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Study on Preparation Technology and Heat Conduction Mechanism of High-Purity & Ultra-Fine Alumina Powder from Scrap Aluminum Cans

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Abstract

In view of the increasing scarcity of bauxite resources in China, the high energy consumption and high pollution of electrolytic aluminum, and the requirements for energy conservation and environmental protection, aluminum recycling and high-value utilization of its derivatives have evolved into a crucial development requirement for the aluminum industry in the future. As an important part of the development of recycled aluminum resources, the high-value application of scrap aluminum cans has always been a hot research topic in various recycled aluminum processing enterprises and scientific research units. The traditional regeneration system of waste cans includes a series of complex technological processes such as pretreatment, paint removal, smelting system and casting system, which is difficult to control in the middle of the process. Most of the recycled scrap aluminum cans are cast and downgraded for later use, except for a part of them used as alloy materials for new cans. In this paper, combined with the research on the preparation of metal aluminum alkoxide, combined with recrystallization heat conduction to further study the effective dissolution or adsorption how to remove impurity elements to obtain high-purity aluminum alcohol salt mechanism research, and thermal effect of alcohols with different carbon chains on the synthesis of high-purity aluminum alkoxide was further investigated. Moreover, the changes in morphology and pore size distribution of hydrolyzed alumina precursor materials under different hydrothermal temperature conditions were discussed by means of the alkoxide hydrolysis-sol-gel process. Eventually, the aluminum alkoxide was obtained by the reaction of waste cans with isopropanol and heavy crystal thermal conductivity, and the high-purity aluminum alkoxide was purified by vacuum distillation. Under the hydrothermal condition of 160°C, the high-purity alumina material with a purity of 99.99% and an original crystal size of 200nm was prepared.

Keywords: Waste cans, Purification of metal alkoxides, High-purity alumina, Water heat treatment, Heat conduction

1. Introduction

Recycled aluminum is an essential way to recycle aluminum resources in China. Currently, the annual production capacity of China's recycled aluminum reaches 10 million tons. With the increasing scrap of China's automobile, household appliances and construction industries, aluminum consumption has ushered in its peak. By 2025, China will become the main source of global aluminum scrap. With the advent of the concept of carbon neutrality and carbon peaking proposed by the Chinese government, and in view of the increasing scarcity of bauxite resources in China, the high energy consumption and high pollution of electrolytic aluminum, and the requirements for energy conservation and environmental protection, aluminum recycling and high-value utilization of its derivatives have evolved into a crucial development requirement for the aluminum industry in the future. China ranks first in the world in terms of the recycling rate of scrap aluminum cans, reaching more than 99.5%. However, less than 20% of the recycled scrap aluminum cans are used to produce new cans, and most of them are downgraded to produce other aluminum processed products. In today's world, aluminum and its alloy products in industrial waste are mainly processed by aluminum remelting and recycling, supplemented by other recycling technologies. The traditional recycling system of waste cans includes a pretreatment, paint removal, smelting system and casting system. Among them, the paint removal and smelting system process have the greatest influence on the traditional regeneration process [1]. For one thing, substances such as hydrocarbons and heavy metal elements contained in the surface paint layer will enter the aluminum melt and produce dioxins, which will not only pollute the environment but also bring difficulties to composition adjustment, increase loss and even reduce the quality of recycled alloy. As a result, the inner and outer surface coatings of the tank must be cleaned as much as possible before smelting. No paint-removing process that boasts of being particularly economical, environmentally friendly and effective has been proposed, resulting in a high cost of paint-removing in general. For another, 3104 aluminum alloy is easy to react with water vapor in the air during high-temperature melting and is oxidized and absorbs hydrogen. Except for about 10%-20% of N_2, O_2, CO, etc., the gases in the liquid aluminum are all hydrogen. Since the saturated solubility of hydrogen in the liquid aluminum alloy is 17 times that in a solid state, about 90% of the hydrogen is dissolved in an aluminum liquid in atomic form. In the process of casting and solidification, hydrogen molecules are squeezed and move to the surface of molten liquid, forming bubbles. When bubbles fail to form on the surface of molten liquid, pinholes will be formed inside aluminum alloy castings, which becomes the source of endangering the internal quality of castings. Meanwhile, aluminum is extremely easy to oxidize during remelting, and about 20% of aluminum slag is produced, resulting in secondary waste.

Longkou Nanshan Aluminum Calendering New Material Co., Ltd., a subsidiary of Shandong Nanshan Aluminum Co., Ltd. (the largest manufacturer of aluminum cans in China), processes and recycles waste cans via a double-chamber furnace, but its capacity and technology are far from solving the problem of rational utilization of a large number of waste cans in China [2]. In this paper, the author cooperated with Nanshan Aluminum Co., Ltd., Tsinghua University Fine Ceramics Center and Institute of Solid State Chemistry and Mechanochemistry, Siberian Branch of Russian Academy of Sciences, and prepared high-purity & ultra-fine alumina powders by adopting metal alkoxide-sol-gel process, thus realizing the high-value product development and application of scrap aluminum cans. Additionally, the synthesis experiments of organic alkoxides with different alloy elements were illustrated. With the aid of a new adsorption-vacuum distillation process, the impurities in different alloy elements were dissolved or adsorbed effectively, and finally high purity aluminum alkyd was obtained. Combined with the comparison of synthesis and purification of different carbon chain alcohols, a process of removing excess alloy elements from waste cans with high efficiency and environmental protection was realized. The high-purity & ultra-fine alumina powder with a purity of 99.99% and original crystal size of 200nm was obtained by the alcoholysis-sol-gel process. See Fig. 1 for the technical route. The production process boasts a host of advantages such as mild conditions, streamlined process, purification treatment, no new hazardous waste and secondary pollutants, low energy consumption, and high added value of products. In the whole production process, alcohol can be recycled to generate part of hydrogen, without industrial waste gas, waste liquid, secondary hazardous waste, etc. Therefore, the process is green and environment-friendly. It opens up a new direction and market for the future green and low-carbon solution to the high-value application of scrap aluminum cans and related waste aluminum products, which is of high social and economic value.

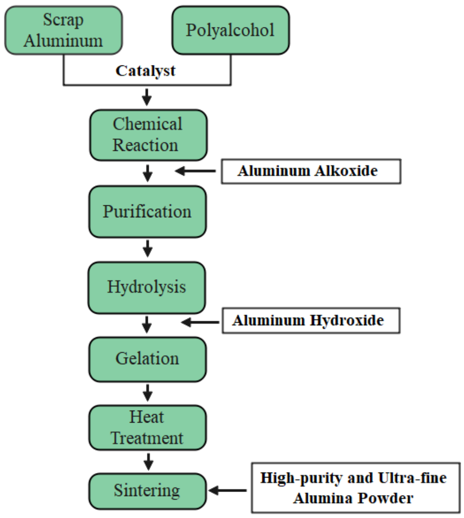

Fig.1 Flow chart of alumina powder preparation

2. Experimental section

2.1. Preparation of high-purity & ultra-fine alumina powder

Firstly, the metal alkoxide-sol-gel process was used to synthesize high-purity & ultra-fine alumina powders. Scrap aluminum cans were cut into aluminum sheets with a size of 3*5mm, and a small amount of aluminum alkoxide was added to the aluminum alkoxide as a catalyst in a molar ratio of 1:4 (aluminum sheet: polycarbon alcohol) to obtain the aluminum alkoxide. Subsequently, aluminum alkoxide was purified by adsorption-vacuum distillation to obtain high-purity aluminum alkoxide. The dispersant PVP was added and mixed with alcohol and water, and the template P123 was added while stirring at 60°C. After hydrolysis and mixing for 1h, the mixture was moved into a 250 ml stainless steel synthesis kettle lined with polytetrafluoroethylene, heated to 160℃ in a closed manner, and hydrothermal for 10h under its own pressure. After the hydrothermal treatment was completed, the alcoholic water was distilled out, the wet solid product was washed with deionized water to remove the alcoholic smell, and dried in the air at 120℃ to obtain the aluminum hydroxide-based precursor. The precursor dry powder was calcined in air at 1250℃ for 3h to remove the organic directing agent and residual organic alcohol, and then a high-purity & ultra-fine alumina sample was obtained.

2.2. Characterization of high-purity & ultra-fine alumina powder

X-ray diffraction (XRD) test of the sample was carried out on the D/max-γb X-ray diffractometer of Rigaku Corporation, Japan, with Cu target, Kα radiation source (λ=0.15406 nm), voltage 40kV and current 40mA. The morphology of the sample was observed by the KYKY-AMRAY-1000B scanning electron microscope (SEM).

3. Results and discussion

3.1. Mechanism analysis of effective purification of main alloy elements in waste cans

Organic metal alkoxides, on the one hand, have a low boiling temperature and are suitable for distillation purification processes, and on the other hand, have the characteristics of high solubility in organic solvents and are easy to react or crystallize in organic solvents. At present, vacuum distillation, extraction and recrystallization are often used to separate and purify organic alkoxides [3]. In the existing studies, the principle of the extraction method is to settle and separate insoluble matter by virtue of the property of metal alcohols soluble in organic solvents. However, this process is time-consuming and is not conducive to large-scale production [4]. The recrystallization method dissolves metal alkoxide containing impurities in an organic solvent and separates insoluble impurities by solvent evaporation, volatilization, and temperature reduction recrystallization to obtain high-purity metal alkoxide. However, this process is inefficient, often accompanied by the precipitation of other metal alkoxides, and it is also not conducive to large-scale production. Vacuum distillation is widely used in industrial production [5,6]. By taking advantage of the low boiling point characteristics of metal alkoxides, high purity metal alkoxides can be obtained quickly and in large quantities by vacuum distillation.

Table 1 shows the alloy composition of aluminum cans. Ordinary aluminum waste cans contain about 95% aluminum, and other main alloy elements are Si, Fe, Mn, Mg, etc. Only metals with a standard redox potential of -3.0V - -1.66V (relative to standard hydrogen electrode) can directly react with alcohols. As shown in Table 2, the standard redox potential of major impurities such as Fe is -0.44V, so the alcohol does not react with metallic iron theoretically. It can also be seen that, except for Al and Mg, other elements Si, Mn, Cu, Mg, Cr, Zn, Ti are not directly reacted with an alcohol to obtain alkoxide.

Table. 1.
Table of the metal composition of various parts of aluminum cans (weight percentage) [7]

Components of each part	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
3104 can body	≦0.60	≦0.80	0.05-0.25	0.80-1.40	0.80-1.30	/	≦0.25	≦0.10	Allowance
3004 can body	≦0.30	≦0.70	≦0.25	1.00-1.50	0.80-1.30	/	≦0.25	/	Allowance
5182 lid	≦0.20	≦0.35	≦0.15	0.20-0.50	4.00-5.00	≦0.10	≦0.25	≦0.10	Allowance
5042 pull ring	≦0.20	≦0.35	≦0.15	0.20-0.50	3.00-4.00	≦0.10	≦0.25	≦0.10	Allowance

Table. 2.
Standard redox potential table of metal elements in aluminum cans

Main metal elements	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
Redox potential	-0.14V	-0.44V	+0.52V	-1.185V	-2.37V	-0.42V	-0.76V	-1.37V	-1.66V

In this paper, compared with the pure aluminum used in the production of high-purity alumina (purity of more than 99.9%, with low content of impurity elements), the scrap aluminum cans used have an overall aluminum content of about 95%, which contains a variety of metal elements. Therefore, it is difficult to effectively remove the impurity elements in alkoxide by simple vacuum distillation. The author puts forward a new adsorption-vacuum distillation method, which can select a suitable adsorbent fixed-bed mode combined with a vacuum distillation process without affecting the purification efficiency of the metal alkoxide.

3.2. Analysis of adsorption mechanism and selection of adsorbent

Adsorption separation has three mechanisms: steric effect, kinetic effect and equilibrium effect [8]. Most adsorption separation processes are based on the equilibrium adsorption of mixtures. The design/selection of adsorbents has always been a complex subject. If it is used for purification, especially for ultra-purification, it needs strong adsorption bonds, which have a high Henry constant and can produce ultra-pure products.

There are two kinds of common adsorption isotherm models in chemical systems, namely the Langmuir adsorption isotherm model of monolayer [9] and the Freundlich adsorption isotherm model of multimolecular layer [10]. The equations are as follows:

$$\text{Langmuir: } C_e/Q_e = 1/(Q_0 b) + C_e/Q_0 \tag{1}$$

$$\text{Freundlich: } \ln Q_e = \ln K_F + C_e/n \tag{2}$$

Where [11], C_e is the equilibrium concentration of adsorbate in solution (mg/L) after adsorption reaches equilibrium; Q_e is the equilibrium adsorption capacity of adsorbate on the unit mass adsorbent (mg/g); Q_0 is the maximum adsorption capacity (mg/g); b is the adsorption strength (g/mg); K_F is the adsorption coefficient, which generally decreases with the increase of temperature; 1/n is the adsorption index. Generally speaking, when the value of 1/n is between 0 and 1, the smaller the value, the better the adsorption performance. When the value of 1/n is between 0.1 and 0.5, it is easy to be adsorbed. When the value of 1/n is greater than 2, it is difficult to adsorb.

The results show that Langmuir isothermal formula is more suitable for describing the surface of the mesoporous aluminum-based adsorbent. It shows that the surface of mesoporous aluminum-based adsorbent is a uniform single adsorption layer. The adsorption process of metal ions in water by mesoporous aluminum-based adsorbent is described by a quasi-second-order kinetic model, and the adsorption of metal ions by mesoporous aluminum-based adsorbent belongs to chemical adsorption [12].

In this paper, a fixed bed model is optimized according to Langmuir isothermal equation to determine the parameters of the adsorbent. If the parameters of the appropriate adsorbent are selected, the isothermal adsorption characteristics can be reflected without complicated calculation,and the selectivity of the adsorbent can be displayed and then its adsorption characteristics can be expressed. Firstly, according to the characteristics of mesoporous aluminum-based adsorbent, the Langmuir isotherm formula shows that when the saturated adsorption pressure is reached, the adsorbent reaches a limit coverage rate and the adsorption surface has uniform energy. Suppose the extended Langmuir equation of the binary mixed system is:

$$q_i = \frac{q_{m1} b_i p_i}{1 + b_1 p_1 + b_2 p_2} \tag{3}$$

The first equilibrium selectivity (α 1,2) in the fixed bed mode is defined as follows:

$$\alpha_{1,2} = \frac{x_1}{x_2} \cdot \frac{y_2}{y_1} \tag{4}$$

Where, X1 and X2 are the mole fractions of the two components on the surface of the adsorbent respectively; y1 and y2 are their corresponding molar fractions in the gas phase. It is generally assumed that component (1) is more easily absorbed. Combining the definition of (4) with the extended Langmuir equation, it can be seen that the adsorption selectivity of the first component is a constant in the whole partial pressure range:

$$\alpha_{1,2} = \frac{qm_1 b_1}{qm_2 b_2} = \left(\frac{K_1}{K_2}\right) \tag{5}$$

Where, the product on the molecule ($q_{mi}b_i$) is equivalent to the initial slope of the isotherm, that is, the Herry constant (k) corresponding to the ith component. Therefore, the adsorption selectivity coefficient is equal to the slope ratio of the adsorption isotherms of the two components at the beginning, that is, K1/K2.

The adsorption capacity of adsorbents mainly refers to the difference in adsorption capacity between high-pressure adsorption and low-pressure desorption of easily adsorbed components. The comparison of the adsorption capacity of the two components can be used as an ideal method to judge the adsorption performance of a special pressure swing adsorption cycle. Therefore, the second factor of the selection ratio of adsorption capacity as a parameter is defined as follows:

$$\mathrm{W} = \frac{\Delta q_1}{\Delta q_2} \tag{6}$$

After two contribution factors to the parameters are defined, the adsorption selection parameters of the fixed bed mode can be defined as follows:

$$S = W \cdot \alpha_{1,2} \tag{7}$$

Or written in expanded form.

$$\mathrm{S} = \frac{\Delta q_1}{\Delta q_2} \cdot \frac{q_{m1} b_1}{q_{m2} b_2} \tag{8}$$

As a result, the above parameters can be used to compare the performance of the two adsorbents. The better the performance of the adsorbent, the higher its S value will be.

In this paper, mesoporous activated alumina with a purity of 99.9% and a specific surface area of 450m^2/g was selected as adsorbent material. On the one hand, alumina adsorbent has high mechanical strength, high-temperature resistance and can adsorb water molecules and heavy metal ions [13]. On the other hand, it is considered that the separated product is high-purity aluminum alkoxide, and no other impurities are introduced to ensure the high-purity alumina product.

3.3 Comparison between adsorption-vacuum distillation and traditional vacuum distillation

In experiment 1, 50g waste cans aluminum sheet and anhydrous isopropanol were used for the synthesis reaction in the presence of a small amount of aluminum alkoxide as a catalyst, and the reaction was completed at 85°C for 2h, and a gray black alkoxide mixture containing impurities was obtained, with obvious impurity particles at the bottom of the flask. Then, with the traditional vacuum distillation device, the organic aluminum alkoxide with the obvious green color can be obtained by vacuum distillation for 1h (high purity aluminum alkoxide is colorless, clear or light blue). It indicates that the traditional vacuum distillation can not effectively remove the impurity elements in the organic metal alkoxide, and even some impurity elements which are difficult to react to are dissolved into the metal alkoxide solution with the synthetic distillation process, resulting in difficult separation.

In experiment 2, 50g scrap aluminum cans and anhydrous isopropanol were used to obtain the metal alkoxide solution after vacuum distillation, and then the temperature was raised to 100-110°C. The adsorbent product was fixed on the fixed bed of the adsorption column by the reactor in Fig. 2 for adsorption separation [14]. Among them, the selected active mesoporous alumina adsorbent product (purity 99.9%, specific surface area 450m^2/g) was used as the adsorbent. The organic alkoxide treated by adsorption-vacuum distillation has no obvious color with naked eyes, and the liquid is colorless and clear. To reduce the influence of dioxin caused by the hydrogenation of paint in waste tanks, a layer of activated carbon/carbon nanotubes can be added at the top as a strong and effective adsorbent for dioxin [15].

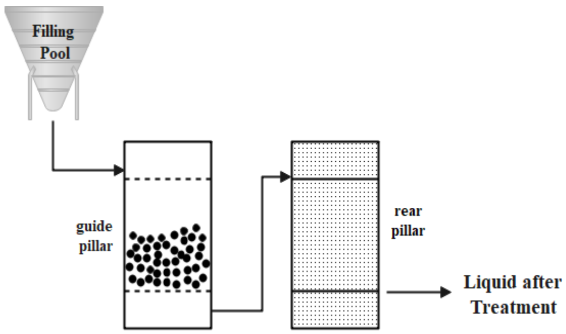

Fig. 2. Flow chart of fixed bed mode adsorption process

3.4 Selection of polycarbon alcohol and its influence on synthesis of alkoxides

The alkoxides synthesized by polycarbonates and waste aluminum from cans, which are A alcohol alkoxide, B isopropanol alkoxide, Csec-butanol alkoxide, Disoamyl alcohol alkoxide and En-hexanol alkoxide from top to bottom. The synthesis time and physical state are shown in the table 3.
Among them, only A alcohol alkoxide and B isopropanol alkoxide are dark green liquid at room temperature, and light green liquid alkoxide can be obtained by vacuum distillation and purification at 210°C/20mmHg. An alcohol alkoxide has a too fast but insufficient response; B isopropanol alkoxide has a moderate reaction rate, with a synthesis time of 3h; En-hexanol alkoxide is a gray-black liquid, with a synthesis time of 2h, so it cannot be purified by vacuum distillation. Disoamyl alcohol alkoxide cannot be effectively synthesized. After comprehensive comparison and consideration of environmental friendliness, aluminum alkoxide of isopropanol can be effectively obtained by the reaction of isopropanol with waste aluminum of cans, which is beneficial to the recovery of isopropanol in subsequent hydrolysis.

Table 3.
Synthesis time and physical state of different alkyds

Serial No.	Alkoxide	Synthesis time	Normal temperature state	Vacuum distillation
1	A alcohol alkoxide	1h	Liquid state	Vacuum distillation is not allowed
2	B isopropanol alkoxide	3h	Liquid state	20mmHg/210°C
3	C sec-butanol alkoxide	4.5h	Solid state	
4	D isoamyl alcohol alkoxide	No response	—	
5	E n-hexanol alkoxide	6h	Solid state	Vacuum distillation is not allowed

3.5. Influence of hydrothermal conditions on particle morphology and pore size distribution of alumina precursor

It is preferred that aluminum isopropoxide alkoxide be hydrolyzed for 1h under the action of the P123 template, with the molar ratio of alkoxide to water being 1:3, and then subjected to hydrothermal treatment at 120°C, 140°C, 160°C and 180°C for 10h respectively. As shown in the SEM image of Fig. 3, it is found that the grain size tends to grow up at high temperatures, and with the increasing of temperature, the grain size tends to be oval or round, and the grain size also increases from more than 10nm to 50-60nm. At the same time, as shown in Fig. 4, the pore size distribution also becomes larger and wider with the increase in temperature. Therefore, to obtain alumina particles with good nano-dispersivity, the hydrothermal temperature should be properly increased, and the pore size and pore size distribution should be controlled. In this paper, the precursor with hydrothermal treatment at 160°C was preferred, and the alumina precursor material with a grain size of 30-50nm, the pore size of 20nm and concentrated pore size distribution can be obtained. Finally, the high-purity alumina material with a purity of 99.99% and a grain size of 200nm was obtained by calcining at 1250°C for 3h to remove the organic directing agent and residual organic alcohol.

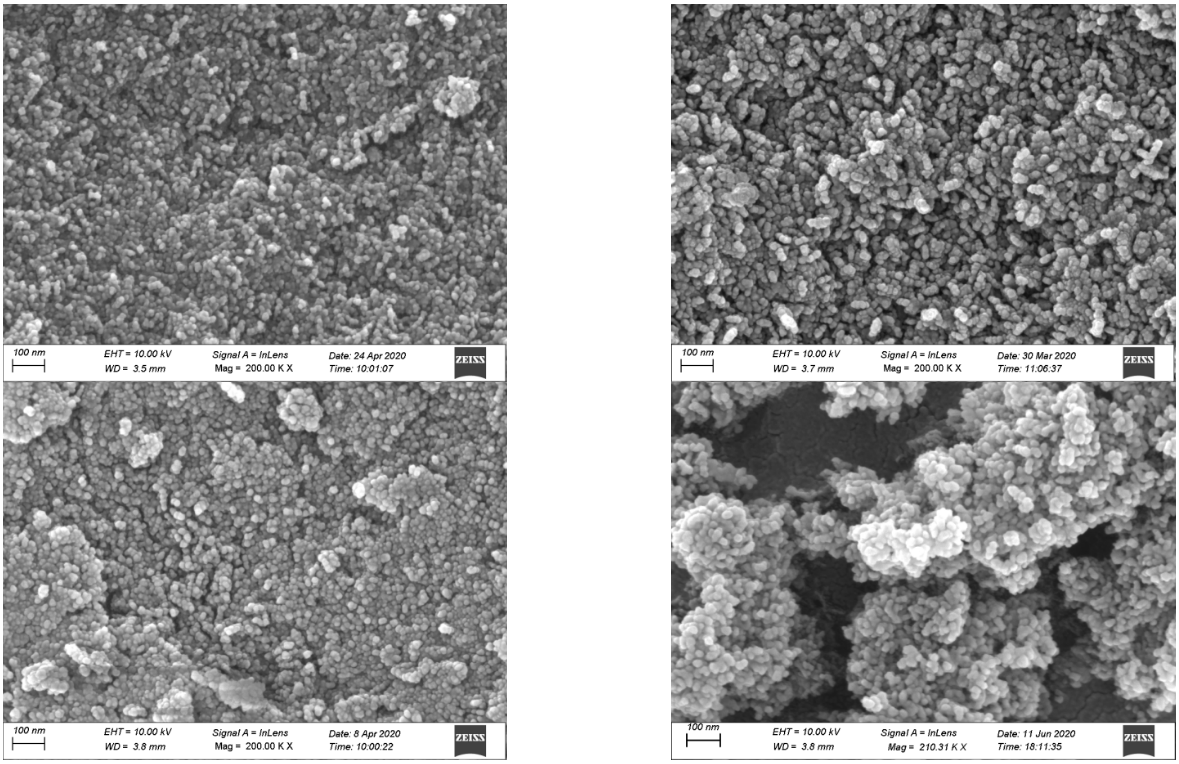

Fig. 3. SEM images of precursor body shape at different hydrothermal temperatures

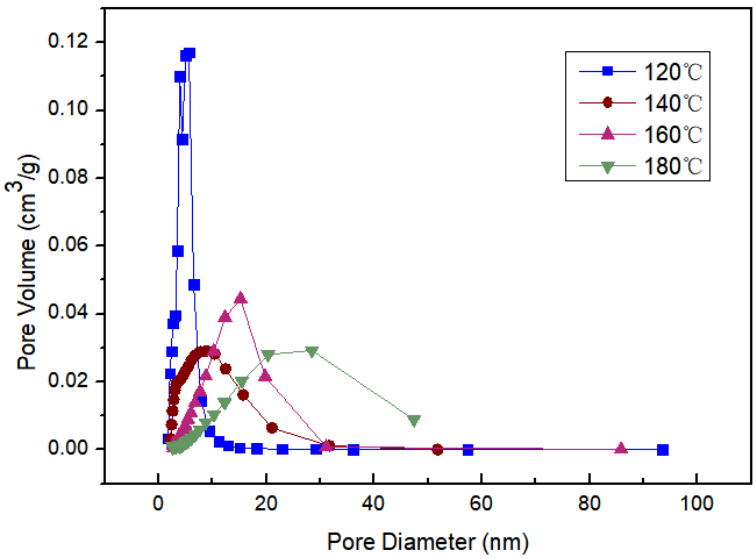

Fig. 4. BET images of pore size distribution at different hydrothermal temperatures

3 Conclusion

1. The traditional recycling system of waste cans has a long process flow and difficult intermediate control, and most recast products are degraded for later use. In this paper, the preparation of high-purity aluminum oxide by metal aluminum alkoxide is proposed, and the mechanism of high-purity aluminum alkoxide is obtained by effectively dissolving or adsorbing impurity elements by a new adsorption-vacuum distillation process instead of the traditional vacuum distillation process is analyzed.

2. With the analysis of the adsorption mechanism and the selection of adsorbents, it is pointed out that Langmuir isothermal equation is more suitable for describing the surface of mesoporous aluminum-based adsorbents. It shows that the surface of mesoporous aluminum-based adsorbent is a uniform single adsorption layer. The adsorption process of metal ions in water by mesoporous aluminum adsorbent is described by a quasi-second-order kinetic model.
3. Based on the influence of multi-carbon alcohol on the synthesis of alkoxide, it is found that isopropanol has high synthesis efficiency, easy distillation and environmental friendliness. Taking into account the influence of different hydrothermal temperatures on the morphology and pore size distribution of alumina precursor particles, it is found that the grain size tends to grow up at high temperatures, and with the rising temperature, the grain tends to be oval or round. At the same time, the pore size will increase with the increase in temperature, and the distribution will be wider.
4. By adopting a new adsorption-vacuum distillation process combined with alcoholysis to prepare alumina, scrap aluminum cans with a purity of about 95% can be impurity-removed to obtain 200nm high-purity & ultra-fine alumina with the purity of 99.99%.

Acknowledgments

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