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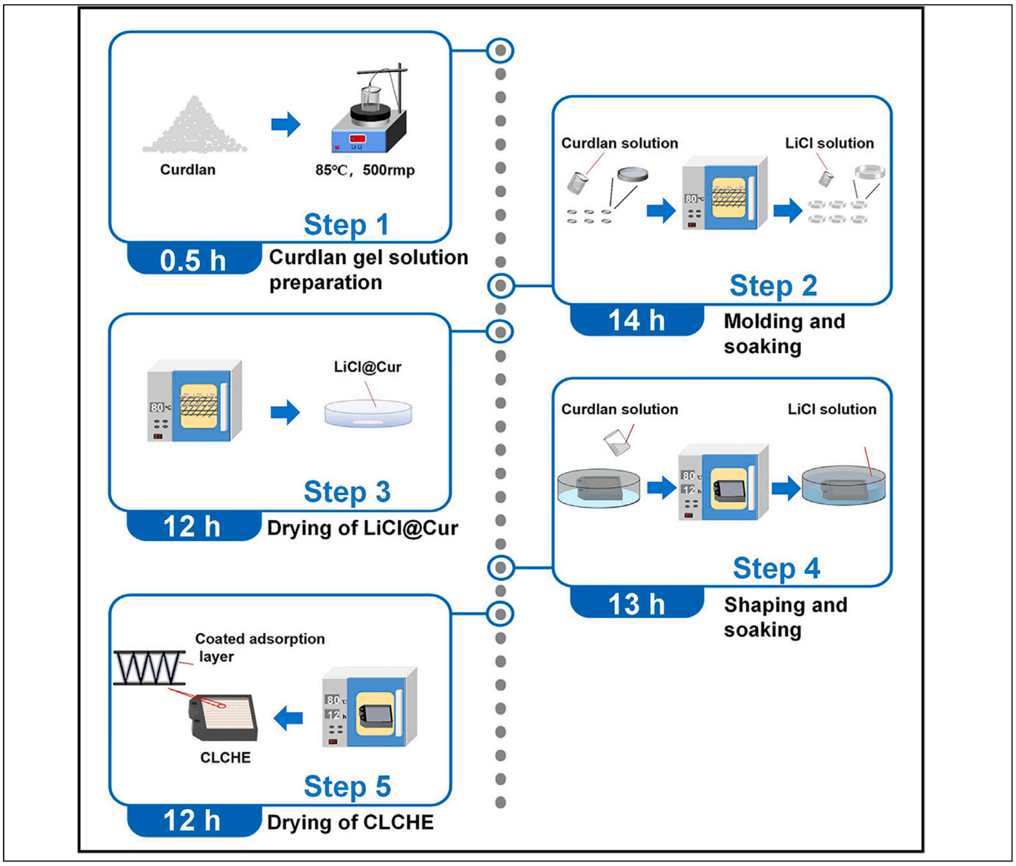

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Highlights

Protocol for preparation of the hydrogel-based composite sorbent

Detailed steps to prepare curdlan hydrogel and LiCl@Cur

An experimental approach for the fabrication of LiCl@Cur-coated heat exchanger (CLCHE)

Desiccant-coated heat exchangers provide a practical solution for the efficient removal of moisture from the air. Here, we present a protocol to synthesize an ultra-hygroscopic polymer to develop a LiCl loaded in curdlan hydrogel (LiCl@Cur)-coated heat exchanger for deep dehumidification. We describe steps for preparing the curdlan gel solution, hydrogel, LiCl solution, and LiCl@Cur. We then detail procedures for preparing curdlan-coated and LiCl@Cur-coated heat exchangers. The coated heat exchanger described in this protocol has a maximum dehumidification capacity of 12 g/kg.

Publisher's note: Undertaking any experimental protocol requires adherence to local institutional guidelines for laboratory safety and ethics.

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Protocol for preparation of LiCl-based ultra-hygroscopic curdlan heat exchanger for dehumidification

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SUMMARY

Desiccant-coated heat exchangers provide a practical solution for the efficient removal of moisture from the air. Here, we present a protocol to synthesize an ultra-hygroscopic polymer to develop a LiCl loaded in curdlan hydrogel (LiCl@Cur)-coated heat exchanger for deep dehumidification. We describe steps for preparing the curdlan gel solution, hydrogel, LiCl solution, and LiCl@Cur. We then detail procedures for preparing curdlan-coated and LiCl@Cur-coated heat exchangers. The coated heat exchanger described in this protocol has a maximum dehumidification capacity of 12 g/kg.

For complete details on the use and execution of this protocol, please refer to Pan et al. (2023).[1]

BEFORE YOU BEGIN

The following protocol describes the specific steps and typical characteristics of the hydrogel-based composite sorbent LiCl@Cur, which has a remarkable sorption ability. The developed adsorbent LiCl@Cur achieved a significant adsorption capacity of 2.65 g/g and a fast kinetic rate of 1.84×10^{-4} s^{-1} at 30°C/30% RH. In addition, this protocol describes a dehumidifier heat exchanger based on an ultra-hygroscopic LiCl@Cur composite polymer, which possesses a maximum dehumidification capacity of 12 *g*/kg and demonstrates the portability and efficiency of dehumidification.

Many experimental studies and explorations on DCHEs have appeared in recent years; researchers coated aluminum sheets with a silica gel-LiCl composite desiccant and found that these coated sheets improved the dehumidification efficiency of a DCHE system.[2] Coating a heat exchanger with aluminum-based metal-organic frameworks (MIL-96 and MIL-100) to obtain a microchannel heat exchanger (MCHE), which had a dehumidification capacity that was more than twice that of a traditional silica-coated heat exchanger.[3] A polyvinyl alcohol-LiCl (50 wt %) composite polymeric desiccant coated on a heat exchanger was found that the DCHE exhibited a 20%–60% higher dehumidification capacity than a DCHE not containing this desiccant.[4] These protocols provide experimental references for designing and developing CLCHE.

Preparation of microchannel heat exchanger

Timing: 13 h

When preparing the CLCHE, selecting a suitable heat exchanger is necessary. Compared with other heat exchangers, microchannel heat exchangers have advantages of high thermal conductivity, energy saving, and large heat/mass transfer capacity. Thus, the following steps describe the pretreatment of a purchased microchannel heat exchanger.

1. Prepare 1 wt % NaOH solution.
   a. Weigh 1 *g* of anhydrous NaOH by using a precision balance and dissolve in 99 mL of deionized water at 25°C.

   Note: Stir the solution while adding the NaOH.

   Note: Wear rubber gloves when weighing and transferring NaOH to avoid injury to your hands.

2. Clean the microchannel heat exchanger with NaOH solution.
   a. Flush the NaOH solution evenly over the fins of the microchannel heat exchanger.

   Note: Flush the solution through each fin channel from top to bottom.

   Note: Wear rubber gloves while transferring and flushing the heat exchanger to avoid injury to your hands.

3. Clean the microchannel heat exchanger with DI water.
   a. Flush the DI water evenly over the fins of the microchannel heat exchanger.

   Note: Flush DI water through each fin channel from top to bottom.

4. Transfer the microchannel heat exchanger to the air-drying oven for 12 h.

   Note: Set the air-drying oven temperature 80°C.

   Pause point: Store the dried microchannel heat exchanger in a dry environment.

KEY RESOURCES TABLE

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Chemicals, peptides, and recombinant proteins		
Curdlan	Shanghai Xintai Food Ingredients Mall	N/A
Sodium hydroxide	Shanghai Aladdin Biochemical Technology Co., Ltd.	CAS: 1310-73-2
Lithium chloride	Adama Sterling Reagent Co., Ltd.	CAS: 7447-41-8
Deionized water	N/A	18 mΩ
Other		
Microchannel heat exchanger	Yancheng City Civilian Electronic Technology Co., Ltd.	PT1120B
Air-drying oven	Shanghai Zuoke Instrument Equipment Co., Ltd.	DHG-9240A
Magnetic stirrer	Shanghai Meiyingpu Instrument Manufacturing Co., Ltd.	524G
Precision electronic balance	Sartorius, Germany, Ltd.	SQP-PRACTUM224-1CN

MATERIALS AND EQUIPMENT

The materials and equipment required for the protocol are shown in Figure 1. This work takes the PT1120B microchannel heat exchanger as the research object, and its picture is shown in Figure 2.

Figure 1. Physical photographs of materials and equipment used in the experiment

(A) The raw material of curdlan, Anhydrous sodium hydroxide, lithium chloride anhydrous, deionized water, and microchannel heat exchanger.

(B) Pipette, insulated gloves, magnetic stirrer, analytical balance and air-drying oven.

The microchannel heat exchanger consists of copper fins, an iron frame, and a flat brass tube. The detailed parameters are listed in the following Table 1.

STEP-BY-STEP METHOD DETAILS

Preparation of curdlan gel solution

Timing: 0.5 h

The following steps describe the detailed process of preparing a gel solution by the thermal induction method (Figure 3A).

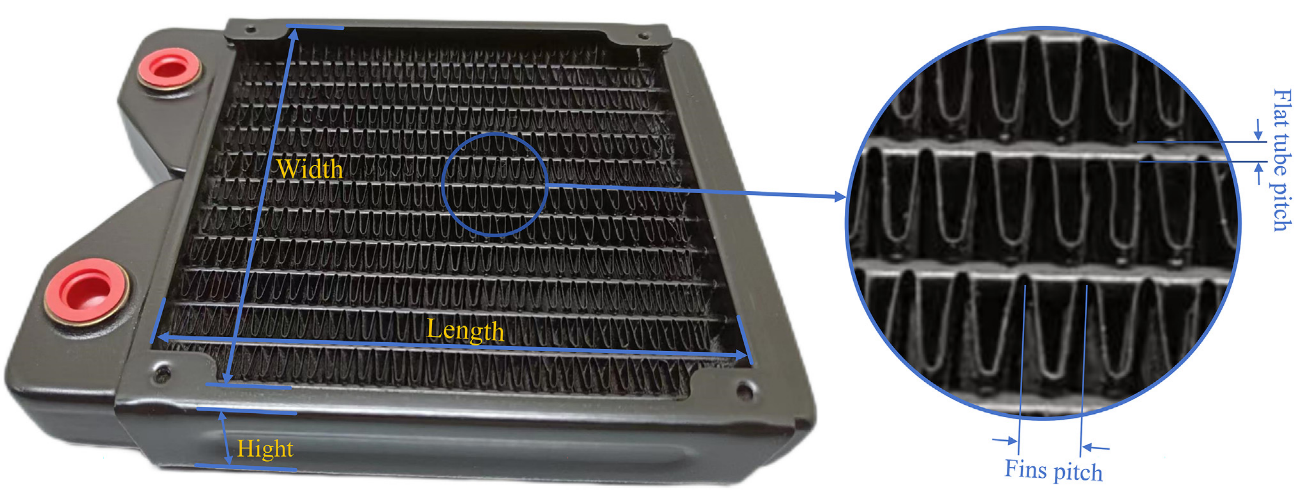

Figure 2. Size and structure of the microchannel heat exchanger

Table 1. DCHE size chart

DCHE parameters	Value
Heat Exchanger Length, L/mm	154
Heat Exchanger Height, H/mm	123
Heat Exchanger Width, W/mm	30
Fin thickness, d/mm	0.1
Fin spacing, ds/mm	2.2
Number of flat tubes, n	12
Inner diameter of copper tube, di /mm	1
Outer diameter of copper tube, do/mm	2

1. Weigh 5 *g* of curdlan and disperse in 95 mL of deionized water at 25°C.
   a. Select a suitable beaker and a magnetic stirring rotor and set the speed to 500 rpm.
   b. Use a spatula and add 5 *g* of curdlan to 95 mL of deionized water slowly.

   Note: Stir the solution while adding the curdlan.

2. Insert a thermocouple pt100 temperature sensor in the beaker and set the final temperature to 85°C and wait for the temperature to rise.

   Note: Stir the solution while increasing the temperature of the solution.

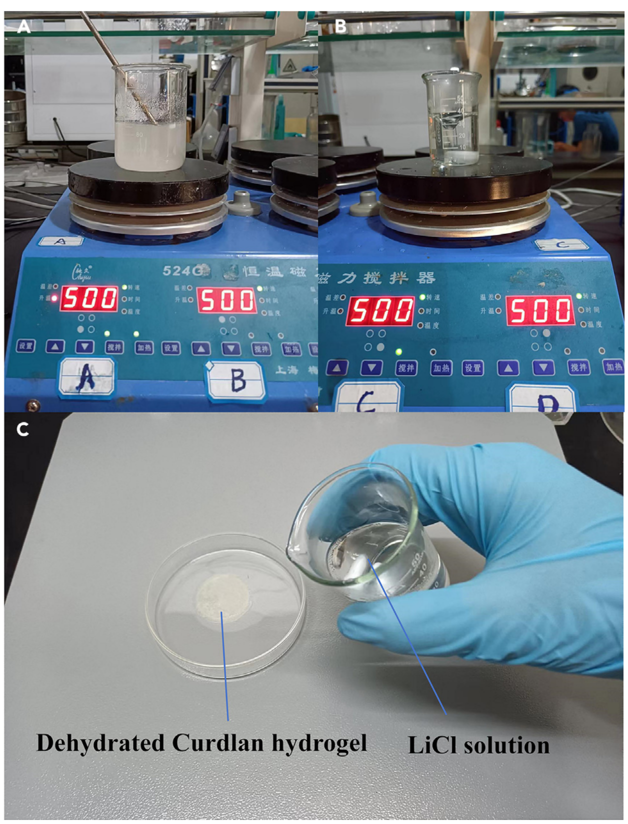

Figure 3. Preparation of LiCl@Cur
(A) Preparation of curdlan gel solution.
(B) Preparation of LiCl solution.
(C) The dehydrated curdlan hydrogels were impregnated with LiCl solution.

3. Complete the preparation of the curdlan gel solution when the temperature is maintained for 5 min at 85°C.

   Note: The curdlan gel solution has a translucent color.

   ⚠ CRITICAL: Temperature changes should always be observed to avoid excessive temperatures that could damage materials and devices.

   Pause point: The prepared curdlan gel solution can be stored at 85°C for 1 h, so it is best to use them as soon as they are prepared.

Preparation of curdlan hydrogel

Timing: 12 h

The following steps describe a method for preparing curdlan hydrogel using a small Petri dish as a mold. The mold shown here is an example. You can set up your own hydrogel mold to suit your research design.

4. Transfer and pour the gel solution prepared in step 3 into the molds (Methods video S1).

   Note: The molds are small petri dishes with a diameter of 36 mm, six in total (or more), each holding 4 mL of solution.

   Optional: Any shape and size can be selected upon requirement.

   ⚠ CRITICAL: Due to the solution solidifying as a gel in a very short time, the solution must be transferred and poured quickly and carefully.

   ⚠ CRITICAL: Insulated gloves and lab coats must be worn while transferring beakers and pouring solutions to avoid burns caused by hot solutions.

5. Allow the solution to cool naturally in the molds for 1 min and the solution solidifies into hydrogels.

   Note: Hydrogels exhibit a translucent appearance and are flexible.

6. Transfer the curdlan hydrogels from the molds to the large petri dishes (Methods video S2).
   a. Use a thin slice (e.g., spatula) to carefully remove the hydrogel from the edge of small petri dishes and transfer them to large petri dishes.

   ⚠ CRITICAL: The slices should not be sharp, or they can easily damage the hydrogels.

7. Transfer the large petri dishes containing the hydrogels to an air-drying oven for 12 h to obtain dehydrated curdlan hydrogels.

   Note: Set the air-drying oven temperature to 80°C.

   ⚠ CRITICAL: Flip the hydrogels every 2 h during the drying process so that the hydrogels do not stick to the wall of the petri dishes and become difficult to remove.

   Pause point: The prepared dehydrated hydrogels can be stored in a dry environment at 25°C for up to one week. The recommended optimal usage is within 24 h.

Preparation of LiCl solution

⏲ Timing: 0.25 h

The preparation of sorbents and coated heat exchangers requires a certain amount of LiCl solution, and the total amount varies according to the experimental needs. The following steps describe a detailed process for preparing a lithium chloride (LiCl) solution.

8. Prepare 15 wt % LiCl solution (Figure 3B).
   a. Weigh 15 *g* of anhydrous LiCl by using a precision balance and dissolve it in 85 mL of deionized water at 25°C.

   Note: Stir the solution while adding the LiCl.

   Note: Wear rubber gloves when weighing and transferring LiCl to avoid injury to your hands.

   ⚠ CRITICAL: The weighing and transfer process must be as quick as possible to minimize water vapor sorption by anhydrous LiCl.

9. Obtain the LiCl solution after stirring for 10 min.

   Note: Observe whether the LiCl is completely dissolved, and the solution is clear and transparent.

   Pause point: The prepared LiCl solution can be stored in a dry and confined environment at 25°C for 48 h. The recommended optimal usage is within 24 h.

Preparation of LiCl@Cur

⏲ Timing: 14 h

The following steps describe a detailed process for preparing LiCl@Cur.

10. Impregnate the dehydrated curdlan hydrogels in LiCl solution for 2 h (Figure 3C).

    Note: The curdlan hydrogels should be completely submerged in the LiCl solution.

11. Transfer the curdlan hydrogels from the step 10 to the air-drying oven for 12 h to obtain the dehydrated LiCl@Cur.

    Note: Set the air-drying oven temperature to 80°C.

    ⚠ CRITICAL: Flip the LiCl@Cur every 2 h during the drying process so that the LiCl@Cur does not stick to the wall of the petri dishes and becomes difficult to remove.

    Pause point: The prepared dehydrated LiCl@Cur can be stored in a dry and confined environment at 25°C for up to one week. The recommended optimal usage is within 24 h.

Preparation of curdlan-coated heat exchanger

⏲ Timing: 13 h

The following steps describe a detailed process for preparing a curdlan-coated heat exchanger.

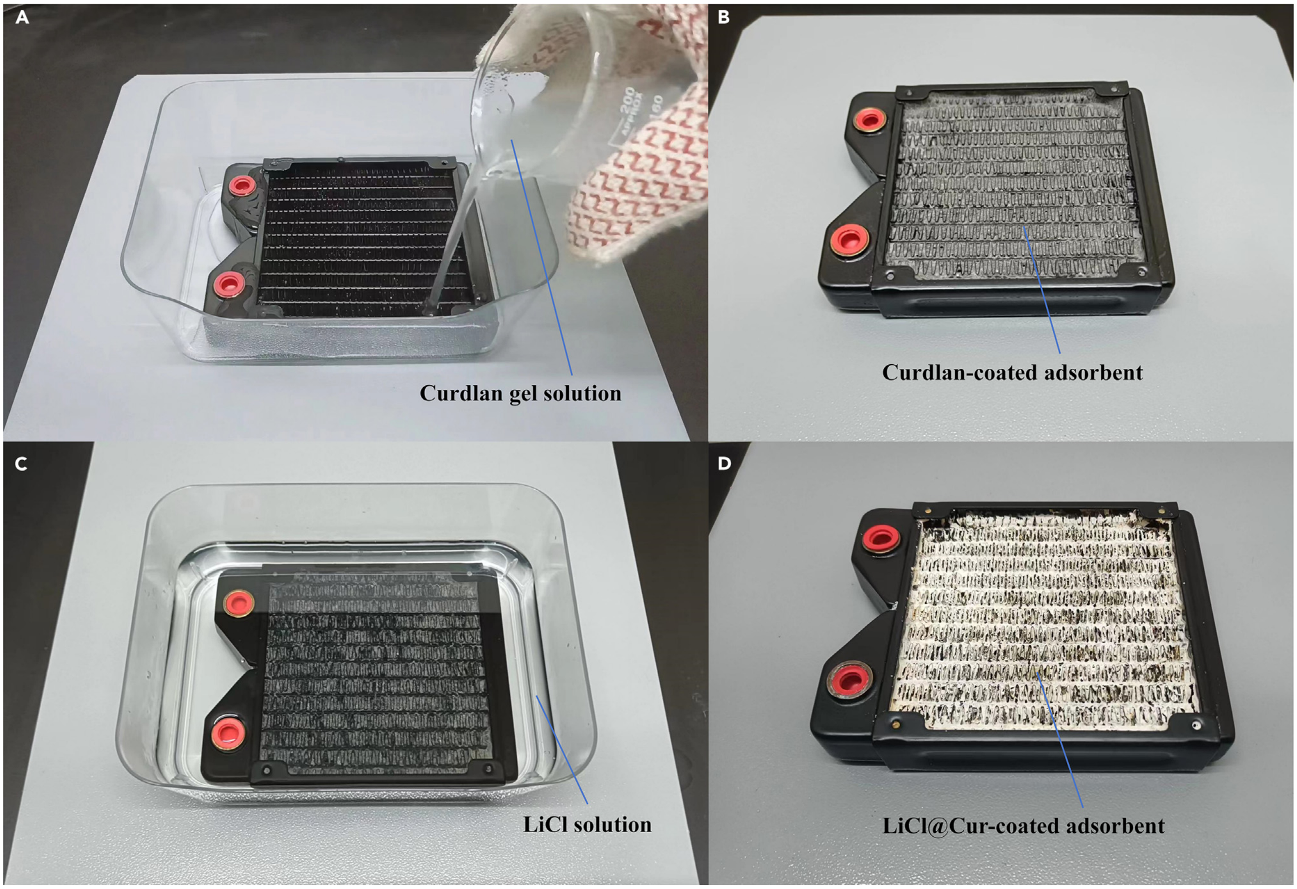

Figure 4. Preparation of CLCHE
(A) Pouring the curdlan gel solution uniformly onto the fins and tube surfaces of the microchannel heat exchanger.
(B) Photograph of the curdlan-coated heat exchanger.
(C) The dried curdlan-coated heat exchanger is impregnated with LiCl solution.
(D) Photograph of the CLCHE.

12. Repeat the preparation of 200 mL, 5 wt % curdlan gel solution according to steps 1–3.

Note: Select one or more suitable beakers and magnetic stirring rotors.

Note: The total amount varies depending on the size volume of the prepared heat exchanger.

⚠ CRITICAL: It is best to prepare enough curdlan gel solution at once and then use it immediately.

13. Pour the prepared curdlan gel solution (85°C, 5 wt %) uniformly onto the fins and tube surfaces of the microchannel heat exchanger (Figure 4A).

Note: Place the heat exchanger in a suitable container to prevent contamination of the lab bench.

Note: Close the inlet and outlet of the heat exchanger when dumping the solution.

⚠ CRITICAL: Due to the solution solidifying as a gel in a very short time, the solution must be transferred and poured quickly and carefully.

⚠ CRITICAL: Insulated gloves and lab coats must be worn while transferring beakers and pouring solutions to avoid burns caused by hot solutions.

14. Allow the solution to cool naturally for 5 min and the solution solidifies on the heat exchanger.
15. Use a thin slice (e.g., spatula) to carefully remove excess hydrogel from the edges and interior of the heat exchanger.
16. Transfer the heat exchanger coated the curdlan hydrogel to an air-drying oven for 12 h to obtain the curdlan-coated heat exchanger (Figure 4B).

Note: Set the air-drying oven temperature to 80°C.

Pause point: The prepared curdlan-coated heat exchanger can be stored in a dry and confined environment at 25°C for up to one week. The recommended optimal usage is within 24 h.

Preparation of CLCHE

Timing: 13 h

The following steps describe a detailed process for preparing a LiCl@Cur-coated heat exchanger.

17. Repeat the preparation of 600 mL, 15 wt % LiCl solution according to steps 8–9.

Note: The total amount varies depending on the size volume of the heat exchanger and the vessel.

18. Transfer the prepared LiCl solution to a suitable vessel (e.g., plastic basin) that can accommodate the heat exchanger.

Note: The temperature of the LiCl solution is maintained at 25°C.

19. Impregnate the prepared dried curdlan-coated heat exchanger in LiCl solution for 1 h (Figure 4C).

Note: The curdlan-coated heat exchanger should be completely submerged in the LiCl solution.

Note: Keep the inlet and outlet of the heat exchanger in a closed state when soaking the heat exchanger.

⚠ CRITICAL: Longer soaking time will corrode the heat exchanger.

20. Wipe the LiCl solution except for the heat exchanger fins and tube surfaces.
21. Transfer the heat exchanger coated the LiCl@Cur to an air-drying oven for 12 h to obtain CLCHE (Figure 4D).

Note: Set the air-drying oven temperature to 80°C.

Pause point: The prepared CLCHE can be stored in a dry and confined environment at 25°C for up to one week. The recommended optimal usage is within 24 h.

EXPECTED OUTCOMES

This protocol allows for the synthesis of LiCl@Cur composite desiccant and the preparation of LiCl@Cur-coated heat exchanger (CLCHE). The expected results are consistent with our previous paper on Cell Reports Physical Science,[1] and some criteria listed below.

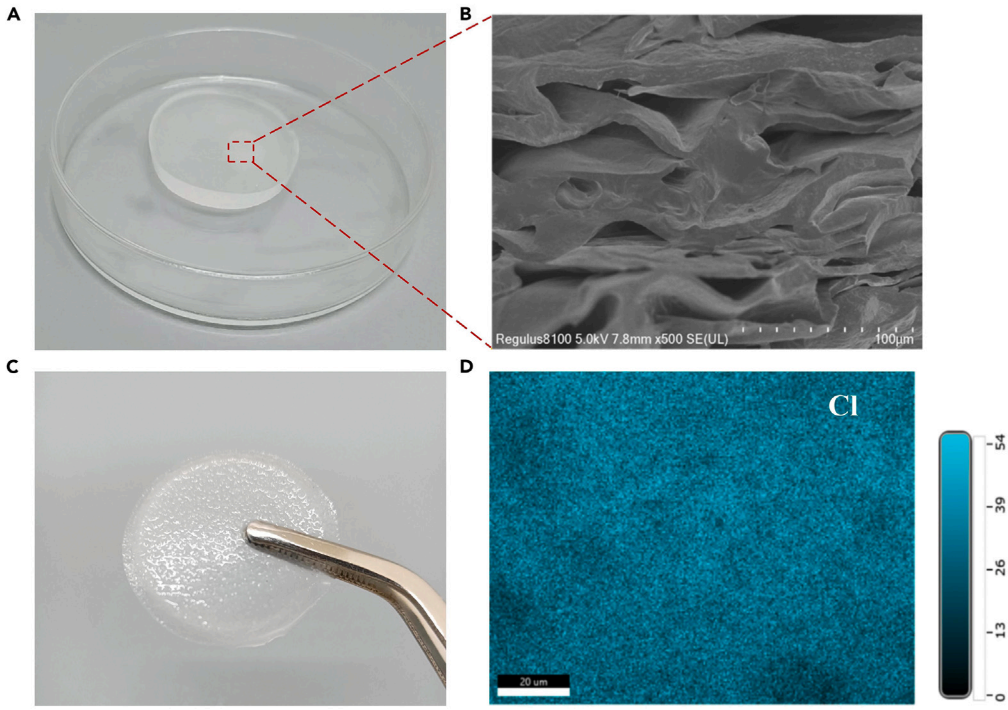

Figure 5. Expected physical and SEM images of curdlan and EDS of LiCl@Cur

(A) Photograph of curdlan.

(B) Scanning electron microscopy images of curdlan.

(C) Photograph of LiCl@Cur.

(D) Elemental mapping of LiCl@Cur (chlorine).

Expected visual outcomes

The curdlan in this work is a thermally irreversible gel with a strong and elastic internal structure, good thermal stability, and strong water storage capacity. Additionally, it can also be machined to the target geometry by changing the shape of the mold. The visual appearance of the curdlan is shown in Figure 5A. Scanning electron microscopy (SEM) shows an ultra-porous hierarchical microstructure of LiCl@Cur surface (Figure 5B); these microstructures facilitate the attachment of hygroscopic salts and the transport of water molecules. By detecting the elements on the LiCl@Cur sample through energy dispersive X-ray spectroscopy (EDS), we can find that numerous chlorine atoms are uniformly attached to LiCl@Cur, which can significantly improve the water uptake capacity (Figures 5C and 5D).

Expected vapor isothermal sorption isotherms of LiCl@Cur

The sorption kinetics of LiCl@Cur during sorption process was tested by STA 449 Netzsch, and the experimental results are shown as red dots in Figure 6. At the low humidity condition of 30% RH and 30°C, LiCl@Cur reveals an excellent water adsorption capacity of 2.65 g/g. In addition, the fitting results obtained by fitting equations are shown as the black line in Figure 6, and LiCl@Cur demonstrates rapid water adsorption kinetics with an adsorption rate coefficient k of $1.84 \times 10^{-4}\ s^{-1}$, which is higher than most adsorbents under the same conditions.

Expected dehumidification results

To demonstrate the dehumidification-regeneration capability and cycle stability of the heat exchanger, three times dehumidification-regeneration cycling tests of CLCHE were repeated at 25°C/80% RH for dehumidification and 80°C for regeneration. The test results showed the maximum instantaneous dehumidification of 12 *g*/kg and the maximum instantaneous regeneration of 52 *g*/kg.

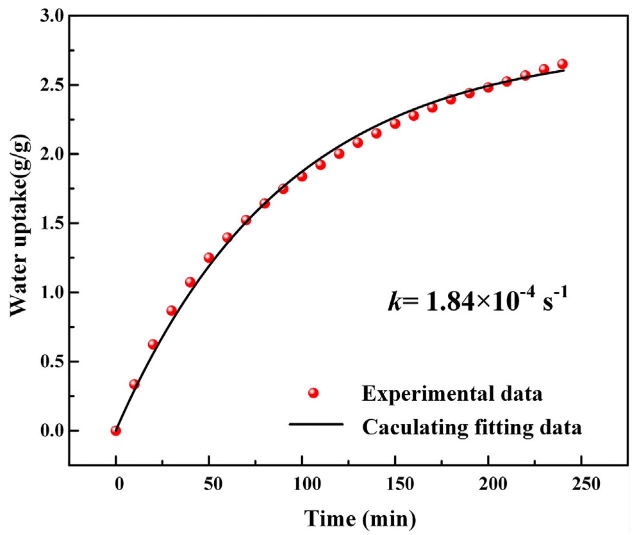

Figure 6. Isothermal sorption kinetics of sorbents at 30°C/30% RH

Moreover, the performance of the heat exchanger did not deteriorate significantly after several tests (Figure 7).

LIMITATIONS

Regeneration temperature is an important factor in determining whether the dehumidification process can be driven by low-grade energy. We tested the desorption performance of LiCl@Cur at different temperatures, as shown in Figure 8. In order to ensure sufficient desorption, the desorption temperature of LiCl@Cur should be above 60°C.

In addition, we performed an experimental analysis of the regeneration temperature of CLCHE (Figure 9). Higher the regeneration temperature, larger moisture content at the outlet of the CLCHE, and shorter regeneration time required. Therefore, we chose 80°C as the regeneration temperature.

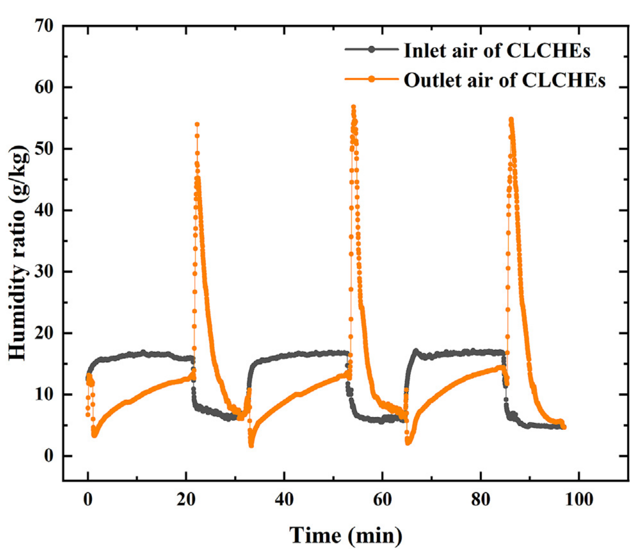

Figure 7. Dehumidification performance test

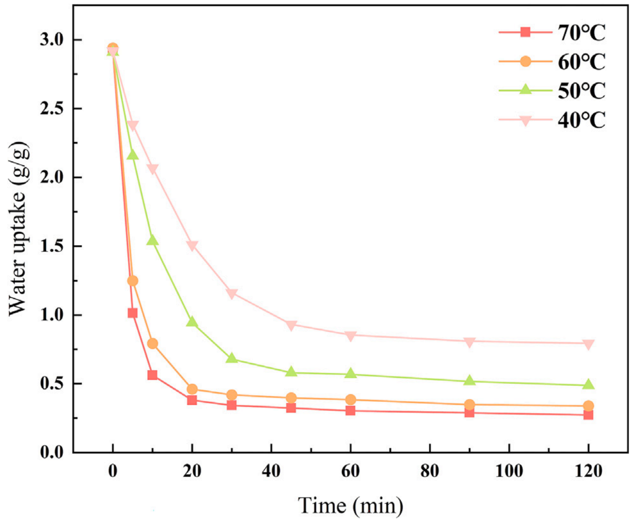

Figure 8. Desorption of LiCl@Cur at different regeneration temperatures

TROUBLESHOOTING

Problem 1

No hydrogel or uneven hydrogel is formed from the curdlan solution in the molds.

Potential solution

Several steps will result in solution polymerization failure. Check the following three points.

- Check whether the raw material of curdlan is expired or not and choose the material within the shelf life.
- Too much or too little of the mass ratio of the curdlan may fail to form hydrogels, so accurately prepare the solution of curdlan (step 1 in curdlan gel solution preparation).
- When preparing a solution of curdlan, the temperature of the solution must reach 85°C, and it can be stirred continuously for another 5–10 min, and the curdlan shows a completely dissolved state (steps 2 and 3 in curdlan gel solution preparation).

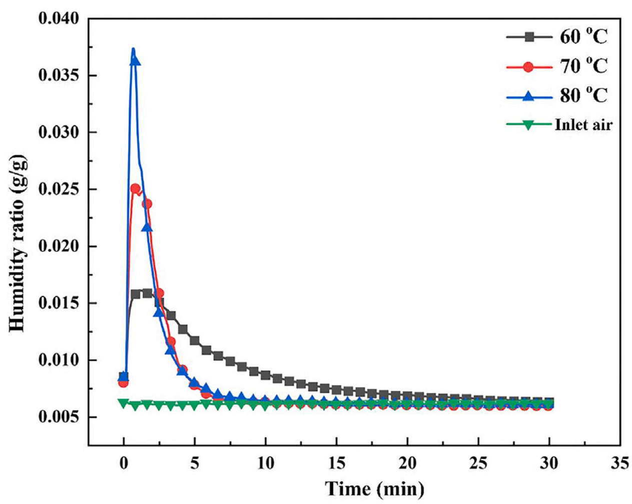

Figure 9. Desorption of CLCHE at different regeneration temperatures

Problem 2

The curdlan hydrogels removed from the molds crumble.

Potential solution

- Use a pick-up device that is not too sharp and carefully remove the hydrogel from molds (step 6 in curdlan hydrogel preparation).
- Prepare multiple samples to increase the success rate (step 4 in curdlan hydrogel preparation).

Problem 3

The water uptake of the sorbent is more than 10% worse than that reported above (Figure 6).

Potential solution

The ideal salt content of LiCl@Cur sorbents is 55 $\pm$ 2 wt %, and the salt content much affects the final sorption performance, so it is important to weigh the difference before and after impregnation and evaluate whether the impregnation process is successful. If not, please check the following two points.

- Carefully check the concentration of LiCl solution when impregnating salt solution. If anhydrous LiCl is exposed in the air for too long, this hygroscopic salt can absorb a large amount of water vapor and form LiCl hydrate. This can lead to weight deviation, reducing the actual content of LiCl (step 8 in LiCl solution preparation).
- Check the impregnation process carefully. Insufficient solution height (less than 2 cm) leads to uneven distribution of LiCl, which affects hygroscopic properties (step 10 in LiCl@Cur preparation).

Problem 4

Hydrogel is coated unevenly, and salt is distributed unevenly on the microchannel heat exchanger.

Potential solution

- Prepare enough gel solution to coat the heat exchanger fins and tubes several times (steps 12 and 13 in curdlan-coated heat exchanger preparation).
- Check the impregnation process carefully. Insufficient solution height (less than 2 cm) leads to uneven distribution of LiCl, which affects hygroscopic properties (steps 18 and 19 in CLCHE preparation).

Problem 5

Excessive deviation between theoretical and experimental values of CLCHE's performance test results.

Potential solution

Due to errors in the measuring instruments (e.g., temperature and humidity sensors), it is necessary to analyze the experimental results to exclude errors.

RESOURCE AVAILABILITY

Lead contact

Further information and requests for resources and reagents should be directed to and will be fulfilled by the lead contact, J.Y. Wang (jywang@usst.edu.cn).

Materials availability

LiCl@Cur and CLCHE generated in this study will be made available on request, but we may require a payment or a completed materials transfer agreement if there is potential for commercial application.

Data and code availability

Any additional information required to reanalyze the data reported in this article is available from the lead contact upon request.

SUPPLEMENTAL INFORMATION

Supplemental information can be found online at https://doi.org/10.1016/j.xpro.2023.102763.

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AUTHOR CONTRIBUTIONS

Z.L., Y.P., and J.W. conceived the experiments. Z.L. and Y.P. designed and optimized the protocol. J.W. and R.W. revised the manuscript and guided the overall project. J.W. and H.Z. funded the project. All the authors discussed and reviewed the manuscript.

DECLARATION OF INTERESTS

The authors declare no competing interests.

REFERENCES

1. Pan, Y., Wang, J., Zhong, G., Gao, P., Zhang, H., and Wang, R. (2023). An ultra-hygroscopic polymer for high-efficiency dehumidification. Cell Reports Physical Science *4*, 101554.

2. Zheng, X., Ge, T.S., Jiang, Y., and Wang, R.Z. (2015). Experimental study on silica gel-LiCl composite desiccants for desiccant coated heat exchanger. Int. J. Refrig. *51*, 24–32.

3. Ge, L., Ge, T., and Wang, R. (2022). Facile synthesis of Al-based MOF and its applications in desiccant coated heat exchangers. Renew. Sustain. Energy Rev. *157*, 112015.

4. Vivekh, P., Bui, D.T., Wong, Y., Kumja, M., and Chua, K.J. (2019). Performance evaluation of PVA-LiCl coated heat exchangers for next-generation of energy-efficient dehumidification. Appl. Energy *237*, 733–750.