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# Occurrence of iron in industrial granitic pegmatite

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# ABSTRACT

Granitic pegmatite is used as raw material for industrial minerals in the production of feldspar and quartz for glass, ceramic and porcelain. One of the most important quality parameters in the feldspar is the content of iron (Fe). At the Lillesand plant, Norway, pegmatite is floated to produce quartz, albite and microcline products. Fe is mostly removed through flotation and magnetic separation, but some Fe is still present in the final products, the amount depending on the raw material source. Rietveld X-ray Diffraction (XRD), Point counting by optical microscopy, and Electron Microprobe Analysis (EPMA) combined with image analysis of Back-Scatter Electron (BSE) images was used to quantify the mineralogy and to map the distribution Fe in the pegmatites. The study showed that Fe is present as mineral inclusions in feldspar, in addition to its occurrence in minor mineral components such as mica and chlorite. The frequency of Fe-mineral inclusions was higher in albite than in microcline, and they were often associated with micro-fractures and areas of alteration. These findings reveal a potential for reducing total Fe<sub>2</sub>O<sub>3</sub> in the microcline products.

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#### 1. Introduction

Granitic pegmatite is mined for the production of quartz and feldspar used in products like porcelain, ceramics, glass and glass fibre. Because of the generally heterogeneous nature of pegmatites, with respect to both grain size and chemistry, it is necessary to blend different raw materials to meet quality specifications. The most important quality parameter is the content of Fe because of its colouring effect in further processes. The quality specifications generally state a maximum Fe content, and may include a requirement of low variation for other elements such as K and Na in the mineral concentrates. To meet the specifications it may be necessary to remove Fe-containing phases in the raw material by mineral enrichment processes. Due to the nature of the enrichment processes and the mineral textures, the Fe content in the resulting mineral concentrates is not necessarily dependent on the Fe content in the raw material. These specifications and requirements point to the importance of understanding the occurrence of Fe in the granitic pegmatite raw material.

This paper presents a case study from the Glamsland pegmatite field, Lillesand, Southern Norway. In recent years, raw material from different deposits, have been blended as feed to the dressing plant. The mineral enrichment process consists of crushing, grinding, and flotation in three steps, drying and high-intensity magnetic separation, before storage and shipping to customers (Hestnes et al.,

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2010). The flotation process consists of three separate circuits: (1)

The aim of this paper is to characterize the occurrence of Fe in key raw material types of the Glamsland pegmatite field in order to understand their different behaviours in the mineral enrichment process and different product qualities. The focus in the investigation will be on the feldspar minerals, since the Fe content of quartz from the Glamsland deposits is known to be stable and low (Sibelco Nordic, 2010). The main objectives of the investigation are to:

- Identify the Fe-bearing minerals and their Fe content.
- Document and quantify the textural relationship between the feldspars and other Fe-bearing minerals.

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Reverse flotation of mica and Fe-bearing minerals, (2) Quartz-feldspar separation, and (3) Separation of Na-rich feldspar from K-rich feldspar. Tailings are produced only in the reverse mica flotation where most of the Fe-bearing minerals are removed. After flotation, the products are de-watered and dried. The quartz product receives no additional handling, while the feldspar products are treated by high intensity magnets to remove Fe-containing minerals that have not been removed by the wet process. Daily operational control only includes semi-quantitative XRF, though experience from the dressing plant has shown that raw materials from different locations behave differently in the mineral enrichment process, and give products of different qualities. This experience is used to map distinct areas of the deposits as suitable for specific product qualities, and thus assist in the blending process of the raw material to the plant. Why the different pegmatite types behave differently during processing is, however, not fully understood.

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Document the variation of Fe as a function of particle size range.
Estimate the total contribution of Fe from the different minerals in each pegmatite type.

The importance of this investigation lies in the ability to utilize the deposits in the best way both economically and environmentally (Baum et al., 2004; Hoal, 2008; Dunham et al., 2011). Depending on the occurrence of Fe in the raw material, it might be possible to improve the enrichment process and increase the feldspar product quality.

## 2. Experimental

#### 2.1. Sample materials

The sample materials used in this study were different raw material types of the Glamsland pegmatite field, Lillesand, Southern Norway. The mineralogy of these raw materials was investigated by Hestnes (2012a,b) and consists of quartz (qtz), albite (ab), and microcline (mc), with minor amounts of muscovite (mu) and biotite (bt). Sulphide minerals like pyrite and pyrrhotite are found in trace amounts and occur as inclusions in the main minerals, or in micro-fractures. Garnet is observed in trace amounts in certain areas of the pegmatite field.

The deposits of importance in recent years of mining have been Lørdagsheia (LH), the C-deposit (C) and Heståsknipen (HK). In these deposits nine types of raw material have been defined (Table 1). Of these, the authors have selected three key raw material types, LRT1 (Pink pegmatite), LRT3 (Green pegmatite) and LRT4 (Red pegmatite), for study based on their mineral enrichment characteristics and the quality of the resulting products. LRT1 is regarded as the highest quality raw material with respect to both processing and final product. However, according to Table 1 this is one of the raw materials with relatively high Fe<sub>2</sub>O<sub>3</sub> content. In contrast LRT3 is known to create problems in the mineral enrichment process and gives a Fe-rich feldspar product that must be blended with less Fe-rich products. LRT4 gives a feldspar product that is very high in Fe, and has to be blended with less Fe-rich products.

#### 2.2. Sample preparation

Each sample was stage-crushed to 100% passing -1.68 mm using a jaw crusher in four steps. This process included sieving of the sample in advance of each step to prevent over-crushing. The sieving product was ground in batches in a polyurethane-coated laboratory mill with an inner diameter of 250 mm and a length of 150 mm. Each grind used 1.0 kg material, 1.0 l of water and a pre-defined selection of steel balls with a total charge weight of 4.0 kg. The ball mill was run with a constant speed of 72 RPM, which is 85% of the critical number of circulation of the mill.

Grinding time was 20 min. After grinding, the pulp was deslimed and dried. When dry, the ground material was sieved into fraction sizes: +600 μm, +500 μm, +425 μm, +300 μm, +212 μm, +150 μm, +75  $\mu$ m and -75  $\mu$ m. To achieve the study objective of determining whether the occurrence of Fe in the investigated pegmatite types varies as a function of particle size range, three fractions: 75-150 μm (+75 μm), 212-300 μm (+212 μm), and 425-500 μm (+425 µm), were investigated in this paper. The sample fractions were prepared as polished thin sections using epoxy resin (EPO-TEK-301, 2012) or ground to powder. The powdered samples were prepared using a micronizing mill with agate grinding elements, adding 10 mL of ethanol as a grinding fluid. All samples were ground for 30 min, extracted from the grinding jar and rinsed with ethanol through a 40 µm sieve to remove oversized particles. When dry, the pulverized sample material was prepared further, depending on the analysis method.

#### 2.3. Methods

This study focuses on Fe-bearing minerals in the raw material types of the Glamsland pegmatite field. The bulk chemistry of the key raw materials was measured using X-ray Fluorescence (XRF), while Rietveld X-ray Diffraction (XRD) and point counting using an optical microscope were used to quantify the mineralogy. Image analysis (IA) on back scatter electron (BSE) images was used to identify Fe-bearing mineral phases related to the feldspar minerals. Mineral chemistry analysis from points and element mapping was done with an Electron Probe Micro Analysis (EPMA).

XRF analyses were performed on powder tablets using an ARL Advant'XP instrument with Thermo software. During analysis the electric current was 70 mA and the voltage was 40 kV, while analysis time was 160 s. The instrument used a program which was specified for the raw material analysed, made by international standards. The results were given as content of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, total-Fe<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, MgO, CaO, and TiO<sub>2</sub>.

Powdered samples were analysed by XRD. The data were collected on a Bruker X-ray Diffractor D8 Advance using 40 kV, 40 mA, and Cu K $\alpha$  radiation of wavelength K $\alpha$ 1 = 1.5406 Å and K $\alpha$ 2 = 1.54439 Å and a K $\alpha$ 1/K $\alpha$ 2 ratio of 0.5. Diffractograms were recorded from 3° to 70° 2 $\theta$ , in 0.021° 2 $\theta$  increments with 0.87 s counting time per increment, and the total analyzing time was 49 min. Output from XRD was compared manually with minerals in the ICDD database using Bruker's EVA<sup>®</sup> software (Madsen et al., 2001; Scarlett et al., 2001) and then exported to Topas for structural refinement (Rietveld, 1967, 1969; Will, 2006; Hestnes and Sørensen, 2011, 2012). This structural refinement was performed using a predefined routine to quantify the main minerals albite, microcline, quartz and biotite (Hestnes and Sørensen, 2011).

Point counting of polished thin sections for the sample size fractions was carried out using a Nikon Optiphot optical microscope with CFW 10× Oculars and magnifications of M Plan 5 DIC 0.1

Table 1	
The chemistry of the defined raw	materials in the Glamsland pegmatite field.

Deposit area	Rock type	Rock type	Raw material	Chemistry (wt.%)							
				SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O3	K <sub>2</sub> O	Na <sub>2</sub> O	CaO	TiO <sub>2</sub>	MgO
Lørdagsheia	LH	Pink pegmatite	LRT1	77.36	13.25	0.65	4.16	4.23	0.88	0.03	0.07
C-deposit	С	Pink pegmatite	LRT2	77.74	13.02	0.69	4.04	4.14	0.98	0.03	0.05
Heståsknipen	HK	Green pegmatite	LRT3	79.53	12.31	0.41	2.01	4.84	1.30	0.01	0.01
C-deposit	С	Red pegmatite	LRT4	79.03	12.32	0.46	4.05	3.86	0.89	0.02	0.02
Lørdagsheia	LH	Pink graphic granite	LRT5	76.64	13.29	0.31	7.55	2.44	0.30	0.02	0.02
C-deposit	С	Pink graphic granite	LRT6	73.64	14.52	0.15	9.37	2.30	0.23	0.01	0.00
Heståsknipen	HK	Pink graphic granite	LRT7	74.43	14.31	0.12	9.18	2.18	0.19	0.00	0.00
Lørdagsheia	LH	White graphic granite	LRT8	76.51	13.36	0.16	6.17	3.29	0.57	0.01	0.00
Lørdagsheia	LH	Mica rich pegmatite	LRT9	76.85	13.39	0.93	3.23	4.67	1.11	0.06	0.14

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