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INVESTIGATIONS OF THE SUITABILITY OF K-FELDSPAR MODIFIED BY MILLING FOR CO_2 SEQUESTRATION

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Abstract: Nowadays, carbon dioxide (CO_2) emissions are one of the main factors of global warming and climate change. Controlling CO_2 levels in the atmosphere and limiting global warming requires urgent action. Some minerals can be used to capture and store CO_2 from the air or other sources. Mechanochemically modified K-feldspar (with KOH, $Ca(OH)_2$, and CaO) was used for *ex situ* and *in situ* CO_2 capture. In the *ex situ* experiment (in a thermoanalytical apparatus at 150 °C, 5 h), infrared spectroscopy indicated that mechanochemically modified K-feldspar was capable of CO_2 sequestration via carbonate formation. The *in situ* CO_2 capture experiment consists of two steps. The first step involved the mechanochemical modification of K-feldspar using $Ca(OH)_2$ and CaO as additives during milling. The second step consisted of direct *in situ* CO_2 sequestration in the milling chamber. X-ray diffraction patterns demonstrated the formation of the calcite phase, and thermal analysis confirmed the decomposition of such created calcite. Elemental analysis has found the binding of approximately 1.6% of carbon, and 5.23 % carbonation ratio of modified feldspar was achieved. In addition, the use of the mineral vermiculite as a natural additive for *in situ* sequestration of K-feldspar was investigated using the above-mentioned analytical techniques.

Keywords: *K-feldspar, vermiculite, mechanochemical modification, milling, CO_2 sequestration, mechanochemical carbonation*

1. INTRODUCTION

It is well-known that CO_2 is the most abundant of the greenhouse gases and is, therefore, the largest contributor to the greenhouse effect. In the last decade, the need and various activities to reduce CO_2 emissions to pre-industrial levels have greatly intensified (What is carbon neutrality and how can it be achieved by 2050?, [Online]).

Mineral carbonation is a natural weathering process in which alkaline earth metals, mainly Ca and Mg react with CO_2 to form stable carbonates. These reactions are exothermic, but in nature, they take place slowly during the weathering of silicate minerals (Pachauri and Reisinger 2007; Seifritz 1990). O´Connor and coworkers (2002) developed an aqueous process of direct carbonation of silicate minerals (olivine, serpentine, enstatite) using pressure and temperature above 150°C as a method for CO_2 storage in solid form. Wang et al. (2014) studied carbonation using natural K-feldspar calcined with phosphogypsum. The first attempts to use mechanical activation (high-energy milling) of various silicate minerals for CO_2 sequestration were performed already 20 years ago (Kalinkin et al. 2003, 2004; Kalinkina et al. 2001a, b). Later, Turianicová and coworkers (2013a, 2014) investigated the carbonation of olivine and vermiculite using mechanical activation. It is known from the literature that mechanical activation causes particle comminution, increases the specific surface area of the minerals and even breaks their crystal structure by the formation of lattice defects, which increases their overall reactivity in subsequent reactions (Baláž 2008).

K-feldspar, a mineral with the specific composition $KAlSi_3O_8$, is a member of the aluminosilicate group. It is widely distributed and abundant in various regions worldwide, including China and Türkiye. Its unique properties make it a significant resource for various industries such as ceramic. Additionally, K-feldspar minerals are emerging as potential candidates for carbon dioxide (CO_2) capture (Guo et al. 2015). For intensification of its carbonation process various additives such as gypsum - $CaSO_4 .2\ H_2O$ or $CaCl_2$ slag were used (Wang et al. 2014; Ye et al. 2014). In order to initiate the chemical reaction of refractory K-feldspar and to introduce alkaline earth metal K or Ca into its crystal structure by mechanochemical modification, it could be used as a milling additive KOH, $Ca(OH)_2$ or CaO respectively in the process of studying CO_2 capture. Vermiculite, a natural silicate mineral with the chemical formula $(Mg,Fe,Al)_3(Al,Si)_4O_{10}(OH)_2.4H_2O$, is another material used in carbon capture studies. It is inherently harmless and has a large specific surface area, high cation exchange capacity, and excellent chemical and mechanical stability. Due to the presence of exchangeable cations (K^+, Ca^{2+}, and Mg^{2+}) inherent to vermiculite, it has demonstrated utility in studies related to the adsorptions (Ma et al. 2024).

The aim of our study was to demonstrate the potential CO_2 sequestration strategies within mineral carbonation of abundant aluminosilicate K-feldspar. *Ex situ* and *in situ* capture of CO_2 on mechanochemically additive-modified K-feldspar during high-energy milling was elaborated, characterized and quantified.

2. Materials and methods

2.1. Materials

K-feldspar or microcline ore used as input material for the experiments was provided by Kale Seramik Company, Türkiye with the following chemical analysis: 70.87%

SiO_2, 16.33% Al_2O_3, 10.6% K_2O, 1.99% Na_2O, 0.34% CaO, 0.15% Fe_2O_3, 0.14% BaO, 0.06% P_2O_5, 0.05% TiO_2, 0.04% MgO, 0.01% SrO. In Figure 1 the X-ray diffraction analysis (XRD) showed in addition to K-feldspar or microcline ($KAlSi_3O_8$), quartz (SiO_2) and albite ($NaAlSi_3O_8$) as well.

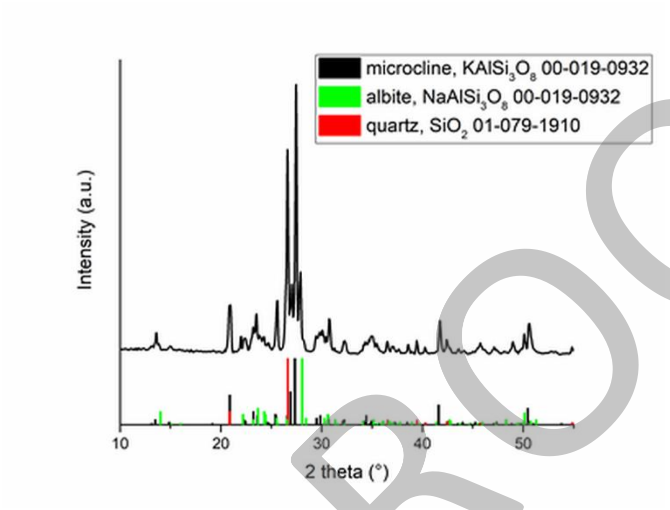

Figure 1

XRD pattern of as-received K-feldspar ore. Reprinted with permission from ref. (Baláž et al. 2024). Copyright 2024 Elsevier

The particle size d_{90}, d_{50}, and d_{10} values were 518, 293, and 121 μm, respectively. For mechanochemical modification of feldspar pure chemicals p.a. KOH (Centralchem, Slovakia), $Ca(OH)_2$, (Centralchem, Slovakia) and CaO (Sigma-Aldrich, USA) were used. Natural vermiculite $(Mg, Fe, Al)_3(Al, Si)_4O_{10}(OH)_2.4H_2O$ from Kuluncak (Malatya, Türkiye) has also been used as an additive to prepare K-feldspar/vermiculite (F:V) composites.

2.1.1. Mechanochemical modification of K-feldspar

Mechanochemical modification of K-feldspar ore was performed in the laboratory planetary ball mill Pulverisette 6 (Fritsch, Germany) with the addition of 1 M of KOH, $Ca(OH)_2$ or CaO (Table 1) under the following conditions: volume of milling chamber-250 mL, loading of the mill-50 balls (10 mm in diameter), the material of milling chamber and balls- tungsten carbide, WC, the total mass of the milling charge-20.18 g, ball-to-powder ratio-20:1, milling atmosphere-air, rotation speed 600 rpm, and milling time 90 min (each cycle of milling lasting 30 min was followed by a cooling break of 15 min).

Table 1

The amounts of added materials for mechanochemical modification of K-feldspar by milling

Sample	Mass of feldspar [g]	Mass of added material + H2O [g]
Feldspar	20.18	---
Feldspar/KOH	16.79	3.39
Feldspar/$Ca(OH)_2$	15.94	4.24
Feldspar/$Ca(OH)_2$ wet	16.85	2.24 + 1.1
Feldspar/CaO wet	16.40	1.65 + 2.1

2.2. CO_2 sequestration of K-feldspar

2.2.1. Ex situ CO_2 sequestration

Mechanically activated K-feldspar and mechanochemically modified samples of K-feldspar with hydroxides KOH, and $Ca(OH)_2$ were subjected to *ex situ* sequestration using thermoanalytical apparatus STA 449 F3 Jupiter (Netzsch, Germany) under dynamic conditions in CO_2 (50 $cm^3.min^{-1}$) by heating up to 150 °C for 5 h.

2.2.2. In situ CO_2 sequestration

The mechanochemically modified samples of K-feldspar with $Ca(OH)_2$ and CaO according to the conditions in *2.1.1* were *in situ* sequestrated using laboratory planetary ball mill Pulverisette 6 (Fritsch, Germany) according to the following conditions: volume of milling chamber-250 ml, loading of the mill-50 balls (10 mm in diameter), the material of milling chamber and balls-tungsten carbide, WC, the total mass of the milling charge-20.18 g, the addition of 10.1 mL H_2O, ball-to-powder ratio-20:1, milling atmosphere-CO_2 (5 $L.min^{-1}$, 3 min flushing), rotation speed 450 rpm, and milling time 30 min.

For testing the sequestration potential of K-feldspar:vermiculite (F:V) composites, the same mill and milling balls (both number and diameter) as specified above were used. The overall sample mass was 18 g and the weight ratio between V and F was modified (namely as-received F and V, and their combinations in 80:20, and 60:40 ratios were used). Before milling, 9 mL H_2O was added and a milling atmosphere of CO_2 (5 $L.min^{-1}$, 3 min flushing) was used. The rotation speed was set to 450 rpm and the milling time was 30 min according to our previous experiments (Turianicová, 2009).

2.3. Characterization techniques

X-ray diffraction measurements (XRD) were carried out in the Bragg-Brentano geometry using a D8 Advance diffractometer (Bruker, Germany), working with CuK_α radiation. ICDD-PDF2 was used for phase matching.

Fourier-transform infrared (FT-IR) spectra were measured using the Tensor 29 (Bruker, Germany) in the frequency range of 4000–400 cm^{-1} with the KBr pellet method. KBr was dried before the analysis at 100°C for 1 h.

Thermogravimetric measurements were carried out using STA 449 Jupiter thermal analyzer (Netzsch, Germany) coupled with a QMS 430C Aëolos mass spectrometer (Netzsch, Germany). The measurements were performed at steady airflow from 45 °C up to 1000 °C with a heating rate of 10 °C/min. Changes in the sample weight and m/z signals (m/z = 18 (H_2O) and m/z = 44 (CO_2)) were constantly monitored.

The elemental analysis (CHNS) was performed by elementary analyser Vario MACRO cube (Elementar Analysensysteme GmbH, Germany) using a thermal conductivity detector. Helium (purity 99.995%, intake pressure 2 bar) was chosen as the carrier gas in all analyses. The purity of oxygen for combustion was 99.995% with an intake pressure of 2 bar A combustion tube was set up at 1150 °C and a reduction tube at 850 °C. Sulphanilamide (C=41.81%, N=16.26%, H=4.65%, S=18.62%) was used as the CHNS standard.

CO_2 mineralization ratio or mechanochemical carbonation ratio was calculated according to literature (Shangguan et al. 2016) based on the weight loss of the *in situ* sequestered mechanochemically modified feldspar samples with $Ca(OH)_2$ and CaO after calcination in a muffle furnace. The calculation was performed according to the formula:

$$CO_2\ carbonation\ ratio\ (\%) = \frac{M_2 - M_3}{M_1} \times 100 \tag{1}$$

where M_2 and M_3 are masses of 1 h calcinated samples at 400°C and 800°C respectively, and M_1 is the mass of the sample before calcination.

3. RESULTS

3.1. *Ex situ* sequestration of mechanochemically modified K-feldspar with hydroxides

The first *ex situ* CO_2 capture tests using mechanically activated K-feldspar without modification were unsuccessful. Therefore, the K-feldspar was subjected to mechanochemical modification with the addition of KOH and $Ca(OH)_2$ in order to break the K-feldspar crystal structure and/or create new phases e.g. $Al(OH)_3$, $CaAl_2Si_3O_{10}$ that would be able to capture CO_2 gas. By evaluating the XRD patterns (see Figure 2) of such modified K-feldspar was found, that during 90 min of milling, only the amorphization of K-feldspar/microcline and albite phases was detected and no new phases were formed when performing milling under neat conditions with both KOH and $Ca(OH)_2$. In the case of milling with KOH, and $Ca(OH)_2$ with H_2O (wet), a WC phase appeared originating from the wear of the milling chamber and balls.

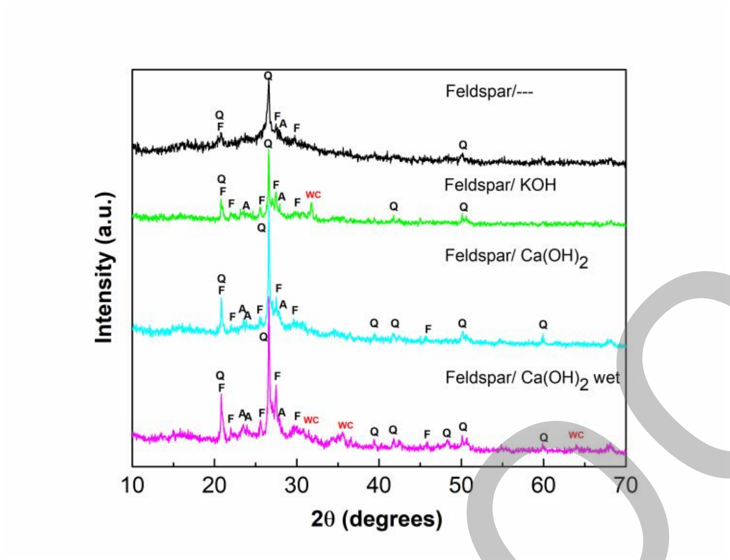

Figure 2

XRD patterns of mechanically activated K-feldspar and mechanochemically modified K-feldspar with KOH and Ca(OH)$_2$. F - KAlSi$_3$O$_8$, A- NaAlSi$_3$O$_8$, Q - SiO$_2$, WC – tungsten carbide

The experimental process of *ex situ* CO_2 sequestration of mechanically activated K-feldspar and mechanochemically modified samples has been carried out according to the conditions in part *2.2.1*. The course of the process is visualized in Figure 3 where TG and DTA curves can be seen. The curves revealed that at the beginning of the experiment, a slight weight increase was observed in the case of K-feldspar/ KOH and K-feldspar/Ca(OH)$_2$ mixtures. This increase in weight might be due to the carbonation process; however, no other effects were detected.

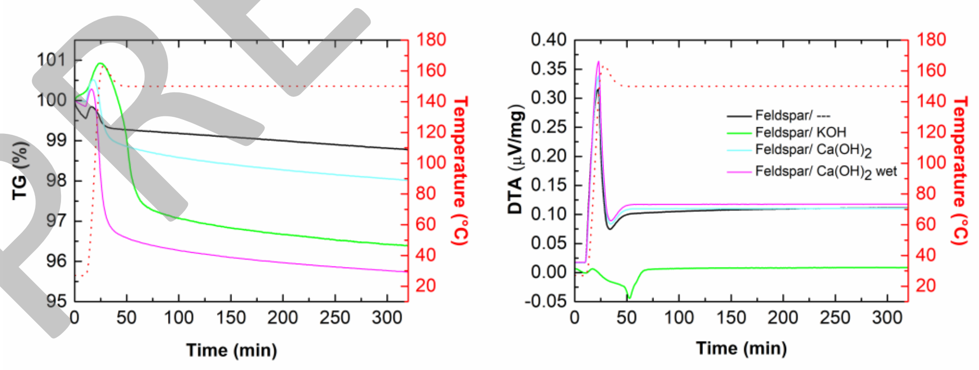

Figure 3

TG and DTA curves of mechanically activated K-feldspar (black line) and mechanochemically modified K-feldspar with KOH and Ca(OH)$_2$ during ex situ CO_2 sequestration

FTIR spectroscopy as another sensitive method for demonstrating CO_2 capture was used. The FT-IR spectra of the samples modified with KOH, $Ca(OH)_2$ under dry and wet conditions after CO_2 exposure in Figure 4 showed evidence of CO_2 binding and carbonate phase formation in all three cases, which can be determined by the peak in the wavenumber region of 1600-1300 cm^{-1}, characteristic for CO_3^{2-} vibrations (Nakamoto 2008). The bands attributed to the carbonate group can be observed as a single or double peak in the range of 1350–1565 cm^{-1}. It was found that peak splitting or merging is related to the alkaline metal with which CO_2 is combined (Turianicová et al. 2013b).

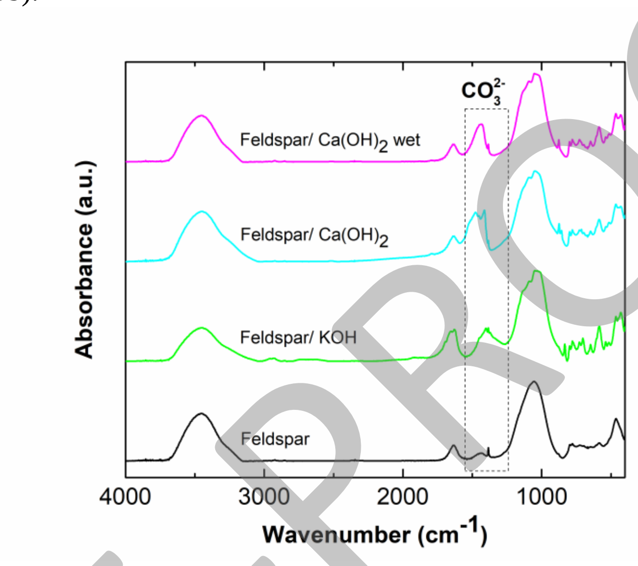

Figure 4

FT-IR spectra of mechanically activated K-feldspar (black line) and mechanochemically modified K-feldspar with KOH and $Ca(OH)_2$ after ex situ CO_2 sequestration

The same behaviour was not detected in the case of unmodified K-feldspar. It means, that probably $CaCO_3$ and K_2CO_3 were formed during mentioned conditions in the case of K-feldspar modified with KOH and $Ca(OH)_2$. However, we assume that the binding of CO_2 proceeds only due to the presence of KOH and $Ca(OH)_2$ and K-feldspar is inactive.

3.2. *In situ* sequestration of mechanochemically modified K-feldspar with $Ca(OH)_2$ and CaO

To intensify CO_2 capture by modified K-feldspar, another investigation strategy was chosen- two-step milling. In the first step, K-feldspar was milled with the addition of $Ca(OH)_2$, or CaO, resulting in a mechanochemically altered K-feldspar, and subsequently in the following second step *in situ* CO_2 sequestration was realized. XRD analysis performed after sequestration confirmed the formation of the calcite phase,

$CaCO_3$ in all three cases (Figure 5). This is the evidence that there is a chemical bond between CO_2 and Ca^{2+} during *in situ* sequestration, i.e. 30 min of milling in a CO_2 atmosphere and the so-called mechanochemical carbonation took place. Moreover, the inactivity of as-received K-feldspar was also detected in this case.

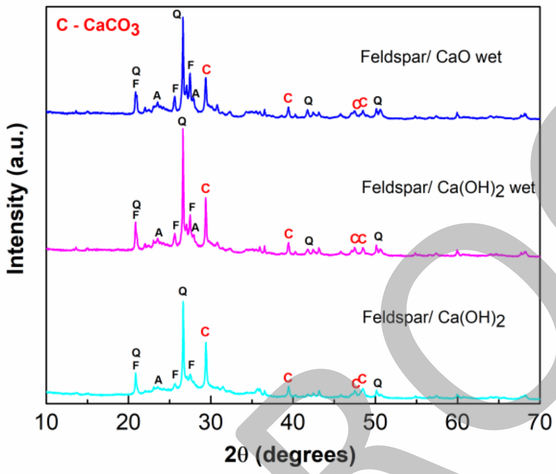

Figure 5

XRD patterns of K-feldspar after two-step milling: mechanochemical modification with $Ca(OH)_2$ and CaO and subsequent in situ CO_2 sequestration

With the aim to confirm $CaCO_3$ formation during milling in a CO_2 atmosphere, the thermal decomposition accompanied by the evolution of gases from the calcium carbonate-containing samples was monitored. Figure 6 compares the thermal behaviour of K-feldspar modified with $Ca(OH)_2$ and CaO after *in situ* mechanochemical carbonation.

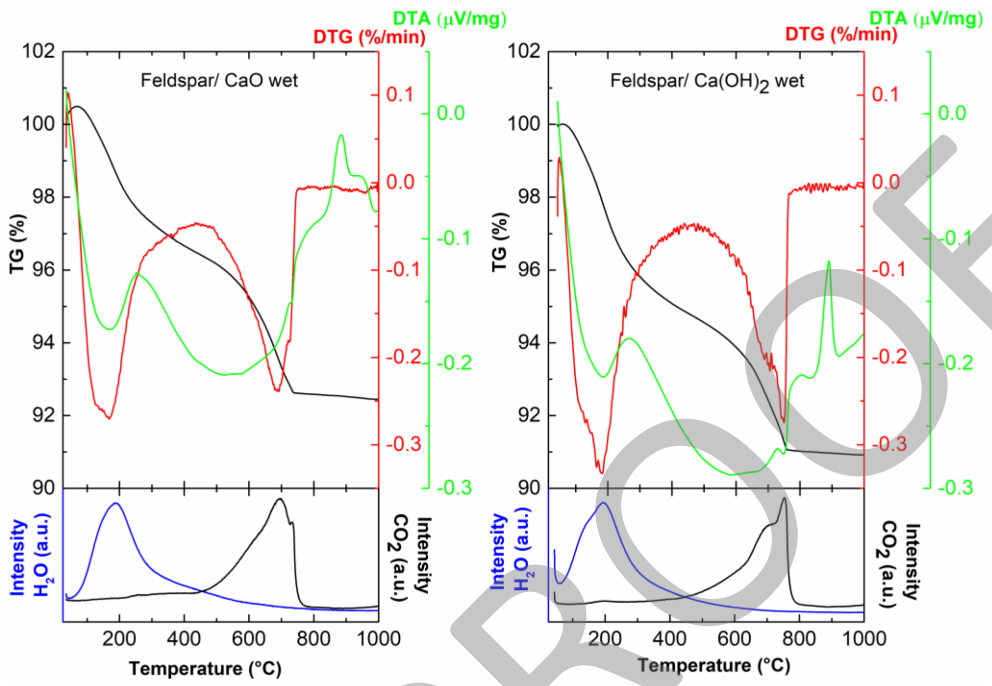

Figure 6

TG/DTG-DTA curves with mass spectrometry analysis of K-feldspar after two-step milling: mechanochemical modification with Ca(OH)$_2$ and CaO and subsequent in situ CO$_2$ sequestration

As can be seen, in both cases, the TG/DTG-DTA curves are similar. As expected, the evolution of H_2O and CO_2 gases has been observed in both cases. While dehydration occurred in the range of 25-400 °C, decarbonation occurred in the range of 500-850 °C, which confirmed the decomposition of the mechanochemically formed calcite phase.

The results of CHNS elemental analysis and the values of CO_2 carbonation (mineralization) ratios of the samples after *in situ* CO_2 sequestration are summarised in Table 2. According to the analysis of the amount of carbon, about 1.2-1.36% C was actually bound in the modified samples after deducting the amount of C of 0.27% corresponding to the unmodified K-feldspar. The contents of N and S were under the detection limit. The mineralization ratio increased up to 7 times compared to unmodified K-feldspar and reached a value of 5.23% comparable to the result achieved by milled K-feldspar (up to 20 h) with the addition of $CaCl_2$-slag, while the CO_2 sequestration was carried out in an autoclave at a temperature of 150°C and a pressure of 4 MPa (Shangguan et al. 2016).

Table 2

CHNS elemental analysis of K-feldspar after two-step milling: mechanochemical modification with $Ca(OH)_2$ and CaO and subsequent in situ CO_2 sequestration and calculated CO_2 carbonation ratio

Sample	C [%]	H [%]	CO_2 carbonation ratio [%]
Feldspar	0.27	1.59	0.74
Feldspar/ CaO wet	1.50	0.71	3.49
Feldspar/ $Ca(OH)_2$ wet	1.63	0.82	4.66
Feldspar/ $Ca(OH)_2$	1.47	0.76	5.23

3.3. *In situ* sequestration of K-feldspar and vermiculite mixtures

In addition to introducing pure artificial chemicals to the K-feldspar, also natural material can be used in this way. It is known that vermiculite mineral is capable of sequestrating CO_2. In order to find a potential synergy and thus the improvement of the CO_2 sequestration ability of K-feldspar, the mixtures of K-feldspar and vermiculite were prepared and subsequently subjected to sequestration. The XRD patterns are provided in Figure 7.

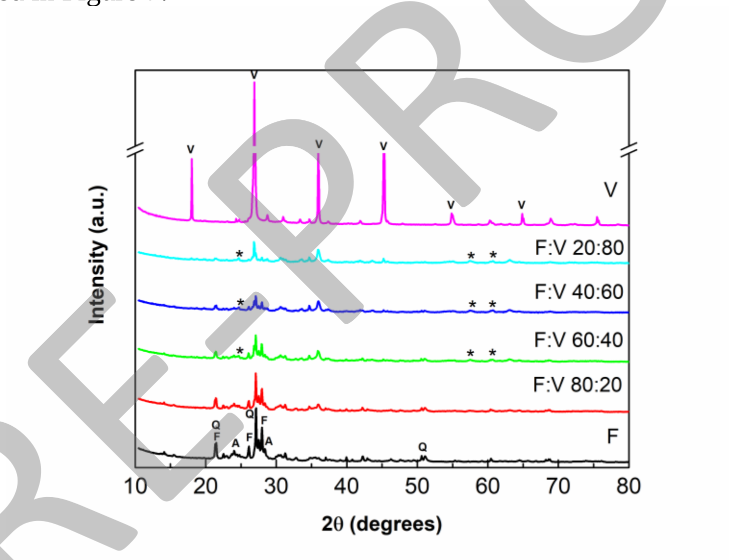

Figure 7

XRD patterns of as-received K-feldspar(F), K-feldspar(F): vermiculite(V) mixtures, and as-received vermiculite(V) after in situ CO_2 sequestration. Specific peaks are marked with an asterisk- see the explanation in the text below)

The XRD pattern of the K-feldspar after CO_2 sequestration is almost completely identical to the starting one described in (Baláž et al. 2024). All diffractions corresponding to microcline or K-feldspar (F), albite (A) and quartz (Q) are visible. Thus, the effect of neither mechanochemical modification nor sequestration is visible. Increasing the content of vermiculite leads to a gradual decrease in K-feldspar diffractions, whereas those corresponding to vermiculite become more pronounced. Interestingly, the four main vermiculite diffractions are still less pronounced than those corresponding to K-feldspar in the F:V 40:60 sample. The diffractions of as-received vermiculite are much more intensive (the counts detected for the most intensive diffraction peak detected for this sample are more than 17 times higher than that of the most intensive one belonging to K-feldspar, and that of other samples are even less intensive) and point to potentially different mechanism involved in CO_2 sequestration. This is further supported by the fact that there are few diffraction peaks (e.g., at 2 Θ = 24.7°, 57.6 ° and 60.7°) that increase in intensity until F:V 20:80 mixture (marked with an asterisk in Figure 7), but they remain in the same intensity when as-received vermiculite was applied. However, no clear diffractions corresponding to carbonate species in either of the samples could be clearly identified via XRD.

However, due to the detection limit of the XRD technique being around 5%, the potential presence of carbon as a result of mechanochemical carbonation was investigated via elemental analysis. The results are provided in Table 3.

Table 3

CHNS elemental analysis of as-received K-feldspar, vermiculite and the mixtures K-feldspar:vermiculite after in situ CO_2 sequestration

Sample [wt.%]	C [%]	H [%]	N [%]	S [%]
Vermiculite 100	0.30	2.21	0.07	0.11
F:V 20:80	0.89	0.55	0.15	0.09
F:V 40:60	0.65	1.15	0.13	0.06
F:V 60:40	0.54	0.55	0.13	0.06
F:V 80:20	0.31	1.57	0.12	0.06
Feldspar 100	0.18	1.24	0.15	0.02

K-feldspar is capable of binding only 0.18% C, whereas, in the case of vermiculite, this value is 0.30%. Interestingly, the composites seem to be more favourable for C binding than as-received vermiculite. Namely, the mixture containing only 20% vermiculite shows the same result and a gradual increase of C content with further increasing vermiculite content can be observed in Table 3. It turns out that K-feldspar can serve the role of the beneficial support to vermiculite being an efficient CO_2 adsorbent, thus a synergy between the two minerals was confirmed in the end. Figure 8 shows the FT-IR spectra of F:V mixtures in four different ratios (20:80 wt.%, 40:60 wt.%, 60:40 wt.%, 80:20 wt.%) after in situ CO_2 sequestration, in the range of 4000–600 cm^{-1}.

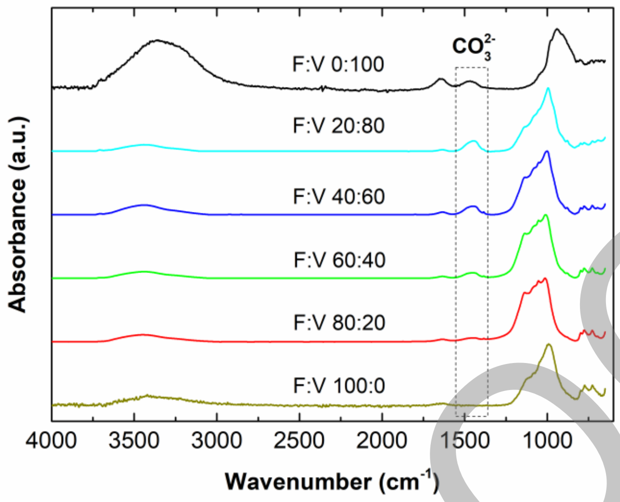

Figure 8
FT-IR spectra of K-feldspar: vermiculite mixtures, F:V after in situ CO_2 sequestration

In the case of K-feldspar mixtures, the intensity of a single carbonate peak with a maximum at approximately 1450 cm^{-1} is present and decreases with an increasing proportion of K-feldspar. Clearly, CO_2 was sequestered by vermiculite. However, it should be noted that the presence of K-feldspar is not negligible. When comparing the CHNS analysis results for as-received vermiculite (100 wt.%) with the F:V mixture (20:80 wt.%), almost 3 times higher amount of carbon was found in the mixture, indicating a greater amount of sequestered CO_2. The interaction of natural and thermally processed vermiculite with CO_2 during milling was also confirmed by Turianicová et al. (2014).

4. Discussion and conclusions

In this paper, three hitherto unused strategies for the potential use of common but refractory K-feldspar mineral for CO_2 sequestration were presented and tested. Before sequestration itself, which took place during the thermal process in a CO_2 atmosphere (*ex situ*) or during milling in a planetary mill (*in situ*), the K-feldspar was mechanochemically modified by milling. KOH, $Ca(OH)_2$, and CaO with and without H_2O were used for its mechanochemical modification. In the *ex situ* strategy, it was found that binding of CO_2 to K-feldspar modified with KOH and $Ca(OH)_2$ occurred and depended only on the added additive. During the second *in situ* strategy of mechanochemically modified K-feldspar with $Ca(OH)_2$, and CaO mechanochemical carbonation and subsequent calcite formation occurred, while a maximum CO_2 carbonation/mineralization ratio of 5% was achieved by 30 min milling. The third *in situ* strategy consisted of adding the mineral vermiculite capable of sequestering CO_2 to the K-feldspar in different weight ratios. The highest content of captured carbon 0.89% was achieved for the K-feldspar:vermiculite 20:80 composite during 30 minutes of milling in a CO_2 atmosphere.

A variety of techniques with new experimental approaches were applied in this case. Both applied strategies (*ex situ* and *in situ*) manifested new possibilities to expand the portfolio of usable materials for mineral decarbonation. Except for artificial chemicals, calcium-based additives also natural material - vermiculite were applied for the modification of K-feldspar. This expanded portfolio of objects under study revealed different mechanisms of CO_2 sequestration. However, despite the particular success caused by the modification of K-feldspar, the application of K-feldspar alone has no beneficial effect on CO_2 sequestration.

Acknowledgements

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