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Carbonation of serpentinite rock from Lithuania and Finland

Inga Stasiulaitiene^a, Johan Fagerlund^b, Experience Nduagu^{b,c},
Gintaras Denafas^a, Ron Zevenhoven^{b*}

^a *Kaunas University of Technology, Department of Environmental Engineering, Radvilenu av. 19, LT-50254 Kaunas, Lithuania*

^b *Åbo Akademi University, Thermal and Flow Engineering Laboratory, Biskopsgatan 8, FI-20500, Åbo / Turku, Finland*

^c *Energy and Environment Systems Group, Institute for Sustainable Energy Environment and Economy,*

University of Calgary, 2500 University Drive NW Calgary, Alberta, T2N 1N4, Canada

Abstract CO₂ mineral sequestration, or mineral carbonation, is presumably the most important CCS alternative when underground CO₂ sequestration is not an option or considered unattractive. The following advantages must be emphasized: leakage-free CO₂ fixation that does not require post-storage monitoring; the overwhelmingly large capacity offered by mineral resources available worldwide; and as recognized more recently, the possibility to operate with the CO₂ containing gas directly, removing the very expensive CO₂ separation step from the CCS process chain. Also, the solid products can be used in applications ranging from land reclamation to iron- and steelmaking. Here, a staged process for mineral sequestration that resulted from a decade of R&D work in Finland is applied to two serpentinite rocks from Lithuania and Finland, respectively. The process involves production of magnesium hydroxide from the mineral, with ammonium sulphate as the extractant (which is recovered downstream), followed by carbonation in a pressurized fluidized bed at 20–40 bar, 450–550°C. Benefits of this route are that 1) the carbonation reaction heat is taken benefit of, 2) only magnesium hydroxide is carbonated, 3) solid residue, magnesium carbonate and iron oxides are obtained as separate streams, 4) no expensive or non-recoverable chemical additives are used and 5) the pressure in the carbonation process is relatively low. The results show that magnesium hydroxide can be effectively extracted from the two serpentinites at somewhat different extraction process conditions, while the magnesium hydroxide particles produced (~170 μm, ~340 μm) can be carbonated for 45–50 % within 10 minutes at 20 bar, ~500°C. Process energy efficiency is similar, or slightly better than (direct, aqueous solution) carbonation processes that were suggested earlier.

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1. Introduction

At many locations carbon dioxide mineralization is a more attractive CO₂ capture and storage (CCS) technique than the underground storage of compressed CO₂, especially for the locations where underground storage is no option. Despite the recognized and documented advantages of the method (very large capacity, no post-storage monitoring needed, exothermic overall process chemistry) [1,2] the

* Corresponding author. Tel.: +358-2-215-3223; fax: +358-2-215-4792
E-mail address: ron.zevenhoven@abo.fi

development work is still in the laboratory demonstration scale. Motivated by the slow deployment of large scale underground storage of CO₂ or simply the availability of large amounts of suitable mineral, R&D progress is being steadily made and reported from an increasing number of research teams and projects worldwide. Still the main obstacle for mineral carbonation is its slow kinetics also when considering direct carbonation of serpentinite in aqueous solutions. In general, aqueous mineral carbonation has been favored over gas-solid carbonation because of its faster kinetics [3,4].

Development work in Finland (where the exothermic carbonation chemistry is the reason for focusing on high temperature, gas/solid carbonation at elevated pressures) has for many years involved cooperation with the Baltic states Estonia and Lithuania. Also in Lithuania significant magnesium silicate resources, i.e. serpentinite rock [5] are available that may be used for large-scale CO₂ mineralization. This material, from the Varena region of south-eastern Lithuania shows similarities with serpentinite rock from Finland's nickel mine at Hitura, central Finland. This paper reports on a study where the carbonation of these two rock types are compared, addressing the production of Mg(OH)₂ and the rate and final level of Mg(OH)₂ carbonation.

2. Lithuanian and Finnish serpentinite

The most interesting rocks for CCS purposes are the serpentinites, due to their abundance and suitable composition, containing up to 40% MgO. Serpentinites are found distributed in the south-eastern part of Lithuania within the Varena Geological Province of the East Lithuanian Domain. Serpentinite bodies are well discernable on the gravity and essentially the magnetic field maps, as they associate with the magnetite (iron ore) concentrations. The chemical composition of the serpentinite samples used in the experiments are shown in Tables 1 and 2. More than a dozen of serpentinite bodies were identified in south Lithuania. Owing to their association with the iron ore deposits these bodies were extensively studied by drilling. They are covered by platform sediments of 280 - 500 m thick – see Figure 1. The depth of the basement increases to the northwest. The depth of serpentinites layer varies from 360 m and continues till 1 – 1.5 km. The volume of the serpentinites in the largest Varena Iron Ore Deposit was estimated 1-2 Gt. Consequently, the sequestration potential was evaluated as high as 0.5-1 Gt. This quantity may provide sufficient resource for storing the volume of CO₂ produced during 200-500 years in the south-eastern part of Lithuania. The other serpentinite bodies identified in the south-eastern Lithuania are much smaller, with the total capacity approximately equal to the Varena Iron Ore Deposit. Thus, the overall resources of Lithuanian serpentinites provide large potential for mineral sequestration of the carbon dioxide.

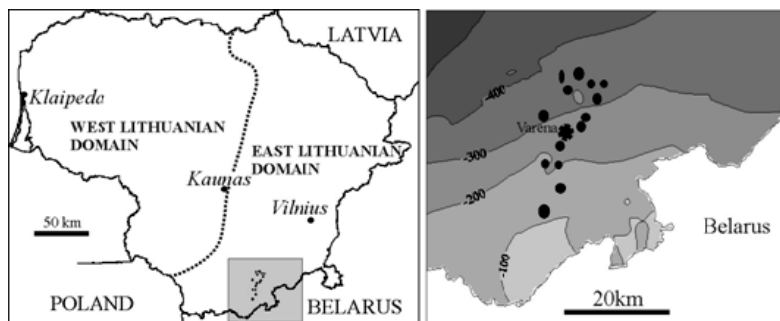


Figure 1 Left – major domains of the crystalline basement of Lithuania. The study area is shaded. Right – distribution of the serpentinite bodies in southern Lithuania. The Varena Iron Ore deposit is marked * [5].

Table 1 Chemical composition of the Lithuanian and Finnish serpentinites studied as oxides, wt-%

Compound	H ₂ O	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	FeO	MnO	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	P ₂ O ₅
Lithuania*	0.2	12.04	0.59	58.5	11.63	0.03	1.26	12.48	0.03	0.12	0.07	0.53
Finland**		32.6			13.4	0.07	0.51	38.1				

* Ref [6]; ** Ref.[7] average of two values

Table 2 Chemical composition of the Lithuanian and Finnish serpentinites studied as elements, wt-%

Element	Mg	Fe	Si	Ca	Mn	Ti	Cu	Ni	S	Al
Lithuania [#]	18.9	12.3	16.0	0.86	0.04	-	0.01	0.01	-	0.12
Finland [#]	21.8	10.1	11.6	0.34	0.08	0.02	0.01	0.02	0.48	0.02

[#] Ref [8-10]

In Finland, millions of tons of poorly documented in situ or hoisted serpentinite or tailed serpentine deposits are located mainly in central Finland. To bind ~ 10 Mt CO₂/a (the reduction required by the Kyoto target set for Finland) by carbonation of serpentinite, ~ 25 Mt/a minerals would be needed. It has been estimated that the serpentinites in Eastern Finland alone could be sufficient for 200-300 years of CCS processing. The minerals or rocks that could be suitable for binding CO₂ in economical amounts are situated especially in those ultramafic rock formations that have already been mined for industrial minerals and metals, such as talc, soap stone, chromium and nickel. These rocks are being constantly piled or dammed as tailings for later use in industry - see Figure 2. Currently available resources of hoisted serpentinite / serpentinite (33-39% MgO) at Finnish Ni, Cr and talc mines are ~ 29 Mt [11,12].

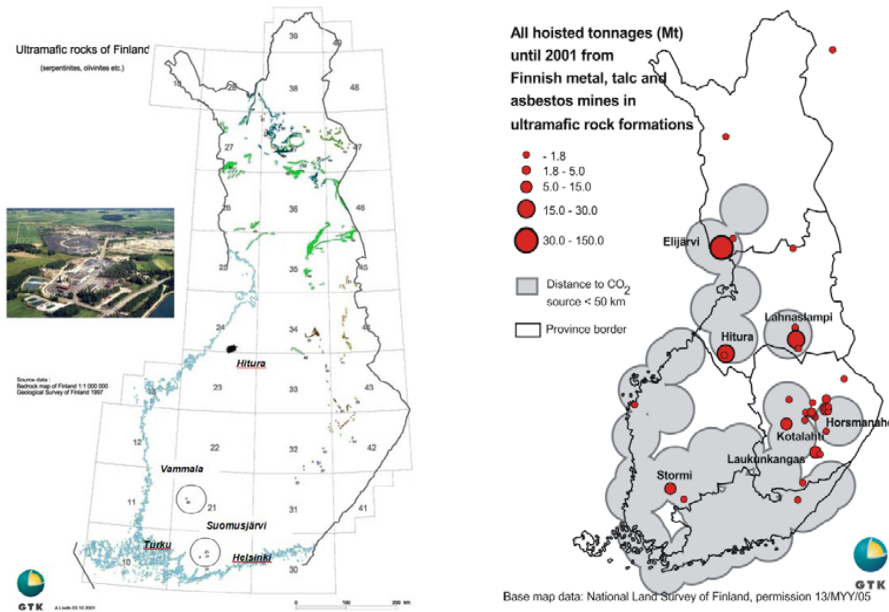


Figure 2 Left – Ultramafic rock in Finland, showing also a photo of the nickel mine at Hitura and its location, and findings in southern Finland at Vammala and Suomensjärvi. Right – Hoisted serpentinites in Finland.

Most work in Finland so far focussed on serpentinite from Hitura (not visible on the 1 km × 1 km accurate map in Figure 2- left!). Current work includes (besides many samples from abroad, supplied by international project consortium partners) serpentinite rock found at Vammala and Suomensjärvi in southern Finland where a significant part of Finland’s CO₂ is produced. These locations are identified on the map and first test results suggest a good quality for CO₂ mineralisation, similar to the Hitura rock material.

3. Methodology and process description

The method for magnesium silicate as currently being optimized at Åbo Akademi University (hereafter: “ÅA”) in Finland implies that a process of extracting the magnesium from the mineral is combined with carbonation of this in a pressurized fluidized bed (PFB) reactor. Schematically the process can be

illustrated by Figure 3. The thick dotted arrows represent the ideal case of heat flows providing energy for step 1 and the inevitable mining activity (crushing and grinding) associated with serpentinite excavation and pretreatment. Although the $Mg(OH)_2$ production is endothermic the consequent carbonation step is exothermic and the overall process could still be rendered energy neutral (or even negative).

The production of $Mg(OH)_2$ is accomplished using a solid/solid reaction with ammonium sulphate (which is recovered downstream for re-use) at 450 – 550 °C followed by precipitation of $Mg(OH)_2$ from an aqueous solution – see Figure 4 – using a 25% NH_3 solution in water. The reaction of serpentine with ammonium sulphate $(NH_4)_2SO_4$ is thermodynamically feasible at temperatures between 200 °C and 550 °C [8-10]. The same extraction route and process conditions were used at Lithuanian serpentinite at KTU, Kaunas (hereafter: “KTU”) and at ÅA. The main difference of the $Mg(OH)_2$ production processes was the furnace equipment used for heating the solid mixture of serpentinite with ammonium sulphate at two locations. Significant amounts of iron oxides are obtained as by-products. Note that while all carbonation tests reported here were done at ÅA, production of $Mg(OH)_2$ from Lithuanian serpentinite was done both at KTU and at ÅA. Finnish mineral was processed only at ÅA.

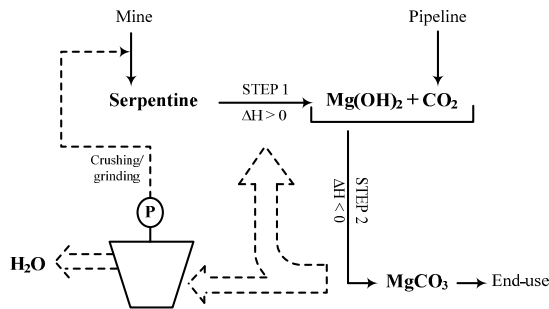


Figure 3 Schematic illustration of a staged gas-solid mineral carbonation process. Step 1 is endothermic while step 2 is exothermic and should provide for (part of) the necessary heat input for step 1.

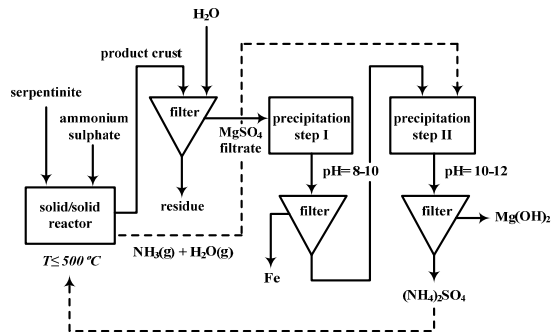


Figure 4 Schematic picture of the $Mg(OH)_2$ production from serpentinite rock [8,15].

Carbonation levels of ~50% of several 100 μm diameter $Mg(OH)_2$ particles had been obtained at ÅA within 10 minutes at pressures > 20 bar (up to 80 bar) and temperatures up to 600°C [13-17], see also below. The experimental setup for carbonation – see Figure 5 - consists of a heated vertical reactor vessel, operated as a fluidized bed with a sintered plate at the bottom (internal diameter 1.4 cm, height 40 cm), a CO_2 preheater, a cyclone for solid particle removal from the exiting fluid and the necessary measuring/controlling equipment for temperature etc.. As a source of CO_2 , a helium pressurized CO_2 bottle was used, allowing for pressures up to 120 bar (CO_2 becomes supercritical above 73.9 bar and 31.1°C).

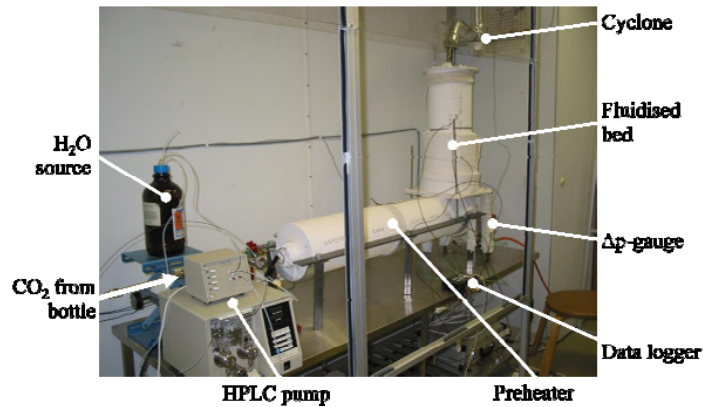


Figure 5 The $\text{Mg}(\text{OH})_2$ carbonation pressurized fluidized bed set-up at ÅA.

4. Results and Discussion

4.1. Production of $\text{Mg}(\text{OH})_2$ from serpentinite rock

The first step of indirect gas/solid serpentinite carbonation ($\text{Mg}(\text{OH})_2$ extraction) was performed with Lithuanian serpentinite both at KTU and at ÅA, but with Finnish serpentinite only at ÅA. The efficiency of Mg extraction at various reactor temperatures was tested. The results of experiments at ÅA for the Lithuanian rock are presented in Table 3. Values for pH for the sequential FeOOH and $\text{Mg}(\text{OH})_2$ precipitation were 8.5-9 and 10.5-11.5, respectively.

Table 3. Fractional amounts of extracted Mg and Fe (i.e. extraction efficiency) for Lithuanian serpentinite in reaction mixture produced at different temperatures [10] $\text{Mg}/\text{Fe} = 1.54 \text{ kg/kg}$

Time (min)	T (°C)	Serpentinite : Ammonium sulphate (g:g)	Mg extraction (%)	Fe extraction (%)
10	430	2 : 3	10.8	5.7
10	480	2 : 4	13.2	4.3
10	480	2 : 4	11.6	4.0
10	520	2 : 3	19.6	10.1
20	500	2 : 3	16.6	2.4
20	535	2 : 3	16.8	2.2
23	535	2 : 3	23.4	3.6

These experimental results show that increasing the reaction time or the temperature are important for the efficiency of Mg extraction. The highest extraction of Mg was achieved at temperature of 535 °C, and a solid/solid reaction time of 23 min.

These results obtained at ÅA were used to find optimal parameters for addition experiment of Mg extraction at KTU, with the aim to verify the influence of different reaction furnaces. Extraction of $\text{Mg}(\text{OH})_2$ was performed at a temperature of 535 °C and duration of 30 min. Substantial effort was paid for optimizing the reaction procedure, aiming at an optimal pH for precipitation of iron as well as achieving a high efficiency of pure $\text{Mg}(\text{OH})_2$ production, indicated by pure white deposits. The production of $\text{Mg}(\text{OH})_2$ initially resulted in somewhat peculiar results, producing material of yellow-ish shade, probably due to inefficiently precipitated iron during the first precipitation step – see Figure 4. This was later confirmed by XRD analysis which showed that besides $\text{Mg}(\text{OH})_2$ also magnesium iron silicate and magnesium iron silicate enstatite were present, noting that it is very unlikely that silicates passed the filter papers in the form of particulates. It was finally determined that the highest efficiency of Fe-removal is achieved at pH value of 9.4 (which is relatively high when compared to what was used for Finnish serpentinite).

After setting the optimal pH values, an amount of ~ 10 g of pure white Mg(OH)₂ was produced. The XRD spectrum identified a pure Mg(OH)₂ material. The samples were further analyzed with the aim to study the differences in properties and CO₂ sorption capabilities – see below. Specific surface area analysis revealed that the sample of produced pure Mg(OH)₂ has the higher (SSA) of 42.18 m²/g, while the one with iron impurities gave 28.11 m²/g [10].

At ÅA, Mg(OH)₂ extraction from Finnish serpentinite (besides several other magnesium silicate-containing rock types) was extensively studied [8-10]: a selection of experimental results is given in Table 4. As for the (later) tests with the Lithuanian and other samples, values for pH for the sequential FeOOH and Mg(OH)₂ precipitation were 8.5-9 and 10.5-11.5, respectively. The SSA of a Mg(OH)₂ produced from Finnish serpentinite was 46.85 m²/g [9].

Table 4. Fractional amounts of extracted Mg and Fe (i.e. extraction efficiency) for Finnish serpentinite in reaction mixture produced at different temperatures [8,9] Mg/Fe = 2.16 kg/kg. *A capita selecta* from a larger data set.

Time (min)	T (°C)	Serpentinite: Ammonium sulphate (g:g)	Mg extraction (%)	Fe extraction (%)
20	388	1 : 1	17.6	15.2
20	535	2 : 3	40.9	10.4
20	493	2 : 1	15.0	2.5
20	500	1 : 0.25	12.2	0
120	400	2 : 2.22	25.0	2.7
60	440	2 : 2.86	35.9	4.1
10	480	2 : 5	43.3	14
10	520	2 : 4	36.3	7.2
30	520	2 : 4	43.6	10.4
60	520	2 : 2.22	29.8	9.4
10	400	2 : 4	11.7	9.9
10	550	2.63 : 2	25.2	5.5
60	400	2 : 5	41.3	9.4
30	400	2 : 5	74.7*	35.6
60	400	2 : 2.86	87.2*	49.5
90	440	2 : 1.67	80.3*	21.8
30	480	2 : 2.22	73.4*	12.4
90	480	2 : 1.67	73.4*	18.3
30	440	2 : 4	71.1*	35.6

* Liquid sample analysis (ICP-OES) several months after the experiment

For the Finnish mineral, the most preferable conditions for extraction of Mg (and Fe) to MgSO₄ (and FeSO₄) are temperatures 400 – 440°C, with 30 – 60 minutes at S/AS = 0.5 – 0.7 kg/kg [10], with 60 – 66% extraction of Mg. Lower temperatures and longer times give a higher (relative) extraction of iron.

The data for Mg extraction from tests with five different magnesium silicate-containing rock types (excluding those marked * in Table 4) can be described by the modelling equation

$$Mg_{\text{extracted}} \text{ (kg / kg rock)} = \left[A \cdot \exp\left(-\frac{E_a}{RT}\right) \cdot t^m - \left(A \cdot \exp\left(-\frac{E_a}{RT}\right) \cdot t^m \right)^2 \right] \cdot \left(\frac{S}{AS} \right)^p \cdot \left(\frac{Mg}{Fe} \right)^q \quad (1)$$

for various minerals with different Mg/Fe (kg/kg) relative amounts. Here, t is time (s), T is temperature (K), S and AS are the amounts of mineral and ammonium sulphate, respectively (kg), and A, E_a, m, p and q are model parameters. For ~ sixty experiments with five different rock types, the model parameters found are A = 0.220 s^{-m}, E_a = 17903 J/mol, m = 0.344, p = - 0.794 and q = 0.362 [9], respectively. However, the R² of the model is 0.80. For the Finnish mineral only (Mg/Fe = 2.158 kg/kg) the result is much better: A = 2.035 s^{-m}, E_a = 29770 J/mol, m = 0.406, p = - 0.827, with R² = 0.99 [9]. Apparently, the Mg/Fe ratio alone is not sufficient for extending the kinetic model to a range of rock types.

4.2. Carbonation of the produced $Mg(OH)_2$

The produced $Mg(OH)_2$ from Lithuanian “Lt” and Finnish “Fi” serpentinite were further carbonated in the pressurized fluidized bed reactor at ÅA as shown in Figure 5. Also tests were made with a synthetic $Mg(OH)_2$ sample, “Sy” commercially available from Dead Sea Periclase (SSA ~ 5 m²/g). Results from the tests are shown in Table 5. Note that quite many carbonation tests were done with the synthetic sample “Sy” [13-17]: here only those that gave > 30% conversion to $MgCO_3$ are given.

The temperature of the carbonation reaction was 490-540°C. Conversion degrees ($Mg(OH)_2$ carbonation to $MgCO_3$ (which was determined by comparing the amount of CO_2 released from the sample, to that which would be obtained for a 100 % pure carbonate of the same weight, [18]) appeared to be very similar for the “Lt” and “Fi” samples tested at 20 bar. The sample containing iron and silicate impurities (#2 in Table 2) had the lowest conversion degree (45%) of the “Lt” samples. The lower conversion efficiency may have occurred for several reasons. First, the SSA analysis revealed a two times smaller SSA compared to the more pure sample. Also, lower purity gives a lower carbonation potential as a result of binding of available Mg to non-reactive complexes.

The duration of experiments is rather short and this shows that pressurized fluidized bed reactor can be operated successfully to rapidly carbonate $Mg(OH)_2$. No difference was noticed between conversion degrees during 9-minute or 3-minute tests. The results also showed no sensitivity to the type of the reactor for heating “Lt” serpentinite with ammonium sulphate mixture, which is not completely surprising since in the end $Mg(OH)_2$ is precipitated from an dissolved $MgSO_4$ -containing aqueous solution.

Similar experiments were made with Finnish serpentinite-derived $Mg(OH)_2$ and the synthetic sample. The synthetic sample performed significantly worse than the $Mg(OH)_2$ produced from the serpentinites, presumably as a result of both a much (10×) lower SSA and a lower purity. The reason why no carbonation degrees higher than 55% have been reached yet is currently under investigation at ÅA – see [13].

Table 5. Pressurised fluidized bed carbonation results using several $Mg(OH)_2$ samples (see also [13-17])

Sample of $Mg(OH)_2$	Sample size (g)	Mean particle size (µm)	Experiment time (min)	Temperature (°C)	CO_2 pressure (bar)	Fluidisation velocity (cm/s)	Conversion degree (%-wt)
Lt1*	3.8	254.5	9	490	20	15.3	52
Lt2*	4.2	255.9	9	490	20	16.0	45
Lt3*	4.0	168.3	9	490	20	12.2	54
Lt4*	4.0	168.3	3	490	20	12.7	53
Fi1	4.0	337.5	10	500	20	12.2	48
Fi2	3.8	337.5	10	500	20	15.1	53
Fi3	3.1	187.5	10	500	20	5.8	32
Sy1	20.0	275	15	500	28	14.1	35
Sy2	4.0	168.5	10	525	35	16.2	46
Sy3	4.0	168.5	10	540	35	6.5	39
Sy4	4.0	168.5	10	525	40	6.2	45
Sy5	4.0	168.5	10	540	40	7.4	31
Sy6	4.0	168.5	10	525	45	6.2	44
Sy7	4.0	168.5	10	510	35	6.6	37

* Lt1 = highly pure sample prepared at KTU, Lt2 = Fe and Si containing sample prepared at KTU, Lt3, Lt4 = highly pure samples prepared at ÅA

5. Conclusions

While two serpentinite rock samples from Lithuania and Finland appear very similar (based on XRD), they have different magnesium and iron contents. This affects the optimal conditions for the production of $Mg(OH)_2$ (and iron oxide by-product). Experiments show that for Mg extraction, the Finnish rock is more reactive (requiring a lower temperature) than the Lithuanian one. $Mg(OH)_2$ reactivity appears to be largely determined by specific surface and the fluidisation velocity in the PFB reactor, and are similar for the two

samples. Reaction times for reaching more than 50% carbonation are of the order of 10 minutes for 170–340 μm particles.

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