Effect of particle size on flowability of Lithium hydroxide powder

Hua Guo\textsuperscript{1a}, Wangyang Yu\textsuperscript{2b}, Jian Zhou\textsuperscript{1c}, Hongjun Li\textsuperscript{1d}, Fangming Shen\textsuperscript{3e}, Jinjian Zhang\textsuperscript{3f}

\textsuperscript{1}School of Mechanical Engineering and Automation, Zhejiang Sci-Tech University, Hangzhou, 310018, China
\textsuperscript{2}Sanhua(Hangzhou) Microchannel Heat Exchanger Co., Ltd, Hangzhou, 310018, China
\textsuperscript{3}Zhejiang Quzhou Yongzheng Lithium Technology Co., LTD, Quzhou, 324000, China
\textsuperscript{a}guohvae@163.com, \textsuperscript{b}yuwongyoung@163.com, \textsuperscript{c}joezhoujian@zstu.edu.cn, \textsuperscript{d}lihongjun@zstu.edu.cn, \textsuperscript{e}shenfangming@yz-li.com, \textsuperscript{f}zhangjinjian@yz-li.com

Abstract: In order to improve the flowability of lithium hydroxide powder, the lithium hydroxide powder in a company was selected under the conveying condition. The degree of circularity was used to quantitatively characterize the micro-morphology of the powder, and the influence of particle size on the flowability of lithium hydroxide powder was studied by the angle of repose method, HR method and Carr flowability index method. The results show that the lithium hydroxide powder gets better with the increasing of particle size. But the flowability of fine particles (≤ 100 \textmu m) is poor, while the flowability of medium particles (100~200\textmu m) and coarse particles (≥ 200\textmu m) is good. Based on the experimental results. It is suggested that the inclination angle of silo should be designed to be no less than 50° and the content of powder with particle size less than 100\textmu m in lithium hydroxide should be controlled to improve the conveying efficiency of lithium hydroxide powder.

1. Introduction

Because of its high energy density, long service life and good safety, high nickel ternary battery has shown great potential in the field of new energy. Lithium hydroxide is an important lithium source for ternary materials with high nickel content. Compared with lithium carbonate, it has obvious advantages in reducing sintering temperature and improving battery charging and discharging performance. Therefore, it is highly praised by many battery manufacturers\textsuperscript{[1-3]}. Lithium hydroxide is affected by its own flowability in the process of transportation and crushing. Lithium hydroxide with good flowability has stronger controllability and stability in the transportation process, while lithium hydroxide with poor flowability will have problems such as blockage and unsmooth transportation, affecting the efficiency of transportation. Therefore, it is of great significance to study the flowability of lithium hydroxide for the optimization of process and the design of conveying device.

At present, in order to improve the lithium mixing uniformity of high nickel ternary materials, battery manufacturers not only require the quality of lithium hydroxide, but also require higher and higher particle size and morphology. The smaller the particle size, the worse the flowability. Wang C H et al.\textsuperscript{[4]} studied the relationship between particle size and flowability of Shenfu bituminous coal by angle of repose method and HR method. The results show that the compressibility of Shenfu bituminous coal is increasing, but the flowability is getting worse with the decrease of particle size. The angle of repose of
Shenfu bituminous coal is greater than 40⁰, and the HR index is between 1.2~2.0. Qi H F et al. [5] used Carr flowability index method to test and analyze three different pulverized coal. It is found in the blanking test that when the particle size is between 36~75µm, the flow mode of pulverized coal is funnel flow. And when the particle size increases to more than 75 µm, the flow mode of pulverized coal changes into integral flow. With the increasing of particle size, the Carr flowability index of three kinds of pulverized coal shows an upward trend. When the particle size increases to 312µm, the Carr flowability index of pulverized coal is greater than 80, and the pulverized coal becomes a free powder which is easy to flow.

Therefore, this paper will study the influence of particle size on the flowability of lithium hydroxide by angle of repose method, HR method and Carr flowability index method. From the experimental results, some measures are put forward to improve the flowability of lithium hydroxide powder.

2. Experimental Part

2.1 Selection and screening of powder

At present, battery-grade lithium hydroxide is mainly divided into two categories: coarse-grained lithium hydroxide and micropowder-grade lithium hydroxide. The particle size of coarse-grained lithium hydroxide is generally between 200~1000µm, while the particle size of micropowder-grade lithium hydroxide is customized according to the downstream application requirements. In this paper, the lithium hydroxide powder in Zhejiang Quzhou Yongzheng Lithium-ion Technology Co., Ltd. was selected under the conveying condition. After that, the powder still needs airflow crushing before it can be packaged.

In order to study the influence of particle size on the flowability of lithium hydroxide powder, in this paper, 75-mesh and 150-mesh quasi-sieve was used to screen the selected lithium hydroxide powder into coarse powders (≥ 200µm), medium powders (100~200µm) and fine powders (≤ 100µm). The microstructure of lithium hydroxide powder with different powder sizes was observed by the JSM-5610LV scanning electron microscope. Subsequently, ImageJ software was used to process the SEM images, and the degree of circularity of each particle was obtained by counting the perimeter and projected area of each powder. The degree of circularity was used to quantitatively characterize the shape of lithium hydroxide powder [6].

2.2 Experimental test

The angle of repose, angle of spatula, tight bulk density and loose bulk density are measured by BT-1000 powder comprehensive property tester. And the degree of proportion is measured by malvin 3000 laser particle size analyzer. The angle of repose and angle of spatula are measured by injection method, while the tight bulk density and loose bulk density are measured by fixed volume method.

3. Results and discussion

3.1 Morphology observation and characterization

It can be observed from Figure 1 that the shape of fine powders is extremely irregular, such as sharp angle, fiber, hexahedron, etc., and the particle size distribution is uneven. However, the shape of medium powders and coarse powders is relatively regular, with sharp-angled particles obviously reduced. But the proportion of nearly spherical powders obviously increased, and the particle size distribution is more uniform than that of fine powders.
Figure 1. Micro-morphology of different granular powders

The larger degree of circularity, the more regular the powder shape. Figure 2 shows the distribution state and size of degree of circularity of different granular powders, in which the degree of circularity concentration is expressed by interquartile spacing (the length of the box). The smaller the interquartile spacing is, the more concentrated the degree of circularity of the powder is, and the smaller the shape difference is. It can be seen from the figure that the average degree of circularity of coarse powders, medium powders and fine powders are 0.873, 0.778 and 0.676 respectively, and the interquartile spacing is 0.081, 0.135 and 0.178 respectively. It shows that the shape of coarse powders is the most regular and the shape difference is the smallest. Medium powders take second place. The shape of fine powders is the most irregular, and the shape difference is significant. Because the lithium hydroxide powder was impacted and sheared in the previous mechanical crushing process, the shape of the crushed powder changed greatly. In addition, the crushing effect decreases with the decrease of particle size, and there are still a large number of irregularly shaped powders in fine powders. It is necessary to adjust the technological parameters of mechanical crushing, such as the rotating speed of ball mill, ball milling time, ball-to-material ratio, etc.

Figure 2. Distribution and size of degree of circularity of different granular powders

3.2 Flowability Test Results and Analysis of Lithium Hydroxide Powder

3.2.1 Angle of repose method

The smaller the angle of repose, the smaller the friction between powders, and the better the flowability of powders[7]. The experimental results of the angle of repose are shown in Table 1 below. The angle of repose of coarse powders and medium powders is not much different, and the angle of repose is between 30° ~ 40°, which shows good flow performance. But coarse powders have the smallest angle of repose and the best flowability. The average angle of repose of fine powder is 46.37°, which exceeds the angle of repose of the other two powders by 10°. It shows that the flowability of fine powders is poor. The
flowability of powder is greatly affected by particle size and morphology\cite{8,9}. It can be seen from the above SEM images that the shape of fine powders is irregular. However, the irregular powder has many actual contact points and large specific surface area. The movement resistance of the powder in the flow process becomes larger, which makes it difficult for the fine powders to flow. With the decrease of particle size, the gravity of powder itself decreases, but the adsorption force between powders increases. Resulting in an increase in the ratio of adsorption force to gravity. Agglomeration between powders is easy to occur, which makes the flow of fine particles more difficult. Therefore, in order to improve the flowability of lithium hydroxide, the inclination angle of the silo should be designed to be no less than 50°.

| Table 1 Test results of angle of repose |
|----------------------------------------|
| Angle of repose | Fine powders | Medium powders | Coarse powders |
| Sample 1(°)    | 46.41        | 36.19          | 33.41          |
| Sample 2(°)    | 45.16        | 35.98          | 34.02          |
| Sample 3(°)    | 47.54        | 36.76          | 33.58          |
| Average(°)     | 46.37        | 36.31          | 33.67          |

3.2.2 HR method

HR method uses two parameters, compressibility and HR, to express the bulk density change of powder before and after compression. The smaller the compressibility and HR, the worse the compressibility of the powder, which indicates that the flowability of the powder is better\cite{10}.

The bulk density of powder mainly depends on the shape, size, distribution and packing mode of particles. It can be seen from Figure.3 that the loose bulk density of fine powders is the smallest, which is 0.721 g.cm\(^{-3}\). Furthermore, the loose bulk density of medium powders and coarse powders is both greater than 0.8 g.cm\(^{-3}\), among which the loose bulk density of coarse powders reaches 0.895 g.cm\(^{-3}\). It shows that the bulk density of lithium hydroxide powder increases with the increase of particle size. However, the maximum value of tight bulk density is 0.994 g.cm\(^{-3}\) of fine powders. Due to the uneven particle size distribution of fine powders, its shape is irregular and different. In the process of testing the tight bulk density, the fine powders will be rearranged under the action of vibration, resulting in a large number of small particle powders filling into the gaps of large particle powders. So that the bulk density of fine powders is significantly increased. However, the particle size distribution of medium powders is uniform and its shape is regular. Even if the powders are rearranged by vibration, the increase of its bulk density is not as much as that of fine powders. Therefore, the tight bulk density of medium powders is the smallest, which is 0.893 g.cm\(^{-3}\).
The results show that when the HR of the powder is less than 1.2 and the compressibility is less than 15%, the powder can show good flowability, and agglomeration will not occur\(^{[11]}\). For this reason, the HR and compressibility of different granular powders were calculated by the experimental results of tight bulk density and loose bulk density. As can be seen from Figure 4, the compressibility and HR of fine powders are both the maximum values, which are 27.46% and 1.38 respectively. It shows that the flowability of fine powders is poor. Nevertheless, the compressibility and HR of coarse powders are 5.29% and 1.06 respectively, which are not different from those of medium powders. It is clarified that coarse powders have the best flowability, but medium powder also shows good flowability. Accordingly, in order to improve the flowability of lithium hydroxide, the ball milling time and speed of mechanical crushing should be adjusted. Thereby controlling the content of lithium hydroxide powder with the particle size less than 100µm.

![Figure 4 Compressibility and HR under different granular powders](image)

**3.2.3 Carr flowability index method**

Carr flowability index method is a comprehensive evaluation method to evaluate the flowability of powder. The higher the Carr flowability index is, the better the powder flowability is. The flowability index of Carr exceeds 80, which indicates that the powder has good flowability\(^{[12]}\).

The test results are shown in Table 3 below. The spatula angle and the angle of repose represent the same flow properties. Among them, the spatula angle of fine powders is 61.52°, which exceeds that of medium powders and coarse powders by 20°, indicating that the flowability of fine powders is poor. The Carr flowability index of medium and coarse powders is 85.5 and 90 respectively, while that of fine powder is only 61. According to the Carr flowability evaluation table\(^{[13]}\), it can be concluded that the flowability grade of coarse powders and medium powders is above good grade, and no flow aid measures are needed. Nonetheless, the flowability grade of fine powders is general, reaching the boundary line where materials are attached and hung. Consequently, in the process of conveying lithium hydroxide powder, it is necessary to take measures to improve its flow performance.

| Test project                  | Fine powders | Medium powders | Coarse powders |
|------------------------------|--------------|----------------|----------------|
| Angle of repose (°)          | 46.31        | 36.33          | 33.67          |
| Compressibility (%)          | 27.46        | 8.4            | 5.29           |
| Angle of spatula (°)         | 61.52        | 38.15          | 35.43          |
| Degree of proportion         | 8.14         | 1.33           | 2.55           |
| Total flowability index      | 61           | 85.5           | 90             |
4. Conclusion

Based on the above different testing methods, this paper draws the following conclusions:

1) The experimental results of repose angle method, HR method and Carr flowability index method show that the particle size of lithium hydroxide powder has great influence on its flowability. Medium powder (100 ~ 200 µm) and coarse powder (≥ 200 µm) all show good flowability, but fine powder (≤ 100 µm) has poor flowability. The angle of repose of fine powders exceeds 100 compared with medium powders and coarse powders. The compressibility and HR of medium and coarse powders are less than 10% and 1.10 respectively, while the compressibility and HR of fine powders are 27.46% and 1.38 respectively. The Carr flowability index of medium and coarse powders exceeds 80, while that of fine powders is only 61.

2) The loose bulk density of lithium hydroxide powder increases with the increase of particle size, but the tight bulk density shows different rules. The maximum tight bulk density is fine powders, which is 0.994 g.cm⁻³. However, the minimum tight bulk density is 0.893 g.cm⁻³, which is medium powders.

3) In order to improve the transportation efficiency of lithium hydroxide in the production process, the content of powder with particle size less than 100µm in lithium hydroxide should be controlled. In addition, the transportation device can be optimized to design the inclination angle of the silo to be no less than 50°.

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