Effect of Operating Conditions on Crystal Growth in an Airlift Loop Crystallizer

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Highlights

- There is a optimum value of V_N, pH and D_d on grain growth size of $Ni(OH)_2$.
- The gas-liquid separation zone has the best micro-mixing effect.
- With the increase of gas flow rate Q_G, the grain size D grow faster.

1. Introduction

Airlift loop reactors have been widely used in industry and paid more attention as crystallizers recently, because of their simple structure, efficient mixing and good mass/heat transfer characteristics [1-3]. Nickel hydroxide, $Ni(OH)_2$, is an important crystalline material for advanced energy conversion and storage, extensively used as cathode material in rechargeable alkaline batteries [4].

In this work, the effects of operating conditions on crystal growth in an airlift crystallizer are studied using $Ni(OH)_2$ as a model. $Ni(OH)_2$ particles prepared in different conditions are measured by XRD. Then, the sizes of crystallites of $Ni(OH)_2$ (D) are gotten by the Scherrer fomula and the variations of D with the volume of ammonium hydroxide (V_N), pH, feeding method, height of draft tube (H_d) and gas flow (q_g) are analyzed one by one. The main conclusions have been elaborated.

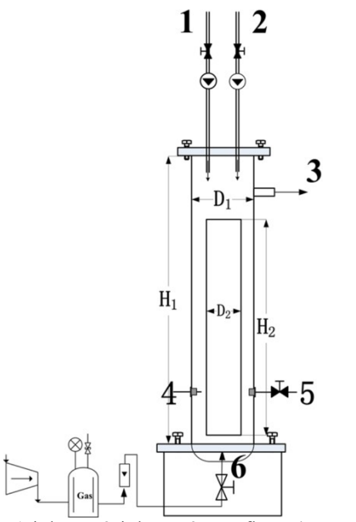
2. Methods

A schematic diagram of the ALR is shown in Fig. 1. The reactor is composed of an outer cylinder of diameter D_1=200 mm and height of H_1=400 mm, a draft tube of diameter D_2=120 mm and height of H_2=300 mm, and an air distributor. The clearance between the reactor bottom and the draft tube is 50 mm.

Before the start of an experimental run, distilled water is heated to the required temperature in an electric kettle and then added into the reactor, and air begins to bubble in through the distributor. After the temperature is stabilized, the NH_3 and NaOH (0.2 mol·L_{-1}) solution is added into the reactor to adjust pH value. Then, $NiSO_4$ is added into the reactor with the feed rate of 55 mL/h and reaction begins to happen. Besides, the NH_3 and NaOH (0.2 mol·L-1) solution is added continuously throughout the whole process to maintain the pH value. Samples are taken every 4 hours for tests including XRD, PSD (particle size distribution), SEM and tap density.

3. Results and discussion

We take the effect of addition of V_N as an example. Figure 2 shows the XRD map changes of nickel hydroxide with V_N. Under different amount of V_N, the crystal shapes of the obtained particles are all beta -Ni $(OH)_2$. With the gradual increase of V_N, the positions of each crystal surface overlap with the standard peaks, and the half width becomes narrower gradually; when the V_N increases to 500 mL, the half width becomes wider and wider. The impurity peak appears near (001) crystal surface, which is the peak corresponding to $NiSO_4·7H_2O$ crystal (111) crystal face. Fig. 3 shows that the size of crystallite varying with V_N, which indicates that D increases firstly and then decreases with the rising of V_N. As for pH and H_d, D varies with them according to the same trend as with V_N. When V_N=300 mL, pH=11.4, H_d=250 mm, the sizes of $Ni(OH)_2$ crystallites get to the maximum value.



1-inlet A; 2-inlet B; 3-overflow; 4-thermometer; 5-sample tap; 6-gas distributor

Figure 1. Schematic diagram of the ALR under investigation.

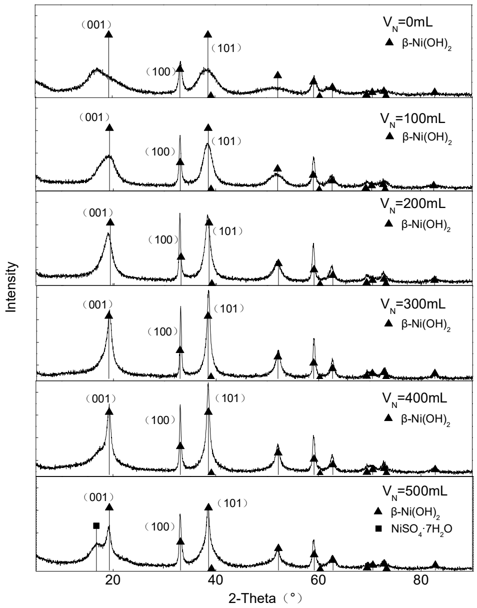
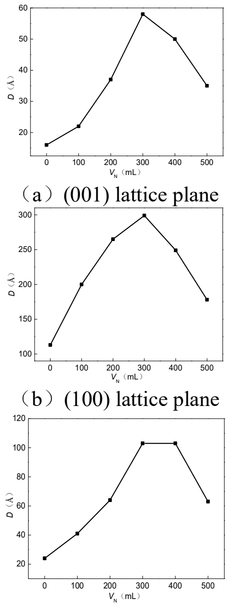


Figure 2. XRD maps of nickel hydroxide with the change of V_N



(a) (001) lattice plane

(b) (100) lattice plane

(c) (101) lattice plane

Figure 3. The size of crystallite varying with V_N.

4. Conclusions

The effects of initial ammonia additions V_N, pH value, feeding method, draft tube length H_d and gas flow rate q_g on the grain growth of $Ni(OH)_2$ in the circulation crystallizer were investigated. The grain size of $Ni(OH)_2$ particles increases first and then decreases with the increase of V_N, pH and H_d value. The micro-mixing in gas disengagement zone is the most efficient, the crystallites grow up quicker when the feeding position is located there. Gas flow has an important effect on the growth of crystallites. The $Ni(OH)_2$ crystallites grow up bigger with the increasing of gas flow rate q_g.

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Keywords

Airlift loop crystallizer; Operating conditions; Crystal growth; Nickel hydroxide