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Water harvesting from air with a hygroscopic salt in a hydrogel-derived matrix

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The extraction of water from air is a promising way to supply fresh water, especially in remote, arid regions. This process can be supported by desiccant materials such as zeolites, metal–organic frameworks, or hygroscopic salts. Here we present a composite material that is able to absorb 660 kg of water per cubic meter of bulk material from air at 10 mbar water vapor pressure and 28 °C. The material consists of calcium chloride incorporated into an alginate-derived matrix. A simple synthesis route leads to spherical beads of the composite with a diameter of approximately 2 mm. This macroscopic structure allows for good vapor permeability through packed beds. The collected water can be released at 100 °C, potentially enabling a solar-driven application. In addition, the synthetic route uses cheap, non-toxic, and easily accessible materials allowing for widespread application.

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It is estimated that around four billion people on the globe suffer from water scarcity¹. Distribution and production of fresh water and the control over water sources are a potential cause for conflicts. Therefore, new ways of fresh water production and improvement of existing techniques are the focus of current research. The utilization of water from lakes and rivers, the exploitation of rainfall runoff and the desalination of salt water are being used extensively to cover the demand of fresh water². Particularly, the desalination of seawater requires high initial investment cost and is mainly feasible on large scale in coastal areas, making it inaccessible for remote regions and decentralized allocation. The use of atmospheric water as source of fresh water has been investigated extensively, but is still underdeveloped^{3–17}. There are two general options for water harvesting from air: 1) Ambient air can be cooled below its dew point and the condensed liquid water can be collected. Large volumes of air and low temperatures at the condenser are required, especially in regions of low relative humidity (r.h.). The cooling of the heat sink for the condensation can be energy consumptive, since a difference in temperature between the condensation unit and the ambience has to be maintained. Additionally, the heat of condensation of the collected water has to be discharged. 2) Humidity can be adsorbed or absorbed (taken up) by desiccant materials, and in a second step desorbed at elevated temperatures followed by condensation of the hot water vapor at ambient conditions. Usually water is taken up during the colder nighttime when the relative humidity is higher and desorbed during daytime. The necessary heat for desorption is ideally provided by solar radiation. The resulting heated water vapor can be brought below its dew point and condensed at ambient temperatures, making an energy expansive auxiliary heat sink redundant. When the ambient air is used as coolant, a sufficient airstream has to be maintained to reject the heat of condensation. Applications for dew point water collection and fog capturing have been presented, but they require high relative humidities, usually above 50%^{3–9}. Those conditions are usually found in regions that offer other possibilities for fresh water production such as water extraction from lakes, rivers or oceans. Only a few applications have been presented that are able to harvest water from air at relative humidity below 30%. These conditions are especially interesting because regions where the relative humidity is low usually offer no efficient ways for fresh water production. Hygroscopic salts^{10–12}, zeolites¹³, porous silica-based materials¹⁴ like MCM-41¹⁵, carbon based composites¹⁶ or metal-organic frameworks¹⁷, such as MOF-801¹⁸, are capable of taking up water at relative humidities below 25%. The adsorption of water on zeolite surfaces at low humidities is accompanied by high adsorption enthalpies of more than 70 kJ mol⁻¹, requiring temperatures above 150 °C for the desorption, making this process energy consumptive^{17,19}. MOFs, such as MOF-801, can readily absorb water from air at around 10% relative humidity

and are able to release it at temperatures below 100 °C. The major problem of materials that harvest water through adsorption on the material surface is the low gravimetric water uptake of around 25 wt.%^{18,20}. However, there are exceptions such as a MOF based on cobalt ions and a bis(1*H*-1,2,3-triazolo[4,5-*b*],[4',5'-*i*])dibenzo [1,4]dioxin (BTDD) linker that, shows a water uptake of around 80 wt.% at 25 °C and 30% relative humidity²¹. Up until now, it has been proven difficult to synthesize most of the suitable MOFs on a scale that allows for significant, cost-efficient water production. Apart from water harvesting through adsorption on the surface, it is possible to harvest water through absorption, e.g., hydration of salts. One of the most promising salts is calcium chloride because of its excellent water sorption properties in combination with a natural and industrial abundance. The salt is able to absorb more than 95% of its own weight in water for the reaction of the anhydrous salt to the hexahydrate. However, hygroscopic salts face the problem of particle agglomeration during hydration, which reduces the gas permeability. Additionally, passivation layers can be formed on the surface of the salt particles, which reduces the absorption capability. Composites of matrix materials that can adsorb water on their surface and hygroscopic salts that absorb water through hydration reactions have shown promising results^{15,16,22,23}. The salt is distributed within the matrix to prevent agglomeration while at the same time increasing the amount of water that can be taken up by the composite. A general problem for pristine, as well as composite materials is the macroscopic structuring. Ideally, a material should be prepared, which can be packed in beds or columns that are penetrable by air with a low pressure loss and high volumetric, as well as gravimetric water sorption capacity.

Here, we present an approach for the encapsulation of CaCl₂ into a polymer-based matrix for water harvesting purposes. A facile ionotropic gelation method is used to synthesize composite beads in the size of around 2 mm. Sodium alginate is chosen as basis for the matrix because of the excellent features of alginate-based hydrogels in terms of macroscopic shaping²⁴. In addition, alginates are a cheap, as well as non-toxic biomaterial that has been utilized in various applications^{25–28}.

Results

Synthesis and characterization. In the present approach, a CaCl₂ solution is used as a Ca²⁺ source for the ionotropic gelation of a sodium alginate solution (see Fig. 1). Droplets of the alginate solution form spherical hydrogel beads upon contact with the gelation solution that contains Ca²⁺ ions. After the initial hydrogel formation, Ca²⁺ and Cl⁻ ions from the gelation solution diffuse into the hydrogel spheres until an equilibrium of the salt concentration between the CaCl₂ solution and the hydrogel is reached. Upon drying of the hydrogel, CaCl₂

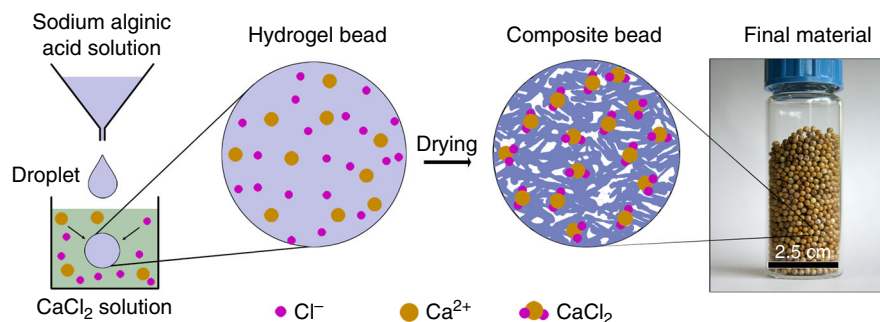


Fig. 1 Scheme for the production of the composite beads. Formation of hydrogel beads with incorporated CaCl₂ and subsequent drying to obtain the final composite beads comprising CaCl₂ and an alginate-based matrix

precipitates within the alginate-derived matrix and a composite is formed. The spherical shape of the hydrogel can be maintained throughout the drying process leading to the generation of spherical beads of an alginate-derived matrix with incorporated CaCl_2 . The size of the composite beads can be altered by the droplet size. The amount of incorporated salt can be adjusted by the concentration of the CaCl_2 solution. Here we used beads in the size of around 2 mm in diameter to obtain a balance between high packing density, easy vapor transport through the packed bed, and limited diffusion length within the individual beads. The final composite, referred to as Alg- CaCl_2 , contains 76 wt.% of salt. This is higher than the amount of salt that can be incorporated through conventional impregnation of matrices, which usually ranges from 30 to 58 wt.% in the case of silica-based materials^{15,18,22}, and can reach up to 68 wt.% in the case of MOFs²⁹.

Figure 2 shows scanning electron microscope (SEM) images of the dried composite. A porous structure was obtained with pores in the range from 1 μm to more than 10 μm in diameter. The performed cyclisation experiments indicate that the hydration of the incorporated salt is possible in a timeframe of 12 h. This leads to the conclusion, that the pores allow diffusion of water vapor within the individual beads. Salt particles cannot be observed in the SEM, indicating a fine distribution of the salt within the matrix. The presence of CaCl_2 within the composite was proven by energy dispersive X-Ray spectroscopy (EDX) and X-Ray diffraction (XRD) measurements (see Supplementary Fig. 1 and Supplementary Fig. 2).

Water sorption performance. Three aspects are of major importance for water harvesting materials: 1) The material has to

adsorb or absorb large amounts of water with respect to weight and/or volume, 2) the harvesting of water should be possible at low relative humidities, and 3) the water should be released at low temperatures. Different experiments were performed to investigate the absorption and desorption behavior of the composite. Additionally, the material was subjected to multiple cycles of water uptake and release to prove the stability of the composite.

Figure 3a shows water sorption isotherms of the fully dehydrated material at 28, 65, and 85 °C. The water vapor pressure was stepwise increased. The hydration to the hexahydrate is directly followed by the deliquescence (formation of a solution) of the salt above around 10 mbar water vapor pressure at 28 °C. At 65 °C and 30 mbar of water vapor pressure a water uptake of 17 wt.% was observed. No mentionable water uptake could be observed at 85 °C. Equilibration times for the data points were set to 600 min below 13 mbar and to 150 min above 13 mbar. The equilibration time for the desorption is set to 150 min throughout the whole measurement. The hysteresis between absorption and desorption is attributed to the short equilibration times that do not allow the system to reach the equilibrium state during desorption. The gain in mass at 10 mbar of 100% translates to a bulk capacity of 0.66 g cm^{-3} (grams of water per volume composite) in a packed bed at 28 °C. Even at 7.5 mbar water vapor pressure, the material is able to absorb 70% of its own weight in water. Under comparable conditions, MOF-801¹⁸ is only able to adsorb approx. 30% of its own weight in water, and zeolite 13X¹⁷ adsorbs approx. 38% of its own weight. Pristine CaCl_2 is theoretically able to absorb 97% of its own weight in water for the hydration to the hexahydrate. However, this is accompanied by agglomeration and a poor permeability of packed beds when the salt is used as bulk.

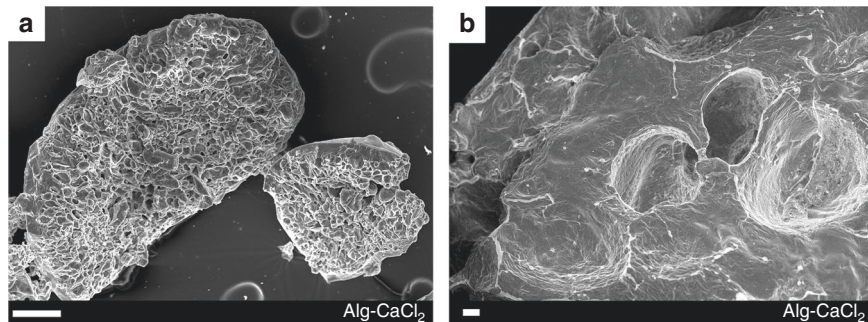


Fig. 2 Scanning electron microscopy images of the composite Alg- CaCl_2 . The composite beads were broken to smaller particles. The samples were coated with a carbon layer prior to the measurements to ensure sufficient electrical conductivity. **a** Overview of particles of the spherical beads (scale bar 100 μm). **b** Pores within the composite (scale bar 20 μm)

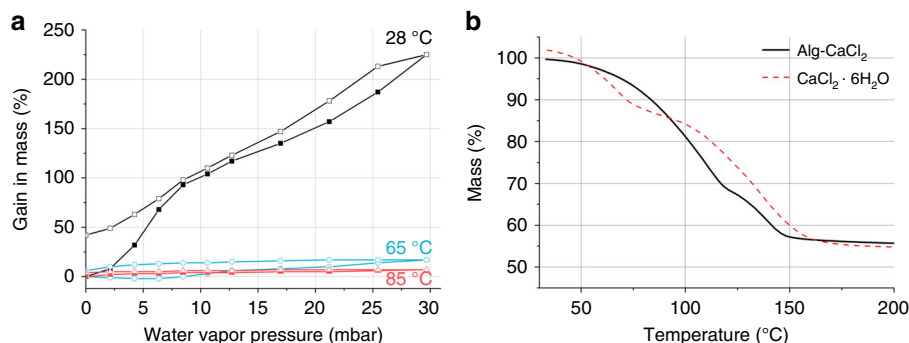


Fig. 3 Water uptake and release. **a** Water sorption isotherms of the composite Alg- CaCl_2 at 28 and 85 °C. The absorption branch is indicated by full symbol, the desorption branch is indicated by empty symbols. **b** TGA of the composite Alg- CaCl_2 and pure $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ with a heating rate of $1 \text{ }^\circ\text{C min}^{-1}$ in argon/oxygen 8/2 atmosphere

Table 1 Comparison of different materials that can be utilized for water harvesting purposes

Name	Matrix	Incorporated salt	Water uptake g/g (Temperature, r.h., water vapor pressure)	Water uptake g/cm ³	Reference
Alg-CaCl ₂	Alginate based	CaCl ₂ (78 wt.%)	1.0 (28 °C, 26%, 10.0 mbar)	0.66 (bulk)	This work
ASLI	Activated Carbon Foam	LiCl ₂ (29 wt.%)	0.5 (25 °C, 30%, 9.5 mbar)	N/A	Wang 2017 ¹⁶
SWS-1L	SiO ₂	CaCl ₂ (24 wt.%)	0.7 (15 °C, 70%, 12.0 mbar)	N/A	Gordeeva 1998 ²³ Aristov 1999 ²²
AC07	Activated Carbon /SiO ₂	CaCl ₂ (64 wt.%)	0.25 (27 °C, 30%, 10.7 mbar)	N/A	Tso 2012 ³¹
MIL-101(Cr)	MOF	CaCl ₂ (68 wt.%)	0.6 (30 °C, 30%, 12.8 mbar)	N/A	Permyakova 2017 ²⁹
Cr-soc-MOF-1	MOF	-	2.0 (25 °C, 70%, 22.2 mbar)	0.38 (crystal)	Abtab 2017 ³⁰
Co ₂ Cl ₂ (BTDD)	MOF	-	0.9 (25 °C, 30%, 9.5 mbar)	0.60 (crystal)	Rieth 2017 ²¹
MOF-801	MOF	-	0.3 (25 °C, 30%, 9.5 mbar)	N/A	Kim 2017 ¹⁸

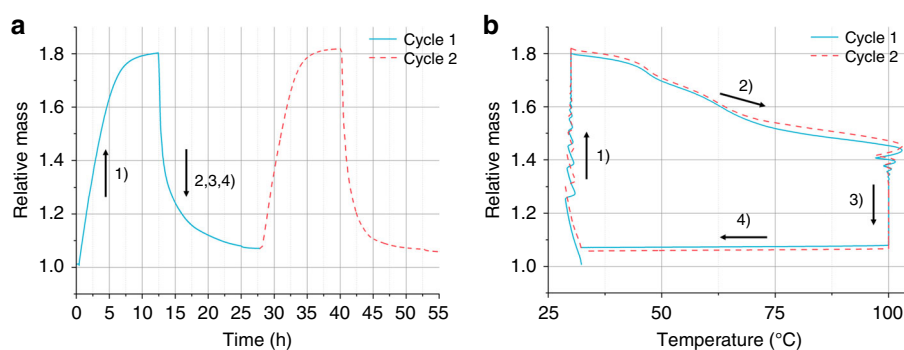


Fig. 4 Repeated hydration and dehydration of the composite Alg-CaCl₂. Hydration is performed at 30 °C and 25% relative humidity for 12 h. Dehydration is performed at 100 °C for 12 h, with an additional heating time of 35 min. After dehydration, the sample is cooled to 30 °C with a cooling time of 70 min and an equilibration time of 2 h, before the second hydration starts. **a** Time-resolved reactions. **b** Temperature-resolved reactions

There are several interesting composite materials with good gravimetric water harvesting capacity as shown in Table 1. The highest water uptake of almost 200 wt.% was achieved with the MOF Cr-soc-MOF-1, however the material only absorbs water above 50% relative humidity, making it unsuitable for water harvesting in arid climates³⁰. A very promising candidate for water harvesting has recently been published by Rieth et al. A Cobalt based MOF with a BTDD linker showed water uptake of around 80 wt.% at 25 °C and 30% relative humidity (9.5 mbar water vapor pressure)²¹. It was presented that the harvested water can be released at 45 °C making a solar driven application feasible. A volumetric capacity of 0.60 g cm⁻³ (grams of water per volume MOF) was calculated from the crystal density. This value is expected to be reduced significantly in a packed bed. In addition, the MOF incorporates a cobalt salt and a dioxin linker, which might be reason for concern regarding fresh water quality. Moreover, it has to be considered that the synthetic approach is more complicated and the price of the cobalt based MOF is significantly higher than the CaCl₂ based material presented here.

The dry composite beads of Alg-CaCl₂ exhibit a bulk density of 0.66 g cm⁻³. Figure 3a shows that 1.0 g of water are absorbed per gram of composite at 10 mbar water vapor pressure at 28 °C. It is, therefore, possible to harvest 660 kg of water per cubic meter of the composite material in a packed bed in one cycle.

Figure 3b shows the thermogravimetric analysis (TGA) of the fully hydrated material with a heating rate of 1 °C min⁻¹. The dehydration behavior of Alg-CaCl₂ is almost identical to the dehydration behavior of pure CaCl₂·6H₂O. Dehydration under the investigated conditions is complete at 150 °C and results in

the formation of anhydrous CaCl₂. The temperature that is required for the desorption of the water from the desiccant material has a major influence on the energy demand of the application. The required temperature directly influences the capital expenditure and the engineering complexity to build a solar system to provide the heat. A lower desorption temperature usually goes along with a cheaper setup.

The composite Alg-CaCl₂ was hydrated and dehydrated repeatedly to investigate cyclisation behavior (see Fig. 4). One hydration/dehydration cycle consists of 4 steps: 1) Absorption of water at 30 °C and 25% relative humidity (10.6 mbar water vapor pressure) for 12 h, 2) heating to the desorption temperature under dry nitrogen with a heating rate of 2 °C min⁻¹, 3) holding time at the desorption temperature for 12 h and 4) cooling down to 30 °C with a cooling rate of approx. 1 °C min⁻¹ and equilibration for 2 h. The dehydration of the composite after the first cycle is not complete. Approx. 6% of the adsorbed water cannot be desorbed under the investigated conditions. The second cycle proves that the absorbed amount of water is still extremely high and that cyclisation is possible with approx. 75 wt.% of water taken up by the material. The experiment has been repeated with slightly different conditions to prove the stability over 8 cycles (see Supplementary Fig. 3).

It has to be noted that the desorption process was performed in dry nitrogen, which does not fully represent application conditions, where ambient air is heated up to desorb the water. In an open system, the desorption behavior is dependent on ambient air temperature and humidity, and has to be investigated according to the respective climate of the intended location of the application.

Discussion

A composite material for water harvesting consisting of an alginate-derived matrix and large amounts of incorporated CaCl_2 was presented, which can be obtained as spherical beads through a facile synthesis route. The spherical structure promotes good gas permeability of packed beds. The presented material is able to adsorb 100% of its own weight in water from air at a water vapor pressure of 10 mbar, which makes it promising for application in arid climate zones. The high water uptake indicated that the incorporated salt is accessible for water vapor, which is attributed to the micro pores of the matrix. More than 90% of the adsorbed water can be released at 100 °C and complete desorption can be achieved at 150 °C. These temperatures can be provided by solar radiation, e.g., vacuum solar tubes. The results show that the presented material is an interesting candidate for water harvesting applications.

The macroscopic structure may allow an application in packed columns. Ambient air can be lead through the material at night to allow for water absorption with a low pressure loss and therefore little energy consumption. At daytime, the temperature of the packed bed can be increased by solar heat and the released water can be condensed at ambient temperature. Calculations from the bulk density of the material and the water uptake indicate that 660 kg of water could be harvested per cubic meter of composite material in a packed bed. We believe that this work may inspire researchers to use the presented material for prototype development to advance in the field of water harvesting from air towards large-scale application.

Methods

Instrumentation. TGA was performed on a Jupiter STA 449 F3 from Netzsch GmbH and Co KG with a STA/DTA sample holder and Al_2O_3 crucibles. Water sorption isotherms were recorded on a SPSx-1 μ Advanced from proUmid GmbH and Co. KG at 23 °C. SEM Images were taken on NTS LEO-1525 from Carl Zeiss company. The samples were coated with a carbon layer by vapor deposition. All experiments were conducted with samples that were dried at 130 °C.

Analysis of the salt content. The ground composite was dried at 130 °C, extracted with water and filtered. The filtrate was subjected to ICP-OES (inductively coupled plasma optical emission spectrometry) to investigate the salt content. The concentration of Ca^{2+} and Cl^- in the filtrate is measured and the amount of CaCl_2 in the composite calculated.

Cyclisation of the composites. The material was stored at 130 °C and loaded into a Jupiter STA 449 F3 from Netzsch GmbH and Co KG equipped with a humidity generator from proumid GmbH and Co. KG in a modified Al_2O_3 crucible (see Supplementary Fig. 4). The airflow was set to 100 ml min^{-1} of nitrogen for all steps. The material was heated to 100 °C for 5 h for full dehydration prior to the experiment. The resulting mass at 30 °C was defined as starting mass.

Analysis of the water sorption and desorption. The material was stored at 130 °C and loaded into a Jupiter STA 449 F3 from Netzsch GmbH & Co KG equipped with a humidity generator from proumid GmbH and Co. KG in a modified Al_2O_3 crucible (see Supplementary Fig. 4). The airflow was set to 100 ml min^{-1} of nitrogen for all steps. The material was heated to the respective temperature (28 or 85 °C) and the humidity was stepwise increased. Equilibration times were set to 600 min below 13 mbar water vapor pressure and to 150 min above 13 mbar water vapor pressure for the absorption. The equilibration time was set to 150 min for the desorption.

Synthesis of the composites. Alg- CaCl_2 was synthesized using a solution of sodium alginate (4.00 g in 200 mL of water) that was added to a solution of CaCl_2 (290.00 g in 400 mL water) dropwise. Droplets were produced with an Encapsulator B390 from Büchi Labortechnik GmbH using a 1000 μm nozzle. The hydrogel spheres were left in the CaCl_2 solution for 12 h, filtered, washed with water and dried at 140 °C for 20 h to obtain the final composite Alg- CaCl_2 .

Data availability. The data that support the findings of this study are available from the corresponding author on reasonable request.

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Author contributions

M.F. directed and oversaw the research project. P.A.K. carried out the experiments and wrote the manuscript. Both authors took part in the interpretation and discussion of the results.

Additional information

Supplementary information accompanies this paper at <https://doi.org/10.1038/s42004-018-0028-9>.

Competing interests: P.A.K., M.F., Michael Steiger, Felix Brieler, Konrad Posern, the University of Weimar and the University of Hamburg filed a German patent application

(Kompositmaterial zur Speicherung von Wasser und/oder Wärme, DE 10 2017 109 618.3) pertaining to the presented synthesis and materials.

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