

Article

Geological Mapping and Characterization of Possible Primary Input Materials for the Mineral Sequestration of Carbon Dioxide in Europe

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Abstract: This work investigates the possible mineral input materials for the process of mineral sequestration through the carbonation of magnesium or calcium silicates under high pressure and high temperatures in an autoclave. The choice of input materials that are covered by this study represents more than 50% of the global peridotite production. Reaction products are amorphous silica and magnesite or calcite, respectively. Potential sources of magnesium silicate containing materials in Europe have been investigated in regards to their availability and capability for the process and their harmlessness concerning asbestos content. Therefore, characterization by X-ray fluorescence (XRF), X-ray diffraction (XRD), and QEMSCAN[®] was performed to gather information before the selection of specific material for the mineral sequestration. The objective of the following carbonation is the storage of a maximum amount of CO₂ and the utilization of products as pozzolanic material or as fillers for the cement industry, which substantially contributes to anthropogenic CO₂ emissions. The characterization of the potential mineral resources for mineral sequestration in Europe with a focus on the forsterite content led to a selection of specific input materials for the carbonation tests. The mineralogical analysis of an Italian olivine sample before and after the carbonation process states the reasons for the performed evaluation. The given data serves as an example of the input material suitability of all the collected mineral samples. Additionally, the possible conversion of natural asbestos occurring in minerals as a side effect of the carbonation process is taken into consideration.

Keywords: olivine; mineral sequestration; geological mapping; mineral deposits; characterization

1. Introduction

The increasing greenhouse gas (GHG) emissions into our atmosphere lead to a serious threat to our future. Especially, the concentration of CO₂, volume-based the most important GHG, is significantly and continuously rising. There are different approaches to reduce CO₂ emissions or to store it by carbon capture and storage (CCS). Another approach is carbon capture and utilization (CCU), the separation of CO₂ out of exhaust gas flows, and the subsequent use in chemical processes. One possible approach of CCU is the mineral sequestration of CO₂. Suitable minerals are mainly calcium or magnesium silicates, which form insoluble carbonates (mainly calcite and magnesite) and silica in an exothermic reaction. This reaction continuously takes place as a natural process of weathering in geological timeframes.

It can be accelerated with high temperature and high pressure in an autoclave [1]. The produced carbonates are stable and will only degas at temperatures above 400 °C [2].

CO₂ sequestration by these minerals can be an opportunity for CO₂ intensive industries, like the cement industry. Due to the mineral composition of limestone, ~500 kg CO₂ are emitted for each ton of cement clinker produced [3]. The goal is the utilization of the emitted CO₂ from cement clinker production processes through CCU and the substitution of primary materials in cement or concrete with the produced carbonate and silica. Possible applications can be, e.g., adding value to concrete mixes as filler material, or partly replacing the pozzolanic supplementary cementitious materials in cement.

To implement the mentioned process, the selection of suitable input material is necessary for ensuring an optimal process flow. Part of this paper is the mapping of possible minerals as input materials in Europe and the characterization of these materials to evaluate the possible use for CCU.

The CO₂MIN project target is to find and characterize the possible feedstock material for mineral sequestration of CO₂, find the optimum conditions for maximum carbonation degree of magnesium silicates and to separate the products of the carbonation reaction to evaluate the possible applications in the cement industry. A theoretical model is created for the evaluation of the process. Besides, a social-economic analysis and a Life Cycle Assessment (LCA) are performed to define the success of the process regarding the carbon footprint and the economic feasibility. After the performance of a sufficient amount of large-scale experiments, their data will allow for global projections of the process capability. The aim of this paper is to provide an overview of the potential of possible feedstock for mineral sequestration of carbon dioxide. Therefore, samples from different European countries have been taken into consideration, mainly from deposits that produce the majority of the European peridotites. Firstly seven producers of peridotites have delivered a 10 kg sample to have a cross-section of peridotites in Europe. After pretreatment of the possible input material and its characterization, a preselection of samples has been made for the carbonation tests. These tests were carried out in an autoclave with high pressure, high temperature and the addition of additives.

2. Materials and Methods

Olivine is no single, clearly defined mineral, but a group of minerals. They are minerals in the series of fayalite-forsterite with their end-members calcio-olivine and tephroite, whereas forsterite is a magnesium silicate and fayalite is an iron silicate. The most common olivine of this solid solution series has a magnesium-iron ratio of 1.6:0.4, with a formula of Mg_{1.6}Fe²⁺_{0.4}(SiO₄). It is of intermediate composition and it has an olive-green color, from which the name is derived [4].

Olivine has two main occurrences:

1. Dunite, which is an ultramafic rock that contains a minimum of 90% olivine with additional minerals, like ortho- and clinopyroxenes, as well as serpentine group minerals, magnesite, chromite, ilmenite, rutile, and magnetite. Additionally, in small volumes, plagioclase, garnet, and spinel can be found in dunites.
2. Serpentinite, a metamorph rock that was built through serpentinization, i.e., through transformation and alteration of ultramafic rocks, which originally contained large amounts of olivine. Serpentinites are mainly composed of serpentine minerals, like chrysotile, clinochrysotile, orthochrysotile, lizardite, antigorite, and others [5].

There are many small deposits of olivine bearing rocks, but only a limited number of deposits represent manufacturing units due to economic factors.

Olivine bearing rocks are normally found within the following geological settings:

- Ultramafic intrusions (e.g., Norway, Germany);
- Ophiolite complexes (e.g., Greece, Italy, Turkey);
- Alpine peridotites (ophiolite or subcontinental mantle) emplaced along thrust faults (e.g., Italy, Spain);

- Rift zones/basalts of mid-ocean ridges (e.g., Iceland);
- Volcanic xenoliths (e.g., German Eifel and Kaiserstuhl, Iceland).

The big deposits normally occur in settings of ultramafic intrusions and ophiolite complexes [6,7].

For possible usage of olivine bearing rocks for sequestration, samples from different European countries, namely Norway, Spain, Italy, Turkey, and Greece, and one sample from Japan, have been collected and investigated regarding their MgO content and phase composition. Besides, German Basalt has been tested due to local proximity. The investigated deposits are marked in Figure 1. As a first investigation step, each producer has delivered one compounded sample of 10 kg. The material has been crushed by jaw crusher, milled by roller mill, sampled, and sieved to produce a fraction below 63 μm for analysis and three different fractions for carbonation tests, as it can be seen with Stopic et al. [1].

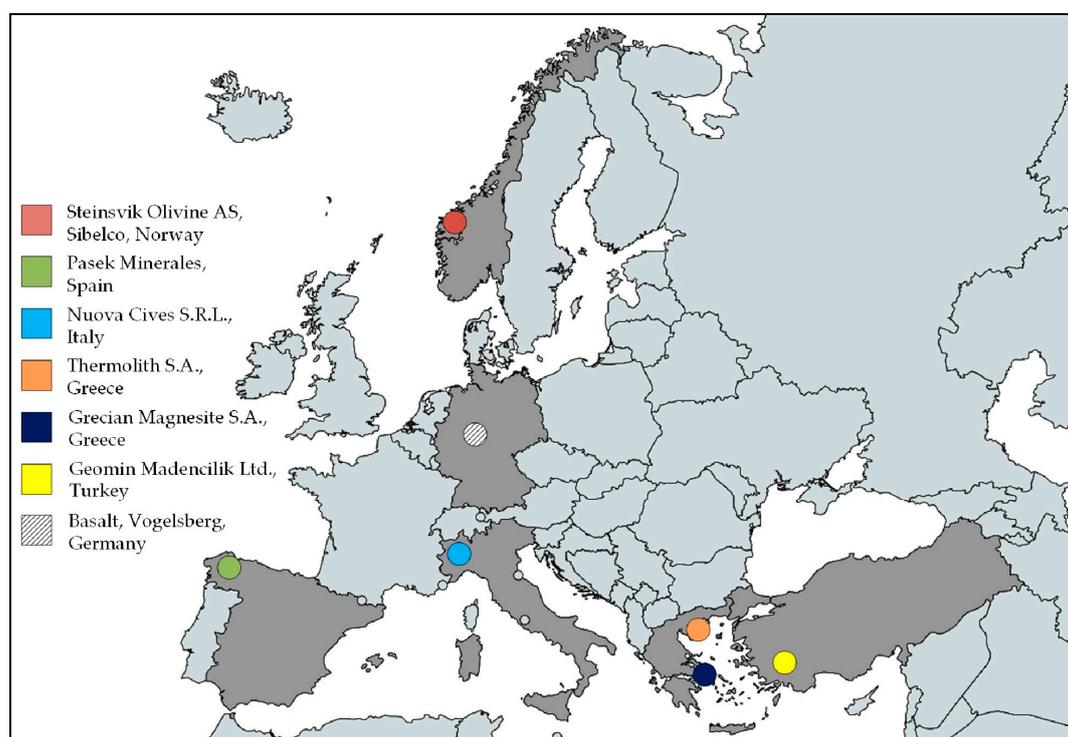


Figure 1. Origin of samples tested for the use in the carbonation process [8].

The main deposits, which are part of the investigation for CO₂MIN project, will be presented below and its locations are marked on the map in Figure 1 with the name of the producing company. The global production numbers of peridotites can be found in Figure 2.

Norway, Spain, Greece, Italy, and Turkey are major olivine producing countries in Europe, with a total amount of between 4200 to 5400 ktpa, which represents more than 50% of the global olivine production of 7800 to 9000 ktpa (Figure 2). The production numbers differ due to published data from O'Driscoll [6] from 2004 and the current numbers from the producers of the investigated samples. The annual production of the seven peridotite producers in Europe plus the Horoman Mine in Japan, which were examined in the CO₂MIN project account for 4595 ktpa, which is between 51% and 58% of the global peridotite production.

Norway is the most important producer of olivine in the world, with a production between 2525 and 3500 ktpa (Figure 2). The material is directly mined as a target material/product. Many deposits are already explored, but not or only periodically mined due to the supply from Aheim Gusdal Mine (mainly mined from Sibelco, but also from Steinsvik) is sufficient for the market. For this reason, only the Aheim Gusdal Mine will be presented in this paper. It is a large olivine deposit situated in the

Almklovdaalen peridotite massif, a large Alpine-type ultramafic body emplaced within the granitoid gneisses of the Western Gneiss Region. The massif contains serpentized ultramafics with a core of dunite. This dunitic core has a large-scale dunite zone with a thickness of up to 300 m with chlorite bearing dunites, as well as 1 to 20 m thick zones of eclogite and chlorite-amphibole dunites [9]. For this paper a sample from Sibelco and from Steinsvik has been investigated but the Steinsvik material has been selected for further examinations.

Different companies mine the deposit, but the biggest mine, the Gusdal olivine pit, produces about 2 ktpa. With this production rate, the proved reserve will last for 150 years with a potential property life of more than 400 years, which makes it the largest commercial olivine deposit in the world. Other mines that are currently producing are located in Steinsvik and Bjørkedal [9].

Olivine bearing rocks in Greece are mainly mined in the north, in Western Macedonia, Northern Euboea (Grecian Magnesite S.A.), the central mainland, and in Chalkidiki (Thermolith S.A.). Most Greek deposits are of industrial interest mainly because of its chromite and PGE (platin-group elements) content utilizing peridotites as by-products.

Vourinos ophiolite complex, a fragment of oceanic lithosphere, is located in Macedonia between Kozani and Grevena. The deposit/complex contains four distinct peridotite varieties from harzburgite to fine and coarse-grained dunite [10].

In central Greece, harzburgite deposits can be found in the Megaplatanos ultrabasic body, which is part of the western Neotethyan ophiolitic belt, situated along the western margin of the Pelagonian zone of Greece, belonging to the Sub-Pelagonian zone. The primary minerals of this harzburgite deposits are olivine (forsterite/fayalite), with 65–80 vol. %, orthopyroxene (15–30 vol. %), clinopyroxene (3–5 vol. %), and Cr-spinel (<5 vol. %). The dominant secondary phase is serpentine with a range of 5 up to 70 vol. % [11].

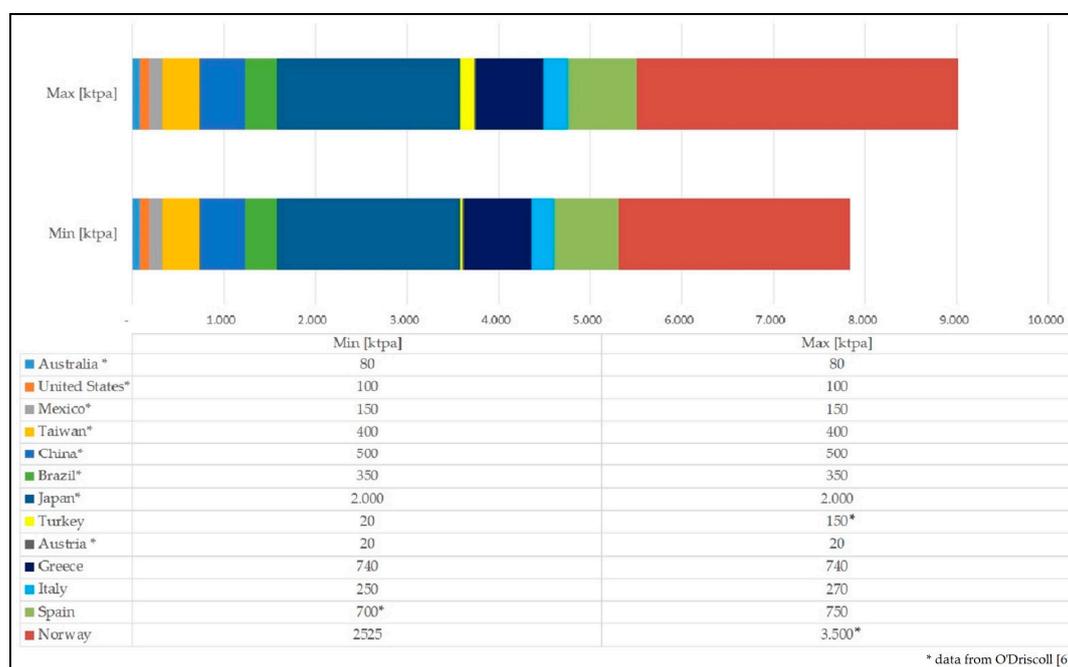


Figure 2. World production of peridotite (Current numbers for the production from the producers of the investigated samples (Figure 1); *: Data from O'Driscoll [6]).

Spain is one of the main producers of olivine bearing dunite. In northwestern Spain (La Coruna District), the proven reserves are estimated at 600 Mt (stated by Pasek Minerale). Ultramafic massifs are located in Sierra de la Capelada and Cabo Ortegal in the Herbaria massif. Ultrabasic peridotites occur in Sierra de Ronda near Málaga [12]. This Ronda peridotite massif is formed of different groups of

mafic rocks, namely lherzolites, olivine-rich lherzolites, harzburgites, and dunites [13]. In comparison to the Herbaria massif, the industrial production of dunite is not of importance at Ronda.

The ultramafic rocks of the Herbaria massif can be described as a heterogeneous enriched upper mantle, where the ultramafic section represents a wedge of lithospheric mantle above a subduction zone, whereas the initial subduction was interoceanic. The dunite has primary mineralization of olivine, orthopyroxenes, and clinopyroxenes and secondary mineralization of Ca-amphiboles classified as hornblendes and serpentines, which derived from different geological processes, like hydrothermal alterations that are promoted by seawater contact [14].

In Italy, olivine/dunite is mined by Nuova Cives S.R.L. from a deposit in the north near Turin in the municipalities of Castellamonte, Vidracco and Baldissero Canavese. This deposit's expected reserves contain about 100 Mt of dunites, with grades of 95% to 97% of olivine [5]. The mafic-ultramafic Ivrea-Verbano Zone in Northwest Italy bears several peridotite massifs, namely Finero, Balmuccia, and Baldissero. Additionally, there are two open-pit mines currently producing: "Bric Carleva" with ~300 Mt resources and "Finero" with ~250 Mt reserves (stated by Nuova Cives SRL).

Dunite rocks and harzburgites are the main sources of olivine in Turkey. There are many Mesozoic ophiolite and peridotite complexes along the suture zones. The important chromite, magnesite, and olivine deposits in Turkey contain peridotite host rocks that consist of harzburgite, verlite, lherzolite, or dunite. Often alteration/serpentinization has taken place within some parts of the peridotite. [15] There are many deposits with a significant amount of olivine bearing rocks, but only a few deposits are mined to date, due to economic and location aspects.

There are no deposits for peridotites in Germany, but some deposits with lower olivine contents mainly in the areas of former volcanic activity as the Eifel, the Rhoen and the Northern Hessian Depression. The olivine occurs as gabbros and in corresponding gangue rocks, dolerites, in olivine bearing basalts, olivine nephelinite, "olivine-bombs", and as xenoliths. The "olivine bombs" in the Eifel can contain nearly pure olivine, whereas the olivine basalts and basanite normally contain up to 10% or a minimum of 10% of olivine, respectively [16].

One sample from Japan has also been analyzed as Japan is the second-largest producer of peridotites for comparison of European olivine sources with worldwide deposits. The sample comes from the Horoman Mine in the Hidaka Province in Hokkaido.

Chemical and mineralogical characterization steps are conducted to establish a basis for a proper assessment of the suitability of primary raw materials from various deposits. To provide the background behind the evaluation of the collected mineral samples as proper input materials for the carbonation in an autoclave, the mineralogical composition of an Italian olivine sample before and after the process serves as an example and it is presented in more detail. Nevertheless, the full extent of process-related parameters as well as further information on both the autoclave reactor and executed experiments are mentioned in prior publications focusing on the process performance [1,17].

3. Results—Characterization

Olivine bearing samples from Norway, Greece, Italy, Turkey, Japan and basalt samples from Germany have been analyzed by XRF and qualitative and semi-quantitative XRD. As in the reaction for the sequestration of carbon dioxide (Figure 3), magnesium silicate reacts with carbon dioxide to magnesite and silica, the magnesium oxide content of the samples has been used as an indicator for their carbonation potential.

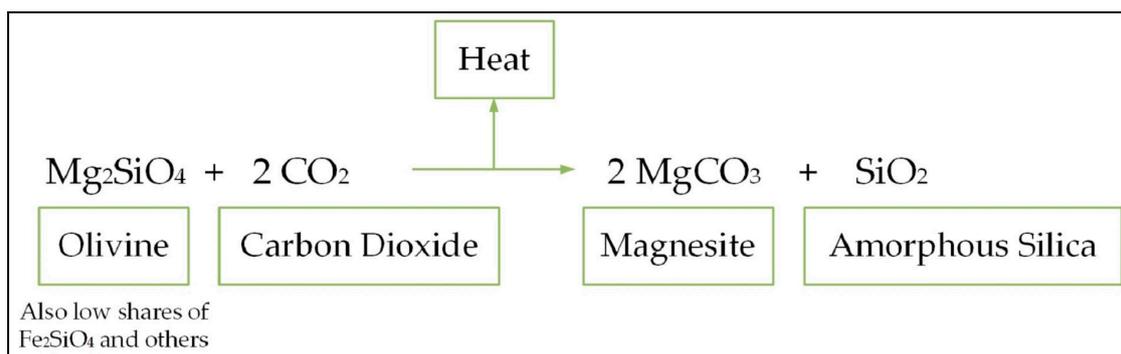


Figure 3. Formula for the exothermic reaction of olivine (forsterite) with carbon dioxide (mineral sequestration).

3.1. Analysis of the Chemical Composition

Before the analysis of the olivine bearing samples of different origin and basalt samples by X-ray fluorescence (XRF) technique using a PW2404 device (Malvern PANalytical B.V., Eindhoven, The Netherlands), the given mineral samples were fused to glass disks by a Claisse LeNeo instrument (Malvern PANalytical B.V., Eindhoven, Netherlands). The respective deviations of the determined values are not further mentioned to enhance the readability since the measuring inaccuracy of the resulting values resides below 0.3% for main components and 1.0% for trace elements. As preparation method grinding by roller mill and wet sieving to a fraction below 63 µm have been used to produce a narrow and precise particle range.

As a first indicator to decide whether the examined minerals exhibit promising carbonation potential, the chemical composition (Table 1) may be compared: SiO₂, Fe₂O₃, and MgO are the main components for all olivine samples analyzed. Furthermore, the share of these major constituents is fairly even with detected amounts between 46–50 wt. % for silica, 7–10 wt. % for ferric oxide and 35–43 wt. % for magnesia. Both the CaO and Al₂O₃ content of all olivine samples fall below 1 wt. %, except for the Italian mineral, which exhibits a value of 2 wt. %. Further components as TiO₂, K₂O, Na₂O, and NiO do not significantly exceed detected amounts of 1 wt. %.

The German Basalt sample exhibits a comparable composition regarding SiO₂ and Fe₂O₃. Since the CaO content of about 10 wt. % does not compensate for the comparatively low MgO amount, both components considered to function as educts for the carbonation process [18] are underrepresented in this comparison.

Table 1. Chemical composition of the examined mineral samples.

Component (wt. %)	Olivine					Basalt
	Italy	Norway	Greece	Turkey	Japan	Germany
SiO ₂	47.5	48.6	46.2	47.4	50.5	49.7
Al ₂ O ₃	2.6	0.5	0.4	0.4	0.8	13.0
Fe ₂ O ₃	10.5	7.8	8.4	9.6	8.1	10.5
TiO ₂	0.1	0.0	0.0	0.0	0.0	1.5
CaO	2.1	0.2	0.5	0.4	0.8	9.8
MgO	35.3	41.1	43.6	40.3	38.0	9.0
K ₂ O	0.2	0.1	0.1	0.0	0.0	1.4
Na ₂ O	0.1	0.0	0.0	0.1	0.1	4.4
NiO	1.0	1.2	0.2	1.2	1.0	0.0

3.2. Examination of the Present Mineral Phases

X-ray diffraction (XRD) method has been applied using a Bruker D8 Advance device in Bragg–Brentano geometry equipped with LynxEye detector (Bruker AXS, Karlsruhe, Germany),

CuK α tube, and nickel filter, in order to investigate the mineralogical composition. The chosen XRD parameters include a measurement range from 5–90° 2 θ in 0.02° steps at 2 s per step.

To enable a comparison of the present mineral phases, as indicated by the components detected via the XRF technique. As this study focusses on natural resources, which are subject to fluctuations, the performance of a semi-quantitative analysis of the mineral phase composition is considered to be sufficient for the aimed process suitability evaluation.

Within the given accuracy range, Table 2 provides information regarding the main mineral phase fractions, as detected in the olivine samples, which includes forsterite, enstatite, lizardite, and, in case of the Italian olivine sample, tremolite.

Unlike the analysis of the chemical composition, the examination of the present mineralogical phases reveals significant differences, which were primarily caused by the presence of larger enstatite fractions. The largest shares of forsterite are found in olivine samples from Norway, Turkey, and Japan (75–80% each); the Greek and Italian olivine samples exhibit 65–70% and 55–60% of forsterite, respectively. Therefore, all of the examined olivine samples constitute promising potential in the carbonation process. For purposes of comparison, the mineralogical composition of the German basalt sample is taken into account additionally, but the comparatively low magnesia amount given in the XRF results also reflects in a rather small forsterite fraction of 10–15%.

Table 2. Mineralogical composition by semi-quantitative examination of the present mineral phase fractions.

Mineral Phase Fraction (%)	Olivine					Basalt
	Italy	Norway	Greece	Turkey	Japan	Germany
Forsterite	55–60	75–80	65–70	75–80	75–80	10–15
Enstatite	15–20	10–15	25–30	10–15	20–25	-
Lizardite	≤5	≤5	≤5	≤5	-	-
Tremolite	10–15	-	-	-	-	-

In a complementary step of the present study, first carbonation experiments have been conducted, autoclaving a previously milled and sieved olivine bearing sample material from Italy within a fraction of 20–63 μm . For these tests, a rocking batch autoclave was operated at 175 °C and 100 bars in an aqueous solution (S/L ratio of 0.2 g/ml) and a CO₂-rich gas phase from 0.5 to 12 h [1]. Figure 4 compares the X-ray powder diffractograms of the initial olivine sample (l.) and the product (r.). Apparently, the main mineralogical phase changes from forsterite to magnesite, whereas the further components undergo only minor modification due to the autoclave process. The presence of magnesite after processing indicates the successful carbonation of the input material.

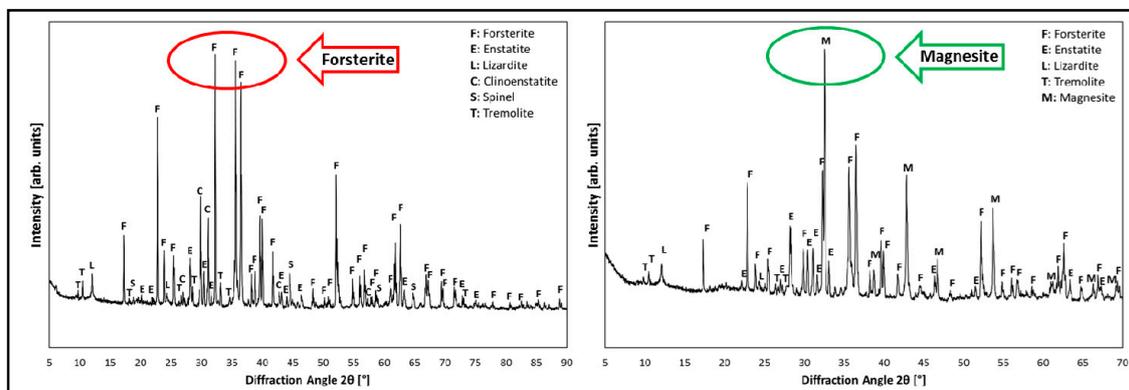


Figure 4. X-ray diffraction (XRD) analysis of Italian olivine sample; initial state (left) and carbonation product (right) [1].

To highlight the contained mineral phase fractions, a semi-quantitative analysis of the X-ray powder diffractograms, as illustrated in Figure 4, is performed by pattern fitting via “*DiffraSuite Topas*” software (version 5.0, Bruker AXS, Karlsruhe, Germany): As presented in Table 3, the fractions of enstatite and lizardite are found to be rather unaffected by the carbonation process, but the amount of forsterite that is present in the semi-quantitative examination has decreased in the order of approximately 20% after the reaction with carbon dioxide, which facilitates the formation of magnesite. The formed fraction of magnesite, about 30%, is also based on the conversion of tremolite, which shows a decrease of approximately 10%.

Table 3. Semi-quantitative examination of the present mineral phase fractions in Italian olivine before and after carbonation.

Mineral Phase Fraction (%)	Olivine (Italy)	
	Input Material	Carbonation Product
Forsterite	55–60	35–40
Enstatite	15–20	15–20
Lizardite	≤5	≤5
Tremolite	10–15	≤5
Magnesite		25–30

Tremolite bears certain risks to health due to its possible occurrence as respirable fibers. Tremolite is characterized by repeating double chains of silica tetrahedral, which is causative for its fibrous habit [19]. The fibrous form of the amphibole tremolite is considered to be of aggravated risk due to its raised bio-persistence compared to chrysotile, which exhibits enhanced bio-solubility in direct comparison. [20,21] While considering these facts, a potential decomposition of natural asbestos could be a significant side effect of the carbonation process. As stated by Ryu et al., the direct aqueous carbonation of tremolite fibers, forming reaction products as calcite, and the triggered morphological modification to a rhombohedral habitus may reduce or even eliminate the hazardous character of tremolite [22].

Still, the investigation by Scanning Electron Microscopy (LEO Type 440i, Leo Electron Microscopy, Cambridge, UK) reveals acicular material (Figure 5), which makes the careful handling of these materials indispensable. It is also indicated that the given carbonation parameters are not capable of enabling a complete reaction process from tremolite to magnesite. Nevertheless, fibrous tremolite has not been observed in the Italian olivine samples, which does not exclude this material from subsequent processing and analysis steps.

The results of the quantitative modal determination via QEMSCAN[®] are shown in Figure 6, enabling a phase assignment to olivine, orthopyroxene, and clinopyroxene (Table 4), which leads to a classification of the investigated samples via QAPF (Quartz, Alkali Feldspar, Plagioclase, Feldspathoid diagram).

Table 4. Quantitative analysis by QEMSCAN[®] of four different samples of olivine bearing rocks from Europe.

Mineral Phase Fraction	Vol. % in Sample			
	Thermolith	Grecian Magnesite	Steinsvik	Nuova Cives
Olivine	67.36	52.21	79.80	59.08
Orthopyroxene	25.93	18.86	11.98	18.98
Clinopyroxene	2.23	3.38	0.89	8.76
Others	4.48	25.55	7.33	13.18

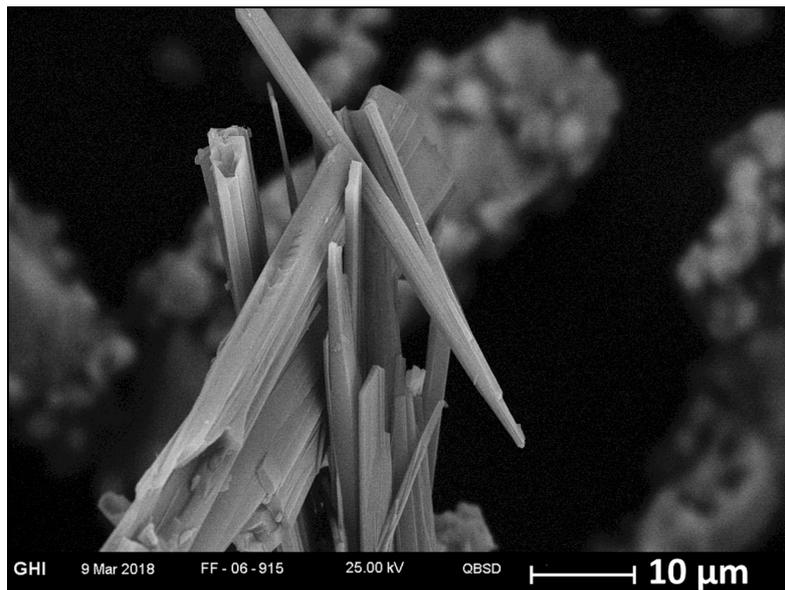


Figure 5. Acicular tremolite as found within the microstructure investigation of an olivine sample after carbonation.

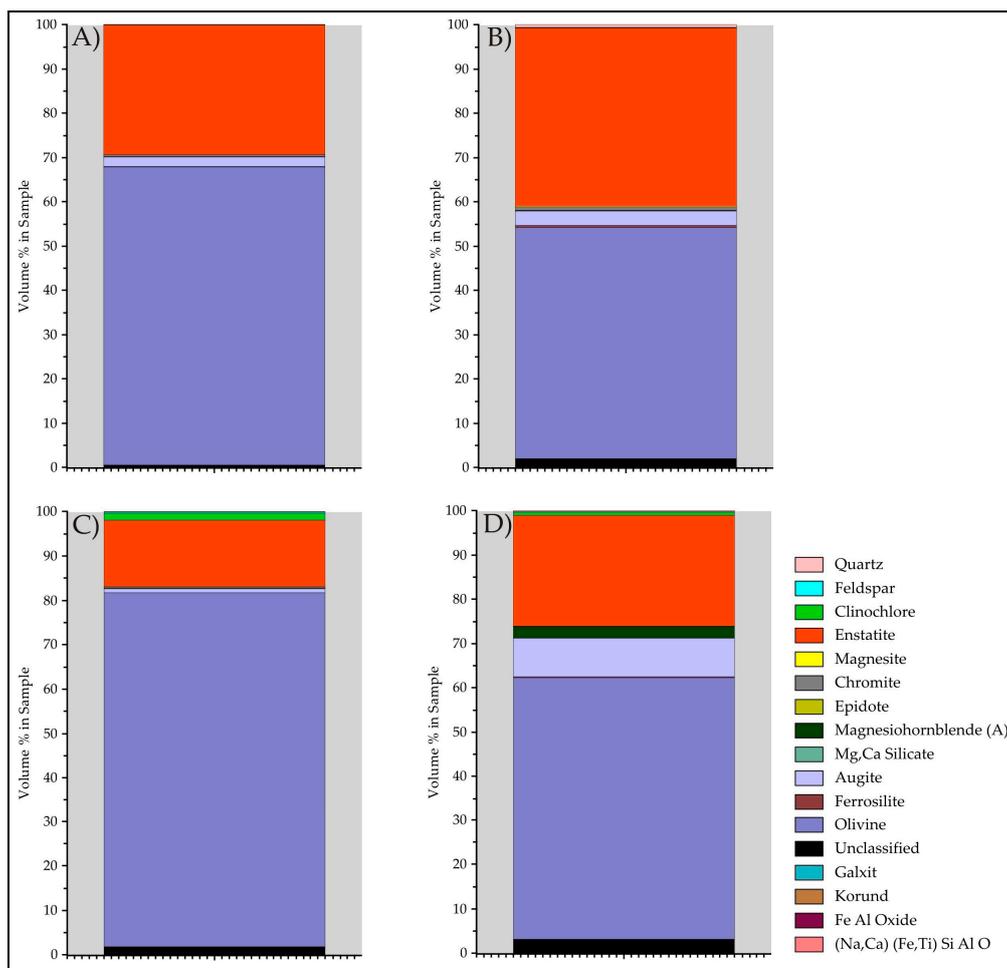


Figure 6. Quantitative Modal determination by QEMSCAN® (A) Thermolith (B) Grecian Magnesite (C) Steinsvik (D) Nuova Cives.

Mineralogically, the olivine bearing materials tested for the CO₂MIN project are neither pure olivine nor dunites, but peridotites, more precisely harzburgites or lherzolites (Figure 7).

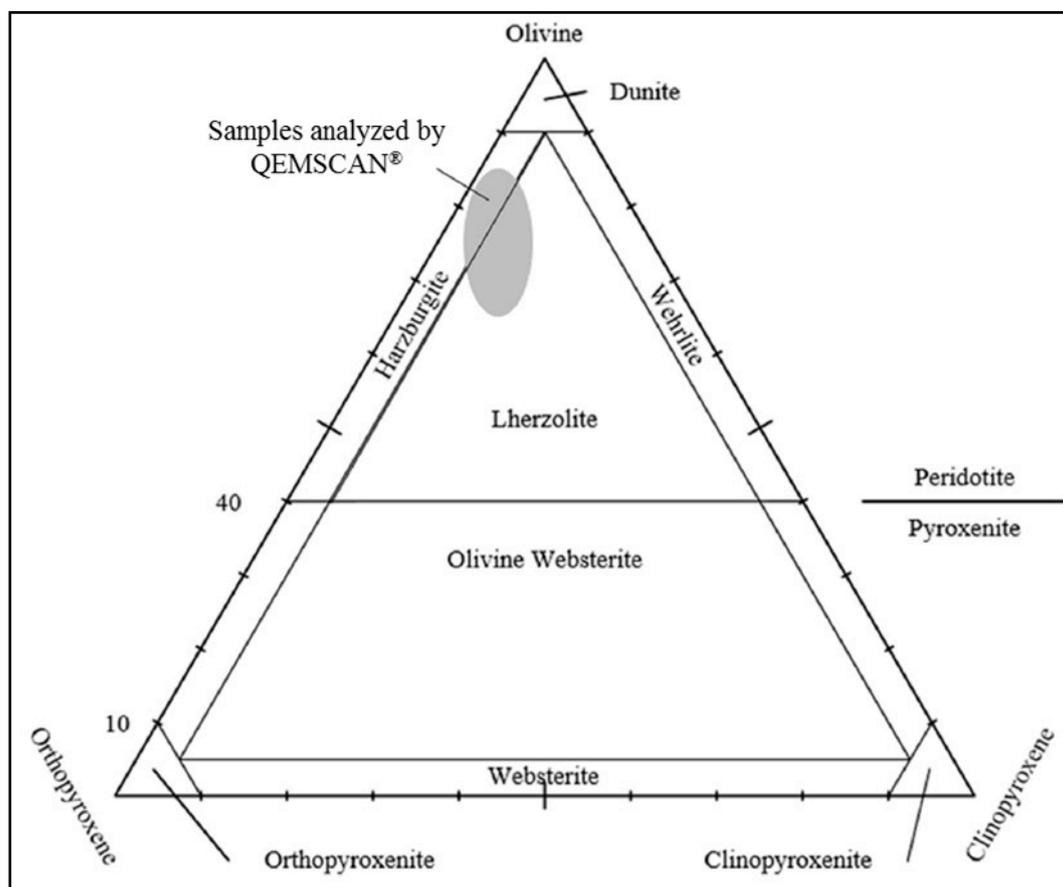


Figure 7. Classification of ultramafic rocks (QAPF) based on the proportion of olivine, orthopyroxene, and clinopyroxene [23].

4. Discussion

As shown by the example of olivine samples within the present study, many parts of Europe, and also Asia, possess mineral resources that are capable of significant carbon dioxide sequestration. Vicariously for regions not exhibiting such olivine deposits, a basalt sample from Germany was also taken into consideration, which merely contained minor fractions of magnesia-rich phases that were found to have larger relevance within the evaluation of the minerals' carbonation potential. Nevertheless, countries, such as Germany, exhibit secondary resources as steel slags, fly ashes, and filter ashes bearing potential for CO₂ sequestration due to their high CaO content. Kelemen et al. have pointed out that mantle peridotite is capable of consuming more than one billion tons of CO₂ per year, in Oman alone, forming reaction products as MgCO₃ and CaCO₃ by an industrial acceleration of natural peridotite weathering [18].

Which of the magnesia-rich phases constituting the main parts of the examined olivine samples, forsterite or enstatite, exhibits the better performance within the carbonation process and whether the mere presence of further mineral phases affects the overall carbonation potential, has to be investigated in future studies. Although the complete carbonation of a synthetic magnesia material to magnesite in a previous step of the study proves the capability of the process, the effects hindering or slowing down the carbonation reaction of magnesia containing phases as Enstatite have to be understood in detail. [1] However, at this stage of the project, the first results that were gathered by Stopic et al. are pointing to forsterite as the more reactive mineral phase within the carbonation process, as shown

by the processing and examination of e.g., Norwegian and Italian olivine samples [1,17]. The next steps are the comparison of the products after carbonation of selected input material, separation of the carbonation products, and also the investigation of residual materials, like slags, fly ash, and red mud as the possible input materials for the sequestration process.

Author Contributions: D.K. performed the geological mapping and was responsible for the preparation of the olivine materials (grinding, sieving) and co-wrote the paper; S.E. supervised and evaluated the XRF- and XRD-analyses and co-wrote the paper with R.T.; J.B. investigated the geology of olivine deposits; P.B. co-wrote the paper, K.M.H. procured the samples for lab tests and characterization and co-wrote the paper; H.W. co-wrote the paper.

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Conflicts of Interest: The authors declare no conflict of interest.

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