Flotation separation of galena from sphalerite using hyaluronic acid (HA) as an environmental-friendly sphalerite depressant

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The application of poly (sodium 4-styrene sulfonate) as an efficient dolomite depressant in the direct flotation of apatite from dolomite

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Abstract

Poly (sodium 4-styrene sulfonate) was tested as a novel and high-efficiency depressant for the selective flotation of apatite and dolomite in this work. The separation performances were evaluated via Micro-flotation tests and the interaction mechanisms were uncovered. Flotation results illustrated that PSS greatly depressed dolomite, while it hardly affected the flotation of apatite. Zeta potential and Fourier Transform Infrared Spectrometer results indicated the adsorption of PSS on dolomite surface was more favorable than that on apatite surface, which impeded sodium oleate to adsorb on dolomite surface and rendered it hydrophilic. XPS results indicated that $-SO_3^-$ groups of PSS strongly complexed with both Ca^{2+} and Mg^{2+} sites on the dolomite surface, but PSS weakly interacted with apatite surface and hardly affected the adsorption of NaOL on apatite surface.

Keywords: Dolomite; Apatite; Depressant; Flotation; Ploy (sodium 4-styrene sulfonate)

1. Introduction

Phosphate ores are indispensable and non-renewable strategic resources, which are extensively utilized in fertilizer, chemical engineering, food industry, and national defense [1, 2]. With the exhaustion of rich phosphate ores, it is urgent to enrich the low-grade phosphate ores to meet the great demand in markets [3, 4]. As the major source of phosphorus, apatite is commonly associated with dolomite and other gangues [5]. The existence of dolomite not only degrades the quality of phosphate products but also increases the reagent consumption in the production of wet phosphoric acid [6]. Therefore, it's critical to remove dolomite from apatite efficiently.

Flotation is considered as the most effective approach to beneficiate apatite from dolomite. However, the efficient separation of these two ores is difficult due to the similar surface characteristics and the interference of inviolable ions [7]. Thus, to beneficiate apatite from dolomite efficiently, more selective flotation reagents such as collectors or depressants are indispensable. In common practice, sulfuric acid or phosphoric acid is generally used to selectively depress apatite during the reverse flotation of apatite from dolomite under acidic conditions [8, 9]. But these techniques suffer from lots of disadvantages like serious environmental pollution and corrosion of equipment [10]. On the other hand, fatty acid-based collectors have been extensively applied in phosphate flotation due to their low price and availability [11, 12], but it's

65 difficult to separate these two minerals because of the poor selectivity of fatty acid-
66 based collectors without depressants.

67   In the past years, considerable investigations had been dedicated to developing
68 novel dolomite depressants. It has been reported that HPMA could selectively depress
69 dolomite but hardly affected the floatability of apatite due to the stronger chelation
70 ability of hydrolytic polymaleic anhydride (HPMA) with Mg^{2+} than Ca^{2+} atoms [13].
71 Yang revealed that the carboxyl groups within poly (acrylic acid-co-maleic acid) were
72 able to coordinate with Mg sites on dolomite, resulting in strong depression effect on
73 dolomite [14]. Zhong pointed out that strong chemical chelating between carboxyl,
74 hydroxyl groups of sodium alginate and Ca sites on dolomite surface caused the
75 depression of dolomite in phosphate flotation process [15]. Adenosine 5'-triphosphate
76 disodium salt (Na_2ATP) was tested as a dolomite depressant in the flotation of dolomite
77 and magnesite. The higher affinity of Na_2ATP toward Ca sites and more exposed
78 calcium atoms on dolomite surface were responsible for the selective depression [16].
79 Li noted that cassava starch (CS) could preferentially adsorb on dolomite surface by
80 means of physical adsorption, which lead to satisfactory separation of dolomite from
81 fluorite [17]. These novel depressants exhibit high selectivity, but they are not applied
82 due to the comprehensive consideration of toxicity, practicality, and cost. Therefore,
83 the development of environment-friendly, low-toxic and selective depressants that
84 could enlarge the floatability difference between dolomite and apatite is still a great
85 challenge.

86   Ploy (sodium 4-styrene sulfonate) (PSS) is a water-soluble polymer with unique
87 effects due to its great water-solubility and flocculation properties (Fig. 1) [18]. PSS is
88 often widely used as a reactive emulsifier in cosmetics as well as water treatment
89 reagents [19], and it has relatively wide applications in sulfur exchange resins
90 (membranes), semiconductors, and heat transfer products [20]. It is well known that -
91 SO_3^- preferentially complexes with Ca^{2+} and Mg^{2+} based on Hard-Soft-Acid-Base
92 theory, but due to the activity discrepancy of metal irons (Ca^{2+} and Mg^{2+}) between
93 apatite and dolomite [21], the adsorption ability of PSS on apatite and dolomite surface
94 may distinguish from each other. Moreover, PSS had very low toxicity compared with
95 other sulfonic acid systems, which could reduce the hazardous waste generated in the
96 flotation process.

97   PSS was tested as dolomite depressant for the direct flotation separation of apatite
98 from dolomite. The flotation performances of PSS on dolomite and apatite were verified
99 via micro-flotation tests. Zeta potential, FTIR, and XPS tests were also performed to

100 uncover the interaction mechanism. The aim of this work was to exploit a novel
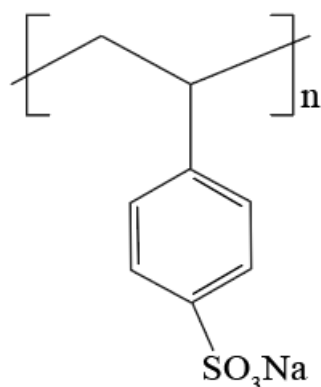101 dolomite depressant to separate apatite from dolomite in direct flotation.

102
103 **Fig. 1.** The moleculare structure of PSS.

2. Experimental

2.1 Materials and reagents

106   In this work, pure apatite and dolomite were acquired from Shanxi Province. After
107 hand-picking, crushing, grinding, and sieving, the apatite and dolomite minerals were
108 sorted into different size fractions. The coarse fraction ($-75 + 38$ μm) was used for
109 micro-flotation tests, while the fine fraction (-10 μm) was applied for Zeta potential,
110 FTIR and XPS measurements. XRD results indicated there were no obvious impurities
111 in these samples (Fig. 2). Chemical assay results suggested the grade of P_2O_5 and MgO
112 in apatite and dolomite was 38.33% and 20.97%, respectively.

113   Analytical grade Poly (sodium 4-styrene sulfonate) (PSS) and sodium oleate
114 (NaOL) purchased from Aladdin were applied as depressant and collector in flotation,
115 respectively. The average molecular weight of PSS was 70000. Analytical grade
116 hydrochloric acid and sodium hydroxide were utilized as pH regulators. Ultra-pure
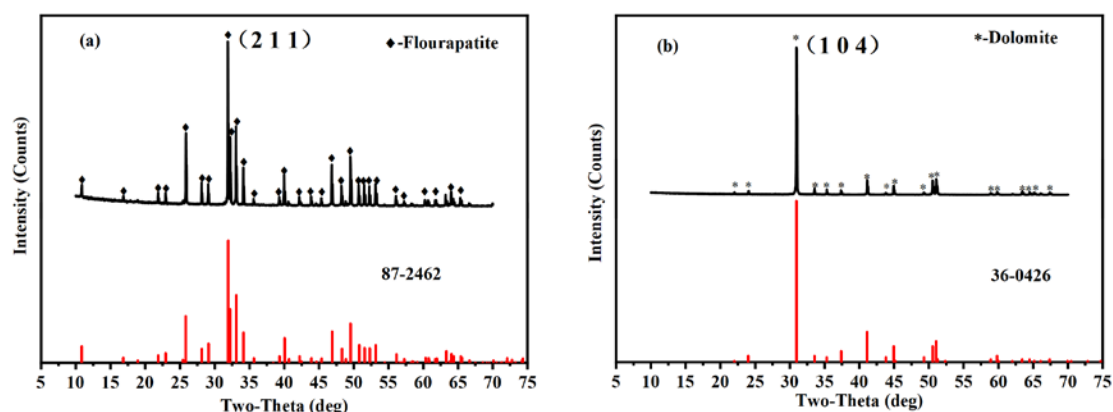117 water was applied in this work.

Fig. 2. XRD results of fluorapatite (a) and dolomite (b).

2.2 Micro-flotation tests

The XFG flotation machine was utilized for micro-flotation tests. 2 g samples were dispersed in 50 mL water homogenously first, then the pulp was transferred into flotation cell and conditioned for 2 minutes. After the adjustment of pH, PSS (200mg/L) and NaOL (3×10^{-4} mol/L) were introduced into the suspension sequentially at the interval of 3 minutes. Then the pulp was aerated for 3 minutes. Finally, the flotation products were filtered, dried, and weighted to calculate floatability.

The mixed mineral samples contained 0.4 g dolomite and 1.6 g apatite were utilized for the flotation test of artificially-mixed minerals. The tests were performed as the same processes mentioned above.

2.3 Zeta potential measurement

Zeta potential was measured using Nano ZS90 (Malvern, England). 0.2 g samples were added into 100ml 0.001M NaCl solution (background electrolyte) and dispersed for 2 minutes. After the adjustment of pH, PSS or sodium oleate were introduced and stirred for three minutes. Finally, 1 mL of supernatant was taken to determine Zeta potential at room temperature.

2.4 FTIR tests

The FTIR spectrum of dolomite and apatite treated or untreated were acquired using Nicolet 6700 (USA). 0.2 g dolomite or apatite was conditioned in various solutions for 15 minutes, the samples were filtered and dried for 24 h. Finally, the mixture of 100 mg KBr and 1 mg sample was measured.

2.5 XPS analysis

AXIS SUPRA (Shimadzu Corporation, Japan) was utilized to obtain the XPS spectrum. Dolomite or apatite was treated in different solutions (pH=10, PSS=200mg/L) for 30 minutes first, then the samples were freeze-dried in the vacuum oven. The collected data were fitted with Avantage software. The binding energy of C 1s was calibrated at 284.80 eV.

3. Results and discussions

3.1 Single mineral flotation

The flotation results of dolomite and apatite at various PSS concentrations are displayed in Fig. 3. Both apatite and dolomite exhibited good floatability without PSS, suggesting it was hard to separate them without depressant. When PSS was introduced, the floatability of dolomite dropped dramatically as PSS concentration increased, while the floatability of apatite was hardly influenced. At PSS concentration of 20mg/L, the floatability difference was expanded to 85%, indicating that PSS could be a potential dolomite depressant that could selectively depress dolomite in the direct flotation of apatite from dolomite.
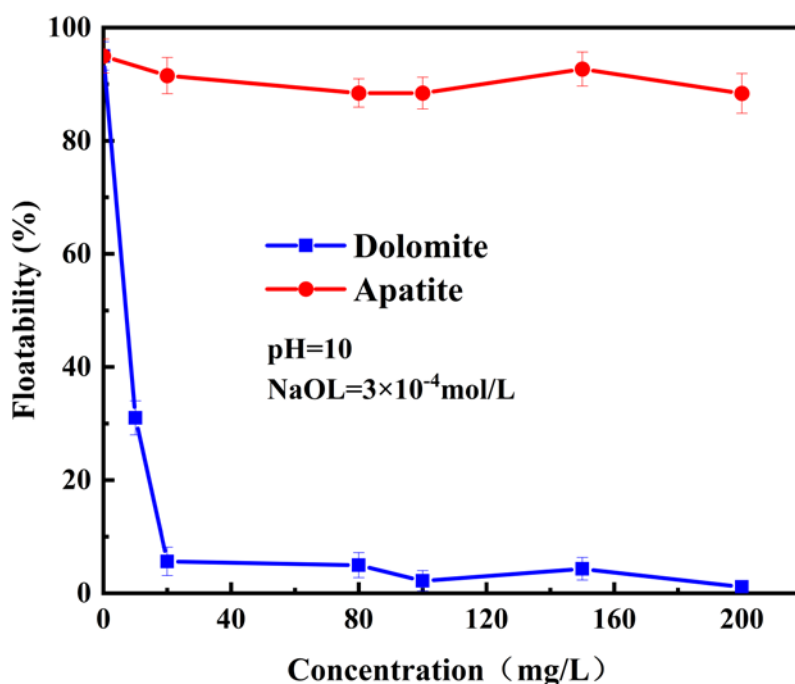

Fig. 3. The floatability of dolomite and apatite at various PSS concentration.

160   Fig. 4 shows the floatability of dolomite and apatite at various pH. PSS depressed

161   dolomite powerfully at broad range (pH 7-12), but barely affected apatite. The

162   floatability difference was enlarged to approximately 80%, which reconfirmed that PSS

163   was an efficient dolomite depressant with good selectivity. Phosphoric acid and sulfuric

164   acid was commonly used as apatite depressant in the reverse flotation of apatite from

165   dolomite However, the application of these reagents was limited at pH 4-5. Moreover,

166   phosphoric and sulfuric acid resulted in many problems in phosphate flotation, such as

167   water eutrophication, corrosion of flotation equipment and waste of collector [22]. Thus,

168   PSS may be a promising dolomite depressant to realize the direct flotation of apatite

169   from dolomite owing to its excellent selectivity and compatibility with fatty acid
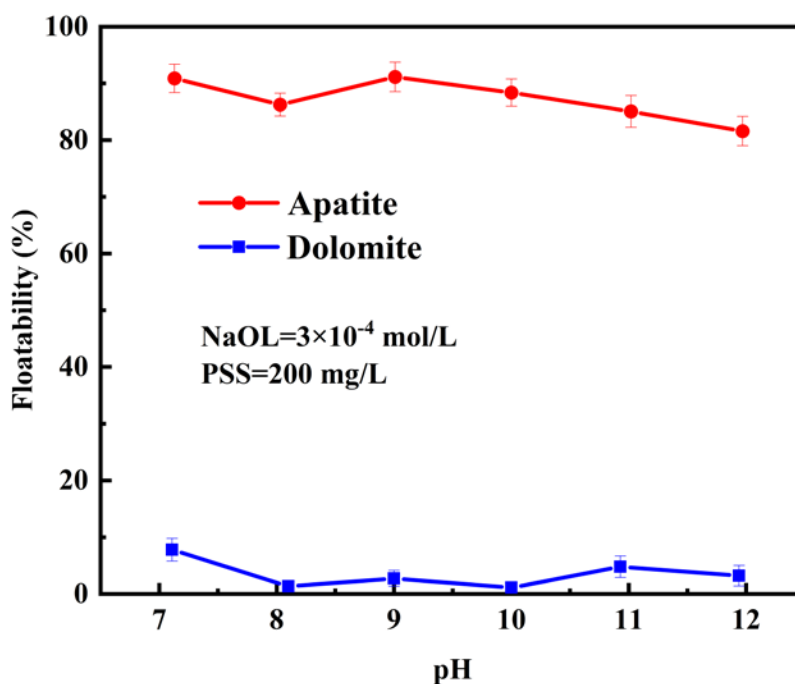
170   collectors.

171

172   **Fig. 4.** The floatability of dolomite and apatite at various pH.

173

174   Fig. 5 illustrates the floatability of dolomite and apatite as affected by NaOL

175   concentration. It could be observed that NaOL didn't influence the selective depression

176   ability of PSS on dolomite at NaOL concentration less than 3×10^{-4} mol/L, suggesting

177   that PSS had good compatibility with fatty acid collectors.
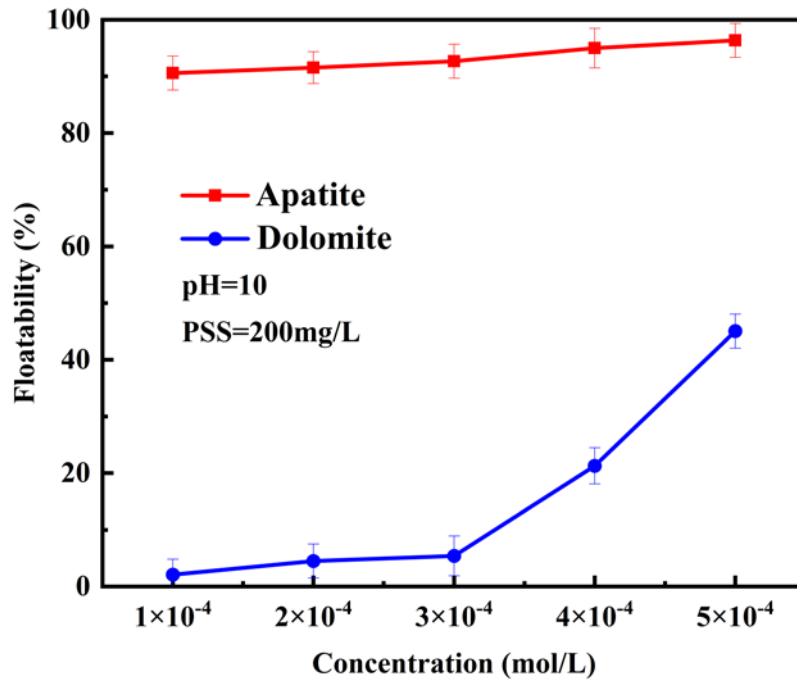
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179   **Fig. 5.** The floatability of dolomite and apatite as a function of NaOL concentration.

180   ## 3.2 Artificially-mixed minerals flotation

181   Artificially mixed minerals flotation tests were also performed to further verifying

182   the selectivity of PSS (Fig. 6). Without PSS, the separation of these two minerals could

183   not be realized. The MgO recovery in concentrate decreased significantly with the

184   increase of PSS concentration, while the recovery of P_2O_5 kept around 90%. Moreover,

185   MgO grade dropped from 4.07% to 0.81% in apatite concentrate, while the P_2O_5 grade

186   in apatite concentrate increased from 30.52% to 35.55%. These results proved that PSS

187   could be a good alternative for traditional apatite depressants to separate apatite and
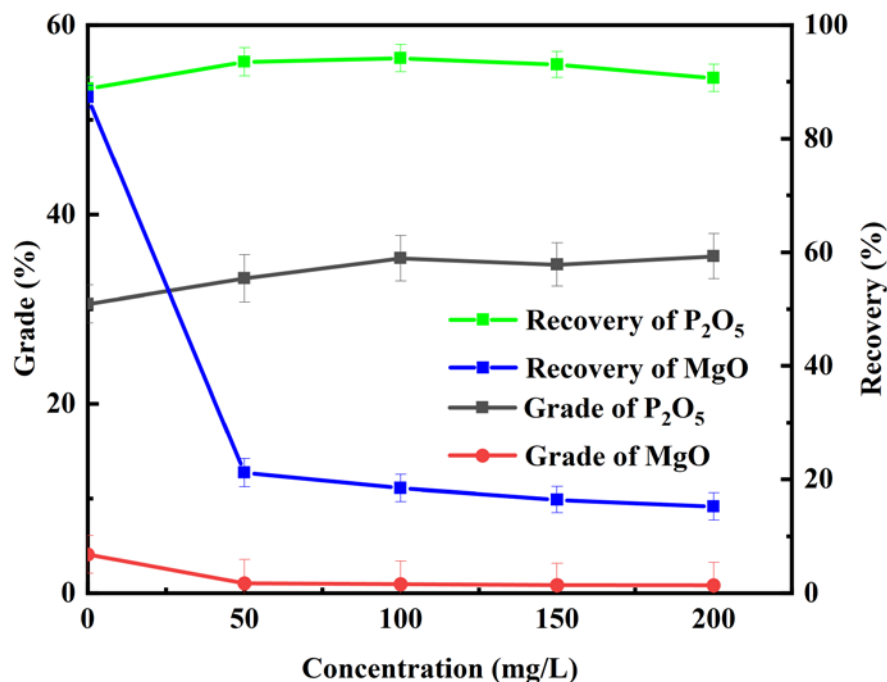
188   dolomite in direct flotation.

189

Fig. 6. The recovery and grade results of artificially-mixed minerals as a function of PSS concentration (NaOL 3×10^{-4}mol/L and pH 10).

3.3 Zeta potential results

Fig. 7 represents the zeta potential of dolomite and apatite under various conditions. When PSS or NaOL were introduced into the dolomite slurry alone, zeta potential moved to the negative direction obviously, which illustrated that both reagents could adsorb on dolomite surface individually (Fig. 7 (a)). When both PSS and NaOL were added, Zeta potential almost coincided with that where PSS was introduced alone, illustrating that the addition of sodium oleate hardly influenced the adsorption of PSS on dolomite surface.

For apatite, zeta potential dropped obviously when PSS or NaOL was added alone, which demonstrated that both reagents could adsorb on apatite surface separately (Fig. 7 (b)). When apatite was treated with both NaOL and PSS, zeta potential results were similar to that of NaOL-treated, illustrating that pre-adsorbed PSS barely influenced the adsorption of NaOL on apatite. In other words, these results suggested that sodium oleate could substitute pre-adsorbed PSS on apatite while it couldn't replace that from dolomite surface, which provided a good explanation for the selective depression effect on dolomite.
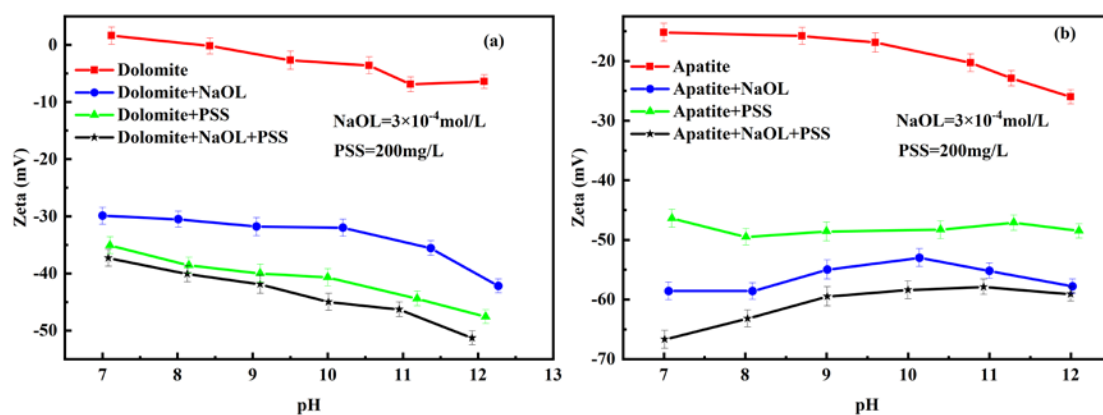
208

209 **Fig. 7.** Zeta potential results of dolomite (a) and apatite (b) at various pH.

210 **3.4 FTIR results**

211   Fig. 8 shows the FTIR spectra of dolomite and apatite. Fig. 8 (a) displayed the

212 spectra of dolomite conditioned with sodium oleate, the peaks located at 2922 cm^{-1} and

213 2848 cm^{-1} were ascribed to -CH$_2$- and -CH$_3$ groups originated from NaOL [23].

214 However, the characteristic peaks of NaOL disappeared after the treatment with NaOL

215 and PSS. Thus, it could be inferred that PSS impeded NaOL to adsorb on dolomite

216 surface, which coincided well with zeta potential results.

217   Fig. 8 (b) illustrated the spectra of apatite treated with various reagents. The

218 characteristic peaks related to NaOL appeared after the treatment with NaOL alone.

219 After being treated with both PSS and NaOL, the peaks corresponding to NaOL still

220 existed at 2922 cm^{-1} and 2852 cm^{-1}, illustrating that PSS hardly affected the adsorption

221 of NaOL on apatite.

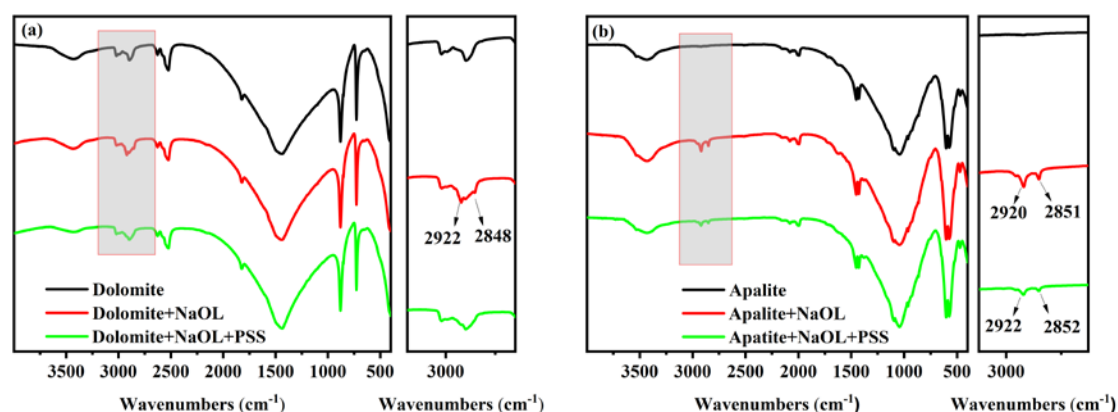
222

223   **Fig. 8** IR spectrum of dolomite (a) and apatite (b) under various conditions (PSS=200

224   mg/L, NaOL=3×10^{-4} mol/L).

3.5 XPS results

Fig. 9 describes the Mg 1s, Ca 2p and S 2p XPS spectra of dolomite surface. The peaks located at 350.80eV and 347.17 eV were described as Ca $2p_{1/2}$ and Ca $2p_{3/2}$ in dolomite (Fig. 9 (a)). After being treated with PSS, the bimodal peaks shifted by 0.20 eV to 351.00 eV and 347.37 eV, which indicated that Ca sites on dolomite might involve in the chemical interaction with PSS [24]. In Fig. 9 (b), the peak belonged to Mg 1s decreased from 1304.05 eV to 1303.85 eV after the introduction of PSS, which meant that Mg sites on the dolomite also underwent a stronger chemical reaction with PSS [25].

For pure PSS, the peaks appeared at 169.62 eV and 168.42 eV were assigned to -SO_3^- groups in PSS (Fig. 9 (c)). When pure dolomite was treated with PSS, the characteristic peaks of SO_3^- groups reappeared at 169.39 eV and 168.19 eV, which indicated that SO_3^- groups in PSS may involve in the chemical reaction with both Ca^{2+}, Mg^{2+} sites on dolomite surface [26].
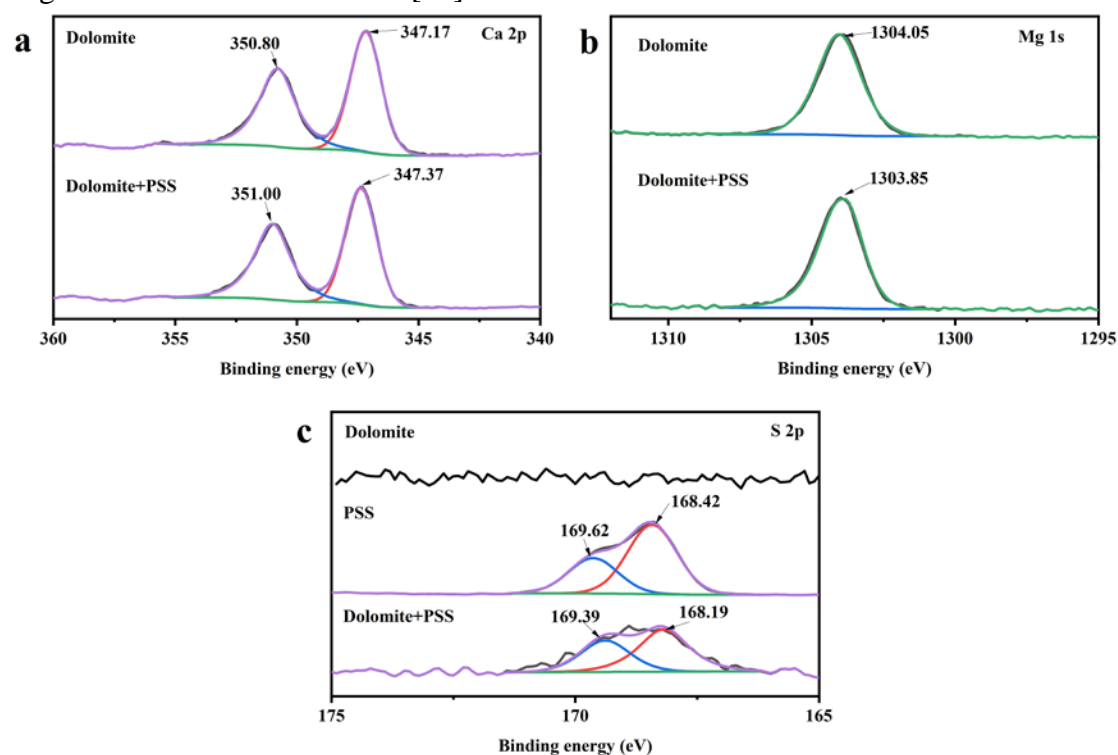
Fig. 9. XPS results of dolomite treated or untreated with PSS: Ca 2p (a), Mg 1s (b), S 2p (c).

Fig. 10 shows the P 2p, Ca 2p, and F 1p XPS spectra on apatite. Fig. 10 (a) displays the Ca spectra of apatite. The characteristic peaks appeared at 350.92 eV and 347.37 eV were considered as Ca $2p_{1/2}$ and Ca $2p_{3/2}$ on apatite, respectively [27, 28]. For PSS-treated apatite, the bimodal peaks shifted slightly to 350.98 eV and 347.43 eV, which

246 indicated that Ca sites on apatite might not involve in the chemical interaction. But
247 anionic groups in flotation reagents are most likely to combine with metal ions on
248 minerals surface, the weak reaction between Ca sites and SO_3^- groups might be the main
249 reason for the adsorption of apatite and PSS [14]. Compared with dolomite, the reason
250 for this great discrepancy in adsorption behavior might be the different activity of Ca
251 sites on apatite and dolomite surface [29].

252   Fig. 10 (b) shows the P 2p spectra of apatite. The peak appeared at 133.34 eV was
253 described as PO_4^{3-} groups on apatite surface. For PSS-treated apatite, the binding
254 energy of P 2p shifted slightly, which demonstrated that PO_4^{3-} groups on apatite did not
255 participate in the reaction with PSS on apatite. Fig. 10 (c) describes the F 1s spectra of
256 apatite. For pure apatite, the F 1s peak located at 684.50 eV. After being treated with
257 PSS, the F 1s binding energy changed insignificantly [30, 31].
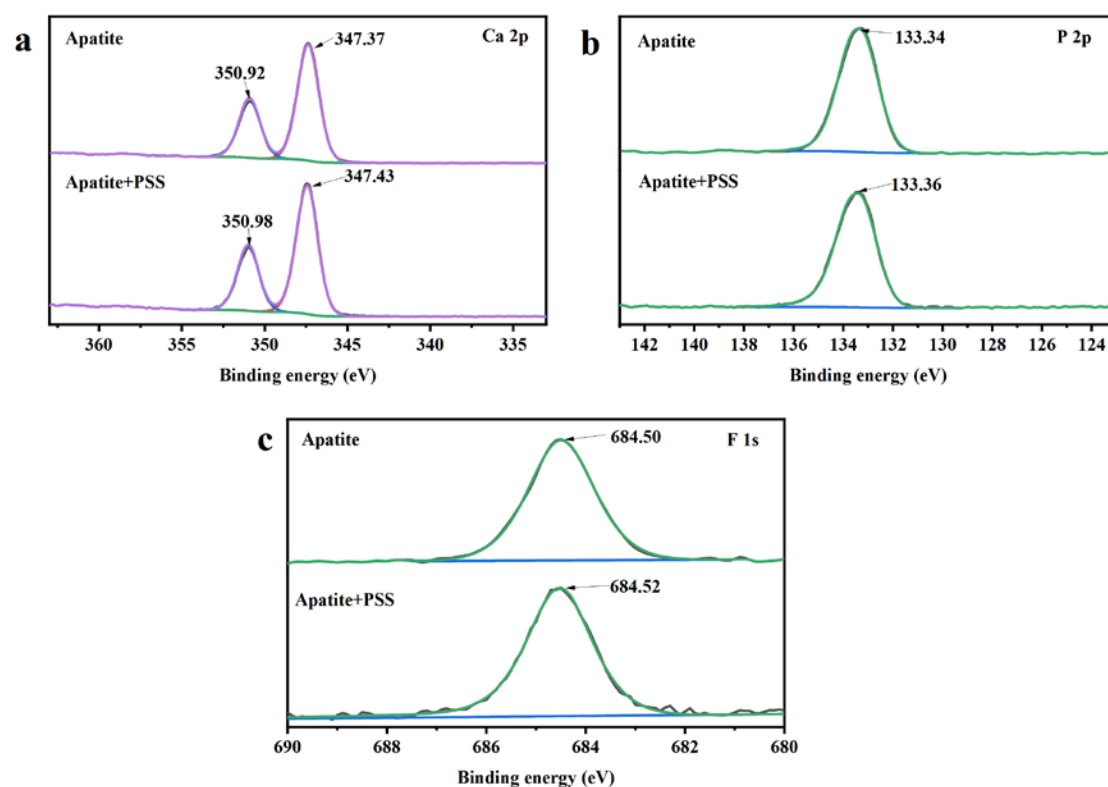
258
259 **Fig. 10.** XPS spectra of apatite treated or untreated with PSS (Ca 2p (a), P 2p (b), F 1s
260   (c)).

261 **3.6 The depression mechanism**

262   Apparently, due to the difference in the crystal structures, the exposed Ca^{2+} density
263 on dolomite was bigger than that of apatite [32]. Moreover, for dolomite, the existence
264 of a large number of Mg^{2+} on the dolomite surface facilitated the adsorption of PSS on
265 dolomite surface. Except that, the steric hindrance and electrostatic repulsion effect

between the PO_4^{3-} and $-SO_3^-$ groups can't be ignored. Due to the larger volume and higher charge of PO_4^{3-} compared with CO_3^{2-}, PO_4^{3-} exhibited high steric hindrance on the adsorption of PSS on apatite surface [33, 34]. Thus, PSS preferentially adsorbed on dolomite surface through the complexation between Ca^{2+}, Mg^{2+} and $-SO_3^-$ groups, which impede the adsorption of NaOL and make the dolomite surface hydrophilic, as proved by XPS and zeta potential results. While PSS weakly adsorbed on apatite surface, which could be replaced by NaOL and restored hydrophobicity.

4. Conclusions

(1) PSS selectively depressed dolomite but hardly affected the flotation of apatite in NaOL system in the pH range of 7-12.

(2) The adsorption of PSS on dolomite was much stronger than that on apatite, which prevented the adsorption of NaOL on dolomite and made its surface hydrophilic.

(3) PSS chemically adsorbed on dolomite surface through the complexation of $-SO_3^-$ groups with Ca, Mg sites. In contrast, PSS weakly adsorbed on apatite surface.

(4) PSS was an efficient and potential dolomite depressant in the upgrading of apatite ores, which may be a good alternative for apatite depressants in future.

Acknowledgements

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