Journal of Ecological Engineering

Journal of Ecological Engineering, 2025, 26(5), 401–409
https://doi.org/10.12911/22998993/200661
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Received: 2025.01.23
Accepted: 2025.03.07
Published: 2025.03.16

In-situ capturing carbon dioxide capture by mechanochemical processing of K-feldspar with calcium oxide

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ABSTRACT

Capturing carbon dioxide (CO_2) emissions is necessary for a multi-faceted approach to combating climate change. It is only one of several essential strategies, including reducing emissions at the source, enhancing natural carbon sinks such as minerals and rocks, and transitioning to renewable energy sources. This pioneering study used abundant and cheap K-feldspar mechanochemically modified with CaO for *in situ* CO_2 sequestration during planetary ball milling. These innovative novel *in situ* CO_2 capture experiments consisted of two simple steps. The first step involved the mechanochemical modification of K-feldspar using CaO and the subsequent second step, direct *in situ* CO_2 sequestration in the same milling chamber during milling. X-ray diffraction patterns demonstrated the formation of the calcite phase, and elemental analysis confirmed the binding of approximately 1.69% carbon, representing a CO_2 mineralization ratio of 6.05% after 60 min of milling with CO_2 gas. Infrared spectroscopy, and thermogravimetric analysis equipped with mass spectroscopy verified CO_2 mineralization of CaO-modified K-feldspar. The morphology of the product after CO_2 capture containing calcite and unreacted K-feldspar was monitored by scanning electron microscopy.

Keywords: K-feldspar, intensive milling, *in situ* CO_2 capture, CaO, calcite.

INTRODUCTION

For more than a decade, the urgent need to reduce emissions of CO_2 and other greenhouse gases, which are responsible for climate change and global warming, has been resonating in the world. There are several political agreements, e.g. The Paris Agreement on Climate Change 2015, which aims to limit global warming below 2 °C to the pre-industrial level (Paris Agreement | Summary & Facts | Britannica, 2024). The Climate Change Conference (COP 28) in Dubai in 2023 called on countries to increase their emission reduction targets and signalled the beginning of the end of the fossil fuel era (COP 28: What Was Achieved and What Happens Next? UNFCCC (n.d.)). In addition to reducing greenhouse gases and achieving climate goals, carbon capture and storage (CCS) is also important for cement, steel, and aviation sectors, which are challenging to decarbonize due to their high CO_2 emissions and lack of alternatives (What is carbon capture and storage? CCS explained, National Grid Group (n.d.)). CCS can help to mitigate emissions from these sectors, playing a vital role in a comprehensive climate strategy (About CO2.Earth (n.d.)). CCS technologies studied over 20 years represented physical (Ban et al., 2014; Belmabkhout et al., 2016; Jansen et al., 2009), chemical (Liu et al., 2020; Sha et al., 2018; Yamamoto et al., 2013) and biological CO_2 fixation (Goli et al., 2016; Latini et al., 2022; Onyeaka et al., 2021).

CO_2 mineralization or carbonation is a safe and stable CO_2 fixation/capture/sequestration through reactions between alkaline earth metal compounds and CO_2 to form stable carbonates.

Initially, almost two decades ago, researchers mainly studied abundant Ca/Mg silicates such as olivine, forsterite, serpentinite, and wollastonite (Huijgen et al., 2006; Lackner, 2002; O'Connor et al., 2002) later also Ca/Mg-bearing industrial waste, slags (Bao et al., 2010; Wang et al., 2014) and even municipal solid waste (Lai et al., 2012) for this purpose. However, existing CO_2 mineralization methods have required calcination of the material at high temperatures, which makes them economically disadvantageous. The first reports on the use of high-energy milling for mechanical activation of silicate minerals for CO_2 carbonation were reported between 2001 and 2004 (Kalinkin et al., 2003; Kalinkin et al., 2004; Kalinkina et al., 2001a, 2001b). Later on, Turianicová et al. investigated the carbonation of olivine and vermiculite using mechanical activation (Turianicová et al., 2013; Turianicová et al., 2014). The main driving idea was to imitate and accelerate the natural weathering process of minerals by high-energy milling, which breaks the mineral crystal lattice together with specific surface growth and overall subsequent chemical reactivity (Baláž, 2008).

K-feldspar, $KAlSi_3O_8$, is an abundant, insoluble potash ore of the aluminosilicate group of minerals with significant worldwide reserves (e.g. in China, and Türkiye), which has attracted interest due to the possibility of extraction of soluble potassium used as a fertilizer (Chen et al., 2024). The K-feldspar crystal lattice contains $[AlO_4]^{5-}$ and $[SiO_4]^{4-}$ tetrahedra joined at the corners through O atoms and forming an infinite 3D structure with K^+ located in channels of the framework, which is typical for tectosilicates (Chen et al., 2024; Skorina and Allanore, 2015). In 2015, Ye et al. published the alternative utilization technology of K-feldspar which was thermally activated with the $CaCl_2$ additive at a temperature of 908 °C. At this temperature and with a suitable amount of $CaCl_2$, K^+ ions were exchanged for Ca^{2+} ions, and anorthite, pseudowollastonite, and wollastonite were formed, which were responsible for the fixation of CO_2 in an autoclave and the subsequent formation of stable calcite (Ye et al., 2014). Such a process using high temperatures during roasting, a temperature of 150 °C and a pressure of 4 MPa during autoclaving was laborious, consisting of process intermediate steps of filtering and drying, which was also time and economically demanding. A year later, Sheng et al. reported in detail that temperature, CO_2 pressure, and reaction time affected the carbonation of potassium-depleted residues of K-feldspar containing wollastonite, pseudowollastonite, Cl-mayenite, anorthite, and quartz. However, they also used an autoclave for experiments with maximum values of 200 °C temperature, 4 MPa pressure, and 120 min reaction time (Sheng et al., 2015).

The use of milling in the investigation of CO_2 mineralization of K-feldspar is very rarely described in the literature. Wang et al. applied a rod mill for wet milling of K-feldspar only for its pretreatment and then for milling calcined mixtures of K-feldspar and $CaSO_4$, which were subjected to CO_2 mineralization in the autoclave at temperatures of 50–150 °C and CO_2 pressures of 0.3–4 MPa (Wang et al., 2014). Shangguan et al. applied the milling-assisted technology using a planetary mill to activate K-feldspar and $CaCl_2$ as an additive before the subsequent CO_2 mineralization process in an autoclave at a temperature of 150 °C and a pressure of 4 MPa (Shangguan et al., 2016). Since the existing CO_2 mineralization methods remain economically costly, it is therefore of the utmost importance to search for new strategies to improve CO_2 carbonation technology.

The aim of this study was to utilize abundantly occurring K-feldspar ore modified with CaO additive for capturing CO_2 gas without using the laborious, expensive and ultimately dangerous autoclaving process. This is the first report on the novel strategy consisting of two-step high-energy ball milling schematically shown in Fig. 2. Namely, the mechanochemical modification of the K-feldspar took place during the first step, followed by *in situ* capture of CO_2 in the second step. To the best of our knowledge, this is a not yet published report on such a strategy. The advantage is the simplicity, time-saving character of the entire process without intermediate steps and its performance in a single device - a chamber of the ball mill. The kinetics of the process, phase analysis, physico-chemical properties, morphology of the products, the amount of captured carbon and CO_2-mineralization ratio were evaluated. Infrared spectroscopy, and thermogravimetric analysis equipped with mass spectroscopy were utilized to verify CO_2 mineralization of CaO-modified K-feldspar.

EXPRIMENTAL

Materials

K-feldspar ore used as input material for the experiments was provided by Kale Seramik

Company, Türkiye together 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. Based on the X-ray diffraction (XRD) analysis shown in Figure 1 in addition to K-feldspar (microcline, $KAlSi_3O_8$), diffraction peaks for quartz (SiO_2) and albite ($NaAlSi_3O_8$) were revealed (Baláž et al., 2024).

The values of particle size of the fractions d_{90}, d_{50}, and d_{10} of K-feldspar ore were 518, 293, and 121 µm, respectively. Reagent-grade calcium oxide powder (CaO, 99.9%, Sigma-Aldrich, USA) was used as an additive for the mechanochemical modification of K-feldspar.

In situ CO_2 capture experimental procedure by two-step milling

Mechanochemical modification of K-feldspar ore with CaO (first step, see Fig. 2) was performed in the laboratory planetary ball mill Pulverisette 6 (Fritsch GmbH, Germany) according to the chemical reaction:

$$2\,KAlSi_3O_8 + CaO + 4\,H_2O = CaAl_2Si_3O_{10} \cdot 3\,H_2O + 2\,KOH + 3\,SiO_2 \quad (1)$$

The following milling conditions were used: 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 powder charged 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). These conditions were chosen based on the Design of Experiments via the Taguchi method from our previous research, as this sample exhibited the highest reactivity for recovery of Al (Baláž et al., 2024). Immediately after the first step (Figure 2), the process of *in situ* sequestration in the same mill and milling chamber followed, so that CO_2 gas with a flow rate of 5 L/min was introduced through the valve of the milling chamber lid and the chamber was flushed for 2 min through the second valve to ensure air-free milling environment. The resulting pressure of CO_2 in the milling chamber before the start of milling was ~30 kPa. The milling conditions were as follows: rotation speed 450 rpm, milling time 30 and 60 min, and addition of 10.1 ml H_2O, in line with the previous CO_2 *in situ* sequestration experiments with olivine (Turianicová, 2009).

Characterization methods

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 and a Bruker LYNXEYE detector. ICDD-PDF2 Database 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.

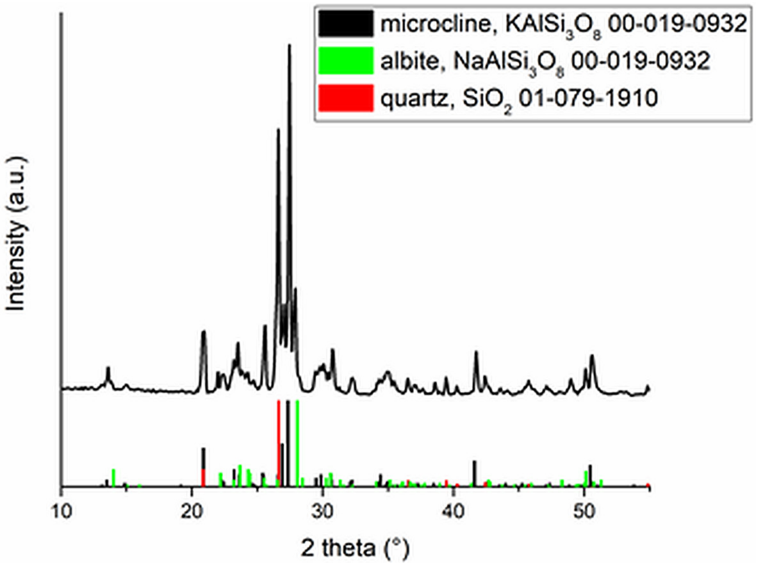

Figure 1. XRD pattern of the raw K-feldspar ore. Reprinted with permission from (Baláž et al., 2024)

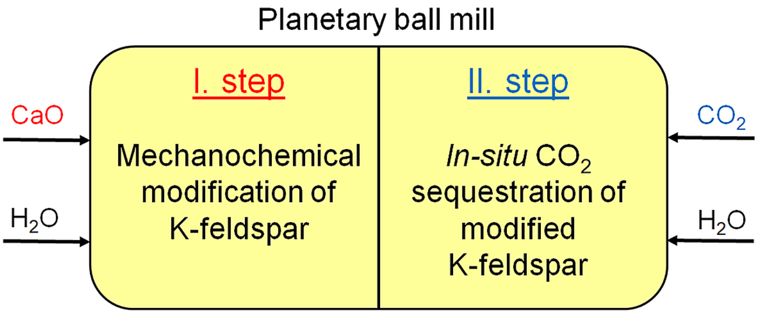

Figure 2. Scheme of *in situ* CO_2 capture procedure by two-step milling

KBr was dried before the analysis at 100 °C for 1 h. Thermogravimetric measurements (TG/DTA) 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.

Elemental carbon, hydrogen, nitrogen, and sulphur were analysed using a Vario MACRO cube (Elementar Analysensysteme GmbH, Germany) with 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 mechanically activated K-feldspar without CaO and mechanochemically modified K-feldspar samples with CaO after calcination in a muffle furnace. The calculation was performed according to the formula:

$$CO_2\ carbonation\ ratio\ (\%) = \\ = \frac{M_2 - M_3}{M_1} \times 100 \tag{2}$$

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

Scanning electron microscopy (SEM) study was performed using a MIRA3 FE-SEM microscope (TESCAN) equipped with the energy-dispersive X-ray (EDX) detector (Oxford Instrument).

RESULTS AND DISCUSSION

The XRD patterns of the mechanochemically modified K-feldspar and a subsequent *in situ* CO_2 sequestration (i.e. after both steps of milling) in Figure 3 confirmed the capture of CO_2 and subsequent mineral carbonation and the formation of calcite, $CaCO_3$ (01-072-1652). With the increase of the *in situ* capture time (second step of milling) from 30 min to 60 min, the intensity of the $CaCO_3$ peaks increased. Further increasing *in situ* capture time would be disadvantageous from an economic point of view of the applicability of the process in practice. By comparing the pattern of K-feldspar without the addition of CaO, where only its significant amorphization occurred, with the addition of CaO, a mechanochemical reaction of K-feldspar with CaO probably occurred leading to the formation of some type of a calcium aluminosilicate such as anorthite, $CaAl_2Si_2O_8$ (041-1486) or scolecite, $CaAl_2Si_3O_{10}.3H_2O$ (01-075-1456)

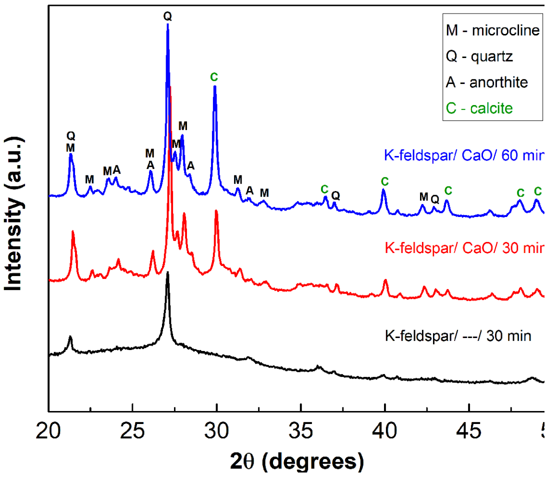

Figure 3. XRD patterns of K-feldspar after two-step milling: mechanical activation and mechanochemical modification with CaO and subsequent *in situ* CO_2 sequestration with milling time 30 and 60 min

according to the Equation 1. However, it could not be proven by XRD analysis due to the overlap of the peaks of these aluminosilicates. The Ca^{2+} cations in these compounds together with H_2O were subsequently able to react with CO_2 gas in the milling chamber during *in situ* sequestration and formed $CaCO_3$. The reaction mechanism of carbonation of Ca-containing minerals was described by Huijgen et al. (Huijgen et al., 2006). According to them, it is a solid-liquid reaction in which gaseous CO_2 forms H_2CO_3 with H_2O, and this differently dissolves Ca^{2+} ions from Ca-containing minerals, and the nucleation and growth of the $CaCO_3$ phase gradually takes place.

Figure 4 compares the behaviour of mechanochemically carbonated K-feldspar after two-step milling for 30 and 60 min during thermal treatment up to 1000 °C. The thermal analysis coupled with mass spectrometry (QMS) indicated similarity between both samples, suggesting that the prolonged milling time did not significantly affect CO_2 sequestration. In both cases, a two-step thermal decomposition occurred, as clearly shown on the TG curves. The similar mass losses of 23.8% and 21.9% are attributed to the release of gaseous products with amu = 18 and 44, corresponding to H_2O (amu 18) and CO_2 (amu 44). These effects are related to the dehydration and decarbonation process, respectively, which was also confirmed by the MS spectra located at the bottom of Figure 4a) and 4b). The *in situ* mechanochemical carbonation occurred under wet conditions, leading to partial formation of calcite (Figure 3). While dehydration occurred up to 200 °C, decarbonation occurred at approximately 750 °C. From the TG and QMS curves (bottom part of Figure 4), it was possible to evaluate the thermal stability of the produced calcite which showed stability in the temperature range of 25–550 °C, and it is assumed that it will also have long-term stability. The endothermic effect around 900 °C visible in the DTA curves could be associated with structural changes in feldspar, most likely resulting from its mechanochemical pretreatment.

Infrared spectroscopy (FTIR) was utilized to confirm the formation of the carbonate phase (calcite) during the *in situ* sequestration process. Figure 5 shows the FTIR spectra of K-feldspar after two-step milling for 30 and 60 min, both before and after thermal treatment. Evidence of CO_2 binding and carbonate phase formation can be identified by the peak in the wavenumber region of 1600–1300 cm^{-1}, characteristic of CO_3^{2-} vibrations (Nakamoto, 2008). As seen in the spectra, this peak is only present when K-feldspar was mechanochemically modified with CaO. The spectra of samples after thermal treatment do not exhibit this peak due to decarbonation induced by heating. The spectra in the range of 1250–400 cm^{-1} showed significant differences after thermal treatment, indicating structural changes. The results of CHNS elemental analysis and the values of CO_2 carbonation (mineralization) ratios of mechanically activated and CaO-modified K-feldspar samples after two-step milling are summarised in Table 1. According to the amounts of carbon, modified K-feldspar samples refer to the binding of around 4–6.5 times more C against

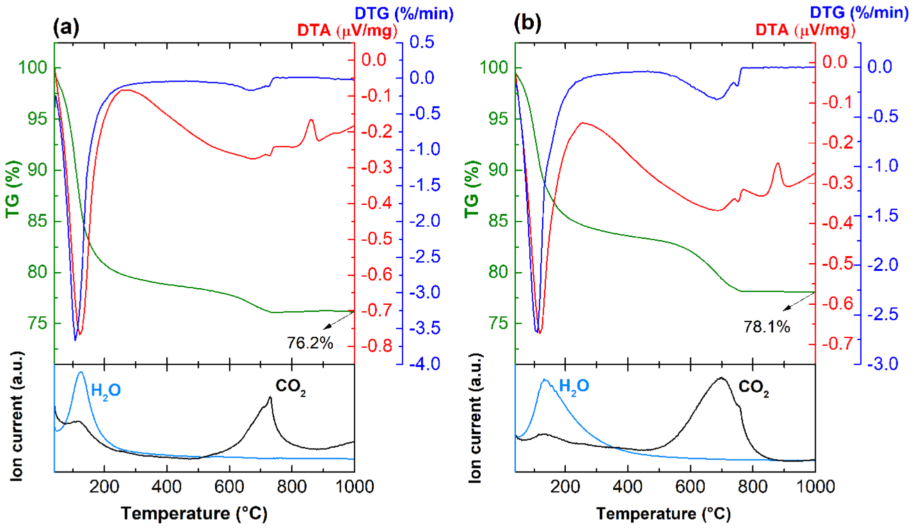

Figure 4. TG/DTG-DTA curves with QMS analysis of K-feldspar after two-step milling: (a) mechanochemical modification with CaO and subsequent *in situ* CO_2 sequestration for 30 min, (b) for 60 min

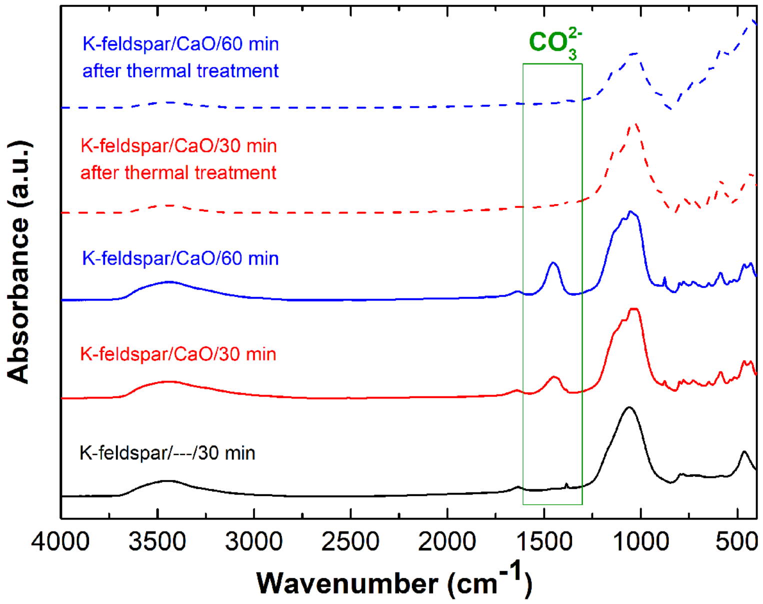

Figure 5. FT-IR spectra of mechanically activated K-feldspar (black line) and mechanochemically modified K-feldspar with CaO and subsequent *in situ* CO_2 sequestration for 30 (red line) and 60 min (blue line) before (solid line) and after (dash line) thermal treatment

unmodified feldspar. The contents of N and S were under the detection limit. The mineralization ratio increased up to 8 times compared to unmodified K-feldspar reaching 6.05%, which is slightly higher than the 5.0% achieved by (Shangguan et al., 2016) for milled feldspar (up to 20 h) with the addition of $CaCl_2$-slag, where CO_2 sequestration was carried out in an autoclave at 150 °C and 4 MPa. On the other hand, our value is slightly lower in comparison to the mineralization ratio of 7.7% reached by (Wang et al., 2014), who calcined feldspar with $CaSO_4$ at 1200 °C for 2 h before autoclaving at 100 °C and initial CO_2 pressure of 4 MPa. The morphology of K-feldspar after mechanochemical modification and subsequent *in situ* CO_2 sequestration was studied with SEM. An SEM image of the K-feldspar/CaO/60 min sample with the highest CO_2 carbonation ratio is displayed in Figure 6a. In order to estimate the chemical composition of the sample,

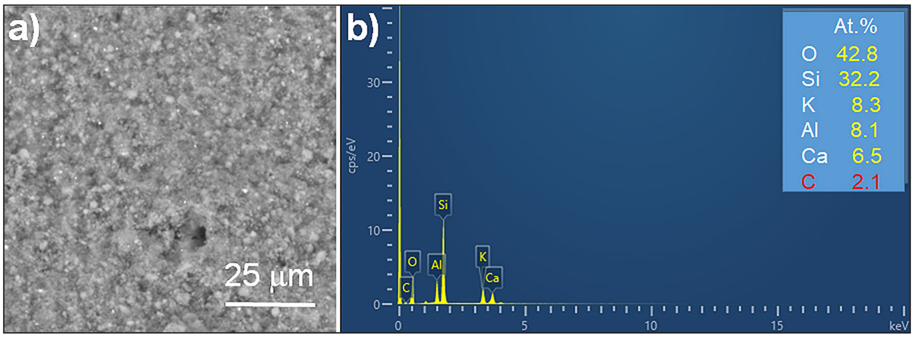

Figure 6. SEM image (a) and EDX analysis (b) of K-feldspar/CaO/60 min

Table 1. Elemental analysis of K-feldspar after two-step milling: mechanical activation or mechanochemical modification with CaO and subsequent *in situ* CO_2 sequestration, and calculated CO_2 mechanochemical carbonation ratio

Sample	C (%)	H (%)	CO_2 mechanochemical carbonation ratio (%)
K-feldspar/–/30 min	0.27	1.59	0.74
K-feldspar/CaO/30 min	1.02	1.50	3.49
K-feldspar/CaO/60 min	1.69	1.24	6.05

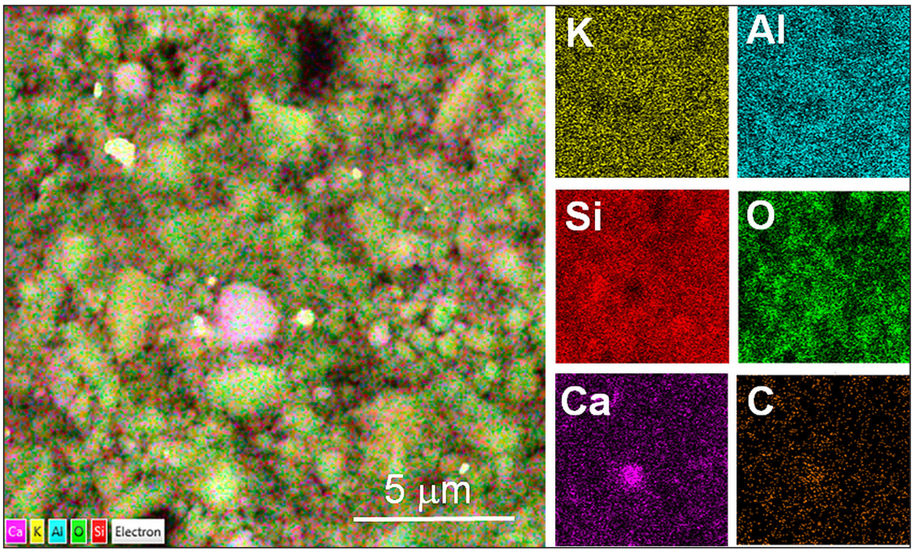

Figure 7. SEM image with element mapping for K, Al, Si, O, Ca, and C of K-feldspar/CaO/60 min

the EDX quantitative analysis was performed as shown in Figure 6b. It resulted in a chemical composition with a K:Al:Si:O atomic ratio of about 1:1:4:5 which corresponded to oxygen-deficient K-feldspar, $KAlSi_3O_8$. The 2.1% of bound carbon, presented as calcium carbonate ($CaCO_3$) roughly matched the CHNS analysis of the given sample (Table 1). The element mapping for Ca and C in Figure 7 also revealed and confirmed the presence of $CaCO_3$ particles in the K-feldspar/CaO/60 min sample.

CONCLUSIONS

In this paper, the new strategy for the potential use of common, and abundant but refractory K-feldspar ore for CO_2 sequestration was presented and tested. The novel mechanochemical processing of K-feldspar consisted of two-step high-energy ball milling, while during the first milling stage, K-feldspar was modified with CaO, and during the second milling stage, CO_2 capture and mechanochemical carbonation were performed. Milling time during the second milling stage had only a slight effect on the amount of sequestrated carbon. The highest amount of carbon 1.69%, and a maximum carbonation ratio of 6.05% was achieved for CaO-modified K-feldspar during 60 min of milling in a CO_2 atmosphere. This new strategy supported the mechanism of CO_2 capture through the formation of calcium aluminosilicate during K-feldspar modification, which was subsequently able to react with CO_2 to form calcium carbonate. Unmodified K-feldspar was unable to capture CO_2 by this process. The proposed simple technology could represent, from an environmental and economic point of view, affordable and scalable processing of K-feldspar, e.g. in the cement industry.

Acknowledgements

The present investigation was supported by the ERA-MIN3 Joint Call 2021 project POTASSIAL (ID # 27) and the Scientific and Technological Research Council of Türkiye (Project 122N039). The authors also gratefully acknowledge the financial support of the İnönü University Scientific Research Projects Coordination Unit through project no FBG-2022-2978. The support of the Slovak Research and Development Agency under the contract No. APVV-23-0372 is also gratefully acknowledged.

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