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Recovery of potassium chloride from blast furnace flue dust

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Abstract: A simple, efficient, economic and environment-friendly recovery process of high-content potassium chloride from blast furnace flue dust (BF flue dust) with abundant potassium content is developed. This process is mainly composed of water-leaching, purification, decolorization, vacuum evaporation and cooling crystallization. In this study, the basic properties of blast furnace flue dust were identified by X-ray diffraction (XRD), inductively coupled plasma analysis (ICP), and laser granulometer (LG). The purity of the KCl products was analyzed by ICP combined with sodium tetraphenylborate (Na-TPB) chemical method and XRD. The particle sizes of the KCl products were characterized by LG and SEM. The results showed that the BF flue dust had a good recovery value with a potassium chloride content of 39.58%. After treated the dust by water-leaching and processed the as-prepared eluent via purification, decolorization and vacuum evaporation, the KCl crystal products were obtained with an yield of 72.77%, 79.52% and 71.09% at 0°C, 5°C and 10°C of cooling crystallization temperature and 2.27, 2.52 and 2.36 of the mass distribution coefficients, respectively. The KCl crystal product exhibited a narrow particle size distribution with a purity of greater than 96.00%.

Key words: BF flue dust; potassium chloride; recovery; characterization; analysis.

1. Introduction

Quite a number of dust and slag are produced as waste materials or byproducts in the iron and steel plant every day^{1, 2}. Since 2013, China has been the one of the biggest crude steel producers in the world with an output exceeding 779 million tons³. It was reported that, in China, for 1 ton of steel production, 20 kg of the BF flue dust would be generated averagely⁴. By calculation, the output of BF flue dust is about 15.58 million tons per year. This BF flue dust is generally dumped into landfills or fields^{5, 6}. As it is well known to all, the BF flue dust contains abundant alkali-metal elements such as potassium and sodium and various toxic

35 heavy-metal elements such as lead, zinc, manganese and copper⁷⁻⁹. Meanwhile, this dust also
36 contains a small amounts of cyanide generated from the coal or the coke which is used as the
37 BF production raw material^{8,10}. Therefore, the traditional dumped-treatment method of the
38 BF flue dust not only can cause environmental pollution and do harm to human beings¹¹⁻¹⁴, but
39 also can lead to the waste of the valuable elements or the recoverable resources. Hence, the
40 dust must be turned into environment-friendly before discharged. In addition, recovering these
41 valuable resources from the BF flue dust can make huge economic benefits.

42 Meanwhile, as the world's second largest consumer, China is extremely dependent on the
43 import of potassium products from international market due to its lacking in potassium
44 resources¹⁵⁻¹⁷. Thus, the recovering of the secondary potassium resources will relieve the
45 contradiction of potassium products supply-demand currently in china and promote the
46 national sustainable development.

47 It has been well demonstrated to be feasible to recycle some metal elements through physical
48 or chemical mineral processing techniques such as hydrocyclonation, magnetic separation,
49 grinding, chemical-leaching, floatation, and high-temperature roasting². So far, several
50 effective ways have been developed to recover potassium chloride¹⁸, mainly including the
51 cold-decomposition and floatation processes, the reverse flotation and cold crystallization
52 process, the hot-melt crystallization and the dilution-cooling and reaction-extraction coupling
53 crystallization processes. For instance, J. Chang¹⁹ reported a method for the preparation of
54 potassium sulphate from the discharged sintering dust. The process was as follows. Firstly,
55 ammonium bicarbonate was used to separate impurity ions from sintering dust such as Ca^{2+} ,
56 Mg^{2+} , Cu^{2+} , Pb^{2+} , Zn^{2+} . Then, ammonium sulphate was added into solution to carry out a
57 double decomposition reaction after decolorization by activated carbon. Finally, industrial and
58 agricultural fertilizer product grade of potassium sulphate and agricultural combined fertilizer
59 $(\text{K}, \text{NH}_4)\text{Cl}$ were acquired by the procedure of concentration and crystallization. Z. Shen *et*
60 *al*²⁰ also reported a method for the recovery of potassium chloride product from sintering dust
61 with a purity of 61.03%. However, the wider applications of these products are limited by the
62 not high enough content or the purity of potassium in the products. Moreover, potassium
63 chloride with the higher content or the purity of potassium has a much more wide range of
64 application than potassium sulphate²¹, such as in the production of basic or additional fertilizer
65 for some crops in agriculture²², medical adhibition and as a raw material of diverse
66 non-chlorine potassium fertilizers industry. For these reasons, the recovered potassium
67 product from the BF flue dust or sintering dust is usually in the form of potassium chloride
68 other than potassium sulphate.

69 Because the BF flue dust usually contains a little amount of cyanide which generated from the
 70 coal or the coke in the raw materials in the iron and steel metallurgic process^{8,10}, it will
 71 deteriorate the quality of potassium chloride. Hence, it is significant to remove the cyanide
 72 compounds from the KCl crystal product to meet with the quality standard regulated by
 73 China²³. It was reported that cyanide could be effectively removed from aqueous solution by
 74 the adsorption of impregnated activated carbons with silver and nickel distributed on their
 75 surface²⁴. However, compared with chemical purification, the adsorption purification of
 76 cyanide by activated carbon is of high cost and a little complicated in operation. Thus, it is
 77 necessary to develop an efficient chemical purification method for the removal of cyanide
 78 from potassium chloride to obtain a high purity of the KCl product.

79 In this paper, a novel and simple process was investigated to recover potassium chloride with
 80 high purity and content by using the BF flue dust from Tangshan Iron & Steel Corporation of
 81 China as raw material. The basic properties of the BF flue dust and the purity of the
 82 recovered product of potassium chloride were analyzed and characterized.

83

84 2. Experimental

85 2.1 Experimental reagents and apparatus

86 The main experimental reagents and apparatus used in the study were listed in table 1 and
 87 table 2, respectively.

88 Table 1 the main experimental reagents used in the research

Reagent	Specification	Manufacturer
Hydrochloric acid	AR	Hunan Huihong reagent Co., Ltd., China
Sulfuric acid	AR	Hunan Huihong reagent Co., Ltd., China
Sodium carbonate	AR	Changsha Xiangke fine chemical plant, China
EDTA	AR	Tianjin Hengxing Chemical Preparation Co., Ltd., China
Sodium hydroxide	AR	Changsha Xiangke fine chemical plant, China
Potassium dichromate	AR	Tianjin Fengchuan Chemical Reagent Co., Ltd., China
Na-TPB or K-TPB	AR	Shanghai Shanpu Chemical Co., Ltd., China
Magnesium chloride hexahydrate	AR	Tianjin Kermel Chemical Reagent Co., Ltd., China
phenolphthalein	AR	Foshan Chemical Demonstration Plant, China
Ferrous chloride	AR	Tianjin Guangfu Fine Chemical Research

 Institute, China

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90

Table 2 the main experimental apparatus used in the research

Apparatus	Specification	Manufacturer
Magnetism Msier	D-971	Zheng Zhou Great Wall Scientific Industry And Trade Co.,Ltd, China
Electronic Scales	ALC-2100.1	BSISL, Switzerland
Water-circulation	SHB-IIIA	Zheng Zhou Great Wall Scientific Industry And Trade Co.,Ltd. , China
Multifunction vacuum Pump		
Three-column Centrifuge	SS600mm	Zhangjiagang Juda centrifuge manufacturing plant, China
Vacuum rotatory evaporator	RE-2000A	Shanghai Yarong Biochemistry Equipment Apparatus Co.,Ltd. , China
Thermostatic cooling tank	HC2010	Chongqing Sida Experimental Instrument Co., Ltd. , China
ICP	Perkin-Elmer OPTIMA 3000	Rhys Scientific Ltd. , USA
XRD	D/Max2550-18KW	Rigaku, Japan
Drying oven	101-2AB	Tianjin Taisite Instrument Co.,Ltd. , China
SEM	JEOL 7500F	JEOL, Japan
LG	Mastersizer 2000	Mastersizer, UK

91 2.2 Physicochemical characterization of the BF flue dust

92 The samples of the BF flue dust from Tangshan Iron & Steel Corporation were used as raw
 93 material in the recovery of potassium chloride. The structural characterization of the BF flue
 94 dust was performed by XRD, and the patterns were analyzed using Search-match software.
 95 Particle size distributions of the BF flue dust and the product of potassium chloride were
 96 measured with a laser granulometer (LG). The contents of the elements of Zn, Fe, Pb, Cu, Na,
 97 Ca and Mg in the dust samples were analyzed by inductively coupled plasma (ICP), the
 98 detection of total-cyanide was identified by China standard method (GB 7487-1987), and the
 99 content of potassium chloride was tested by sodium tetraphenylborate (Na-TPB) chemical
 100 method.

101 2.3 The fundamental of the recovery flow of potassium chloride

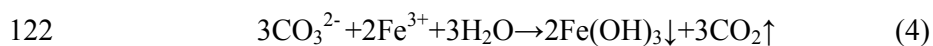
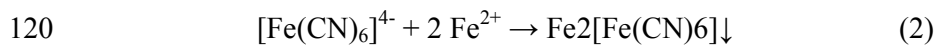
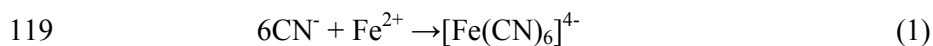
102 As we know, the chloride compounds of potassium, sodium, calcium and magnesium can
103 easily be dissolved in water and the carbonate compounds of Ca, Mg, Fe, Pb and Cu are
104 insoluble in water. Therefore, water-leaching can be used to separate potassium, sodium,
105 calcium and magnesium chloride from the metal elements of Fe, Pb and Cu.

106 In addition, it is found that both ferro- and ferric-cyanide complexes are insoluble in water and
107 exhibit extreme stability under most environmental conditions^{25,26}. Hence, this property can be
108 used to eliminate cyanide effectively from the leached aqueous solution by adding ferrous
109 chloride into the eluent.

110 Furtherly, by chemical precipitation via adding sodium- or potassium-carbonate into the
111 eluent, the elements of Ca, Mg, Fe, Pb and Cu will be effectively separated from sodium and
112 potassium in the eluent. Thus, the eluent will be purified for the impure metal elements
113 except sodium. Meanwhile, by adding activated charcoal into the eluent, the chroma and the
114 trace metal such as iron and copper in the eluent will be removed.

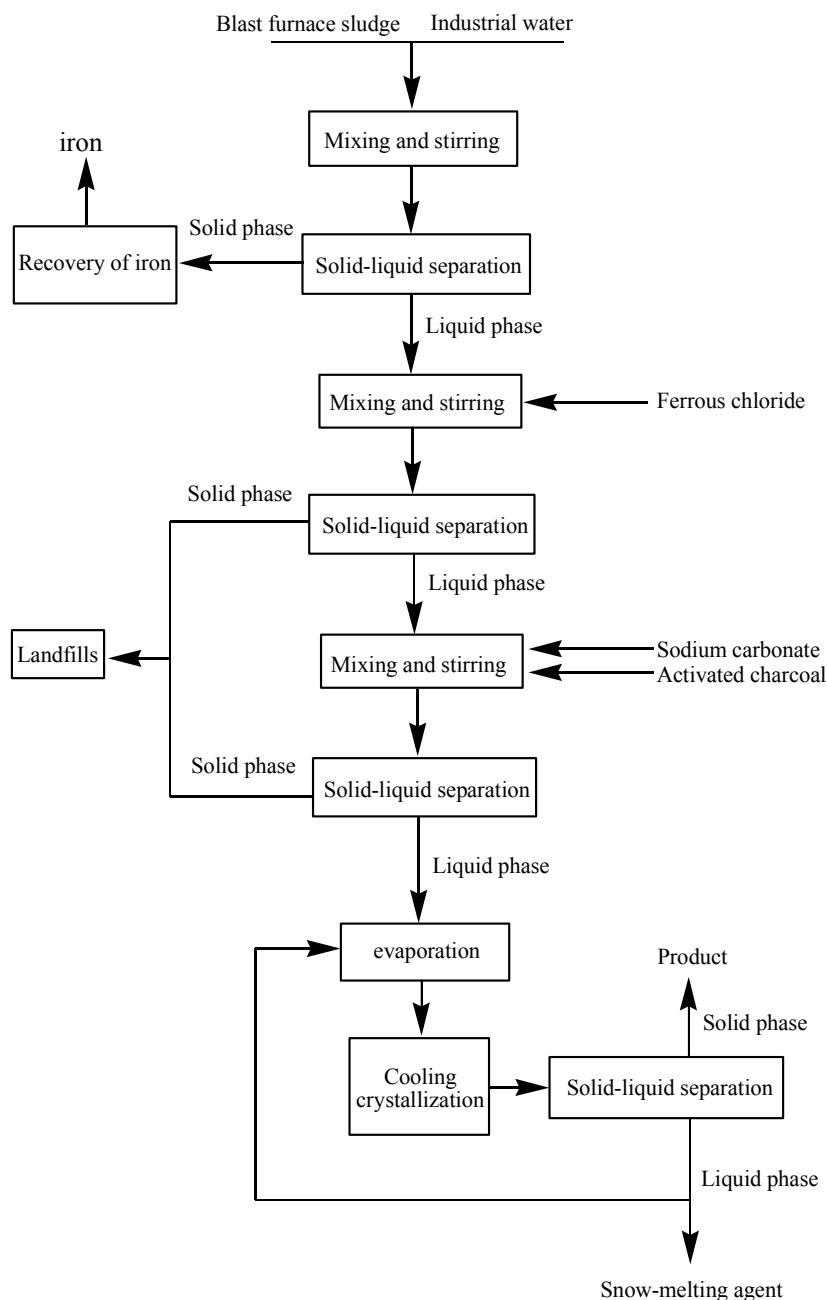
115 Finally, by evaporation and cooling-crystallization, the desired product of potassium chloride
116 will be separated from the sodium chloride in the purified eluent.

117 The chemical reaction equations for the removal of cyanide and the impure metal elements
118 are shown as follows.



123 Where M= Ca, Mg, Fe, Pb, Cu and Zn.

124 According to the above ideologies, a flow chart for the preparation of the potassium chloride
125 product was designed as shown in Figure 1.



126

127

Fig.1 Flow chart of experimental process

128 2.4 Recovery of potassium chloride from the BF flue dust

129 All the reagents used in the experiments were of analytical purity without further purification.

130 On the basis of the previous probing experiments, 2.0 kg of the BF flue dust was mixed with

131 8.0 L of water, stirred with the speed of 200 r/min at room temperature and filtered after 20

132 min. Then, the obtained eluent was treated by ferrous chloride with 2.5 times of theoretical

133 quantity and stirred for 30 min. After filtration, a certain amount of sodium carbonate was

134 added into the eluent until pH was up to 8.0. Then, activated charcoal of 2.5 gram per liter of

135 the eluent was added for decolorization. The purified eluent was filtered after 25-40 min.
136 Finally, 700 mL of the purified eluent (with KCl of 51.80 g/L or 0.65 mol/L, and NaCl of
137 4.02 g/L or 0.069 mol/L) was used for vacuum evaporation by rotary evaporator ^{27, 28} in each
138 run.

139 When a certain concentration of the condensed eluent was reached, the condensed liquor was
140 cooled in thermostatic cooling tank at 0 °C, 5 °C and 10 °C with the stirring speed of 120 r/min
141 for 4 h ^{29,30}, respectively. Then, by filtration, the dried crystal of KCl and the residual mother
142 liquor was analyzed via Na-TPB method to determine the content of potassium chloride.
143 Several runs of the repeat experiments were carried out to ascertain the reliability of the
144 process. The as-prepared products were also identified and characterized by XRD, LG, SEM,
145 and ICP, respectively.

146 **2.5 Characterizations of the KCl product**

147 **2.5.1 XRD Characterization**

148 The structural characterization of the KCl product was performed by XRD. The X-ray
149 patterns of the samples which powdered to 300 mesh were acquired in the 2 θ -range from 10°
150 to 90° with a scan step of 0.05°/2 θ and a fixed counting time of 1 sec for each step. Finally,
151 the patterns were analyzed by Search-match software.

152 **2.5.2 Granulometric analysis**

153 The particle size distributions of the KCl products were measured with a laser granulometer
154 (LG) by dry analysis.

155 **2.5.3 SEM Characterization**

156 The products were examined by a SEM instrument for the granulometric distributions and the
157 particle shape.

158 **2.5.4 ICP analysis**

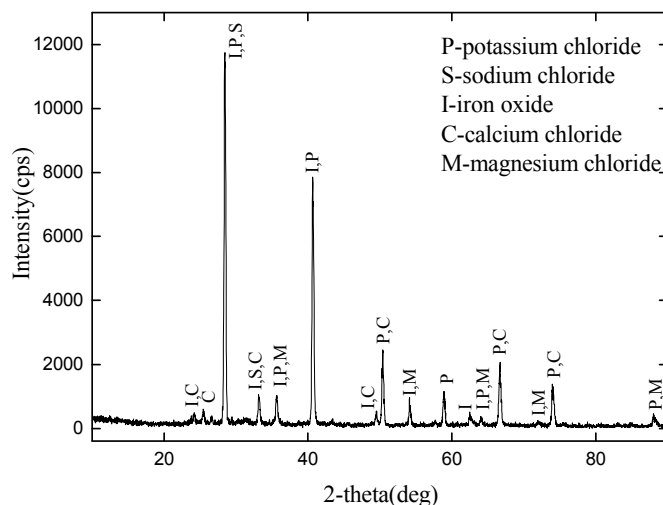
159 The products were analyzed with an ICP instrument to determine their contents and
160 compositions.

161 **3. Results and discussion**

162 **3.1 Physicochemical characterization of the BF flue dust**

163 As shown in Fig.2, the XRD pattern of the BF flue dust sample is presented by five major
164 phases: iron oxide³¹, potassium chloride, sodium chloride, magnesium chloride and calcium
165 chloride. The strong diffraction peaks of potassium chloride illustrate its high content in the BF
166 flue dust. In particular, the characteristic peaks of other potassium-containing compounds have
167 not been discovered, probably due to the different compositions of iron ore. These results are
168 identical with the data obtained by ICP analysis. Thus, potassium chloride is the only form of

169 potassium-containing compounds in the BF flue dust, which provides the theoretical
170 foundation for the further research to recover high content potassium chloride from the BF flue
171 dust.

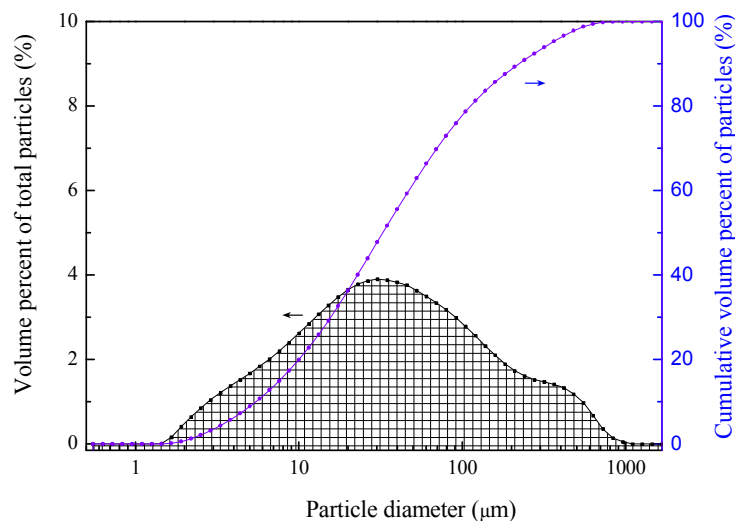


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173

Fig.2 XRD pattern of the BF flue dust

174 The granulometric distribution analysis of the BF flue dust sample is shown as in Fig.3 and
175 Table 3. It exhibits a wide and non-uniform distribution of particle sizes, possibly owing to the
176 capture technology of the BF flue dust in the ferrous metallurgy. The mean particle size P50
177 (the mass percentage of the particles at 50%), P10 and P90 of the BF flue dust is 37.47 μm ,
178 6.27 μm and 254.64 μm , respectively. Combined with the small specific surface area of the
179 dust which shown in Table 3, a conclusion can be draw that such granulometric distribution of
180 the BF flue dust is beneficial to the leaching of soluble potassium compounds without the
181 additional of dispersant or surfactant.



182 Fig.3 Granulometric distribution of the BF flue dust

183 Table 3 The Granulometric characteristic parameters of the BF flue dust

184

Granulometric Characteristic parameter	Value
Median diameter, μm	6.63
Volume mean diameter, μm	91.44
Superficial area mean diameter, μm	16.87
Specific surface area, m^2/g	0.36

185 The element contents of the BF flue dust by chemical analysis combined with ICP method
 186 were demonstrated in Table 4. The results showed that the metal content in the BF flue dust
 187 was 41.00% of Fe, 20.72% of K and 4.39% of Mg, respectively, including a small amount of
 188 Pb(0.65%), Cu(0.12%), Zn(0.03%), Ca(0.51%), Na(0.55%) and Total-cyanide(0.03%). This
 189 result indicates that the BF flue dust is a ferric oxide mixture with a high content of potassium,
 190 implying it is of great potential values of recovering.

191 Table 4 The main metal element constituents of the BF flue dust

Element	TFe	K	Pb	Cu	Zn	Ca	Na	Mg	Total-cyanide
Content, wt%	41.00	20.72	0.65	0.12	0.03	0.51	0.55	4.39	0.03

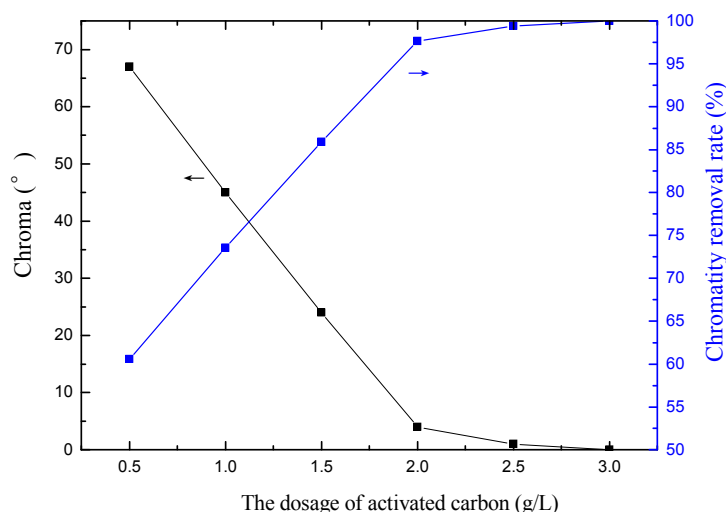
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193 3.2 The effect of operation conditions on the KCl recovery process

194 3.2.1 The effect of the dosage of activated charcoal on decolorization

195 The dosage of activated charcoal is a key factor in the adsorption for the decolorization of the
 196 eluent. The relationship between the dosage of activated charcoal and the chroma of the eluent

197 was studied and the result was shown as in Fig.4. It can be found that the decolorization
198 efficiency becomes better with the increase of the amount of activated carbon. Particularly,
199 when 2.00g /L of activated charcoal is used to dispose the eluent, the chroma of the eluent
200 decreases from 170 °to 4 ° and the removal rate of chroma is up to 97.65%, possibly owing to
201 the increase of active molecular particles in the sorbent. Moreover, when the dosage of
202 activated charcoal is increased to 2.50g /L, the chroma in the eluent reduces to near 0.
203 Therefore, 2.50g/L was chosen as the optimum parameter of the dosage of activated charcoal
204 in decolorization.



205

206

Fig.4 the relationship between the dosage of activated charcoal and the chroma

207

(Experiment conditions: decolorization time=30 min; agitation speed=120 r/min;

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temperature=room temperature)

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3.2.2 The effect of time on decolorization

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Decolorization time is also a significant factor which has an important influence on the

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adsorption effect of activated charcoal. As shown in Fig.5, the chroma decreases with the

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increase of the decolorization time. The chroma of the eluent declines from 170 °to 17° at 25

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min of adsorption time. Correspondingly, the chroma removal rate reaches to 90%. Whereas

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at 35 min of adsorption time, the removal efficiency of the chroma reaches to the highest

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value of 90.59%. Thus, 30 min was chosen as the proper decolorization time from the

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viewpoint of cost and the treatment capacity in industrial practice.

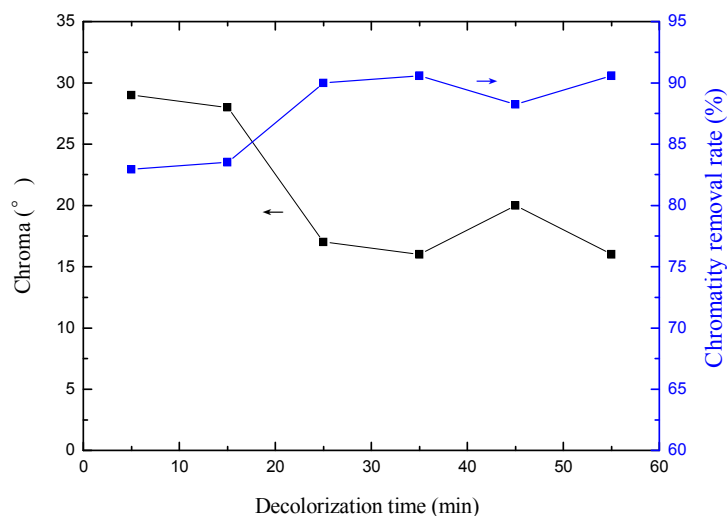
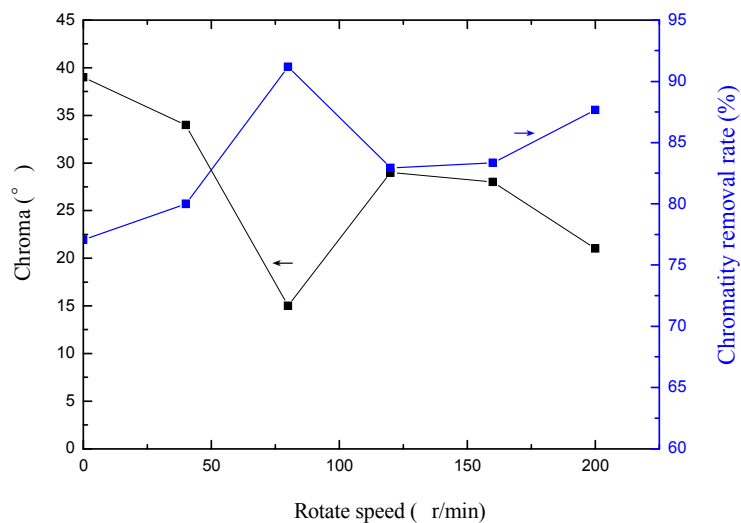


Fig.5 the relation between decolorization time and chroma

(Experiment conditions: dosage of activated charcoal=2.00g/L; agitation speed=100r/min; temperature=room temperature)

3.2.3 The effect of agitation speed on decolorization

The speed of the agitator is a non-ignorable factor which does impact on the dosage of activated charcoal. As shown in Fig.6, the whole tendency of the chroma in the eluent decreases with the increase of the stirring speed of the agitator. The residual chroma in the eluent markedly decreases from 170° to 15° at the stirring speed of 80 r/min and the removal efficiency of the chroma reaches to 91.18%, probably due to the increase of the turbulence and mixing degree. Therefore, 80 r/min was selected as the suitable agitation speed.



228

229 Fig.6 the relation between the stirring speed and the chroma
 230 (Experiment conditions: dosage of activated charcoal=2.00 g/L; decolorization time= 5min;
 231 temperature=room temperature)

232 To validate the reliability of the selected operation conditions for the KCl recovering process,
 233 a further experiment was conducted under the conditions of the dosage of activated charcoal
 234 at 2.50 g/L, decolorization time at 30 min, agitation speed at 80 r/min and the temperature of
 235 25°C. The result shows that the chroma of the eluent decreases from 170 °to 2 ° with a
 236 removal efficiency of 98.82%, indicating that the selected operation conditions is befitting to
 237 purify the chroma of the eluent.

238 After decolorization and purification, the element contents in the purified eluent were
 239 analyzed by ICP with other chemical methods. The results are shown as in Table 5. Table 5
 240 indicates that the purified eluent is mainly composed of K with few of Na, Ca and Mg, and
 241 the elements of Fe, Pb, Cu, Zn and the cyanide (CN) are not detected in the eluent,
 242 manifesting that the impurities are effectively removed by the separation process.

243 Table 5 The main element contents in the purified eluent

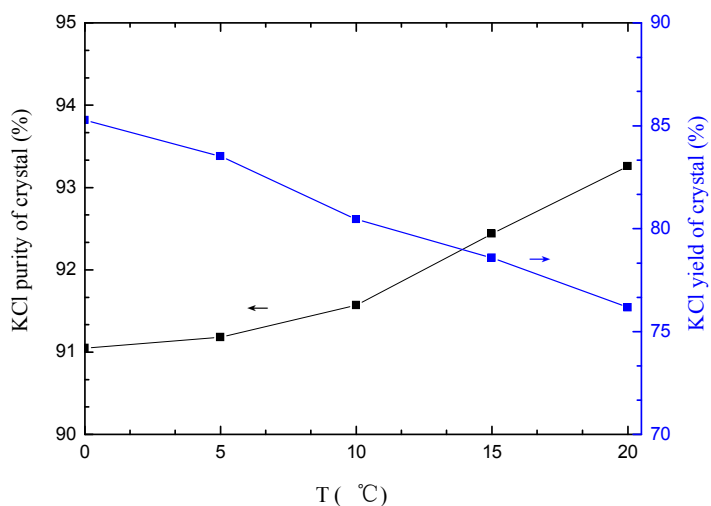
Element	Fe	K	Pb	Cu	Zn	Ca	Na	Mg	CN
Concentration, g/L	Not detected	50.69	Not detected	Not detected	Not detected	0.08	0.03	0.55	Not detected

244

245 3.2.4 The effect of temperature on the KCl crystallization process

246 In the KCl crystallization process, the influences of temperature on the purity and the yield of
 247 the KCl crystal are shown as in Fig.7.

248 Fig.7 reveals that the potassium chloride purity of the crystal increases with the increase of the
 249 cooling crystallization temperature, whereas the yield of the KCl crystal decreases with the
 250 increase of the temperature. Therefore, to get more potassium chloride product from the eluent,
 251 the cooling crystallization temperature should be lower. Contrarily, the cooling crystallization
 252 temperature must be higher for the purpose of obtaining a high content of potassium chloride.
 253 Aimed at getting a higher content of potassium chloride to suffice the quality demand
 254 regulated by the KCl product market, a higher cooling crystallization temperature at the range
 255 of 10°C to 15°C may be of feasibility, but the yield of the KCl product will be sacrificed a
 256 little.



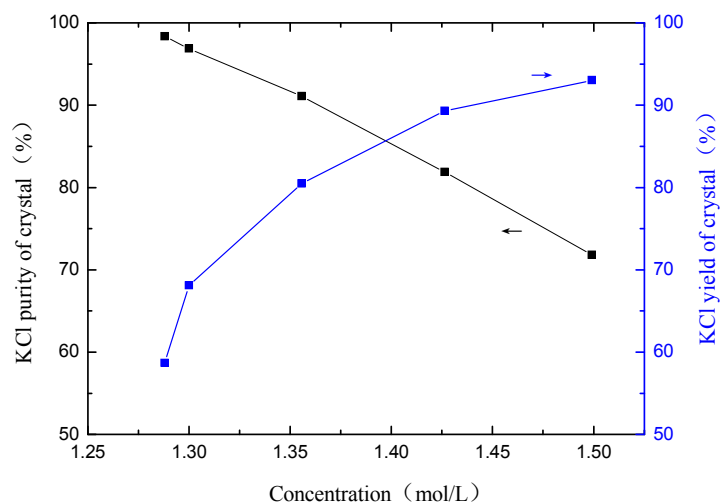
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258 Fig.7 the influence of crystallization temperature on the KCl purity and yield
259 (Experiment conditions: mass distribution coefficient= 2.27; initial KCl crystallization
260 concentration=1.29 mol/L or 96.2 g/L; crystallization time=4.0 h)

261 3.2.5 The effect of KCl crystallization concentration on the crystal recovery

262 The influence of the initial KCl crystallization concentration in the condensed eluent on the
263 purity and the yield of the product crystal are demonstrated as in Fig.8.

264 Fig.8 indicates that, at a certain cooling crystallization temperature, the potassium chloride
265 purity of the product crystal decreases with the increase of the initial crystallization
266 concentration of KCl in the condensed eluent, whereas for the yield, it rises up. This implies
267 that the content of sodium chloride in the crystal product will be increased with the increase
268 of the initial KCl crystallization concentration, which will leads to the deterioration of the
269 crystal product quality.



270

271 Fig.8 the influence of KCl crystallization concentration on the crystal purity and yield

272 (Experiment conditions: mass distribution coefficient= 2.27; crystallization

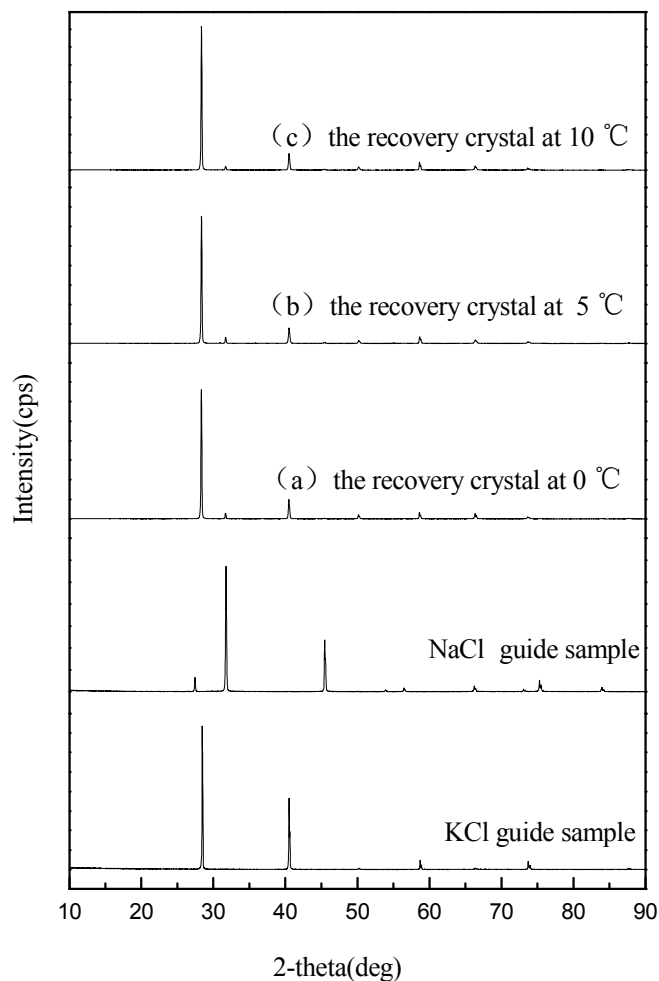
273 temperature=10°C; crystallization time=4.0 h)

274 3.3 Characterizations and Analysis of the KCl product

275 3.3.1 XRD results of the KCl products

276 The potassium chloride crystals obtained at different crystallization temperature with the
277 distribution coefficient (the mass of KCl in the crystal to that in the mother liquor) of 2.27
278 and the initial KCl crystallization concentration of 1.33 mol/L or 99.2 g/L, were characterized
279 by XRD and the results were shown as in Fig.9.

280 Combined with the standard XRD spectrum of potassium chloride and sodium chloride, it can
281 be seen from Fig.9 that the characteristic diffraction peaks at $2\theta=28.38^\circ$, 40.47° , 50.15° ,
282 58.61° , 66.35° , 73.66° and 87.66° are ascribed to potassium chloride. Whereas for the
283 characteristic diffraction peaks at $2\theta = 24.24^\circ$, 31.69° , 45.39° , 66.39° , 75.19° and 83.88° ,
284 they are ascribed to sodium chloride. Hence it can be draw a conclusion that the recovered
285 products contain a great amount of potassium chloride with very few amount of sodium
286 chloride or other impurities.



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Fig.9 XRD patterns of the recovery crystals, NaCl and KCl guide sample

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A further analysis to the recovered crystals was conducted by ICP combined with Na-TPB or K-TPB method and the results are shown as in Table 6. From Table 6, it can be seen that the recovered crystal is mainly composed of potassium chloride (97.08%) with very few amount of sodium chloride(1.78%) and other impurities (0.08% of Ca, 0.04% of Mg) by ICP method. Whereas by chemical analysis method for the same sample, it reveals that the recovered crystal is compose of 96.77% of potassium chloride, 1.91% of sodium chloride, 0.10% of Ca and 0.08% of Mg. Therefore, it can be concluded that the recovery crystal product is of high content and purity of KCl, Na- or K-TPB method is of similar accuracy compared with ICP method, and the quality of the KCl product is satisfactory to the demand of national product standard of China regulated by GB6549-2011.

300

Table 6 The analysis results of the product by ICP and Na-, K-TPB methods

Analysis method	KCl	NaCl	Ca	Mg	Ca+Mg
ICP, %	97.08	1.78	0.08	0.04	0.12*
Chemical Analysis	96.77	1.91	0.10	0.08	0.18*
	(Na-TPB)	(K-TPB)	(ICP)	(ICP)	
GB6549-2011 ²³	≥91.94	≤2.00	≤0.5	≤0.4	≤0.2

301

Where the “*” in Tab.5 represents the value from calculation.

302

3.3.2 Granulometric distributions of the KCl products

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The granulometric distributions of the crystal products obtained at cooling crystallization temperature of 0°C, 5°C and 10°C, correspondingly with the distribution coefficients of 2.27, 2.52 and 2.36 are demonstrated in Fig.10, respectively.

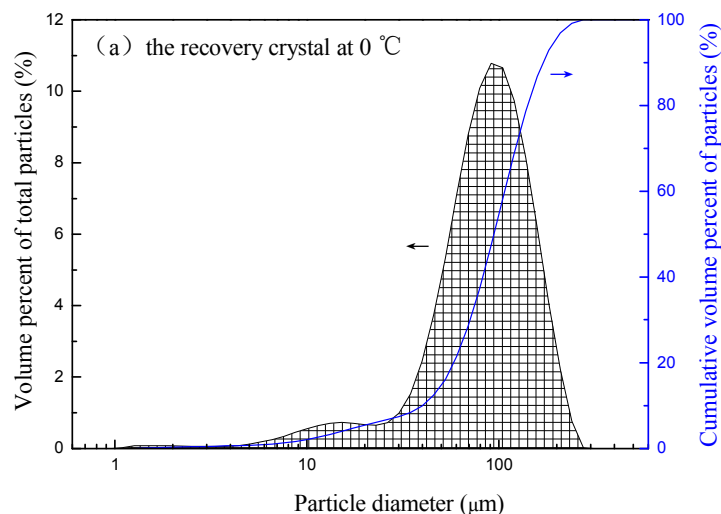
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Fig.10 reveals that the mean particle size of P50, P10 and P90 of the product is 94.29 μm, 39.91 μm and 169.48 μm at cooling crystallization temperature of 0°C, 98.58 μm, 38.46 μm and 178.82 μm at 10°C and 107.74 μm, 44.18 μm and 194.59 μm at 15°C, respectively.

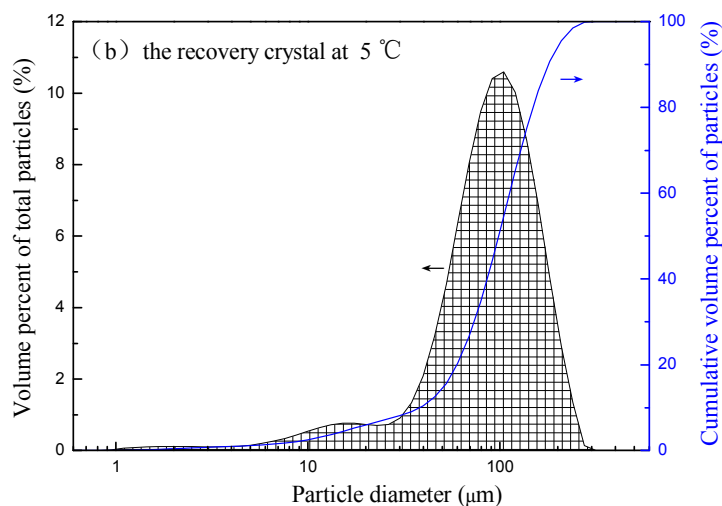
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From Fig.10, it also can be seen that all the particles of the crystals obtained at different cooling crystallization temperature exhibit a narrow size distribution, indicating that the particles have a considerable uniform size and the crystal product is of a good extrinsic quality³².

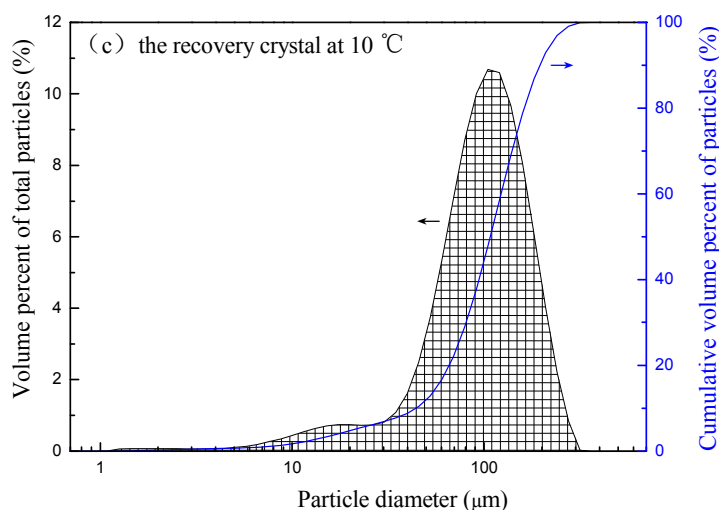
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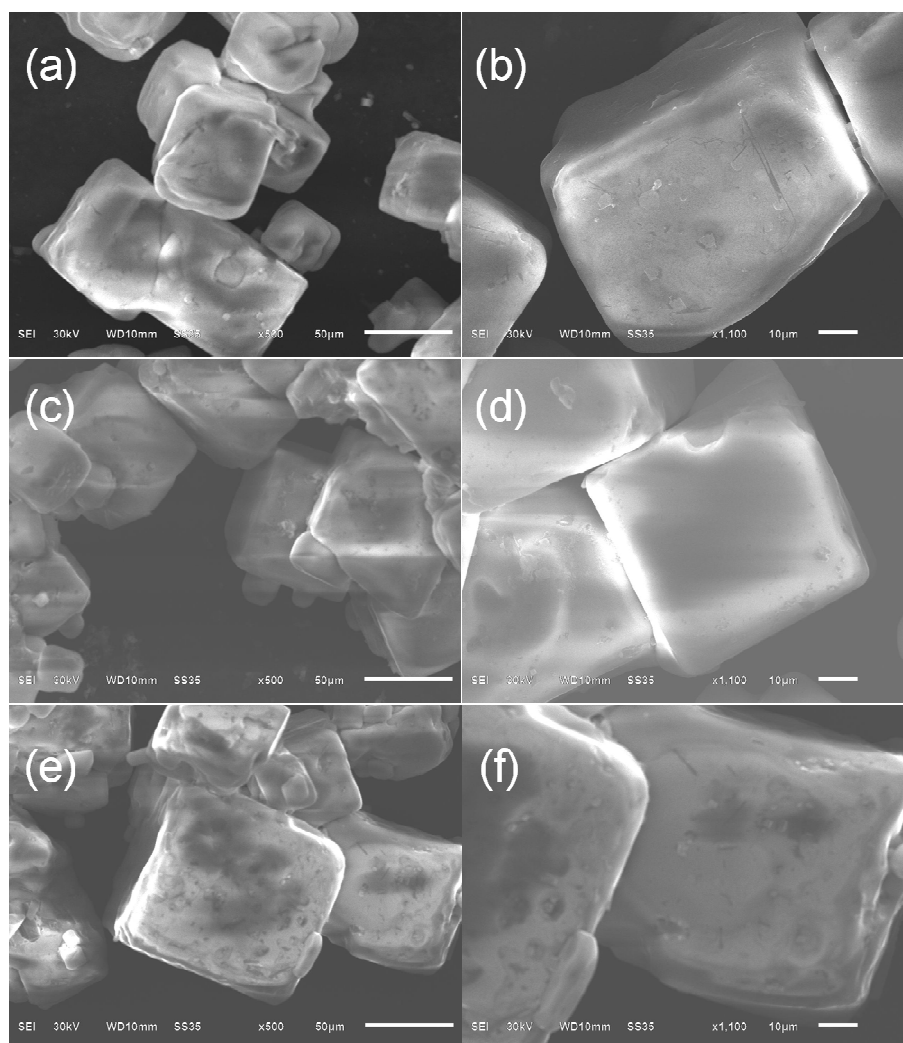
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316 Fig.10 Granulometric distributions of the products at cooling crystallization temperature
 317 of (a) 0□, (b) 5□ and (c) 10□

318 3.3.3 SEM results of the KCl products

319 The SEM diagrams of the products at crystallization temperature of 0□, 5□ and 10□ are
 320 shown as in Fig.11, respectively. The SEM characterization testifies that the principal
 321 crystalline forms of the products at different temperature are all cubic and the size of the
 322 particles is comparatively homogeneous. Interestingly, it can be observed from the figure that
 323 the crystals become larger and larger with the rising of the crystallization temperature. It was
 324 reported that the products with small particle size would be easy to consolidation, which
 325 would affected the stability and quality of the product³³. Therefore, the cubic potassium

326 chloride crystal with a bigger particle size is beneficial to the stabilization of the products³⁴. In
327 conclusion, crystallization process operated at a higher temperature level is of advantages to
328 the apparent quality of the KCl products.



329
330 Fig.15 SEM images of the products at cooling crystallization temperature of (a) and (b): 0°C;
331 (c)-(d): 5°C and (e)-(f):10°C

332 4. Conclusions

333 In brief, a simple, efficient, economic and environment-friendly recovery process of high
334 content potassium chloride from the BF flue dust is developed. This process is mainly
335 composed of water-leaching, decolorization, purification, vacuum evaporation and cooling
336 crystallization.

337 The physicochemical characterization revealed that the BF flue dust contained a high content
338 of potassium chloride which being of recovery value.

339 The effects of the dosage of activated charcoal, the adsorption time and the agitation speed on

340 the decolorization efficiency were investigated. By single-factor experiment, the appropriate
341 operation conditions for the decolorization of the eluent from the water-leaching of the BF
342 flue dust were selected as follows: 2.50 g/L of activated charcoal, 30 min of adsorption time
343 and 80 r/min of stirring speed. Under these conditions, the removal efficiency of the chroma
344 in the eluent was 98.82%. By a further chemical precipitation of sodium carbonate, the
345 impure elements of Ca, Mg, Fe, Pb and Cu in the eluent were effectively separated from K
346 and Na.

347 In the KCl crystallization process, the influence of temperature and the initial KCl
348 crystallization concentration of the condensed eluent on the purity and the yield of the KCl
349 crystal product were studied. The results showed that the cooling crystallization temperature
350 at 10°C to 15°C was of feasibility, and the purity of the KCl crystal product decreased whereas
351 the yield increased with the increase of the initial crystallization concentration of the KCl in
352 the condensed eluent.

353 The KCl crystal products obtained at different cooling crystallization temperatures and mass
354 distribution coefficients were analyzed by ICP or chemical methods and characterized by
355 XRD, SEM and LG, respectively. The qualitative XRD combined with the quantitative ICP or
356 chemical analysis results showed that, the recovered KCl products contained a great amount
357 of potassium chloride with a purity of greater than 96.00% , and very few amount of sodium
358 chloride or other impurities were detected in the products, implying that it is satisfactory to
359 the national product standard of China regulated by GB6549-2011. The SEM and LG analysis
360 revealed that the KCl crystal product exhibited a narrow particle size distribution with P50,
361 P10 and P90 of 94.29-107.74 μm , 38.46-44.18 μm and 169.48-194.59 μm , respectively,
362 indicating that the KCl crystal products have a good extrinsic quality, and crystallization
363 process operated at a higher temperature level is beneficial to the stabilization and the
364 apparent quality of the products.

365

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