



Direct CO₂ capture from ambient air by K₂CO₃/alumina composite sorbent for synthesis of renewable methane

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Abstract

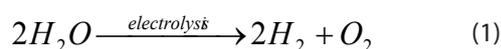
A composite sorbent was synthesized by impregnating cylindrical granules of mesoporous γ -Al₂O₃ with an aqueous solution of K₂CO₃. Three fractions of the composite with the characteristic grain size of 1–2 mm, 3–4 mm and 4–6 mm were tested for direct carbon dioxide capture from ambient air in a continuous-flow system. It was shown that dynamic CO₂ absorption capacity of the composite sorbent increases significantly with decrease of the characteristic grain size, which indicates that the process is limited by mass transfer. The effect of temperature on thermal regeneration of the composite was also investigated. The change of regeneration temperature from 250 to 300°C enhanced CO₂ absorption capacity for all the fractions studied. XRD in situ study showed that the reason of this enhancement is thermal decomposition of potassium dawsonite KAlCO₃(OH)₂ formed during synthesis of the composite material. The composite sorbent demonstrated a good stability of its CO₂ absorption characteristics, therefore it should be considered as a promising material for direct carbon dioxide capture from air.

Keywords: Carbon dioxide, potassium carbonate, alumina, composite material, absorption, regeneration, air, grain size effect

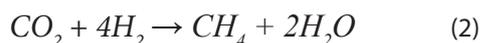
Introduction

One of the main problems of power systems based on renewables, such as wind and solar energy, is volatility of the power supply. Chemical energy storage systems have the potential to become a sustainable and realistic solution to this challenge, which enables an offsetting in the time gap between power production and its consumption.

The surplus electricity generated by renewables can be used for water electrolysis (1) in order to produce hydrogen:



Hydrogen is considered to be a perspective fuel for low-carbon economy, but there are still unresolved safety problems regarding its transportation and storage. Therefore, it is reasonable to use the generated hydrogen for production of a conventional hydrocarbon fuel, e.g., methane, which can be obtained by Sabatier process [1]:



The synthetic renewable methane, which corresponds to chemically

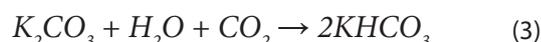
normal natural gas, can be injected into the existing gas network or stored and utilized on demand.

It is very important for sustainable development that renewable methane can be potentially produced using carbon dioxide captured from ambient air. It is well known that CO₂ is the major anthropogenic greenhouse gas, which contributes to global climate change. Carbon dioxide capture is usually discussed in the context of capturing CO₂ from large point sources, such as flue gases from industries and coal-fired power plants. For small distributed sources of CO₂, i.e., transport vehicles, capture at the emission source is not favorable from the practical point of view. Therefore, capturing carbon dioxide directly from the atmosphere ("direct air capture", DAC) is currently under consideration as an option for stabilizing global CO₂ concentrations [2,3]. Direct CO₂ capture from ambient air offers more flexibility compared to conventional source point capture, because CO₂ capture units can easily be located close to CO₂ recycling center. The DAC technology has a potential to be "carbon negative", but only if a renewable energy source is used to drive the process. Incorporation of the DAC unit into the energy

storage system offers an opportunity to use anthro-pogenic carbon dioxide as a valuable feedstock for the production of renewable methane, which can be utilized in the heating market or as a fuel for transportation.

Carbon dioxide capture from ambient air is more challenging than conventional carbon capture from flue gases because of very low CO₂ concentration in air (~400 ppm) and the necessity to operate in the presence of moisture excess at ambient temperature and pressure. The most developed DAC technologies are based on carbon dioxide absorption by aqueous alkali solutions. The major drawback of this approach is that the regeneration of these solutions is generally multi-stage and energy intensive.

Potassium carbonate is a well-known solid inorganic chemisorbent, which reacts with atmospheric CO₂ in the presence of water vapor forming potassium bicarbonate [4]:



The main problem with using bulk potassium carbonate for CO₂ absorption purposes is low carbonation rate [5,6]. This problem can be solved by dispersing K₂CO₃ particles inside a porous support material. Composite materials "potassium carbonate inside a porous matrix" are known to be effective sorbents for capturing carbon dioxide from wet flue gases [5-9]. Recently, it was also shown that K₂CO₃/γ-Al₂O₃ composite is a promising material for CO₂ capture directly from ambient air [10]. However, the room for improvement still exists and more in-depth study is needed in order to optimize absorption properties of the composite material. In this paper we will discuss some factors affecting CO₂ absorption capacity by K₂CO₃/γ-Al₂O₃ material under conditions of direct CO₂ capture from ambient air with thermal regeneration in the temperature-swung absorption (TSA) cycles.

Materials and methods

Composite sorbent preparation and characterization

Cylindrical γ-Al₂O₃ granules with diameter of 3 mm and typical length of 4-6 mm were produced by JSC «Angarsk Catalysts and Organic Synthesis Plant». The alumina is a mesoporous material with the average pore diameter of 15 nm.

The composite sorbent K₂CO₃/γ-Al₂O₃ was prepared by dry impregnation method, described in detail in [9]. The pores of the alumina matrix were filled with 40 wt.% aqueous solution of potassium carbonate. After the matrix and the solution contacted for about 1 h, the granules were dried at 90°C for 24 h and then calcinated at 200°C for 2 h. The resulted product was ground in a mortar and sieved to obtain the desired particle size range fractions: 1-2 mm, 3-4 mm and 4-6 mm.

Specific surface area and pore volume of samples of the γ-Al₂O₃ porous matrix and the composite sorbent were determined from nitrogen adsorption isotherms measured at 77 K on a Nova 1200e Surface Area and Pore Size Analyzer (BET method). Before the measurement, the samples were degassed at 150°C under vacuum for at least 3 h.

The phase composition for the composite sorbent was determined from powder X-ray diffraction (XRD) patterns, which were obtained on a Bruker D8 Advance diffractometer using Cu K_α radiation. Diffraction intensities were measured with the LynxEye position sensitive detector (2.9° angular range). XRD patterns were collected in 2θ range 10-70°, with 0.02° step size and 2s collection time. Variation of the phase composition of the composite sorbent owing to heating and cooling of the composite sorbent in air were studied by XRD analysis *in situ* by Siemens D-500 X-ray diffractometer equipped with graphite monochromator on the diffracted beam using Cu K_α radiation. Diffraction patterns were detected in the 10°-50° (2θ) region by points with the 0.05° (2θ) step. Measurements at elevated temperatures were carried out in a special X-ray chamber under ambient atmosphere. The sample of the as-synthesized composite sorbent was placed in the chamber and heated in air. Temperature of the chamber was gradually increased from 50°C up to 350°C by the step of 50°C. A diffraction pattern for the heated sample was measured after holding the sample at given temperature for about 15 minutes. Then the sample was cooled down to the room temperature, and a diffraction pattern was detected 12 hours after. Phase analysis was performed using ICDD PDF-2 and ICSD data bases. The size of the coherent scattering domain (CSD) for constituent phases was estimated using by Fundamental parameter approach [11].

CO₂ absorption and desorption experiments

Processes of carbon dioxide absorption from ambient air and consecutive thermal desorption were studied in a cyclic mode (TSA test) in the experimental apparatus schematically shown in Figure 1. Installation of four absorbers connected in parallel was used for simultaneous testing the sorption properties of several samples with different size fractions (Table 1). Heating control and redistribution of the gas stream to the reactors were carried out automatically using the controllers MVU-8 (OWEN, Russia) and Thermodat-13K2 (Thermodat, Russia). The composite sorbent was placed into a cylindrical glass tube

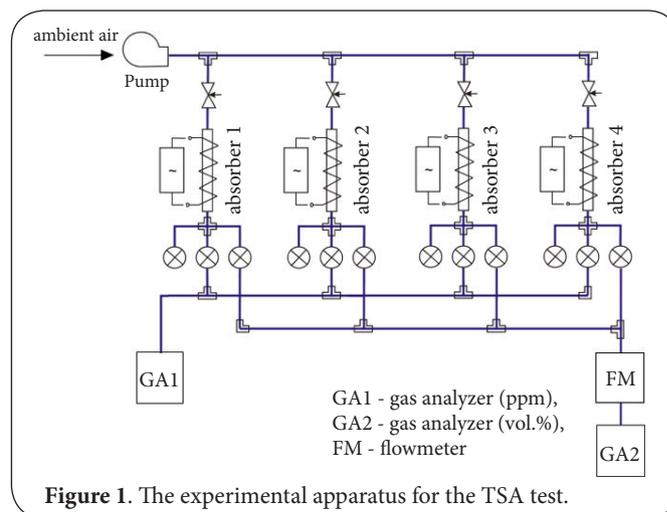


Figure 1. The experimental apparatus for the TSA test.

Table 1. Samples of the $K_2CO_3/\gamma-Al_2O_3$ composite for TSA tests.

Absorber	Fraction, mm	Mass, g
1	1-2	2.64
2	1-2	2.69
3	3-4	2.59
4	4-6	2.56

absorber 6.5 mm ID located inside an electrical heater. Indoor air was pumped through the reactor continuously using the air pump FIAC CCS 100-335. Pressure drop at the absorber inlet was maintained constant during the experiments. Air flow rate was controlled at the absorber outlet by the flow meter SMC PFM510-F01-2.

A single TSA cycle comprised 2 steps: 1) CO_2 absorption from ambient air for 6 h; 2) the composite sorbent thermal regeneration for 2 h. Two regeneration temperatures were used in TSA cycles: 250 and 300°C. The next CO_2 absorption step started right after the regeneration, as the absorber was cooling down to room temperature. Outlet concentrations of CO_2 during regeneration step were measured by the gas analyzer Test-1.1 (BONER, Russia) in the range from 0 up to 5 vol. % with accuracy of 0.05 vol. %. The gas analyzer OPTOGAZ-500.4C (OPTEC, Russia) was used for measuring outlet concentration of CO_2 during cooling (sorption) steps in the range from 0 to 2000 ppm with relative error of 3%. The summary of experimental conditions of TSA tests is presented in **Table 2**.

Table 2. The experimental conditions of the TSA tests.

Sorption step	
Step duration	6 hours
Air flow rate	20-24 ml/s
Desorption step	
Heating rate	12 K/min
Step duration	2 hours
Air flow rate	20-24 ml/s
Air flow direction	Concurrent
Regeneration temperature	250°C; 300°C

CO_2 outlet concentration curves $c(t)$ [vol. %] measured during the regeneration step were used for calculation of specific CO_2 flow $Q(t)$ [$cm^3/s/g$] and the absorption capacity of the material a_v [cm^3/g]:

$$Q(t) = \frac{U(t) \cdot c(t)}{m}, \quad (4)$$

$$a_v = \frac{1}{m} \cdot \int_0^t U(t) \cdot c(t) dt, \quad (5)$$

where $U(t)$ [cm^3/s] is the air flow rate and m is the mass of the

composite sorbent [g].

The value of the absorption capacity was also calculated in wt. % (a_m), using the ideal gas approximation:

$$a_m = \frac{a_v}{V_m} \cdot M(CO_2) \cdot 100\% \quad (6)$$

where V_m is the molar volume of the ideal gas ($2.44 \cdot 10^4$ cm^3/mol for the standard conditions: ambient temperature and pressure of 298 K and 1 bar, respectively), $M(CO_2)$ is molar weight of CO_2 (44 g/mol).

Results and discussion

CO_2 absorption and desorption behaviors for different fractions of the composite sorbent

The effect of the grain size on CO_2 absorption and desorption behavior of the composite was investigated in the TSA cycles with $T_{reg} = 250^\circ C$. One can see that the minimal value of outlet CO_2 concentration during absorption differs significantly for the fractions with different characteristic grain size (**Figure 2**). The lowest prebreakthrough level of CO_2 concentration (about 200 ppm) was reached by 1-2 mm granules. In comparison, for 4-6 mm granules the minimal CO_2 concentration detected during the absorption run was as high as 450 ppm. Nevertheless, one can see that, irrespective of the grain size, at the end of the absorption step outlet CO_2 concentration becomes equal to the inlet one, which indicates that the CO_2 absorption rate becomes negligible.

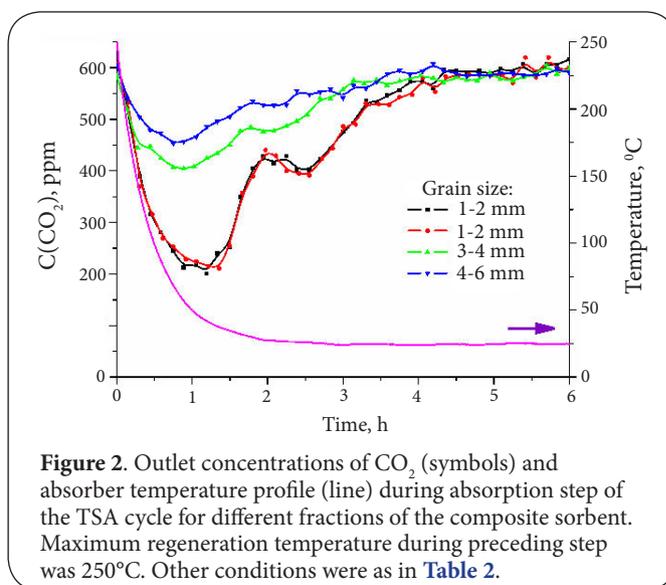


Figure 2. Outlet concentrations of CO_2 (symbols) and absorber temperature profile (line) during absorption step of the TSA cycle for different fractions of the composite sorbent. Maximum regeneration temperature during preceding step was 250°C. Other conditions were as in **Table 2**.

The desorption curves detected during thermal regeneration step with $T_{reg} = 250^\circ C$ (**Figure 3**) show that the grain size also affects the maximal value of CO_2 concentration, which is reached after 8-9 minutes of heating at the temperature of about 150°C. It should be noted that repeatability of the CO_2 absorption and desorption behavior was proved using the

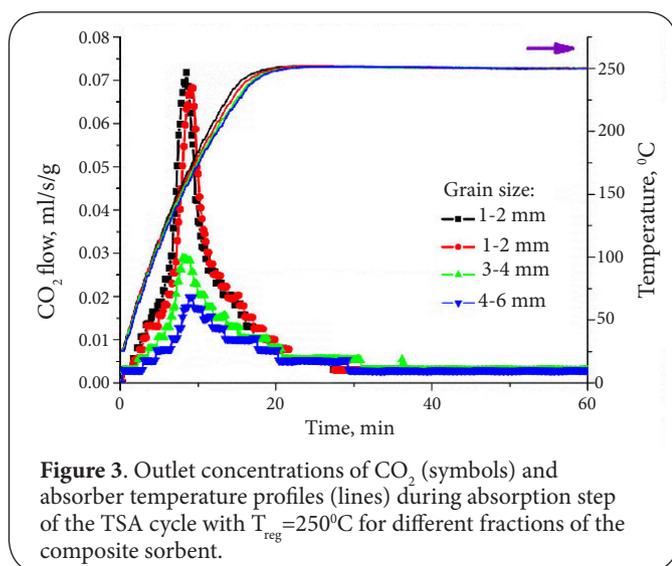


Figure 3. Outlet concentrations of CO₂ (symbols) and absorber temperature profiles (lines) during absorption step of the TSA cycle with T_{reg}=250°C for different fractions of the composite sorbent.

granules of 1-2 mm grain size placed inside two different absorbers.

The values of CO₂ absorption capacity, which were calculated from desorption curves, were found to be fairly stable for all the fractions of the composite for ten consecutive TSA cycles with T_{reg}=250°C (Figure 4a). Ultimately, the decrease in the grain size from 4-6 mm to 1-2 mm resulted in increasing CO₂ absorption capacity from 10 to 25 cm³/g.

It was shown that all the fractions of the composite showed a gain in CO₂ absorption capacity as a result of increasing regeneration temperature from 250 to 300°C (Figure 4b). The absolute value of CO₂ absorption capacity in the TSA cycles with T_{reg}=300°C was still strongly influenced by the grain size. The best result was obtained by the sample consisted of granules with 1-2 mm sizes, which demonstrated CO₂ absorption capacity a_v of 34 cm³/g (a_m ≈ 6 wt. %) and the utilization extent of potassium carbonate about 85%.

The grain size effect on the integral CO₂ absorption capacity of the material indicates that either the absorption or the desorption step of the TSA cycle is limited by the mass transfer within the grain. As a result of this limitation, the part of the supported alkali component in the larger granules is apparently unable to absorb CO₂ from air during the cyclic process. In order to study this effect in more depth we compared some characteristics of the big granules of the composite sorbent before and after TSA cycling.

Chemical composition of the composite sorbent

Potassium loadings, which were determined by means of atomic absorption spectroscopy for the samples of the composite sorbent before and after TSA experiments, turned out to be 12.5 and 12.3 wt. %, correspondingly. These values are equivalent to 21.8-22.1 wt. % of K₂CO₃. Thus, the TSA cycling procedure does not lead to the significant loss of the supported alkali component.

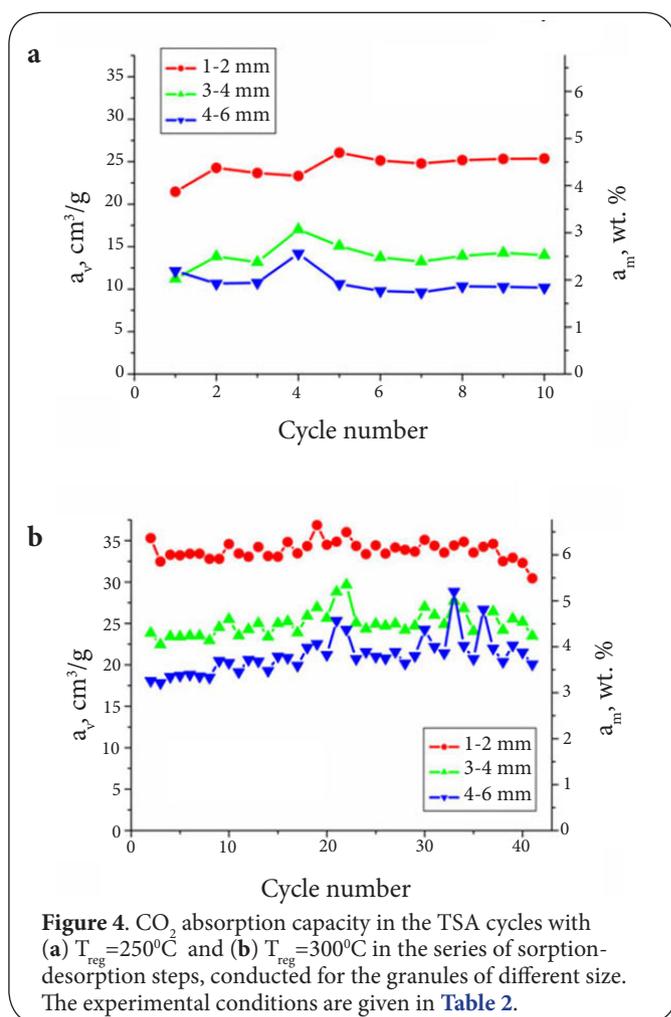


Figure 4. CO₂ absorption capacity in the TSA cycles with (a) T_{reg}=250°C and (b) T_{reg}=300°C in the series of sorption-desorption steps, conducted for the granules of different size. The experimental conditions are given in Table 2.

Porous structure of the composite sorbent

Porous structure of the host matrix and the composite before and after TSA cycling with T_{reg}=250°C was characterized by BET method. The alumina matrix used for synthesis of the composite material is a mesoporous material which demonstrates a bimodal pore size distribution (PSD) with one peak centered at 4 nm and another one characterized by a maximum at 19.5 nm (Figure 5). Modification of mesoporous alumina with the alkali chemisorbent predictably led to the decrease in both the specific surface area and pore volume in comparison to those for pure γ-Al₂O₃ (Table 3). The PSD for the as-synthesized composite sorbent differs noticeably from the one for the unmodified alumina (Figure 5) as the first peak at 4 nm decreases and the second peak shifts from 19.5 to 16 nm. The likely explanation for the latter effect is that the supported alkali component covers the inner surface of γ-Al₂O₃ as a thin layer resulting in pore narrowing.

The composite sorbent after TSA cycling is characterized by lower values both the specific surface area and the total pore volume compared to those for the as-synthesized material (Table 3). The comparison of PSDs for the composite

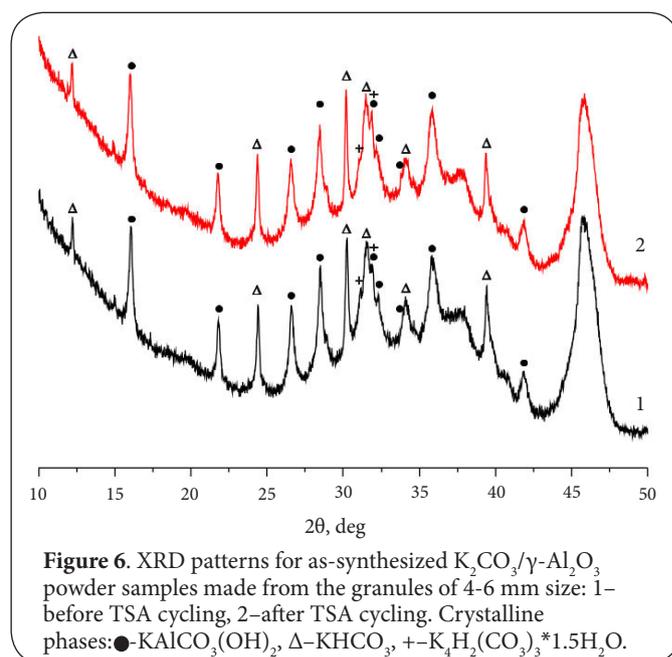
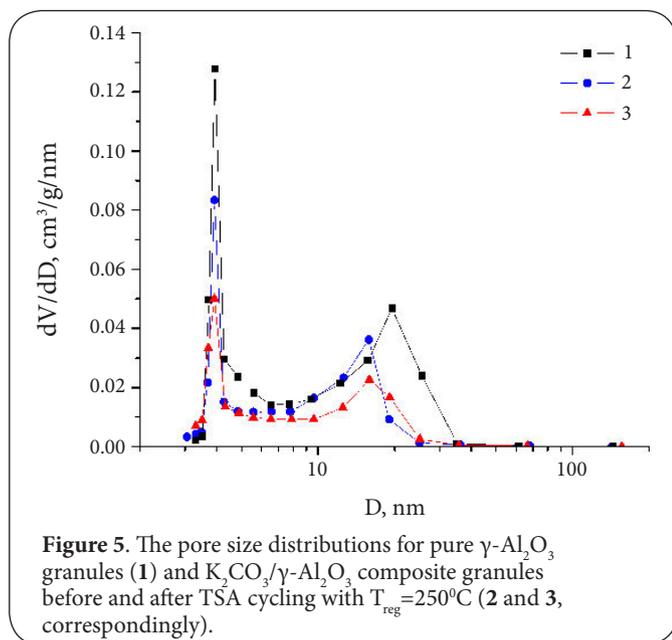


Table 3. Porous structure of $\gamma\text{-Al}_2\text{O}_3$ and $\text{K}_2\text{CO}_3/\gamma\text{-Al}_2\text{O}_3$ granules.

Sample	Surface area, m^2/g	Pore volume, cm^3/g
$\gamma\text{-Al}_2\text{O}_3$	197	0.72
$\text{K}_2\text{CO}_3/\gamma\text{-Al}_2\text{O}_3$ (before TSA cycling)	99	0.35
$\text{K}_2\text{CO}_3/\gamma\text{-Al}_2\text{O}_3$ (after TSA cycling)	80	0.29

before and after TSA cycling shows that used material has less mesopores with the characteristic sizes of 4 nm and 10-16 nm, but slightly more mesopores with the characteristic size about 19 nm. This change in the PSD is likely be caused by redistribution of the active alkali component inside the porous media of the alumina matrix.

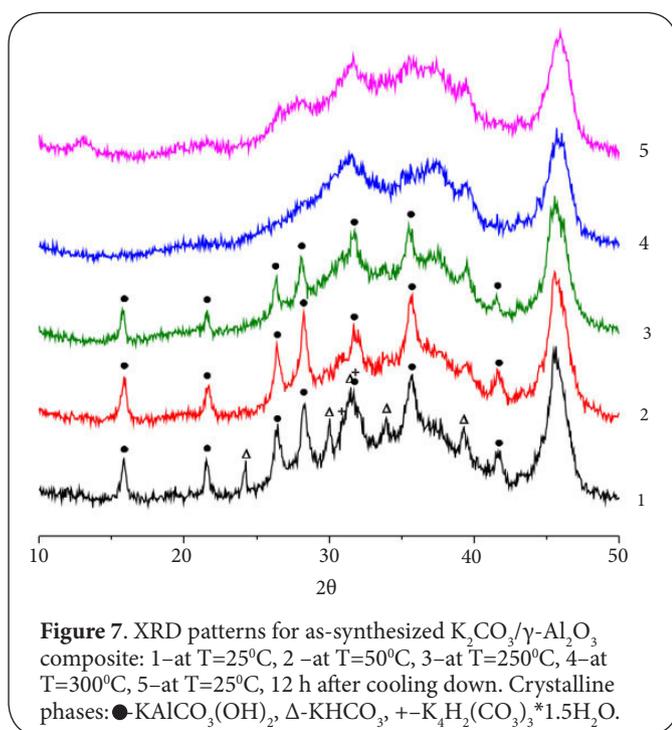
Phase composition of the composite sorbent

Crystalline phases of potassium-containing species for the composite sorbent have been characterized by means of XRD analysis (Figure 6). The main diffraction peaks observed for the sample of the composite at ambient temperature were attributed to KHCO_3 (Kalicinite, P2/a, ICDD PDF-2 #12-0292) and $\text{KAICO}_3(\text{OH})_2$ (potassium dawsonite, Cmcn, ICSD #153303). Additional peaks at $2\theta=30.98$, 31.76 and 33.68° were assigned to mixed potassium carbonate-hydrocarbonate phase $\text{K}_4\text{H}_2(\text{CO}_3)_3 \cdot 1.5\text{H}_2\text{O}$ (Pbam, ICDD PDF-2 #20-0886). Widths diffraction maxima corresponding KHCO_3 phase for the both samples are narrow and compatible with the widths of a standard sample that indicates the coherent scattering domain (CSD) size is >100 nm. The CSD size of $\text{KAICO}_3(\text{OH})_2$ phase is 18 nm for the as-synthesized material and 14 nm for the composite after TSA cycling with $T_{\text{reg}}=250^\circ\text{C}$. The results of

XRD analysis indicate that the crystals of potassium dawsonite and bicarbonate seem to be spatially separated in the granule. Potassium dawsonite has the CSD, which is comparable with the average pore diameter of the alumina matrix. Therefore, the $\text{KAICO}_3(\text{OH})_2$ phase is likely to be dispersed inside the pores, on the internal surface of the alumina matrix, as it was previously stated in [12]. On the contrary, the CSD size for potassium bicarbonate phase is >100 nm, which is higher than the typical pore sizes for the mesoporous alumina matrix (see Figure 5). Thus, the KHCO_3 crystals are likely to be located on the external surface of the grain.

In order to explore structural transformations of the composite sorbent in the course of TSA cycling, XRD *in situ* experiment was carried out. The characteristic XRD patterns are presented in Figure 7. When the as-synthesized composite was heated from 25°C to 50°C , the reflections corresponding to potassium bicarbonate vanished. This temperature is too low for thermal decomposition of potassium bicarbonate, therefore the reflections disappearance is likely to be caused by dissolution of bicarbonate in the water adsorbed on the alumina surface. The aqueous solution is obviously more mobile than the crystalline phases, so the redistribution of the active alkali component inside the granule, which apparently affects the PSD of the composite, seems to be the result of the fluid-aided dissolution-precipitation of the supported components.

At 250°C intensities of the reflections corresponding to potassium dawsonite phase seem to be lower than at the room temperature, however, thermal destruction of $\text{KAICO}_3(\text{OH})_2$ is not complete until it is heated up to 300°C . It was previously reported that decomposition of $\text{KAICO}_3(\text{OH})_2$ phase occurred in the temperature range of $260\text{--}320^\circ\text{C}$ with formation of crystalline K_2CO_3 [13]. However, diffraction maxima, chara-



characteristic for K_2CO_3 crystalline phase, were not detected at elevated temperatures. It indicates that potassium carbonate produced as a result of thermal regeneration of the composite is likely to be X-ray amorphous. The XRD pattern detected 12 h after the composite was cooled down to the room temperature reveals that potassium-containing components of the composite still remain X-ray amorphous. However, the halo at $25-29^\circ$ indicates a likely formation of highly dispersed potassium dawsonite phase, which is in line with the results of our previous study [10].

$K_2CO_3/\gamma-Al_2O_3$ composite as a material for DAC combined with methanation process

There are several reasons why the $K_2CO_3/\gamma-Al_2O_3$ composite material should be considered as a promising adsorbent for CO_2 capture from ambient air with subsequent methane production. First of all, this composite material absorbs CO_2 directly from ambient air without any pretreatment and is thermally stable in multiple TSA cycles. In comparison, zeolites cannot absorb CO_2 directly from air due to their high affinity to moisture [14], while supported amine adsorbents suffer from degradation in the presence of oxygen-containing gases [15,16].

The composite sorbent based on potassium carbonate is also favorable for application in the processes combining CO_2 capture and methanation, because this material enables production of concentrated CO_2 stream during desorption stage, which is required for the effective methanation process. As it was estimated in [10], realization of the DAC/methanation process at ambient pressure means that the equilibrium CO_2

pressure over the sorbent must be above 0.17 atm in order to comply with optimal gas composition during combined desorption-methanation step. Using thermodynamic data for the reaction of potassium hydrocarbonate decomposition [17], it was calculated that the partial CO_2 and H_2O pressures of 0.17 atm are reached at the equilibrium reaction temperature of $135^\circ C$, while the further increase in the regeneration temperature results in the higher equilibrium CO_2 pressure.

The efficiency of renewable energy storage sequence, which includes direct air capture by $K_2CO_3/\gamma-Al_2O_3$ composite material and the subsequent methanation process, was estimated to be about 50%, taking into account that the heat released due to the reaction of methanation (2) can be utilized for thermal regeneration of the sorbent [10]. Straightforward implementation of the DAC/methanation process will be fulfilled experimentally in the nearest future.

Conclusions

Three different fractions of the composite sorbent $K_2CO_3/\gamma-Al_2O_3$ were tested in the cyclic process of direct CO_2 capture from ambient air with thermal regeneration of the material. It was found that dynamic CO_2 absorption capacity of the composite increases with decrease of the characteristic grain size, which indicates that the process is likely to be limited by mass transfer.

The change of regeneration temperature from 250 to $300^\circ C$ led to increase of absorption capacity for all the fractions of the composite. As a result, for 1-2 mm fraction absorption capacity reached the level of 6 wt. %. The XRD *in situ* experiment showed that the reason behind this effect is thermal decomposition of $KAlCO_3(OH)_2$ crystalline phase, which is likely to produce highly reactive K_2CO_3 species as a result of heating.

$K_2CO_3/\gamma-Al_2O_3$ composite should be considered as a promising material for the capture of air-borne CO_2 and subsequent methane production using renewable energy sources.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

Authors' contributions	JVV	VSD	TYK	AGO
Research concept and design	✓	✓	--	✓
Collection and/or assembly of data	✓	✓	✓	--
Data analysis and interpretation	✓	✓	✓	✓
Writing the article	✓	✓	✓	✓
Critical revision of the article	✓	✓	✓	✓
Final approval of article	✓	✓	✓	✓
Statistical analysis	✓	✓	--	--

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