AA06 - The Effects of Suspended Solids in Green Liquor on the Quality of Product During the Precipitation Process

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Abstract

Based on the analysis of the chemical and mineralogical composition of suspended solids in green liquor, and the impurities distribution in different size aluminum hydroxide, the main theory that suspended solids in green liquor deciding product quality was explored. The results demonstrate that higher suspended solids will degrade quality of product. Meanwhile, four consecutive cycle precipitation tests were carried out to verify the relative discoveries.

Keywords: Green liquor, suspended solids concentration, quality of product.

1. Introduction

The alumina factory of China produces alumina with diaspore bauxite mainly, during the process of alumina production by Bayer process, green liquor is obtained after 265 ℃ digestion and subsidence filtration [1]. While subsidence filtration can not achieve complete separation of solids and liquid phase, namely some suspended solids still being contained in the green liquor, which will directly enter aluminum hydroxide and result in chemical impurity of final product. Currently, all alumina refineries have required that suspended solids level is less than 0.015~0.030 g/L in green liquor, many of them can not fulfill control to this index, which have caused the standard exceeding of impurity content in their products. According to *GB/T 4294-2010 Aluminum hydroxide* and *GB/T 24487-2009 alumina*, it is required that the contents of SiO_2, Fe_2O_3 and Na_2O in first grade alumina product must be less than 0.02 %, 0.02 % and 0.5 % respectively [2]. At the stage, all alumina refineries have conformed to the principle of high quality and high price, chemical quality unqualified of aluminum hydroxide and alumina will degrade product, which will affect economic price of the product [3].

2. Equipment and Method

2.1 Test Equipment

Main equipment used in this test include precipitator with mechanical agitation and thermostatic water bath heating, analytical balance, sieve shaker, vacuum filtration apparatus, etc.

Analysis involved in the tests including: Chemical component of suspended solids in green liquor was analyzed using X-ray fluorescence spectrophotometer PW2403, mineral composition of green liquor suspended solids were analyzed using X-ray diffraction instrument, SiO_2 and Fe_2O_3 content in aluminum hydroxide was analyzed using 723N visible spectrophotometer, Na_2O was analyzed using atomic absorption spectrometer SOLAARM6, and sodium aluminate solution component was analyzed using chemical titration method.

2.2 Test Method

Component analytical method for green liquor and suspended solids: green liquor from a production site of alumina, followed by vacuum filtration, chemical and mineral composition analysis after cleaning and drying the filter cake.

Component analytical method for impurity in seed of aluminum hydroxide: seed of aluminum hydroxide from production site of alumina, analyze impurity component content after blending, washing and drying, then conduct granularity screening of the sample, analyze the impurity component by adopting aluminum hydroxide from different particle size.

Method and condition for precipitation test: the green liquor was obtained from a production site and filtered. The solids were discarded. Treated and untreated green liquor was added into a precipitator preheated to the set temperature followed by the addition of aluminum hydroxide while stirring for the duration of the scheduled precipitation temperature and time. Solids separation from the slurry followed. A portion of the solids was retained for analysis while the rest was used as the precipitation seed crystals for next cycle. A total of four precipitation cycles were performed. The slurry solids content was 830 g/L, the precipitation time was 45 hours, the precipitation initial temperature was 65 °C and the end temperature was 55 °C.

3. Result and Discussion

3.1 Analysis Result of Suspended Solids in Green Liquor

The chemical component analysis result of suspended solids in table 1 shows that the suspended solids are 50 % fine red mud and 50 % calcium oxide and alumina suspended solids containing fine red mud that enter the precipitation system will bring in detrimental impurities that will cause a raise of impurities in the alumina product.

Mineral component analysis of suspended solids is shown in Table 2. Main components in the suspended solids are garnet, cancrinite and $3CaO \cdot Al_2O_3 \cdot CaCO_3 \cdot 11H_2O$. Among them cancrinite and partial hydration garnet may be directly brought in by red mud fine particles. The rest may be the outcome of reaction between alumina and milk of lime additive, which is the filter aid used in the filtration process prior sodium aluminate precipitation.

Table 1. Chemical Composition of Suspended Solids, %.

Composition	Al_2O_3	SiO_2	Fe_2O_3	TiO_2	K_2O	Na_2O	CaO	MgO
Content	27.98	8.21	1.86	1.39	0.55	2.27	32.17	0.64

Table 2. Mineral Composition of Suspended Solids, %.

Composition	Hydro Garnet	Cancrinite	$Ca(OH)_2$	Diaspore	Perovskite	$3CaO \cdot Al_2O_3 \cdot CaCO_3 \cdot 11H_2O$	$CaCO_3$
Content	55	15	2	2	2.5	22	1

3.2 Analysis Result of Impurity Component for Aluminum Hydroxide Seed

The chemical component and suspended solids content in green liquor from an alumina refinery is shown in table 3, which is also the analytical result of four times' precipitation test liquor. The chemical component of impurities for aluminum hydroxide is shown in table 4, which is the impurity analytical result of initial seed in precipitation test. By analyzing, suspended solids of the alumina plant have exceeded the standard considerably and the SiO_2 content of aluminum hydroxide seed has also been as high as 0.017 %.

Table 3. Chemical Component and Suspended Solids Content in Green Liquor, g/L.

Serial number	Na_2O_T	Al_2O_3	Na_2O_K	α_k	Suspended solids
1	179.36	181.3	154	1.40	0.058
2	184.71	183	160	1.44	0.066
3	183.07	175.2	159	1.49	0.084
4	176.53	172.8	154	1.47	0.176

Table 4. Chemical Component of Aluminum Hydroxide Seed, %.

Composition	SiO_2	Fe_2O_3	Na_2O
Content	0.017	0.013	0.23

Granular screening was conducted of the aluminum hydroxide seed in table 4. The impurities content in different granular levels can be seen in Table 5, Figure 1, Figure 2 and Figure 3. By analyzing, SiO_2 and Fe_2O_3 have mainly distributed in aluminum hydroxide fine particles above 325 mesh. Fine suspended solids have directly entered aluminum hydroxide seed with sodium aluminate solution. They have caused the impurity levels of fine particles in the product to increase resulting in the aluminum hydroxide standard in the product to exceed its specification.

Table5.Chemical Component in Different Granular Grades for Aluminum Hydroxide, %.

Granular grade distribution (mesh size)	SiO_2	Fe_2O_3	Na_2O	Weight ratio
0~100	-	-	-	0.24
100~170	0.006	0.012	0.24	11.8
170~200	0.011	0.012	0.22	36.8
200~325	0.014	0.012	0.21	26.2
-325	0.033	0.021	0.22	24.9
Before screening	0.017	0.013	0.23	100

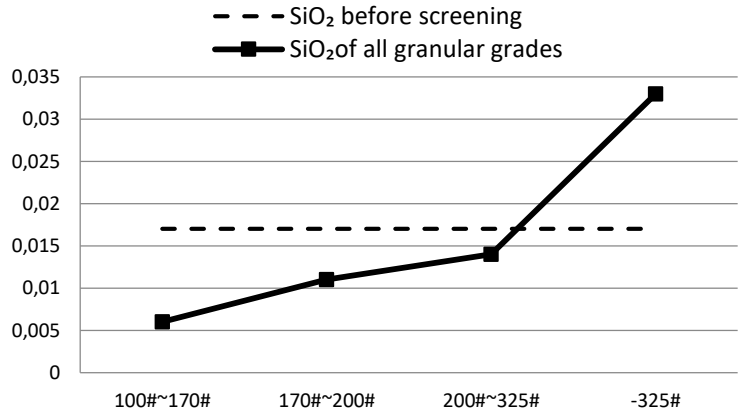

Figure 1. SiO_2 Content in aluminum hydroxide from different granular grades.

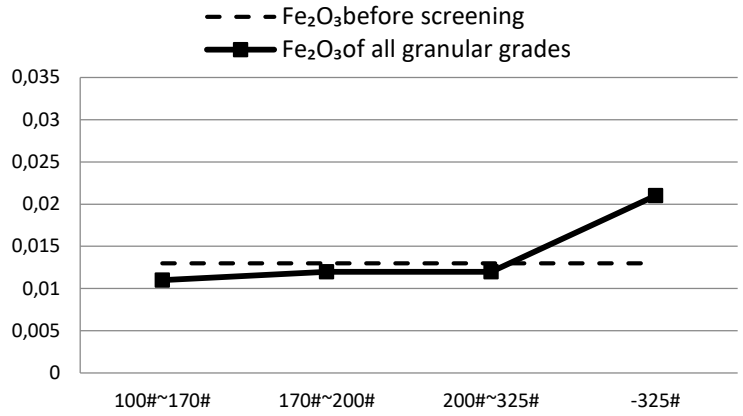

Figure 2. Fe_2O_3 content in aluminum hydroxide from different granular grades.

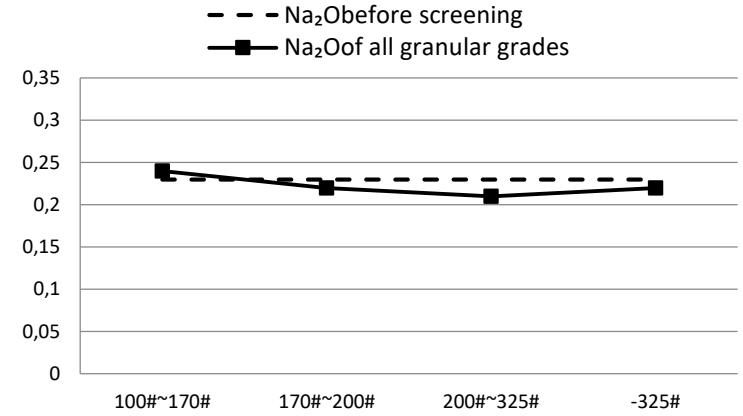

Figure 3. Na_2O content in aluminum hydroxide from different granular grades.

3.3 Analysis of Different Suspended Solids Content from Precipitation Test

Raw materials are shown in table 3 and table 4, and 4-reclycle precipitation test result of different suspended solids are displayed in table 6, with the variation tendency of impurity elements in aluminum hydroxide in the test process being shown in figures 4, 5 and 6. It can be seen that after four cycles, the SiO_2 in aluminum hydroxide without suspended solids has declined from 0.017% to 0.015%, while the variation of Fe_2O_3 is not obvious. However, during the precipitation process of high-suspended solids, product SiO_2 increases rapidly, which can reach 0.020% in the second period and even approach 0.030% towards the end. Finally, during the test process, Fe_2O_3 has arising tendency, but rather slow, with little effect on product quality; however, during the overall test process, Na_2O has generally no change.

Table 6. Chemical component of product with different suspended solids contents during the precipitation process.

Serial number	Suspended solids (g/L)	Product SiO_2 (%)	Product Fe_2O_3 (%)	Product Na_2O (%)
1-1	0.058	0.017	0.013	0.23
1-2	0	0.017	0.013	0.23
2-1	0.066	0.020	0.014	0.24
2-2	0	0.019	0.014	0.24
3-1	0.084	0.025	0.015	0.23
3-2	0	0.017	0.015	0.23
4-1	0.176	0.030	0.018	0.25
4-2	0	0.015	0.015	0.24

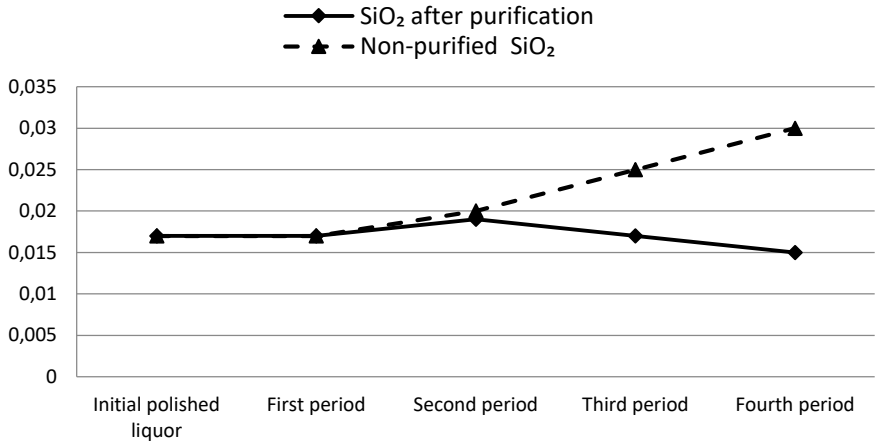

Figure 4. SiO_2 content comparison analysis for products of different suspended solids.

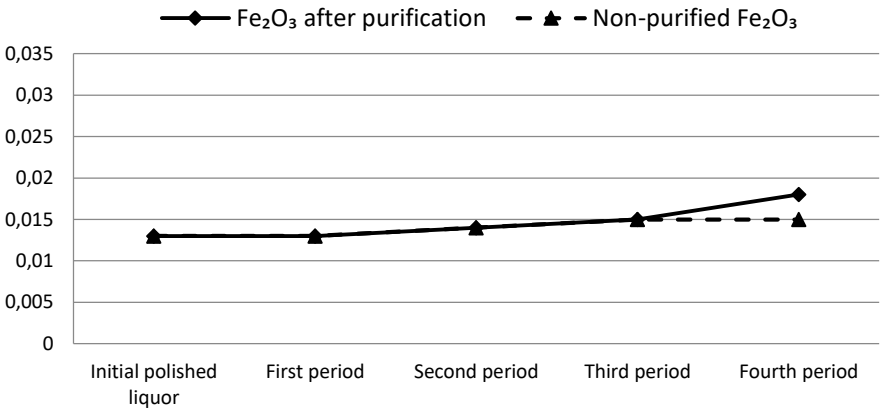

Figure 5. Fe_2O_3 content comparison analysis for products of different suspended solids.

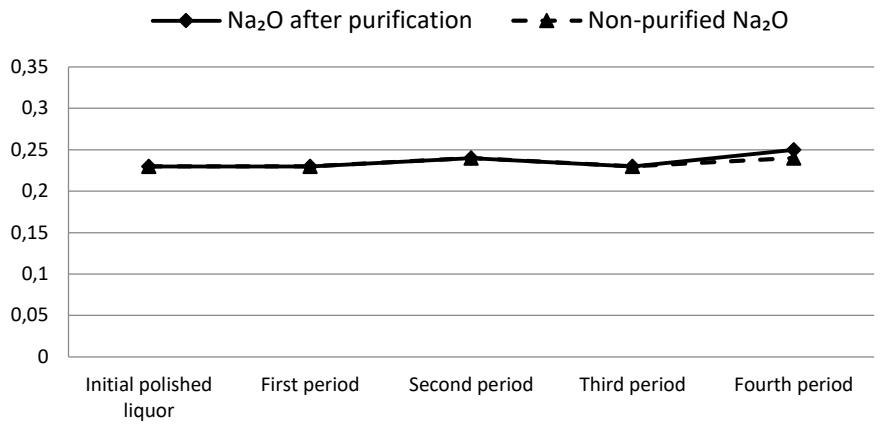

Figure 6. Na_2O content comparison analysis for products of different suspended solids.

4. Conclusions

From the analytical results of chemical component and mineral composition for suspended solids in green liquor, it can be seen that 50 % is fine red mud and the other 50 % is the compound of calcium oxide and alumina. The suspended solids will cause the content of SiO_2 and Fe_2O_3 to raise if it enters the aluminum hydroxide.

From the component analysis of aluminum hydroxide impurities from different granular grades, it can be seen that during the precipitation process, suspended solids in green liquor can directly enter the fine particles of aluminum hydroxide with sodium aluminate solution, which could cause the standard of impurity element content of fine particles in the product to exceed specifications, resulting in the degradation of product quality.

From four-cycle precipitation tests for different suspended solids content, it is demonstrated that suspended solids, will cause a sharp increase of SiO_2 and Fe_2O_3 content in the aluminum hydroxide, however, Na_2O content will show little change.

5. References

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