Preparation of Lithium Carbonate by Pyrolysis in a Rotating Packed Bed Reactor

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Highlights

- Rotating packed bed (RPB) reactor was used to prepare well-dispersant fine Li_2CO_3 powders by $LiHCO_3$ pyrolysis.
- Ethanol is the best dispersant to prepare Li_2CO_3 powders with small particle size distribution in the experiment.
- RPB reactor shows a great potential in the preparation of well-dispersed Li_2CO_3 particles.

1. Introduction

Lithium carbonate (Li_2CO_3) powders can be used as long-term lithium therapy for acute mania, raw materials for lithium conductor synthesis, and additives for low melt-point ceramics or glass manufacture. Several technologies have been used to prepare Li_2CO_3, such as recrystallization, precipitation, electrolysis, bicarbonation-decomposition method. Among them, bicarbonation-decomposition method presents a promising future owing to its simple operation, low cost, high efficiency, and low pollution. However, Li_2CO_3 crystals have a serious tendency to form aggregates in the suspension, and the phenomenon is more obvious on heat transfer interface in the preparation process. To obtain well-dispersed fine particles, three requirements should be considered in the preparation process: (a) a high degree of supersaturation, (b) uniform spatial concentration distributions, and (c) the same growth time for all crystals.

Rotating packed bed (RPB) is a novel reactor that takes advantage of the centrifugal force to intensify the processes limited by mass transfer and mixing rate. Direct contacting of heat exchange in RPB reactor can also intensify heat transfer due to violent renewed interface between gas and liquid phase. The liquid can be split into tiny droplets, ligaments, and thin films in the RPB reactor. This will be very helpful to provide a homogeneous supersaturation environment for the nucleation and growth of Li_2CO_3 particles in the gas-liquid reaction and precipitation process. Besides, the same and short residence time of reactants in RPB reactor ensures same growth time and rapid removal of Li_2CO_3 particles to obtain well-dispersed fine particles. What's more, the heating steam directly contacting with $LiHCO_3$ solution in RPB reactor offers an attractive approach for energy efficient utilization compared to conventional heat exchangers.

In this work, Li_2CO_3 was prepared in an RPB reactor by the method of $LiHCO_3$ pyrolysis. Steam was used for the heat medium and directly contacted with $LiHCO_3$ solution. The objective of this study is to select an optimal dispersant and investigate the effects of different operating conditions on the particle size, which is significant for the further optimization and industrial scale up.

2. Methods

The reaction for Li_2CO_3 preparation by $LiHCO_3$ pyrolysis is described as follows:

$$2LiHCO_3(\text{aq}) = Li_2CO_3(\text{s})\downarrow + CO_2(\text{g})\uparrow + H_2O(\text{l})$$

Ethanol, polyethylene glycol (PEG), and polyvinyl alcohol (PVA) were selected as dispersants.

3. Results and discussion

Figure 1 shows X-ray diffraction (XRD) patterns of the product from the different dispersants. No impurity peaks were observed, indicating that $LiHCO_3$ pyrolysis method can obtain pure Li_2CO_3 crystal. The

particle size distribution (PSD) curves is shown in Figure 2. The median particle size of Li_2CO_3 produced by the dispersant of ethanol and PEG is both about 2-5 μm which is smaller than that produced by PVA and none dispersant. The size of particles produced by ethanol and PEG are much more homogeneous compared with the latter. Figure 3 illustrates the typical SEM images of the products obtained by using different dispersants. It is obvious that all the products are rod-like crystal particles, and the size of the particles produced by ethanol and PEG is much smaller than that produced by PVA and without dispersant. Figure 4 shows the effect of the rotational speed on the median particle size of Li_2CO_3 particles.

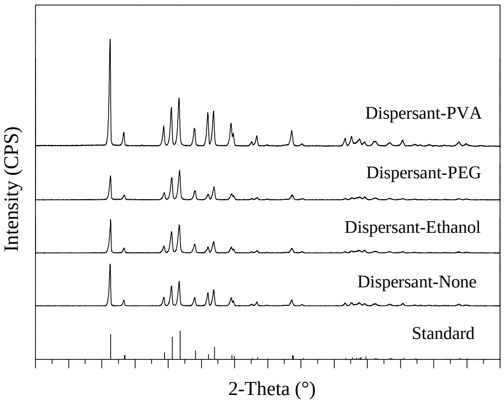

Figure 1. XRD patterns of the product

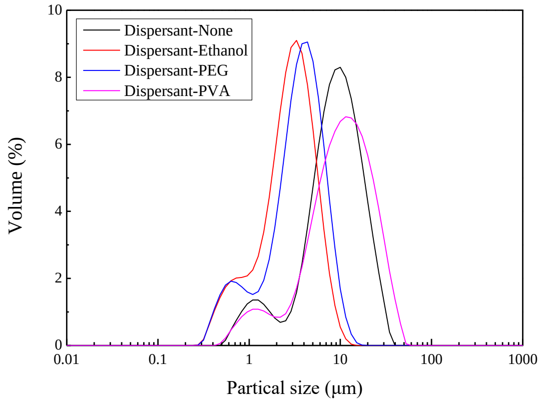

Figure 2. PSD curves of the product

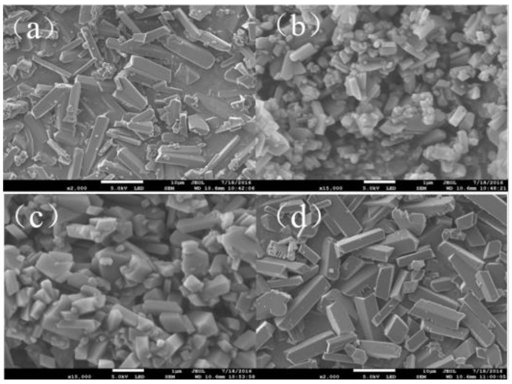

Figure 3. Morphologies of product
(a) none, (b) ethanol, (c) PEG, (d) PVA dispersants

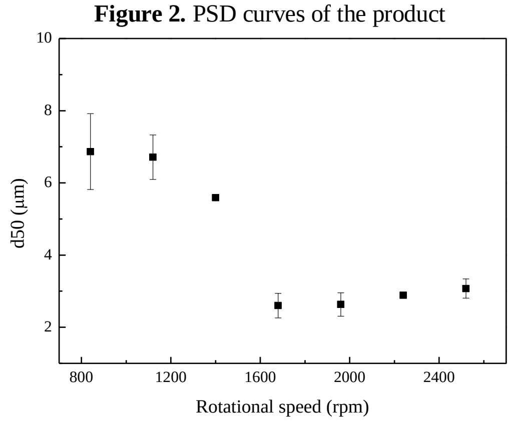

Figure 4. Effect of rotational speed on d50 of particles

4. Conclusions

A RPB reactor was applied to prepare Li_2CO_3 by the $LiHCO_3$ pyrolysis. It was found that the dispersant plays an important role in the Li_2CO_3 preparation process which would affect its particle size distribution. Experimental results shows that ethanol is the best dispersant to prepare Li_2CO_3 particles with small particle size distribution. The particle size distribution of Li_2CO_3 is mostly sensitive to the volume fraction of ethanol, rotational speed, and preheating temperature. The increases of these parameters could lead to the decrease of the median particle size. Nevertheless, the flow rates of feed solution and steam have nearly no effect on the particle size. Considering the product properties and energy consumption, the optimal operation parameters are volume fraction ψ=10%, rotational speed N=1680 rpm, flow rates of feed solution Q_L=450 mL/min, water steam Q_G=2 kg/h, and T=45 °C.

References

[1] Y.Z. Sun, X.F. Song, J. Wang, J.G. Yu. Cryst. Res. Technol. 46 (2011) 173–177.
[2] H. Zhao, L. Shao, J.F. Chen. Chem. Eng. J. 156 (2010) 588–593.
[3] J.F. Chen, L. Shao, F. Guo, X.M. Wang. Chem. Eng. Sci. 58 (2003) 569–575.

Keywords
Lithium carbonate; Pyrolysis; Dispersant; Rotating packed bed reactor.