MICROWAVE DESORPTION OF ZEOLITE FOR RENEWABLE ENERGY PROCESSES

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Abstract

This study investigates microwave-assisted desorption of zeolites as a innovative and energy-efficient method for adsorption applications in renewable energy systems. Comparative experiments using various zeolites reveal that microwave desorption significantly enhances drying efficiency compared to conventional oven methods. The results highlight the critical roles of initial water content, ion composition, and temperature control in optimizing energy use. These findings support the potential of microwave desorption for CO_2-neutral storage and purification processes, offering a promising pathway toward more efficient and decentralized energy systems.

Keywords: Zeolite, microwave desorption, thermal energy storage, renewable energy, energy efficiency, sorption, CO_2 reduction.

1. Introduction

The generation of renewable gases, including synthetic natural gas (SNG) and hydrogen (H2), is progressively reliant on sophisticated purification and separation methodologies to guarantee gas quality and process efficiency. Sorbents, including zeolites, activated carbon, and functionalized polymers, are utilized extensively throughout the production process, from the preparation of feedstock to the upgrading of gas and the application of catalysis.

In the gas purification stage, zeolites are employed to remove water vapor, NH_3, and other polar contaminants or even unipolar CO_2 from biogas, syngas, or hydrogen streams. Their high selectivity and thermal stability make them ideal for pressure swing adsorption (PSA) and temperature swing adsorption (TSA) processes. For instance, zeolite 13X has been shown to effectively adsorb moisture and CO_2 from wet flue gas streams, while activated carbon and metal oxides are used for sulfur compound removal (Donkers, et.al. 2017; Wang, et.al. 2007)

In the gas separation phase, zeolites such as 5A and 13X are used for hydrogen purification, while metal-organic frameworks (MOFs) and alkali-promoted hydrotalcites offer high selectivity for CO_2 separation. Studies by Boer et al. (2023) and Faria et al. (2022) have demonstrated the effectiveness of these materials in cyclic adsorption processes for CO_2 capture.

However, conventional thermal desorption methods for regenerating these sorbents are often energy-intensive and slow due to the poor thermal conductivity of granular materials. Microwave-assisted desorption offers a promising alternative, as microwaves can penetrate sorbent beds and heat materials volumetrically, enabling faster and more energy-efficient regeneration—provided the dielectric properties of the sorbent are favorable.

This technique is particularly relevant for non-flammable gas environments, such as CO_2 and H_2O separation, where microwave processes can be safely applied without hazard in case of spark formation or electrical discharge. Recent research has explored microwave desorption in sorption-enhanced methanation (SEM) and water gas shift reactions, highlighting its potential to improve process efficiency and reduce energy consumption (Li, et.al. 2019)

For example, Gómez et al. (2023) optimized zeolite-catalyst mixtures for SEM applications, while Xu et al. (2023) demonstrated high-purity hydrogen production using sorption-enhanced water gas shift with intermediate-temperature CO_2 sorbents. Purdue and Qiao (2018) provided molecular insights into moisture adsorption on zeolite 13X, supporting its role in vapor management.

This article investigates the potential of microwave desorption for zeolite regeneration in renewable energy systems, focusing on its thermodynamic advantages, operational constraints, and integration into existing gas purification and synthesis workflows.

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2. Materials and Method

2.1. Microwave Desorption and Technical Innovation

Thermal regeneration of sorbents like zeolites is challenged by their poor thermal conductivity, which limits conventional heating approaches in packed beds. To address this, microwave-assisted desorption techniques are gaining traction. Microwave heating enables volumetric and selective energy transfer, particularly efficient in hydrated zeolites due to the high dielectric loss of water (Clark and Sutton, 1996; Menéndez et al., 2010). Studies have demonstrated that this method enhances regeneration rates, reduces energy losses, and maintains the structural integrity of the zeolite bed (Petkovska and Tsiakaras, 2000; Tang et al., 2005).

Innovative application of this method involves coupling microwave heating directly with photovoltaic (PV) power sources for sustainable zeolite regeneration. By synchronizing magnetron operation with PV maximum power point tracking, the system enables efficient, off-grid, and dynamic desorption processes, paving the way for cost-effective and carbon-neutral energy storage and drying technologies.

Two different measurement techniques were employed, including a multimode microwave oven with a wave stirrer and internal weight measurement and temperature control, and conventional drying in a hot air oven. A weighing device was incorporated into the design of the microwave oven in order to facilitate the monitoring and recording of the weight loss of the material sample. The magnetron has a nominal output of 1000W, with typical conversion losses of 35%. The emitted wave has a frequency of 2.45GHz. Three infrared temperature sensors are positioned above the material sample, allowing for the monitoring of the temperature of the zeolite bed. A power meter is employed to quantify the electrical power consumption of the microwave. Comparative measurements in a conventional drying oven are conducted to facilitate a comparison of the efficiency of the two desorption routes. The measurements in the drying oven were determined with other sample weights due to the size of the oven. The electrical power was only measured during the desorption process, and the preheating of the empty oven was not taken into account in efficiency comparison. Alle material moisture measures are mentioned as amount of water compared to the dry mass of the samples. The dry mass was established by exposure of the material in a oven at 300°C for min 4 hours. Details of the measurement setup were already published (Zettl, et.al 2024).

2.2. Sorbents and gases

A variety of different zeolite types were used to study their desorption behavior under the influence of microwaves: commercial LTA Type like 4A, 4A binderless, 3A and 5A (purchased from company CKW and SILKEM). The granule size is between 1.5-2.5 mm. Material treatment in the form of salt impregnation (2% LiCl wet impregnation) was done for some zeolites.

Table 1: List of investigated sorbents

Material[1]	Company	Type
YBF, YK, NaY-10B	CWK[2]	Varieties of Faujasit (Y-Typ)
13XBF, 13XK	CWK	Varieties of Faujasit NaMSX
4ABF, 4AK	CWK	Varieties of LTA-Typ
5AK	CWK	LTA zeolite, potassium modified
3ABF, 3AK	CWK	LTA zeolite, potassium modified
4A SILKEM	SILKEM[3]	LTA-Type
Clinoptilolite	IPUS[4]	Natural zeolite, heulandite
Thompsonite	ZMM Canada[5]	Natural zeolite
KC-N BASF	BASF	Sorbead® R, silica gel

[1] Letters BF refer to the binderfree variants, K indicates attpugite as binder
[2] Chemiewerke Bad Köstritz, https://www.cwk-bk.de/
[3] SILKEM, https://silkem.si/de/
[4] IPUS, https://www.ipus.at/
[5] ZMM Canada, https://zmmcanadamineralscorp.com/

To determine the CO_2 adsorption capacity of zeolite samples, first, the samples are completely desorbed at 250°C for a period of 6 to 12 hours to remove all gases. Immediately afterward – while still hot – the samples are weighed and transferred to a pressure vessel. In the vessel, the samples are then pressurized with CO_2 at 1 bar. Adsorption normally takes place for several hours until the sorbents reach room temperature. After the adsorption phase, the samples are removed and weighed again. The difference from the initial mass corresponds to the amount of CO_2 absorbed. For desorption, the samples are then stored in room air. The water vapor contained in the air displaces the adsorbed CO_2, as zeolites have a significantly higher affinity for water than for CO_2. Alternatively, water absorption can be prevented by heating the zeolite with a microwave immediately after extraction and then recharging it with CO2.

3. Results and discussion

3.1. Water desorption

A series of desorption experiments were conducted using different sorbents. All materials exhibit an initial heating phase, followed by a subsequent dehydration phase. The termination of experiments occurred after a duration of 30 minutes, or in instances where the materials attained a water content that fell below 2%. The microwave power was decreased once the temperature in the reaction bed attained 200°C, with the objective of averting excessive heating that could potentially result in a thermal runaway event. As illustrated in Figure 1, the material's water content, temperature, and drying efficiency are demonstrated for two distinct materials. Drying efficiency refers to the proportion of the microwave's electrical energy consumption that directly leads to water desorption from a sample, thus causing a measurable weight loss, relative to the total consumption. This only takes into account the energy required to overcome the evaporation enthalpy and the adsorption enthalpy of the water, but not energy required for heating the sample or other losses (e.g., heat conduction, radiation losses, ambient heating).

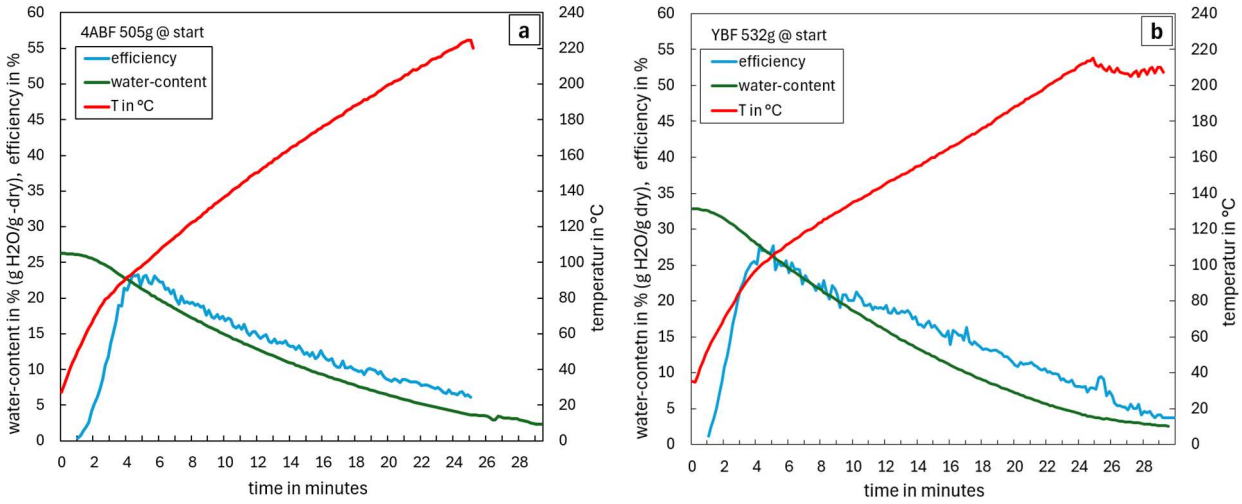

Fig. 1: Desorption measurements (a) zeolite 4A binderfree LTA type (b) zeolite Y-Faujasite

Figure 1 shows the desorption curves of two zeolites. 4ABF (left, initial mass 505 g) starts with 26% water content at 20°C and is dried to approximately 5% at 220°C after 24 minutes. The stored residual heat dries the material to 2% water content. The efficiency is initially low because the heating requires energy, reaching a peak of 23% after 5 minutes and decreasing again to 7% as desorption progresses. Fig 1 right shows a similar behavior for the desorption of zeolite YBF, the water content at the beginning is higher (33%), the maximum efficiency (26%) also.

In general, the desorption process is very similar for all zeolites, with varying maximum efficiency values. To test the influence of free ions in the crystal lattice on heating, several materials (13XBF, YK, 5AK, 4AK, and 3AK) were impregnated with LiCl salt. The salt was dissolved in water and impregnated into the zeolite. The goal was to achieve a 2% salt content by weight in the zeolite after drying.

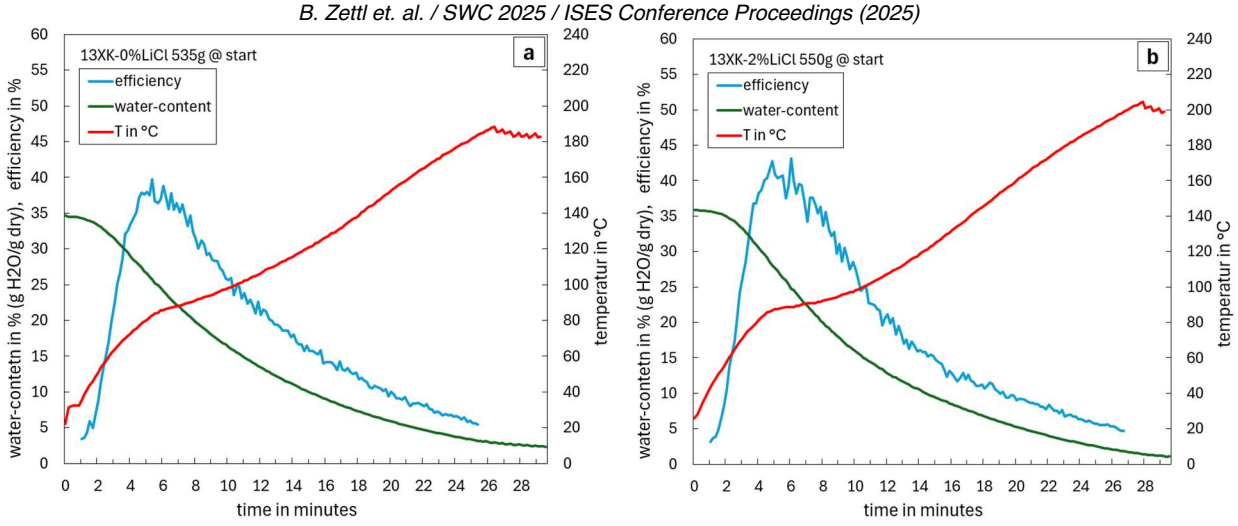

Fig. 2: Desorption measurements (a) zeolite 13XK (b) 13XK salt impregnates

Figure 2 shows a comparison of the desorption of untreated (left) and impregnated 13XK zeolite (right). The curves are very similar; the drying rate, peak efficiency, and the temperature reached indicate only a very slight influence of the impregnation. Similar results were also achieved with the other impregnated zeolites.

The first phase of the drying process is characterized by high efficiency up to approximately 100°C (peak efficiency). Subsequently, efficiency decreases, which is due to the decreasing moisture content in the material. A clear correlation between the maximum efficiency and the material type could not be found.

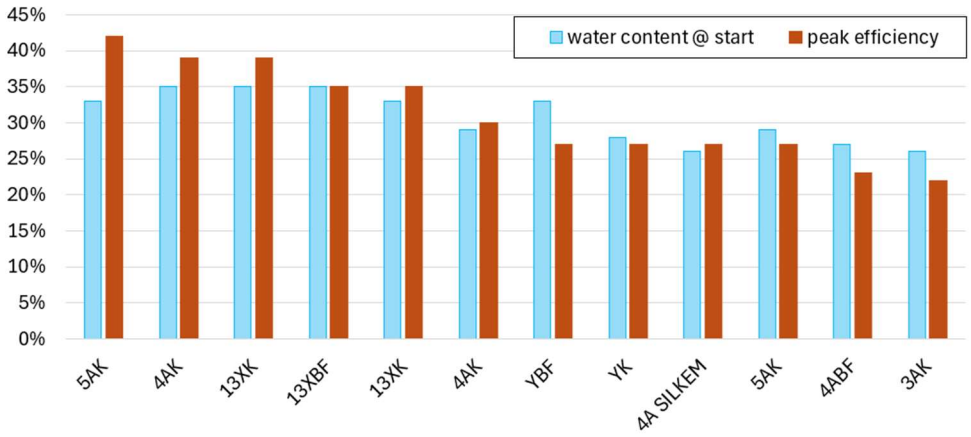

Fig. 3: Peak efficiency and water content (at start of the desorption process) for different sorbents (repeated date for zeolite 13XK and 5AK from different experiments).

Figure 3 shows the relationship between peak efficiency and the initial moisture content of the material. This comparison reveals a certain correlation between the two parameters. Although an influence of the material type cannot be ruled out, it is not clearly visible in the available data.

High maximum drying efficiency values were observed particularly frequently at high initial moisture levels. Zeolites with a particularly high pore volume, such as 13XBF, 13XK, YBF, and YK, can adsorb a significant amount of water and exhibit high values. However, this can also be demonstrated by adding water to zeolites with an otherwise lower efficiency. To demonstrate this, zeolite 5AK was moistened to 39% well beyond its pore capacity (Fig 3-left). Free water is therefore present in the macropores of the binder and on the surface of the granules. This water content also leads to a good initial efficiency (43%), although the drying efficiency is comparatively low at higher temperatures (110°C to 170°C).

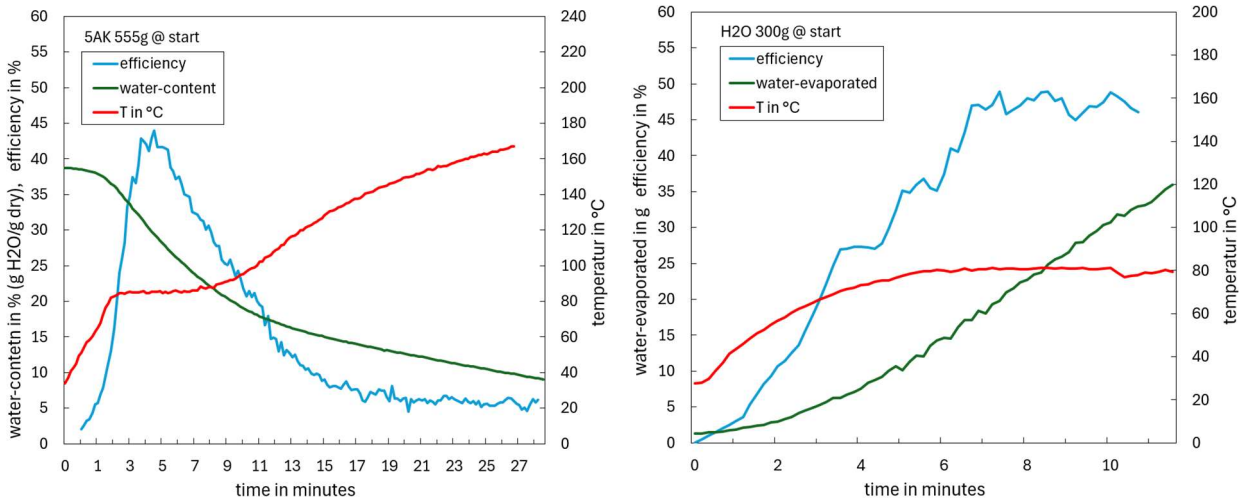

Fig. 3: Water evaporation caparison: overhydrated zeolite 5AK (left) and liquid water (right)

The maximum efficiency that can be measured with pure water is shown in Figure 3- right. After initial heating of water in a very shallow dish with the same diameter as used for zeolite desorption, the temperature rises to 80°C, at which point the water evaporation rate reaches its maximum. 47% of the total electrical power consumed is used for evaporation at this time, which represents the technical limit for maximum efficiency in this setup. The initial drying efficiency of the 5AK material with its high free water content almost reaches this value.

Table 1: Average desorption efficiency for water desorption in zeolites in the temperature range from 110°C to 180°C

Material	Mass at start in g	Efficiency 110°C-180°C
YBF	532	17,6%
4ABF	505	14,6%
YK	512	14,1%
4AK	539	13,8%
13XBF	540	13,1%
4A SILKEM	497	12,5%
3AK	535	12,5%
13XK	530	11,7%
5AK	555	6,6%

The second phase of the desorption process (after passing the peak efficiency) extends over a temperature range of approximately 110°C to 180°C. In this phase, a material-dependent efficiency can be observed. Repeated experiments with different initial moisture contents but the same material show comparable drying efficiencies between 110°C to 180°C. A common explanation in the literature is that at low residual moisture levels, microwave absorption depends primarily on the availability of mobile ions in the crystal structure of the zeolites. These ions, together with the remaining water content, largely determine the heating of the material. Table 1 lists the material-specific efficiency of the tested zeolites. YBF has the highest efficiency at 17.6%, while 5AK has the lowest at 6.6%.

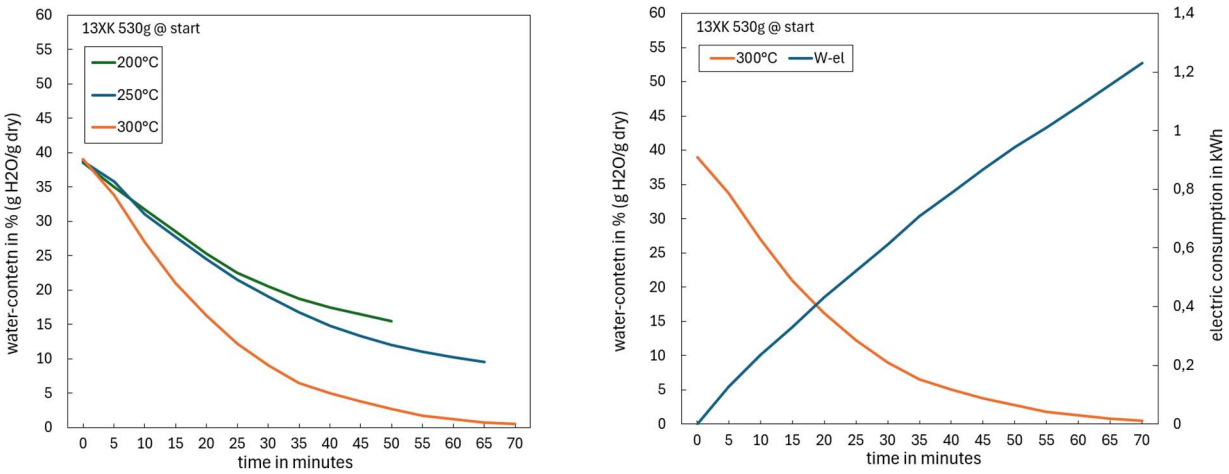

Fig. 4: Desorption measurements in conventional drying oven: zeolite 13XK at different temperatures (left) and related power consumption at 300°C (right)

For comparison with conventional oven drying, samples with similar starting masses were dried in a conventional drying oven (brand Nabertherm, with N_2 purge of 2 nl/min) at different temperatures. The starting mass, the sample weight at regular intervals, and the energy consumption were recorded (oven heating not included). Figure 4 (left) shows the weight loss of zeolite 13XK over approximately 60 minutes of oven exposure at 200°C, 250°C, and 300°C. The higher the temperature, the higher the drying rate and the achieved final moisture content. Figure 4 (right) shows the electrical energy consumption from the start of drying. With higher sample water content, the increase is somewhat greater than with lower moisture content. The data analysis shows that the average drying efficiency is 10.8% at 200°C, 8.8% at 250°C, and 4.9% at 300°C of the electrical consumption. Higher heat losses and slower heating compared to microwave desorption characterizes the process.

3.2. CO_2 desorption

The adsorption of pure CO_2 gas at an overpressure of 1 bar (equivalent to 2 bar absolute) achieved values between 2.5 and 5.4 mmol/g for the synthetic zeolites tested (Fig. 5). In contrast to water adsorption, where the mass or volume of adsorbed water is often specified, the amount of substance in millimoles per gram of sorbent (mmol/g) is usually used for characterization in the case of CO_2. The peak value of 5.4 mmol/g corresponds to a weight fraction of 25%. The desorption of CO_2 can, in principle, also occur without microwave radiation, in a cold state by storage in room air. This is because water molecules have a significantly stronger adsorption enthalpy due to their high dipole moment and can displace the adsorbed CO_2.

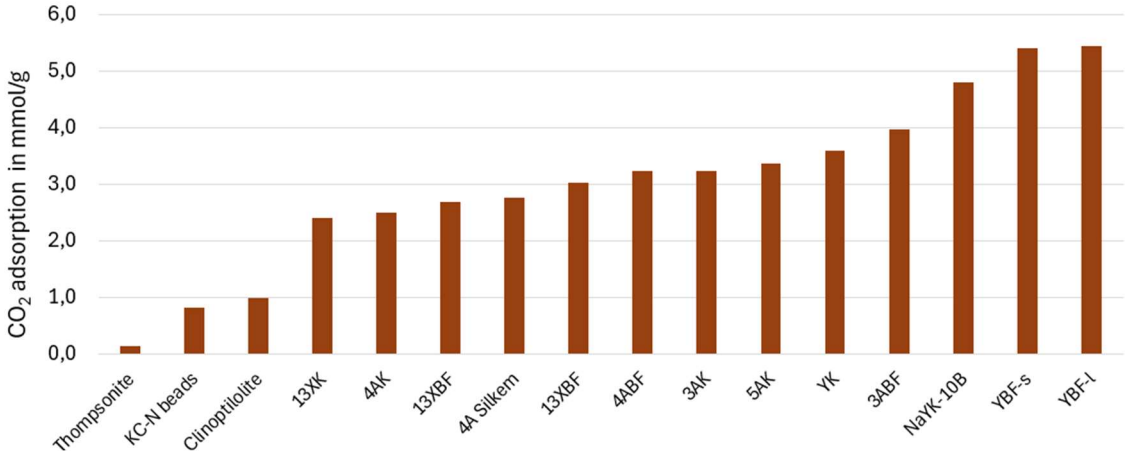

Figure 5: CO_2 adsorption in different sorptive materials contained within a pressurized vessel at 1 bar gauge pressure

However, for efficient CO_2 reloading, it is energetically more advantageous to first heat the zeolite to specifically release the CO_2 and then cool it in an airtight container. This process regenerates the sorbent with less energy, as CO_2 binding is weaker than that of water. The comparatively low reaction enthalpy can be explained by the fact that water undergoes a phase change (gas → liquid) during adsorption, releasing a high reaction enthalpy. CO_2, on the other hand, remains gaseous within the zeolite pores even in the adsorbed state because, unlike water, it has a much lower critical temperature (31°C instead of 374°C). The desorption of CO_2 therefore occurs faster and with less energy than water.

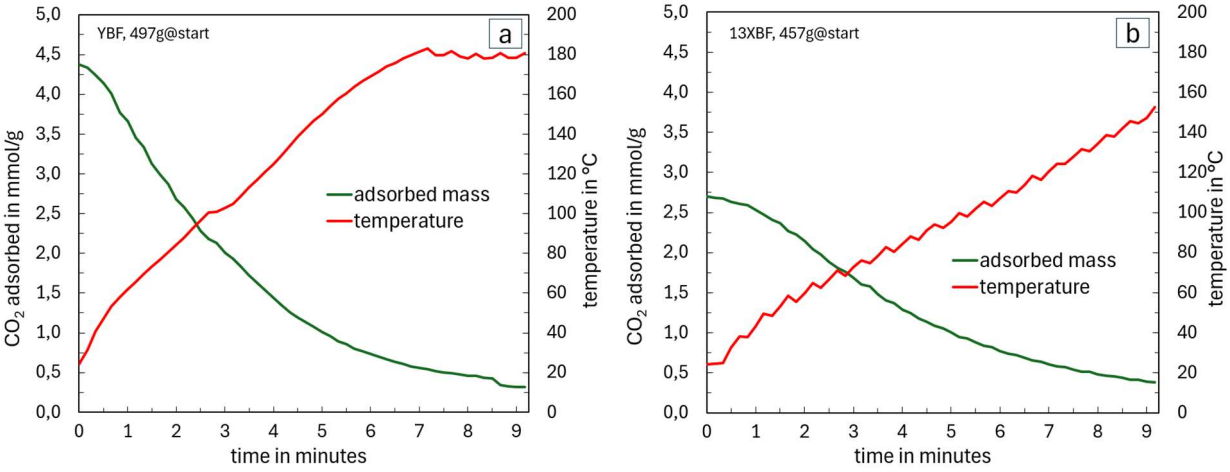

Figure 6: (a) Desorption of CO_2 from type YBF zeolite (binderfree), (b) from 13XBF at lower microwave power

Figure 6 shows the mass loss and temperature increase during CO_2 desorption from zeolites YBF and 13XBF. Zeolite YBF exhibits a significantly higher CO_2 adsorption capacity than 13XBF and heats up significantly more rapidly, even without the presence of water molecules (compare Fig. 6b). After just 7 minutes, all of the CO_2 is released, and at the same time, a temperature is reached at which no water adsorption occurs. A comparison of Fig. 6a with the water desorption of YBF (Fig. 1b) shows that heating occurs faster during CO_2 desorption and requires less energy therefor.

4. Conclusion

This study demonstrates the potential of microwave-assisted desorption as a highly efficient and sustainable method for regenerating zeolitic sorbents in thermal energy storage and gas purification applications. By integrating microwave heating with photovoltaic power sources, the process enables in principle off-grid operation and significant energy savings. The experimental results highlight the influence of material type, initial moisture content, and ion composition on desorption efficiency. Furthermore, the comparison with conventional oven drying confirms the superior performance of microwave techniques in terms of speed and energy utilization. Key Findings are:

- Microwave desorption significantly improves drying efficiency compared to conventional oven methods.
- Binderless zeolites (e.g., 4ABF, YBF) show higher desorption efficiency due to better heat transfer and active surface.
- Initial water content strongly correlates with peak desorption efficiency.
- LiCl impregnation has only a minor effect on desorption performance, despite increased ionic mobility.
- Zeolite YBF achieved the highest average efficiency (17.6%) in the 110–180 °C range.
- Overhydrated samples (e.g., 5AK with 39% water) can temporarily reach near-maximum efficiency (~43%).
- Microwave heating allows volumetric energy transfer, reducing thermal gradients
- Reduced microwave power at material temp >200°C avoids hot spots with thermal runaway
- CO_2 desorption is faster and less energy-intensive than water desorption due to lower adsorption enthalpy.

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