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Solid-State Synthesis of Li_2ZrO_3 and the Thermochemical Modelling of the Process

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Abstract: In the present study, the solid-state synthesis of lithium zirconate (Li_2ZrO_3), a promising high-temperature CO_2 sorbent, was investigated. The effects of calcination temperature and duration on the synthesis process were analyzed, and the thermochemical modelling of the reaction was done to predict the optimal experimental conditions. Lithium carbonate (Li_2CO_3) and zirconium oxide (ZrO_2) were utilized as raw materials, and the reactions were performed under an air atmosphere with temperatures ranging from 650°C to 1050°C. X-ray diffraction (XRD) and scanning electron microscopy (SEM) analyses revealed that nearly pure Li_2ZrO_3 could be achieved at temperatures above 750°C for reaction durations of at least 2 hours. The findings also highlighted the formation of monoclinic-Li_2ZrO_3 at elevated temperatures. This study demonstrates the efficiency of the solid-state synthesis method for producing Li_2ZrO_3 and provides insights into the thermochemical behaviour of the process, facilitating its potential application in CO_2 capture technologies.

Keywords: Lithium zirconate, solid state synthesis, thermochemical modelling.

Li_2ZrO_3'ün Katı Hal Sentezi ve Prosesin Termokimyasal Modellenmesi

Özet: Mevcut çalışmada, ümit verici bir yüksek sıcaklık CO_2 sorbenti olan lityum zirkonatın (Li_2ZrO_3) katı hal sentezi araştırılmıştır. Kalsinasyon sıcaklığının ve süresinin sentez süreci üzerindeki etkileri analiz edilmiş ve optimum deneysel koşulları tahmin etmek için reaksiyonun termokimyasal modellemesi yapılmıştır. Lityum karbonat (Li_2CO_3) ve zirkonyum oksit (ZrO_2) hammadde olarak kullanılmış ve reaksiyonlar 650°C ile 1050°C arasındaki sıcaklıklarda hava atmosferi altında gerçekleştirilmiştir. X-ışını difraksiyon (XRD) ve taramalı elektron mikroskobu (SEM) analizleri, en az 2 saatlik reaksiyon süreleri boyunca 750°C'nin üzerindeki sıcaklıklarda neredeyse saf Li_2ZrO_3 elde edilebileceğini ortaya koymuştur. Bulgular ayrıca yüksek sıcaklıklarda monoklinik-Li_2ZrO_3 oluşumunu da vurgulamıştır. Bu çalışma, Li_2ZrO_3 üretiminde katı hal sentez yönteminin verimliliğini göstermekte ve sürecin termokimyasal davranışına ilişkin öngörüler sunarak, CO_2 yakalama teknolojilerindeki potansiyel uygulamasını kolaylaştırmaktadır.

Anahtar Kelimeler: Lityum zirkonat, katı hal sentezi, termokimyasal modelleme.

Article

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1.Introduction

Carbon capture technology is becoming increasingly important to reduce the accumulation of CO_2 generated during industrial emissions and energy production in the atmosphere. This technology involves the separation of carbon dioxide from emission sources and its storage or reuse. The main methods include chemical absorption, physical adsorption, membrane gas separation and gas hydrate techniques. High-temperature solid sorbents for carbon capture, especially lithium-based ceramic materials such as Li_2ZrO_3, have attracted great attention due to their low energy consumption and high CO_2 selectivity (Guo et al., 2017; Zhang et al., 2019). Various techniques such as solid-state synthesis, sol-gel and hydrothermal methods are used in the production of such materials. Solid-state synthesis attracts attention with its simple process steps, low cost and high efficiency, while the hydrothermal method allows high-purity materials to be obtained at lower temperatures. Increasing research is focused on new combinations and methods to improve the carbon capture performance of these materials and increase their applicability at industrial scale (Afandi et al., 2024; Xiao et al., 2013).

Li_2ZrO_3 has two crystal structures. These are tetragonal (t-Li_2ZrO_3) and monoclinic (m-Li_2ZrO_3) phases. t-Li_2ZrO_3 is metastable and it transforms to m-Li_2ZrO_3 at 900 ºC (1173 K). Li_2ZrO_3 has an important place as a high temperature CO_2 sorbent because of its features like fast uptake rate, high capture capacity, ease of regeneration and good stability (Guo et al., 2017).

It has been observed that Li_2ZrO_3 has remarkable CO_2 capture capacity at high temperatures (Garcia et al., 2017; Guo et al., 2017). Theoretical maximum uptake value is 28.75% by wt. and the CO_2 capture reaction of Li_2ZrO_3 is given with Equation 1. Many countries around the world seek to reduce carbon emissions by 45% by 2030 in order to comply with the Paris Agreement, which aims to limit the rise in average global temperature below 2°C. To minimize CO_2 emissions, several measures have been used worldwide, including carbon taxation/trading programs, renewable energy, efficiency improvements, and carbon capturing equipment. However, fossil fuel use is unavoidable for the industry because of high energy generation, which results in large volumes of electricity created at once. Thus, carbon capturing technology is expected to assist industry, as CO_2 emissions are unavoidable during combustion (Afandi et al., 2024). Researchers have been actively investigating a number of strategies to lower the atmospheric concentration of carbon dioxide in order to address the pressing need to lessen the impact of its emissions. Technologies such as absorption, adsorption, membrane gas separation, and gas hydrate methods have emerged as potential solutions, aiming to capture carbon dioxide directly from the air or industrial emission sources to minimize its impact on the greenhouse effect (Mete et al., 2024). Although low temperature CO_2 adsorption has been of great interest in recent years to capture CO_2 after combustion, on the other hand high temperature CO_2 capture by using solid sorbents avoids the need to cool or quench the combustion gases (Garcia et al., 2017; Guo et al., 2017). The use of solid loop systems provides both high CO_2 adsorption capacity and selectivity. Lithium-based ceramics were investigated to perform well in four processes including CO_2 capture from fixed flue gases after high-temperature combustion, methane vapour reform, sorption increased alcohol reform, and CO oxidation combined with subsequent CO_2 chemicalization (Zhang et al., 2019).

$$Li_2ZrO_{3(s)} + CO_{2(g)} \rightarrow Li_2CO_{3(s)} + ZrO_{2(s)} \quad (1)$$

Solid state synthesis process and other liquid phase processes are production methods which are used to synthesize Li_2ZrO_3 (Guo et al., 2017). Those methods used so far for the synthesis of Li_2ZrO_3 are solid state, sol-gel method, soft chemistry route and hydrothermal method (Xiao et al., 2013). The solid state reaction is a conventional synthesis route which does not require solvent (or any liquid phases) in which solid starting materials, usually Li_2CO_3 and ZrO_2, are mechanically mixed and heated at high temperatures to complete the reaction (Xiao et al., 2013). This method has some advantages, for instance high efficiency, simple operation process and low cost (Natalia et al., 2018). Indeed, the solid-state synthesis reaction is the reverse of the CO_2 capture reaction of Li_2ZrO_3 and, it is given with Equation 2.

$$Li_2CO_{3(s)} + ZrO_{2(s)} \rightarrow Li_2ZrO_{3(s)} + CO_{2(g)} \quad (2)$$

The liquid phase method is also used for the preparation of Li_2ZrO_3, in which soluble Zr and Li compounds are raw materials. A complex solution is obtained which is dried to form Li_2ZrO_3 (Xiao et al., 2013). The sol-gel method has been used for decades for the synthesis of fine-grained materials. It has been proven in recent years that pure tetragonal-Li_2ZrO_3 nanocrystals can be obtained by soft chemistry using relatively low calcination temperatures. Hydrothermal method is used for controllable synthesis of materials at low temperatures and high purity (Zhang et al., 2019).

The production of Li_2ZrO_3 by solid state synthesis is achieved by mechanical mixing and heat treatment of zirconium oxide (ZrO_2) and lithium carbonate (Li_2CO_3) at high temperatures. In this method, the initial lithium carbonate molar ratio should be slightly higher in practice. Because lithium is easily sublimated at high temperature, the use of excess lithium carbonate as a raw material is importantly suggested (Yin et al., 2010). Li_2ZrO_3 has been paid attention because of its remarkable CO_2 capacity at high temperatures. The use of Li_2ZrO_3 was first reported by Nakagawa and Ohashi in 1998 as a high temperature CO_2 adsorber (450-600 ºC). At temperatures between 450°C and 650°C, Li_2ZrO_3 exhibits remarkable sorption kinetics and a sorption capacity of 0.287 g CO_2/g sorbent (Benzesik et al., 2022). This compound has an important potential of CO_2 adsorption capacity (~28% by weight) and small volume change during CO_2 sorption/desorption cycles (Yi and Eriksen, 2006). Nair et al. compared the performance of lithium zirconate prepared using tetragonal and monoclinic zirconia (Nair et al., 2004). As a result, it was observed that tetragonal lithium zirconate reacts with CO_2 significantly faster. Ida et al. developed a double-shell model to describe the reaction mechanism of Li_2ZrO_3 prepared by powder mixing. According to this model, Li_2CO_3 forms an outer shell on the particle leaving the ZrO_2 layer behind. Lithium and oxygen ions are emitted from the ZrO_2 and Li_2CO_3 layers for further reaction. The rate limiting step is the diffusion of lithium and oxygen ions from the ZrO_2 and Li_2CO_3 layers (Ida and Lin, 2003). Natalia et al. prepared Li_2ZrO_3 as a starting material by reaction of Li_2CO_3 and ZrO_2 from zircon sand in solid state. The lithium zirconate prepared in this study was observed to be in a single stage without any impurities (Natalia et al., 2018). Li_2ZrO_3 based sorbents in different compositions from Li_2CO_3, K_2CO_3 and ZrO_2 mixtures were synthesized by solid state reaction method. It has been observed that the Li_2ZrO_3 compound with the addition of Li_2CO_3/K_2CO_3 showed remarkable high temperature CO_2 sorption (Wang et al., 2014).

In this study, solid state synthesis conditions of Li_2ZrO_3 were investigated. Experiments were conducted to understand the effects of the calcination temperature and duration on the synthesis of Li_2ZrO_3. Moreover, thermochemical simulation of the synthesis reaction was modelled to predict experimental conditions.

2. Materials and Methods

2.1 Materials

For the solid-state synthesis of lithium zirconate, Li_2CO_3 (D90: 12.1 µm, 99.99% pure by mass, Merck) and ZrO_2 (<106 µm average particle size, 99.5% pure by mass) were used as raw materials. Synthesis experiments were conducted in Al_2O_3-based crucible.

2.2 Methods

Before the solid-state synthesis experiments, thermochemical modelling studies were done by means of HSC Chemistry 6.12 software (Roine, 2023). The software was used for the prediction of synthesis temperature range of the Li2ZrO3. “Equilibrium Compositions Module” of the software was used to plot the thermochemical modelling graphics. This module utilizes the Gibbs energy minimization method and GIBBS solver was used for the calculations.

Raw material mixtures were prepared by using TURBULA three-dimensional shaker mixer with respect to Equation 2 for 1 mol ZrO_2 and for 1.1 mol Li_2CO_3. Excess Li_2CO_3 was used to prevent the sublimation of Li-based compounds (Izquierdo et al., 2018). Before synthesis experiments, raw materials were dried in drying oven for 2 hours. In the first series of experiments, prepared powder mixtures were calcined (heat-treated) at increasing temperatures from 650 ºC to 1050 ºC for 3 hours in a muffle furnace. In the second series, synthesis experiments were carried out at the temperature of 850 ºC for increasing durations from 1 hour to 4 hours. All experiments were done under an air atmosphere and for a heating rate of 10 ºC·min^{-1}. After waiting times, samples were remained in the muffle furnace to cool down to the room temperature. Samples which were taken out of the furnace were weighed to calculate weight loss by mass because of the formation of the CO_2 in the reaction products of Equation 2. Thus, it was predicted that experiments having higher conversion rates would result in higher weight loss values after synthesis experiments. The flow-chart of the experiments was given in Figure1.

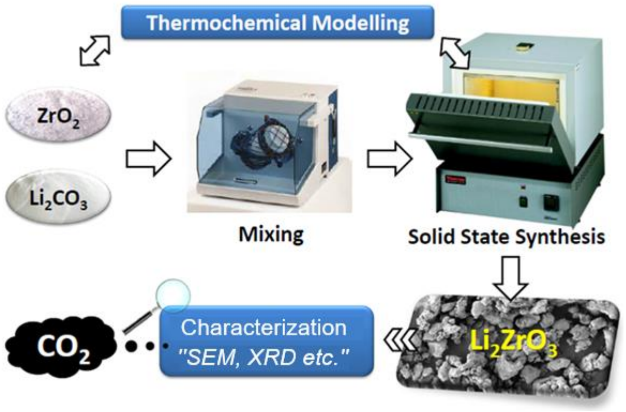

Figure. 1. Flowchart of the experimental studies.
Şekil 1. Deneysel çalışmaların akış şeması.

Synthesized samples were characterized by using X-ray diffraction spectrometry (XRD, Rigaku D/Max 2200/PC). Also, micrographs of the sample which was synthesized at 850 ºC for 2 hours were taken by using scanning electron microscope (SEM, Carl Zeiss, Gemini 300). In addition, the particle size (Beckman Coulter LS 13 320) of the obtained samples was analyzed.

3. Results and Discussion

3.1. Thermochemical modelling results

Thermochemical simulations were conducted to predict the formation conditions of Li_2ZrO_3 from Li_2CO_3 and ZrO_2 with increasing temperature with respect to Equation 2 (Figure 2). The formation of Li_2ZrO_3 began approximately at 500 ºC, and a slight amount of Li_2O was observed to arise, which addresses the sublimated fraction, in reaction products at the temperatures greater than 850 ºC.

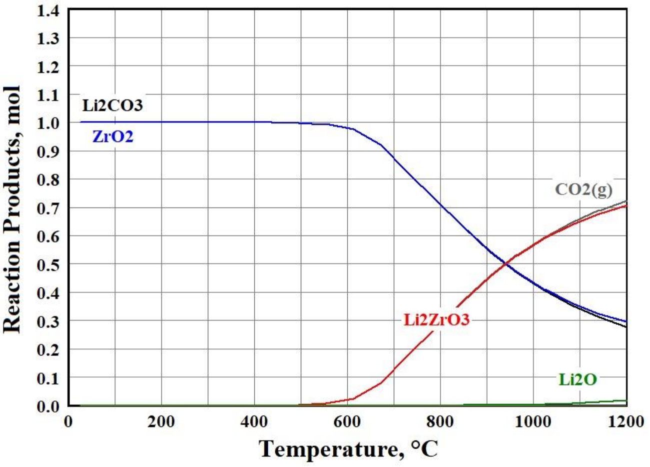

Figure 2. Thermochemical simulation of Li_2ZrO_3 formation from Li_2CO_3 and ZrO_2 at increasing temperatures.
Şekil 2. Artan sıcaklıklarda Li_2CO_3 ve ZrO_2'den Li_2ZrO_3 oluşumunun termokimyasal simülasyonu.

3.2. Results of synthesis experiments

Weight loss values of samples after calcination experiments are given in Figure 3 and Table 1. The weight loss was 14.69% for the samples calcined at 650 ºC for 3 hours and, it reached equilibrium at the temperatures equal to and greater than 750 ºC for 3 hours. After 750 ºC, weight loss values changed between 24.44% (at 750 ºC) and 26.82% (950 ºC). For the reaction durations lower than 3 hours, weight loss values were slightly lower but still close to results for 3 hours. The data shows that the synthesis reaction reaches equilibrium at the temperature greater than 750 ºC and for reaction durations equal to and greater than 1 hour.

Table 1. The change of weight loss values (%) after calcination experiments.
Tablo 1. Kalsinasyon deneyleri sonrası ağırlık kaybı değerlerinin değişimi (%).

Duration, h	Temperature, ºC				
	650	750	850	950	1050
1			23.89		
2			24.25		
3	14.69	24.44	25.99	26.82	26.01
4			24.72		

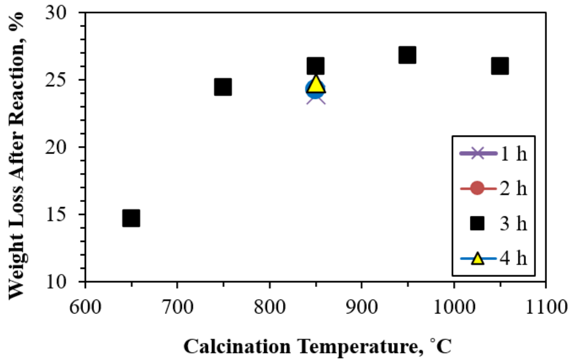

Figure 3. The change of weight loss values (%) after calcination experiments at increasing temperatures for 3 hours and for reaction durations from 1 hour to 4 hours at 850 ºC.
Şekil 3. 850 ºC'de 3 saat artan sıcaklıklarda ve 1 saatten 4 saate kadar reaksiyon sürelerinde kalsinasyon deneyleri sonucunda ağırlık kaybı değerlerinin (%) değişimi.

XRD patterns of selected synthesis products were given in Figure 4 and in Figure 5. XRD patterns supported weight loss values after the reactions. With the increase in reaction temperature and duration, almost pure Li_2ZrO_3 samples were obtained. Particularly the samples synthesized at 850 ºC and 950 ºC for reaction durations of greater than 2 hours consisted of Li_2ZrO_3 with a slight amount of ZrO_2. Moreover, the formation of monoclinic-Li_2ZrO_3 from tetragonal-Li_2ZrO_3 was observed with increasing reaction temperature, particularly at the temperatures equal to and greater than 750 ºC (Xiao Q. et al (2011).

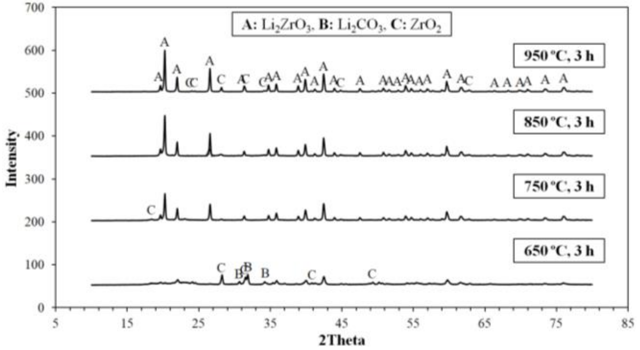

Figure 4. XRD patterns of Li_2ZrO_3 samples synthesized at the temperatures from 650 ºC to 950 ºC for 3 hours.
Şekil 4. 650 ºC ile 950 ºC arasındaki sıcaklıklarda 3 saat boyunca sentezlenen Li_2ZrO_3 numunelerinin XRD desenleri.

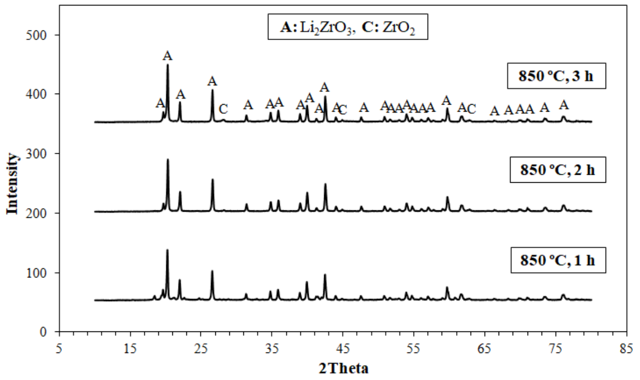

Figure 5. XRD patterns of Li_2ZrO_3 samples synthesized at the temperature of 850 ºC for increasing durations from 1 hour to 3 hours.
Şekil 5. 850 ºC sıcaklıkta 1 saatten 3 saate kadar artan sürelerde sentezlenen Li_2ZrO_3 numunelerinin XRD desenleri.

SEM micrographs of the sample synthesized at 850 ºC for 3 hours were given in Figure 6. As seen in the micrographs, synthesized Li_2ZrO_3 powders were in micron size. SEM analyses revealed that Li_2ZrO_3 samples exhibited irregular particle distribution and aggregation tendency. At higher magnifications, cracks and voids resulting from gas release were observed on the particle surfaces.

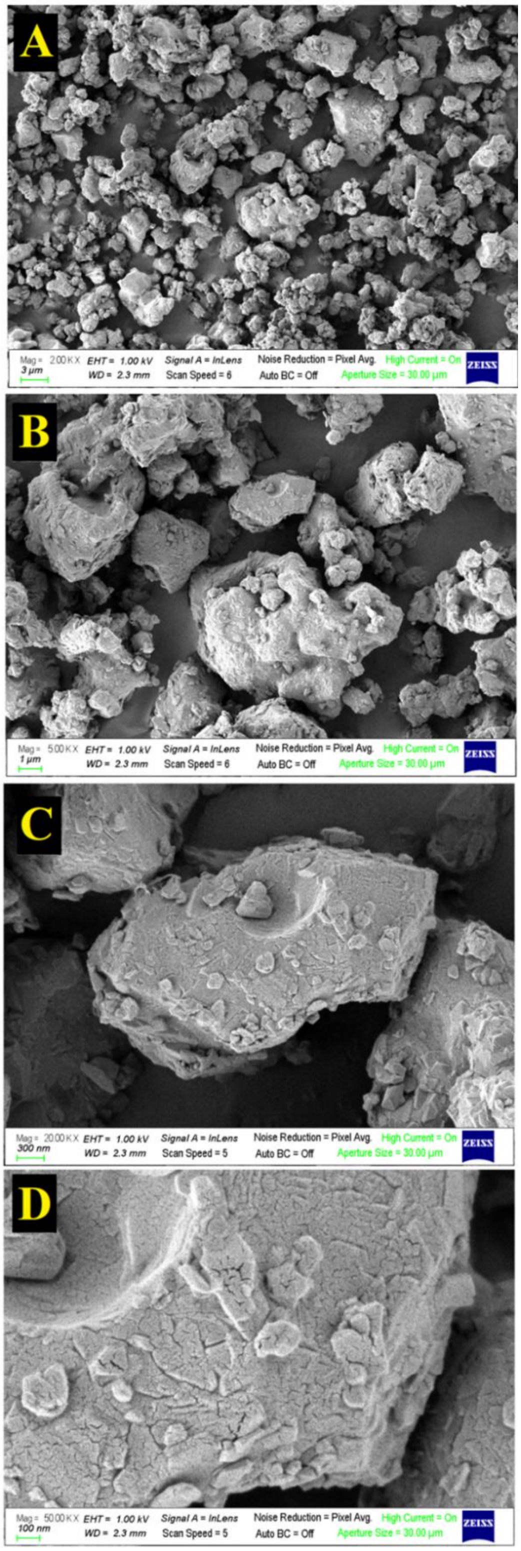

Figure 6. SEM micrographs of the Li_2ZrO_3 sample synthesized at 850 ºC for 3 hours: (A) 2k X, (B) 5k X, (C) 20k X and (D) 50 k X.
Şekil 6. 850 ºC'de 3 saat sentezlenen Li_2ZrO_3 numunesinin SEM mikrografları: (A) 2k X, (B) 5k X, (C) 20k X and (D) 50 k X.

The particle size distribution of the sample synthesized at 850ºC for 3 hours was concentrated between 2-60 µm, and the average particle diameter by volume was determined to be approximately 17.62 µm (Fig 7). The average particle size of the sample synthesized at 950 ºC for 3 hours was determined to be 20.39 µm (Fig 8). In addition, the particle size distribution was between 6-60 µm. This shows that the particle size increases with the synthesis process carried out at higher temperatures. Moreover, macrographs of synthesized samples were shared in Figure 9.

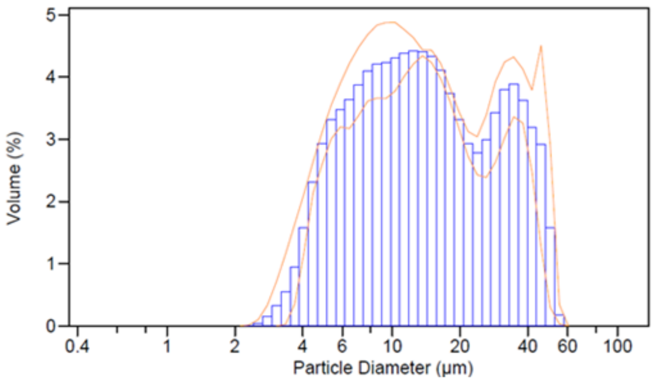

Figure 7. Particle size distribution of Li_2ZrO_3 sample synthesized at 850 ºC for 3 hours.
Şekil 7. 850 ºC'de 3 saat boyunca sentezlenen Li_2ZrO_3 numunesinin partikül boyut dağılımı.

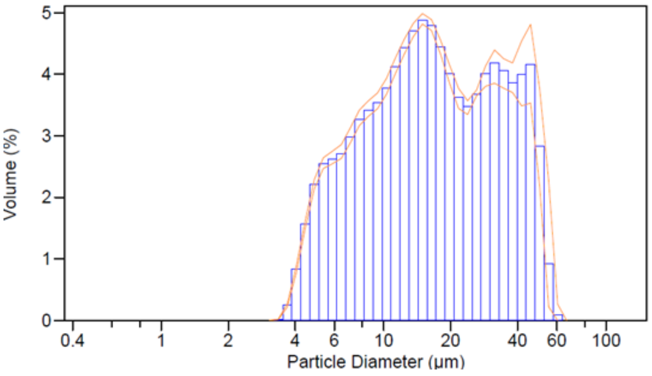

Figure 8. Particle size distribution of Li_2ZrO_3 sample synthesized at 950 ºC for 3 hours.
Şekil 8. 950 ºC'de 3 saat boyunca sentezlenen Li_2ZrO_3 numunesinin partikül boyut dağılımı.

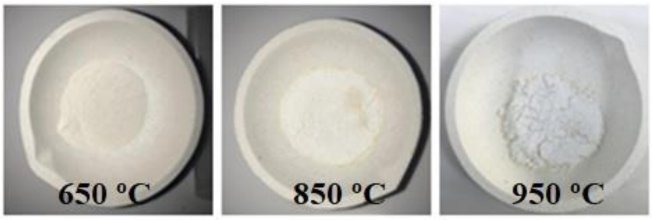

Figure 9. Macrographs of Li_2ZrO_3 samples synthesized at 650 ºC, 850 ºC and 950 ºC for 3 hours.
Şekil 9. 650 ºC, 850 ºC ve 950 ºC'de 3 saat sentezlenen Li_2ZrO_3 numunelerinin makrografları.

A moderate average particle size was observed in this study on the synthesized Li_2ZrO_3 samples. It is a well-known phenomenon that finer or sub-micron particle sizes are favourable to foster CO_2 uptakes capacities of the high temperature sorbents. This study was mainly designed to reveal the optimum Li_2ZrO_3 solid state synthesis conditions and, it is suggested that further studies should be conducted with finer particle size raw samples and following CO_2 uptake tests to entirely discuss the effects of the solid-state synthesis conditions on the CO_2 uptake.

4. Conclusion

This study successfully demonstrates the optimization of synthesizing lithium zirconate (Li_2ZrO_3) through a solid-state method using lithium carbonate (Li_2CO_3) and zirconium oxide (ZrO_2) as precursors. The experiments revealed that nearly pure Li_2ZrO_3 could be synthesized under optimal conditions of calcination temperatures above 750°C and reaction durations of at least 2 hours. The thermochemical modeling supported these findings, indicating that Li_2ZrO_3 formation begins at approximately 500°C, with equilibrium achieved at higher temperatures. The synthesized Li_2ZrO_3 samples exhibited moderate particle sizes. Smaller, sub-micron particles are known to enhance CO_2 uptake in high-temperature sorbents. This study focused on optimizing Li_2ZrO_3 synthesis conditions, recommending further research with finer raw materials and CO_2 uptake tests to fully explore synthesis effects.

5. Acknowledgements

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6. Conflicts of Interest

The authors declare no conflict of interest.

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