



Precipitating sandy aluminium hydroxide from sodium aluminate solution by the neutralization of sodium bicarbonate

Yan Li ^{a,b}, Yifei Zhang ^{a,*}, Chao Yang ^a, Yi Zhang ^a

^a Key Laboratory of Green Process and Engineering, Institute of Process Engineering, Chinese Academy of Sciences, Beijing 100190, China

^b Graduate School of the Chinese Academy of Sciences, Beijing 100039, China

ARTICLE INFO

Article history:

Received 19 December 2008

Received in revised form 24 March 2009

Accepted 25 March 2009

Available online 31 March 2009

Keywords:

Precipitation

Sodium aluminate solution

Sodium bicarbonate solution

Aluminium hydroxide

ABSTRACT

In the sinter process for alumina production, it is still difficult to produce sandy aluminium hydroxide by the carbonation of sodium aluminate solution. Therefore, a precisely controllable and feasible process for the precipitation of sandy aluminium hydroxide from the supersaturated sodium aluminate solution neutralized by sodium bicarbonate aqueous solution is proposed for the first time, in which the neutralization reagent (NaHCO_3) can be efficiently recycled by the carbonation of the produced sodium carbonate aqueous solution. The influencing factors of the precipitation, i.e. the pH value, the initial NaOH concentration, the temperature and the addition rate of neutralization agent, were investigated. All the aluminium hydroxides precipitated in the research were identified as gibbsite type. The gibbsite products had regular globular morphology and narrow size distribution whose minimum residue on a 44 μm mesh was 92–94%.

© 2009 Elsevier B.V. All rights reserved.

1. Introduction

The Bayer process and the sinter process are commonly used for the production of alumina from bauxite ores, in which alumina is obtained by the calcination of aluminium hydroxide, and the quality of the alumina largely depends on that of the aluminium hydroxide. The Bayer process is basically used for the extraction of alumina from the bauxite ores with the mass ratio of alumina to silica (A/S) above 9 (Misra, 1986). In the process, aluminium hydroxide is precipitated from the supersaturated sodium aluminate solution by supplying large quantity of gibbsite seed particles, and the spent caustic solution is recycled to extract alumina from the bauxite. The sinter process is widely used to process the poor-grade diasporic bauxite ores with A/S below 7 in China and Russia by sintering the bauxite ores with sodium carbonate aqueous solution. In the process, the supersaturated sodium aluminate solution is obtained by leaching the sintered bauxite pre-mixed with sodium carbonate solution. The precipitation of aluminium hydroxide is promoted by bubbling carbon dioxide gas into the supersaturated sodium aluminate solution, i.e., the carbonation process (Misra, 1986, Zhao et al., 2004), meanwhile the caustic in the sodium aluminate solution is converted to sodium carbonate which is recycled to extract alumina from the bauxite in the sintering process (Bi, 2006).

Before 1970s, alumina was produced in two different grades, i.e. flourey and sandy. During the last decades, the sandy alumina was more desirable because of the increased concerns of environmental

influence and energy cost in electrolytic aluminium industry (Arnsward et al., 1995, Gnyra and Brown, 1975). In order to produce eligible sandy alumina with the minimum residue of 90% on a 44 μm mesh, aluminium hydroxide obtained from sodium aluminate solution must have a minimum residue of 92–94% on a 44 μm mesh (Satapathy and Padhi, 1990). The eligible sandy aluminium hydroxide can be obtained by the Bayer process, however, in which the crystallization of aluminium hydroxide has a lower recovery ratio about 50% during the unusually long precipitation period about 48–72 h (Addai-Mensah and Ralston, 1999). For the sinter process, the decomposition ratio of the sodium aluminate by the carbonation is over 90% within 4 h, but the carbonation process is difficult to produce eligible sandy aluminium hydroxide with low fine particle content and narrow size distribution (Wang et al., 2005). In the gas–liquid reactive crystallization process of the carbonation, the low solubility of the precipitated aluminium hydroxide crystals and the relatively high concentration of carbon dioxide near the interface in the solution result in high local supersaturation, and further lessen the size of the precipitated aluminium hydroxide particles. Wachi and Jones (Wachi and Jones, 1991) also presented the high fine particle content was induced by the high resistance of gas–liquid interfacial mass transfer and the high interfacial supersaturation. Up to now, there is no successful example for producing sandy aluminium hydroxide from the sodium aluminate solution by the carbonation process.

For the precipitation of sandy aluminium hydroxide in the sinter process, a precisely controllable and feasible precipitation process of supersaturated sodium aluminate solution neutralized by sodium bicarbonate aqueous solution but carbon dioxide is proposed in this research. In the novel process, part of the sodium carbonate solution produced in the precipitation can react with carbon dioxide to

* Corresponding author.

E-mail address: yfzhang@home.ipe.ac.cn (Y. Zhang).

regenerate sodium bicarbonate (Hou, 1942), whilst the other sodium carbonate solution can be used to extract alumina from bauxite ores by the sintering process (Misra, 1986; Bi, 2006). Compared to the gas-liquid reactive crystallization of the carbonation process, the liquid-liquid reactive crystallization using sodium bicarbonate aqueous solution to neutralize the sodium aluminate solution is generally quite controllable, especially in modulating the supersaturation of the solution, and has significant advantages in the production of the precipitate with narrow size distribution and qualified morphology.

The reactions taking place in the proposed process are as follows:

1. HCO_3^- neutralizes free hydroxyl in the supersaturated sodium aluminate solution.



2. $\text{Al}(\text{OH})_4^-$, the dominating aluminate ions in the supersaturated sodium aluminate solution under the condition of $\text{pH} > 10$ (Li et al., 2005), decomposes to OH^- and $\text{Al}(\text{OH})_3$ precipitate.



In the above process to produce aluminium hydroxide, both reactions of the neutralization (1) and the decomposition (2) mainly take place in the bulk solution. The progress of the reaction (2) crucially depends on the reaction (1) which is a fast reaction of neutralization and its progress can be easily controlled by the addition of sodium bicarbonate aqueous solution. It suggests that the supersaturation of the aluminate solution, as well as the rate of the precipitation could be crucially operated to produce qualified sandy aluminium hydroxide with narrow size distribution and qualified morphology. So that the influencing parameters of the precipitation process, i.e. pH value of the solution, caustic soda concentration, precipitation temperature and the addition rate of sodium bicarbonate aqueous solution, were investigated.

2. Materials and methods

Precipitation experiments were performed in a 400 mL cylindrical stainless steel crystallizer with a mechanical stirrer, a pH electrode and an inlet for sodium bicarbonate aqueous solution. The crystallizer was immersed in a water bath thermostatically controlled within ± 0.5 °C.

Aluminium hydroxide (AR), sodium hydroxide (AR), sodium bicarbonate (AR) and deionized water were used in the experiments. Sodium bicarbonate aqueous solution was prepared by the dissolution of the sodium bicarbonate in deionized water, whilst sodium

Table 1
Starting solution specifications and experimental conditions for precipitation experiments.

Expt number	C (g/L, NaOH)	MR	Addition rate of NaHCO ₃ (mL/min)	A_{eq} (g/L, Al(BOH) _{3B})	Precipitation temperature (°C)	Final pH
1	154.8	1.5	1	95.0	70	10
2	193.5	1.5	0.5	130.6	70	11
3	193.5	1.5	1	130.6	70	11
4	193.5	1.5	2	130.6	70	11
5	172.9	1.5	1	110.9	70	10.2
6	192.3	1.5	1	129.4	70	10.5
7	200.0	1.5	1	137.2	70	10.8
8	216.8	1.5	1	154.9	70	10.9
9	229.7	1.5	1	169.3	70	11.2
10	193.5	1.5	1	86.0	50	11
11	193.5	1.5	1	106.6	60	11
12	193.5	1.5	1	130.6	70	11
13	193.5	1.5	1	158.3	80	11
14	193.5	1.5	1	130.6	70	12
15	193.5	1.5	1	130.6	70	13

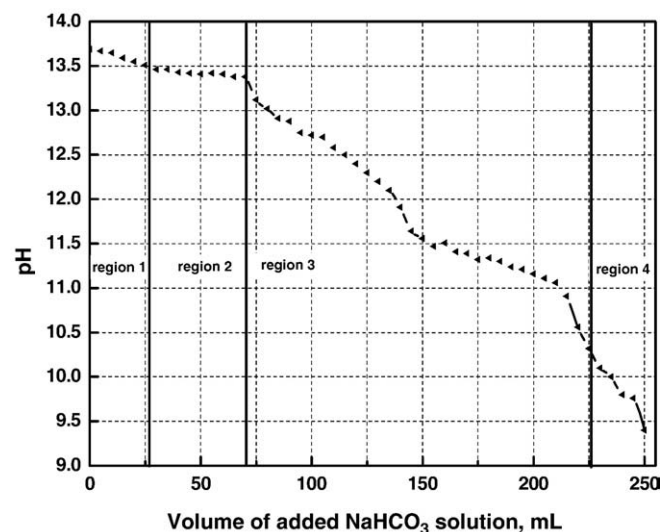


Fig. 1. Titration curve obtained at 70 °C.

aluminate solution by the dissolution of aluminium hydroxide into hot alkaline solution. The NaOH concentrations of the prepared sodium aluminate solution varied while the molar ratios of NaOH to $\text{Al}(\text{OH})_3$ (MR) were constant at 1.5. The concentrations of the aluminate solutions are listed in Table 1, in which C is the caustic concentration in g NaOH /L, A is the concentration of aluminium hydroxide in g $\text{Al}(\text{OH})_3$ /L, while A_{eq} , the equilibrium concentration of $\text{Al}(\text{OH})_3$ at the crystallization temperatures, is calculated by the method proposed by the reference (Misra and White, 1971).

50 mL of the supersaturated sodium aluminate solution with a certain NaOH concentration in the crystallizer was agitated at 300 rpm (sufficient to bring the slurry in full suspension). When the sodium aluminate solution was heated to a certain temperature within ± 0.5 °C, a measured volume of freshly prepared sodium bicarbonate aqueous solution was added to the crystallizer at constant rate, e.g. 1 mL/min, until the set pH value reached. After the start of the addition of sodium bicarbonate solution, the samples of the slurry were taken and filtered, and the $\text{Al}(\text{OH})_3$ content of the filtrate was analyzed by the ICP-AES method. To obtain the purified samples of the precipitates, the filter cakes were re-dispersed in hot deionized water, filtered and dried at 80 °C for 24 h.

The phase of the precipitates was identified by X-ray diffraction, using the Siemens D5000 X-ray diffractometer with $\text{CuK}\alpha$ radiation. The morphology of the precipitates was observed by the scanning electron microscope images, using a JEOL JEM 2100F instrument operated at 200 kV. The particle sizes distribution of the precipitates was measured by a Malvern Mastersizer-Laser particle size analyzer.

3. Results and discussion

3.1. Titration curves

The titration of the supersaturated sodium aluminate solution by sodium bicarbonate aqueous solution at the rate of 1 mL/min was carried out (Expt.1 in Table 1). The titration curve (pH vs V_{NaHCO_3}) at 70 °C is shown in Fig. 1. Different from the reference (Panias and Krestou 2007), there are four distinct regions in the diagram. In Region 1 ($\text{pH} > 13.46$), the spontaneous chemical reaction between the added HCO_3^- ions and the free hydroxide ions in the supersaturated sodium aluminate solution takes place. At the end of Region 1, or the start of Region 2, aluminium hydroxide begins to precipitate, which is indicated by the change of the color of the solution from clear to opaque, and the obvious increase of the buffering capacity of the aluminate solution. In Region 2 ($13.38 < \text{pH} < 13.46$), because of the

Download English Version:

<https://daneshyari.com/en/article/212930>

Download Persian Version:

<https://daneshyari.com/article/212930>

[Daneshyari.com](https://daneshyari.com)