth Flotation:

The main purpose of the froth flotation circuit in the proposed Engebø processing plant is to efficiently separate the pyrite from the minerals so that the remaining minerals can be

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upgraded further into a rutile concentrate. In order to facilitate this separation, a collector and frother are added in the flotation process. Since pyrite is a sulphide and the rest of the minerals in the flotation feed are mostly silicates and oxides, Xanthates are used as the collector. Xanthates readily attaches to sulphide minerals preferentially and makes the sulphide mineral surface hydrophobic.

Usually, if many sulphide minerals are present in the ore, Copper sulphate is added as an activator. However, metallurgical testwork has shown that no activator is required for floating the pyrite in the Engebø ore.

There is no need of any depressants in floating pyrite in the Engebø ore as the xanthates are selective enough to achieve the required separation as there are no other sulphides present in the ore. Even at dosages that are 20 times the current expected dosage, there are no minerals other than pyrite that reports to the froth phase.

No modifiers are required in the flotation process as the flotation is conducted at natural pH. The flotation process uses DowFroth400 (Polypropylene Glycol) as a frother to stabilise the froth.

Reagent Dosages and Tonnages in plant:

The feed to the froth flotation unit will be 12.2% of the ROM process feed mass. At nominal operation, this will roughly be 22.2 tonnes per hour on a dry solids basis reporting to the flotation circuit feed. The relative mass splits, solids percent, and tonnages to the different streams from the flotation circuit can be seen in Table 2.

Table 2: Froth Flotation Stream Characteristics in the Proposed Engebø processing plant

Solids Density	% Solids	tph dry solid	Rel % mass
FF Feed	31.1	22.2	100%
Flotation Tailings	31.3	21.3	96%
Froth Phase	26.0	0.9	4%

The flotation collector will be added to this flotation feed at a dosage of 10grams of collector per tonne of feed ore. The frother will be added to the flotation cell at a dosage of 15grams of frother per tonne of feed ore.

Engebø deposit consists of various ore types. During the metallurgical test programmes, froth flotation has been used effectively to separate the pyrite from the other minerals on all the ore types.

Reagent attachment in the froth flotation operation

The SIBX and the frother are added to the froth flotation feed ore stream in the conditioning tank and flotation cells respectively. The collector will attach to the mineral surface in order to facilitate efficient flotation. There are two streams that exit the froth flotation circuit as depicted in the schematic (Figure 2).

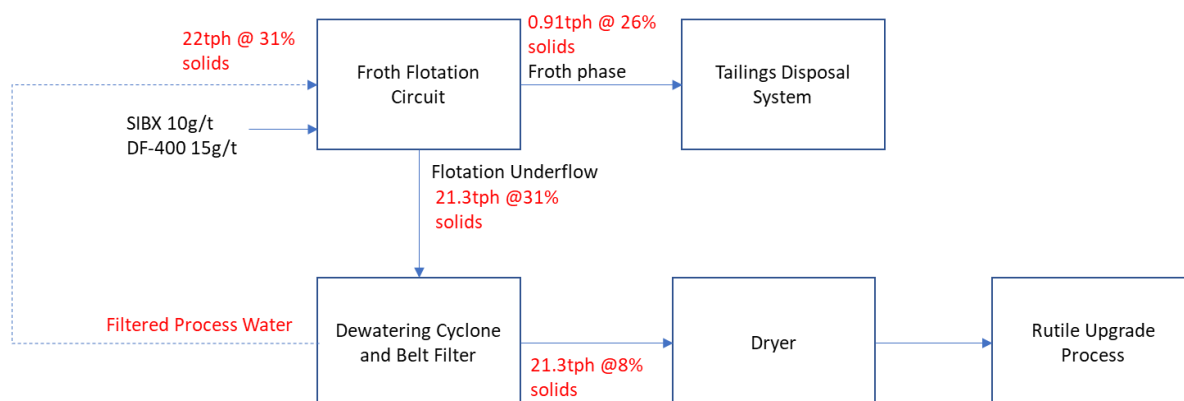


Figure 2: Schematic representing the mass and water flow in the froth flotation circuit in proposed Engebø processing plant

They are the froth phase and the flotation underflow stream. The froth phase is pumped to the tailings co-disposal system whereas the remaining underflow slurry is pumped to a dewatering cyclone and a horizontal vacuum belt filter where the solid phases are separated from the water phase. The water from the belt filter and the dewatering cyclone is re-circulated back into the froth flotation cell feed water splitter box. Hence, the only exit point of the water into the tailings is through the froth phase (either attached to the mineral surface or as unattached xanthate or breakdown products in the water phase).

Any xanthate attached to the non-pyrite minerals in the flotation underflow will be broken down while drying and will not report to the fjord. The remaining xanthate in the water phase in the flotation underflow will either report back to the flotation circuit and build up in concentration which can potentially aid in reducing the Xanthate dosage, or will be adsorbed onto the belt filter, or will breakdown over time. The xanthate attachment tests are further described in sections below. It is envisaged that at least 90% of the xanthates will attach to the pyrite and report to the tailings via the froth phase.

Tailings Disposal

Mass flow and relative mass flow rate

The tailings stream that reports to the co-disposal system and eventually to the fjord roughly constitutes 78.3% of the mass to entering the beneficiation plant (roughly 21.7% of the mass reports to either the garnet product or the rutile product) under nominal operation.

Table 3: Tailings stream constituents in the proposed Engebø processing plant (Figure 1)

Stream	Mass %	Rel .Mass %
CG Tailings (Stream 1)	7.3	9.3%
FPP Tailings (Stream 2)	4.0	5.1%
FG Tailings (Stream 3)	29.2	37.3%
FG Undersize (Stream 4)	0.8	1.0%
SCP Tailings (Stream 5)	28.0	35.8%
FF Tailings (Stream 6)	0.5	0.6%
RUP Tailings (Stream 7)	8.5	10.9%
Total	78.3	

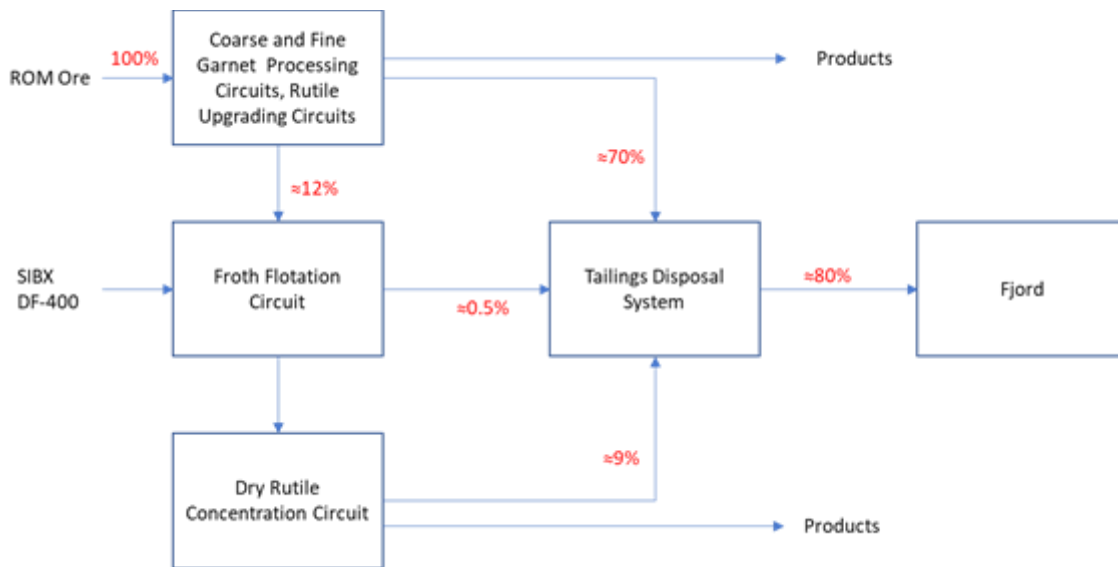


Figure 3: Relative mass flow to the froth flotation circuit and the tailings in the proposed Engebø processing plant

Co-Disposal System:

The tailings from the process is mixed along with sea water in the tailings co-disposal system before being discharged in the fjord.

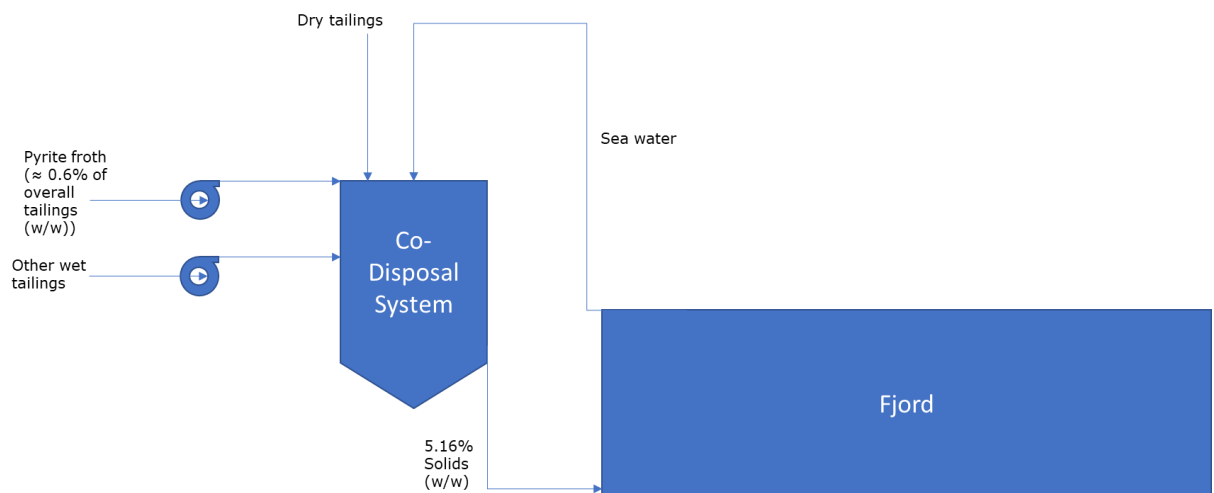


Figure 4: Schematic of the proposed co-disposal system in the Engebø processing plant

Under nominal operation, the only major source of flotation reagent entering this system is from the pyrite froth stream. The overall tailings are mixed with sea water to a solids density of 5.16% (w/w) before being discharged at the bottom of the fjord.

Reagent Concentration Calculations:

Based on the above knowledge, a conservative estimate can be made to calculate the concentration of the flotation reagent at the discharge point in the fjord from the co-disposal system.


Table 4: Calculations estimating the maximum reagent concentration that will be observed in the co-disposal pipeline if all reagents report to the co-disposal system unattached

Stream	Value	Units	Basis
Feed to thickener	34.2	tph	
Feed to Flotation Unit	22.2	tph	LIMN Model; Nominal tonnage
Solids in tailings	145.5	tph	LIMN Model; Nominal tonnage
Process Water in disposal system	46.7	tph	COWI Calculation
Sea Water in disposal system	2626.3	tph	COWI Calculation
Total Water in disposal system	2673.0	tph	COWI Calculation
% Solids in disposal system	5.16%		COWI Calculation
Reagents			
SIBX	10	gpt	Nominal dosage
DF400	15	gpt	Nominal dosage
Magnafloc 5250	10	gpt	Nominal dosage
Dosage			
Grams of SIBX added	222.2	gph	100% of SIBX reports to the tailings
Grams of DF400 added	333.3	gph	100% of DF reports to the tailings
Grams of Magnafloc 5250 added	342.5	gph	100% of Magnafloc 5250 reports to the tailings
Reagent Concentrations (mass/mass) basis			
Concentration at discharge SIBX	0.083	ppm	100% of SIBX reports to the tailings
Concentration at discharge DF400	0.125	ppm	100% of DF reports to the tailings
Concentration at discharge M5250	0.128	ppm	100% of Magnafloc 5250 reports to the tailings
Specific gravity			
Specific gravity of sea water	1.03		Measured Average
Specific gravity of process water	1.00		COWI Calculation
Specific gravity of total discharge water	1.03		COWI Calculation
Volume of total water in disposal system			
Volume of total water in disposal system	2602	m ³ ph	Based on sg of total discharge water (1.7% process water + 98.3% discharge water)
Volume of total water in disposal system	2,602,488	lph	m ³ ph to lph (x 1000)
Reagent Concentrations (mass/volume) basis			
Concentration at discharge SIBX	85	µg/l	Assuming 100% of SIBX reports to the water phase of tailings*
Concentration at discharge DF400	128	µg/l	100% of DF reports to the water phase of the tailings
Concentration at discharge M5250	132	µg/l	Assuming 100% of Magnafloc 5250 reports the water phase of to the tailings**

*A >90% attachment of the SIBX to particles is expected giving <8.5µg/L as the actual concentrations in the tailings. Additional dissolution take place in the fjord upon discharge

**Magnafloc is expected to be attached to the fine particles due to flocculation effects

The calculations in Table 4 pertaining to collector concentration at the bottom of the fjord during nominal operation are conservative: It is assumed that all the xanthate that is added to the flotation cell will report to the tailings without being attached to any mineral though the water in the froth phase i.e 100% of SIBX reporting to the co-disposal system in the aqueous phase. In reality more than 90% will be attached as argued in the next section.

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At nominal operation, 145.5 tonnes per hour of dry solid equivalent will be deposited in the tailings in the form of a slurry at 5.16% (w/w) solids. 46.7 tonnes per hours of process water will report to the tailings. This corresponds to ≈3,492 tonnes per day of dry solids and ≈1,120 tonnes per day of process water in the tailings. On a yearly basis, this corresponds to ≈1,274,580 tonnes of tailings of dry solids and 409,092 tonnes of process water.

The froth phase only constitutes of 4.1% of the mass of the feed reporting to the flotation unit. Only 4% of the water in the flotation unit will report to the water phase in the froth. It is very unlikely that all the reagents will be concentrated in this water, especially if the reagent is dissolved in aqueous phase in the flotation cell.

Xanthates attachment is necessary for the flotation process to work successfully as pyrite is seldom naturally hydrophobic. If there is no reagent attachment, the process will not work, and it is easily observed by the mass reporting to the froth phase. Previous testwork history has shown that pyrite is readily floatable which empirically suggests that Xanthates will always attach selectively to the pyrite.

All the water in the froth underflow is either re-circulated back into the flotation unit or evaporated in the dryer. There is a possibility of the reagent attaching to the filter cloth or being destroyed during the re-circulation process.

The amount of xanthate reporting along with the tailings will depend on how it adsorbs to the different minerals and especially pyrite, the kinetics of this adsorption, and how any un-adsorbed reagent interacts with the rest of the minerals in the tailings. Several studies were conducted to understand the adsorption of Xanthates on the mineral surface.

Xanthate adsorption studies:

Tests were conducted to understand adsorption of xanthates on the mineral surface. The main tests and outcomes are described in the following sections.

Aquateam


Tests were conducted at Aquateam in Norway in early 2019 to understand the adsorption and binding of xanthates. At this time, since the exact flotation feed sample was not available, a sample from different streams from the process were combined to be used for this test. The combined sample had within in what would have reported to the flotation feed but was from a preceding stage in the process. (Stream A, Stream B, and Stream C in Figure 1)

During the time of the test, the reagent dosages were in the process were not optimised and a collector dosage of 100g/t and a frother dosage of 40g/t test were used. The dosage rate was 10 times higher than optimal dosage.

Since the sample used was not the flotation feed, the amount of collector needed to be added was calculated based on the difference in sulphur grade of the sample and flotation feed

The test followed a procedure to determine the adsorption as would have been seen in the flotation cell, and then was followed by leakage and binding tests. The leakage tests followed a standard procedure of dilution to L/S 10.

The results from the tests showed that a SIBX adsorption of 92% was achieved in the initial test and after step 2 mixing with sea water, the overall adsorption was 80%.

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Core Group, Brisbane

Laboratory flotation tests were conducted at Core Group in Brisbane using bench scale and pilot scale flotation equipment. Additionally, the xanthate in the solution were analysed to estimate xanthate and DF-400 reagent attachment to the minerals in the froth flotation feed.

Solution samples were taken from 1kg batch tests from two different Feasibility Programs , and from a larger scale test unit utilising a 40kg sample from a Feasibility study program. These samples correspond to Stream 8 as depicted in Figure 1. Since this stream is the actual stream that will report to the flotation stage, these tests use representative samples using relevant lab scale metallurgical equipment that mimics the process. Additionally, two samples with known concentrations (1% SIBX, and 10% SIBX) were analysed along as reference samples.

The solution samples were shipped to Levay and Company. The reference samples from the two batches indicated that the SIBX purity was close to 87% to 92%, which is in alignment with the stated purity of xanthates. This also suggests that the SIBX had not degraded during the shipment.


The results from the xanthate analysis showed that all the solutions analysed were below the limit of detection of 0.05 mg/L. The 1kg tests were dosed at 5g/t SIBX and 40kg sample test was dosed in total with 15g/t of SIBX. Even though the dosage was higher than nominal dosage of 10g/t for the 40kg test, SIBX was undetectable in all the solutions. This will translate to >95% of SIBX attachment to the mineral surfaces.

SINTEF

Additional adsorption study was conducted at SINTEF using samples from a definitive feasibility study program that represents the flotation feed. These samples correspond to Stream 8 as depicted in Figure 1. This study included a scope to analyse SIBX in a much more precise manner to understand the adsorption and desorption kinetics compared to the Aquateam tests. A collector dosage of 10g/t of feed was used. A procedure similar to the Aquateam test procedure was used, however, instead of a L/S10 dilution with sea water, a dilution to what will be observed in the co-disposal system (5.16% solids) was used. This is depicted in Figure 1 (Co-Disposal system - 'D'). The tests were conducted by agitating the sample in a beaker and by introducing air bubbles into the beaker. The results suggested that up to 92% of SIBX can be adsorbed on the mineral surface. The results also suggested that adsorption can take place even after sea water dilution provided that the collector molecules can encounter a suitable mineral particle.

It is important to mention that the test will not directly emulate the co-disposal conditions even though the sample was diluted to the right solids density. This is due to the fact that a much higher mass enters the co-disposal system and the proportion of the froth phase is much lower compared to the overall mass. Since the froth phase could not be separated in the SINTEF tests, and since all the remaining tailings sample were not available for making a representative tailings sample, the sample was diluted to the appropriate solids density and used as a proxy for the actual tailings. This roughly resembles the co-disposal system and what will be discharged to the fjord prior to further mixing in the fjord system. The discharge pipeline to the bottom of the fjord is depicted in Figure 4.

After the discharge of the tailings to the fjord, further dilution is expected to take place in the fjord from sea water. This will further dilute the reagents in the water phase in the fjord. The

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further dilution process was modelled by SINTEF using a numerical DREAM (Dose related Risk and Effect Assessment Model) model.

Annual Reagent Consumption

The Engebø rutile and garnet comminution and beneficiation facility is designed to process 1.5 million tonnes of ore per year as per the current feasibility study. The annual consumption of reagents is directly proportional to the run-of-mine tonnage that is processed through the beneficiation plant. Apart from the tonnage, a few other factors that affect the reagent consumption are:

Variability of the ore

The Engebø deposit can to some extent be defined as a heterogenous ore body with a measurable variability associated with the mineral composition, mineral grain size and mineral association. Mining operation will aim to selectively mine the ore that will be economical to process at a profit. The variability will translate to changes in mass flow within the processing plant over time. Since the reagents are dosed based on the tonnage of feed to a unit process, this will affect the annual reagent consumption over time. It may so happen that the reagent consumption will be higher than an average consumption for a few years of mining, and lower than an average calculated consumption for the remaining years due to this variability.

In general, a higher-grade ore which is more profitable will be mined in the initial years of operation to improve the economics of the project. As the mine progresses through operation over time, the grade of the gradually decreases; some operations usually trigger exploration work to increase ore body knowledge in order to increase the life of mine. In cases where the mining is conducted in phases (for example and open pit phase and an underground phase), there can be an increase in grade after the first phase. These factors can influence reagent consumption.

The operating factor of the plant

An operating factor for a facility is an indication of the percentage of time the production will be at instantaneous design rate and expected quality. It is therefore dependant on the amount of time a facility is available for operation, the percentage of the available time that is utilised, the achieved rate of operation/production, and the quality of the product. If the operational targets are met, more ore would be processed through the plant resulting in a higher reagent consumption. If there is more down time


Market Conditions and Economy of the project:

Engerbø processing facility aims to produce a premium grade rutile product and garnet products for the abrasive and waterjet cutting markets. The prices for these end products are intricately related to the world economy. A change to the price of these product may result in either a change in strategy during operation or may even result in design of the operation. All these factors will be needed to taken into consideration while applying for a permit

Engerbø Ore Reagent Consumption:

There are three reagents that are used in the Engebø process facility; the flotation reagents (SIBX and DF400) and the flocculant (Magnafloc 5250).

The consumption of the flotation reagent is related to the mass proportion reporting to the flotation unit. The collector is dosed at a rate of 10g/t of feed at nominal operation, and the

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frother is dosed at a rate of 15g/t. At nominal operation this will correspond to approximately 2 tonnes of SIBX per year and 3 tonnes of DF400 per year

Magnafloc is used to flocculate fine material in the thickener to clean the process water for recirculation within the process plant. The flocculant consumption is dependent on the amount of material that reports to the thickener. A flocculant dosage of approximately 10g/t is recommended and the consumption will vary based on the amount of feed reporting to the thickener. A yearly consumption of 4 tonnes per annum should be sufficient to operate the thickener successfully.

Discussion and Conclusion


Emulating the exact process conditions that is expected to take place in the industrial process is difficult as the overall tailings constitute of various streams that need to be combined. The method of analysing and interpreting the actual reagent pathway into the fjord will require an interdisciplinary approach that combines knowledge and laboratory equipment in various scientific disciplines such as mineral processing, analytical chemistry, organic chemistry, toxicology, marine biology, modelling and simulation of marine systems to name a few. Effort has been made by Nordic Mining, along with DNVGL, to coordinate and understand this reagent pathway into the fjord.

Xanthates are selective collectors that preferentially attach to sulphide minerals over silicate and oxide minerals. Although it is difficult to analyse the adsorption of collector to mineral surfaces directly in the solid phase, it is an acceptable assumption that most of the xanthates selectively adsorb to the sulphide minerals, and that any adsorption to non-sulphide minerals is nonselective. This can be empirically validated metallurgically in that even at much higher collector dosages of 10-20 times the current envisaged dosage of 10g/t, there is only a minimal increase in mass pull to the froth phase. It is fair to assume that the xanthate either enters the tailings in a solution phase, or with the solids phase from the flotation froth.

Since the water from the flotation circuit is re-circulated within the circuit, the froth phase is the only part of the circuit where the xanthate can reach the tailings. The recirculating water in the flotation circuit also provides opportunity to reduce the xanthate dosage if the xanthate does not adsorb effectively to the pyrite and remains in the solution.


Tests conducted at Core Group have suggested that most of the xanthate is attached to the pyrite. Since these tests are conducted using actual flotation equipment using actual samples, these results would be very pertinent to understanding the adsorption of xanthates and how it relates to the metallurgical performance. The use of actual flotation equipment would rule out any unforeseen variables or mixing inefficiencies in the beaker and agitator lab units. The results show xanthate levels below lower limit of detection in all filtrate samples. This corresponds to a >95% xanthate adsorption onto mineral surfaces. The use of reference samples increases the confidence in this analysis.

The reaction kinetics of xanthates onto sulphide surface can be reduced if the sulphide surface is oxidised. In actual operation, the delay between sample comminution and milling and froth flotation will be less than 24 hours. This would, ideally, result in better adsorption and flotation of the pyrite. Some of the slower adsorption kinetics in tests can be possible due to long storage of sample.

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Analysis conducted at SINTEF with the relevant sample, but on makeshift lab equipment gave xanthate adsorption of 92%. Tests conducted using relevant samples and industrial equipment suggests xanthate adsorption on to the mineral surface of >95%.

For an annual consumption of chemicals, 2 tonnes per annum of SIBX, 3 tonnes per annum of DF400, and 4 tonnes per annum of Magnafloc5250 is recommended.

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