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Extraction of magnesium from four Finnish magnesium silicate rocks for CO₂ mineralisation - Part 1: Thermal solid/solid extraction

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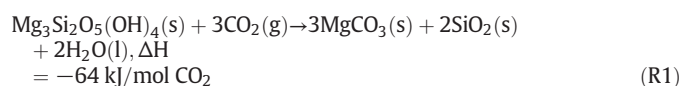
ABSTRACT

The only option for carbon capture and storage (CCS) in Finland is mineral carbonation, which has been extensively researched at Åbo Akademi University (ÅA). Finding suitable minerals for mineral carbonation in different regions of Finland will keep transportation of either CO₂ or the actual mineral at a lowest possible level. Four different rocks from different regions in Finland have been compared with respect to composition and possibility to extract magnesium. This paper presents experiments applying thermal solid/solid extraction. It was found that a mixture of ammonium sulphate (AS) and ammonium bisulphate (ABS) could extract a significant part of magnesium compared to tests using only AS or ABS. Serpentinite rock is also the best option for magnesium extraction while diopside and magnesiohornblende are much less reactive. Continuous mixing during the experiment in a rotary tube and adding some water to the solid/solid mixture gives better extraction results. The binding capacities of CO₂ in the serpentinites with respect to the magnesium extractions achieved in this study are 240 kg CO₂/ton rock (Serp-A) and 207 kg CO₂/ton rock (Serp-B).

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1. Introduction and background

Finland's annual emission allocations are based on the reduction obligation of 16% of the emission levels of 2005 by the year 2020 (Official Statistics of Finland, 2015). According to VTT (2012) it should also be possible to cover 85–100% of the Finnish energy use without emissions of carbon dioxide (CO₂) by 2050. This puts demands on technologies for carbon capture and storage (CCS) as well as increased energy efficiency. The only option for CCS within Finland's borders is mineral carbonation, which has been intensively researched at Åbo Akademi University (ÅA) (Fagerlund, 2012; Nduagu, 2012; Romão, 2015; Sjöblom and Eklund, 2014). Starting with an extraction reaction of mainly magnesium from silicate rock it becomes possible to sequester CO₂ through the overall (exothermic) reaction (Nduagu et al., 2012);



One mineral found to be suitable for mineral carbonation via the ÅA route is serpentinite. One of the studied serpentinites is originating from the Hitura ultramafic complex located in the central part of Finland. The complex consists of mainly porous serpentinites found to be reactive

enough to be suitable to use in the ÅA route. This rock will be presented further here together with another serpentinite rock from the Vammala region as well as a diopside and a magnesiohornblende, all from southern parts of Finland. The locations of the different rocks are shown in Fig. 1. An issue for mineral carbonation is to find suitable minerals in different regions of Finland, preferably as close to large CO₂ emitting sources as possible. This part 1 focuses on possible suitable mineral reserves found in the regions of Hitura, Vammala, Salittu and Parainen and the extraction of magnesium from these rocks by thermal solid/solid extraction. Part 2 (Erlund et al., 2016) focuses on aqueous solution leaching methods (Sanna et al., 2014; Styles et al., 2013; Zhao et al., 2015) of the same rocks.

2. Material and methods

2.1. Materials

Four different rocks have been studied. One is the also earlier studied mine overburden from the Hitura nickel mine consisting mainly of serpentine-rich rock and magnetite (Serp-A), (Nduagu et al., 2012; Romão, 2015). Earlier tests resulted in extractions of magnesium of up to 70% (Nduagu, 2012; Romão et al., 2013). This serpentinite from the now closed Hitura nickel mine was chosen since a possibility could be to apply CO₂ sequestration by mineral carbonation to a lime kiln located at a steel making plant in Raahe. The distance between Raahe and the Hitura nickel mine is approximately 140 km. The nickel mine was closed

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Nomenclature

Abbreviations

ÅA	Åbo Akademi University
AAS	Atomic Absorption Spectroscopy
ABS	Ammonium bisulphate
APS	Ammonium pyrosulphate
AS	Ammonium sulphate
CCSM	Carbon Capture and Storage by Mineralisation
CCS	Carbon Capture and Storage
Mg-Horn	Magnesiohornblende from Salittu region in Finland
Serp-A	Serpentinite from Hitura nickel mine in Finland
Serp-B	Serpentinite from Vammala region in Finland

Chemical compounds

CO ₂	Carbon dioxide
CaSO ₄	Calcium sulphate
CaSO ₄ ·2H ₂ O	Gypsum
FeCaSi ₂ O ₆	Hedenbergite
Fe ₃ O ₄ , FeO·Fe ₂ O ₃	Magnetite
Fe(OH) ₂	Iron(II)hydroxide, Ferrous hydroxide
Fe(OH) ₃	Iron(III)hydroxide, Ferric hydroxide
FeSO ₄	Iron (II) sulphate, Ferrous sulphate
MgCO ₃	Magnesium carbonate
Mg ₅ (CO ₃) ₄ (OH) ₂ ·4H ₂ O	Hydro magnesite
MgCaSi ₂ O ₆	Diopside
MgSO ₄	Magnesium sulphate
MgSi ₂ O ₅ (OH) ₄	Serpentine
NH ₄ HSO ₄	Ammonium bisulphate, ABS
(NH ₄) ₂ Mg ₂ (SO ₄) ₃	Synthetic efremovite
(NH ₄) ₂ SO ₄	Ammonium sulphate, AS
(NH ₄) ₂ S ₂ O ₇	Ammonium pyrosulphate, APS

in 2013 as a result of the decreasing nickel price. The dimension of the ultramafic complex is about 0.3 × 1.3 km (Belvedere Resources, 2016).

Rock from close to the Vammala mine was earlier studied by Mäkelä (2011) and Zevenhoven et al. (2012). The authors suggest that more studies of the rock should be done in order to optimize the extraction of magnesium. The rock used in this study (Serp-B) is, however, originating directly from the mine. A benefit with this rock type is the location close to CO₂ emitting sources located in southern part of Finland. The distance between Vammala and a lime kiln in Parainen, for example, is also approximately 140 km.

A concentrated diopside rich fraction of the wall rock of a lime stone quarry was also studied (diopside). The rock was concentrated using density separation. Diopside has a density about 3.3 g/cm³ (Mindat, 2016), which is a higher density than the rest of the rock. However, it should be mentioned that the diopside rich fraction only corresponded to 10% of the whole wall rock sample. The benefit of being able to carbonate this rock would be the short (zero) distance from the CO₂ emitting source (i. e. the lime kiln) and for the company to utilize mined side rock. Lu et al. (2011) and Frost and Beard (2007) mention the option for a reaction with diopside in an acidic environment, resulting in serpentinite as product. This was tested more thoroughly in part 2 of this paper (Erlund et al., 2016). Diopside belongs to the pyroxene group with silicon-oxygen tetrahedral chains. In the diopside rich fraction - obtained after the density separation of the wall rock from the limestone quarry - approximately half of the magnesium is substituted by iron, see Table 1 and Table 2. Thus, twice the amount of diopside would be required for the same amount of magnesium extraction compared to pure diopside. The pyroxene with magnesium substituted by iron is known as hedenbergite (CaFeSi₂O₆) and the Mg-Fe substitution giving the diopside-hedenbergite series also results in changes in the Si-O chain configuration (Deer et al., 1992).



Fig. 1. Locations of the rocks used in this study.

The Salittu open pit is processing peridotite, a rock type mainly containing olivine and/or pyroxene (Länsi-Suomen ympäristölupavirasto, 2009). Olivine is known to be another suitable mineral besides serpentinite for mineral carbonation (IPCC, 2005). The Salittu mine is also located in southern part of Finland. However, no pyroxene nor olivine were found at all in the rock used in this study (Mg-Horn) when looking at the X-ray diffraction analyses (XRD) in Table 1. Plagioclase and amphibole seems to dominate in the rock samples used here.

Table 1 shows XRD results for the rock types studied while Table 2 presents the X-ray fluorescence (XRF) analyses of the same particle size fractions. The diopside and the Mg-Horn contain much less magnesium than Serp-A and Serp-B. The diopside contains quite a lot of Ca and could with respect to that be used instead of Mg to bind CO₂. This requires that the Ca is not already bound to carbonates, however, which could be released as CO₂ when treated.

Comparison of the binding capacities between the rocks and contents of valuable minerals in the context of mineral carbonation (Mg, Ca) are presented below, showing that the contributions of interesting (“carbonatable”) elements are comparable in all rock types tested (Fig. 2). Serp-A and Serp-B offer the possibility to bind a significant

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