Electronic Supplementary Material (ESI) for RSC Sustainability.

Supporting information

Fast and simple preparation of micro particles of $KHCO_3$ by freeze - dissolving method with single solvent or additional antisolvent

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1. Selection of dispersion time of ultrasonic disperser

The Master Sizer 2000 was employed for the measurement of stable samples in a liquid suspension. The primary influence factors on the accuracy of these measurements are inadequate sample preparation, including issues such as caking, dissolution, and reunion of samples. The ultrasonic disperser was used for durations of 5 and 15 min to test the influence on the measurement results. The results presented in Table S1 show the consistence in repeated measurements, with 5 or 15 min ultrasonic treatments.

Table S1 Particle size analysis results of micro particles obtained by different methods with c=0.3 g $KHCO_3$/ g water

Samples	Time(min)	D10(μm)	D50(μm)	D90(μm)
FDss	5	20.2	45.7	88.5
	15	21.9	45.6	87.9
FDas	5	10.2	26.3	63.3
	15	11.2	24.6	60.6
FDry	5	13.2	28.5	74.5
	15	12.5	26.8	77.8

2. Estimation of the energy consumption

The energy consumption of the three methods were roughly calculated based on the operations in the lab (Table S2). The main energy consumption for the freeze-drying is the vacuum pump and compressor. Due to long time operation in FDry, the total energy consumption was about 40000 kJ. The FDas and FDss was fast completed with energy mainly consumed in water bath, with only about 200-300 kJ. The operation temperature of the FDas was below ice point, whereas FDss could be performed at the room temperature. Hence, the energy consumption of FDss was slightly less than that of FDas, which were both two order lower than the energy consumption of FDry method.

Table S2 Energy consumption for FDry/FDas/FDss with products in order of g scale

	FDry [a]		FDas [b]		FDss [c]	
Operation	Power(w)	Time(min)	Power(w)	Time(min)	Power(w)	Time(min)
Vacuum pump	400	1440				
Compressor	100	1440				
Water bath compressor			200	5		
Pump for filtration			200	5	200	5
Drying oven			500	5	500	5
Production, transfer and storage of liquid nitrogen(kJ)	30.8		30.8		30.8	
Total energy consumption(kJ)	43230.8		300.8		240.8	

[a] FDry: freeze-drying. [b] FDas: freeze-dissolving in antisolvent. [c] FDss: freeze-dissolving in saturated solution.

An air separation plant uses 1.976 kJ of electricity to produce 1 g of liquid nitrogen (based on the report from Maziak UK company [1]). In this work, the first step of the FDss, FDas and FDry methods required same process, which is about 13 g liquid nitrogen for producing 1 g micro particles, requiring 25.7 kJ energy to produce the liquid nitrogen. If the energy consumption of liquid nitrogen transportation and storage is estimated to be below 20 % of the energy for producing liquid nitrogen, the production of 1 g micro particles required about 30.8 kJ in total for producing, and transferring and storage. The energy

was one order lower and several orders lower than the operation energy consumption for Freeze-dissolving and Freeze-drying method shown in Table S2, respectively. Therefore, the conclusion will remain the same. The energy consumption of FDss and FDas method only about 1% of energy consumption of FDry method, with or without considering the first step of the formation of frozen ice particles.

Notes and references

1. How on-site nitrogen generation reduces your operating costs. Accessed September 4, 2023. Available at: https://maziak.co.uk/news-and-media/how-on-site-nitrogen-generation-reduces-your-operating-costs?__cf_chl_rt_tk=7KYLpfjYpi7oZPW8mxGYV201G.TEl8xf9Bm4otgMYWk-1693807407-0-gaNycGzNDWU