

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

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

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33 **Abstract**

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

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

46 **1. Introduction**

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

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

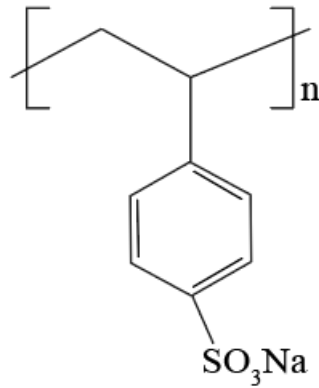
65 difficult to separate these two minerals because of the poor selectivity of fatty acid-
66 based collectors without depressants.

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 ions (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.



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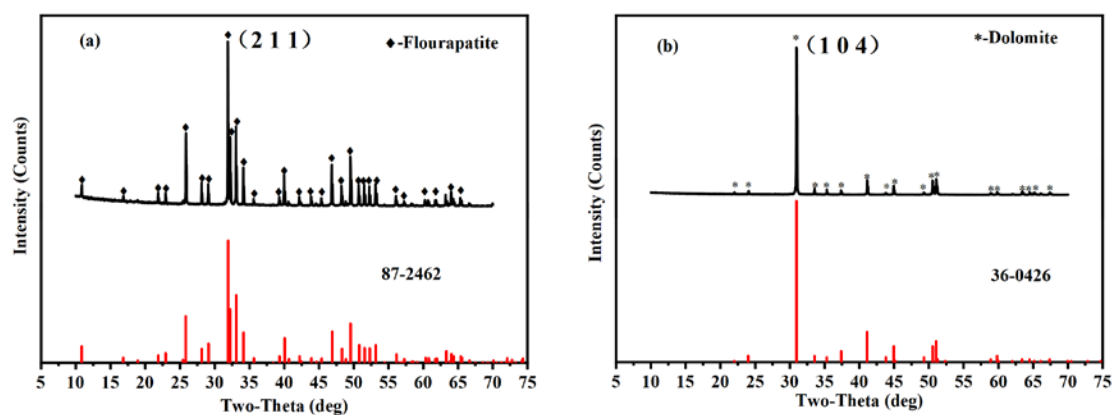
Fig. 1. The molecular structure of PSS.

104 **2. Experimental**

105 **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.



118

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

120 **2.2 Micro-flotation tests**

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

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

130 **2.3 Zeta potential measurement**

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

136 **2.4 FTIR tests**

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

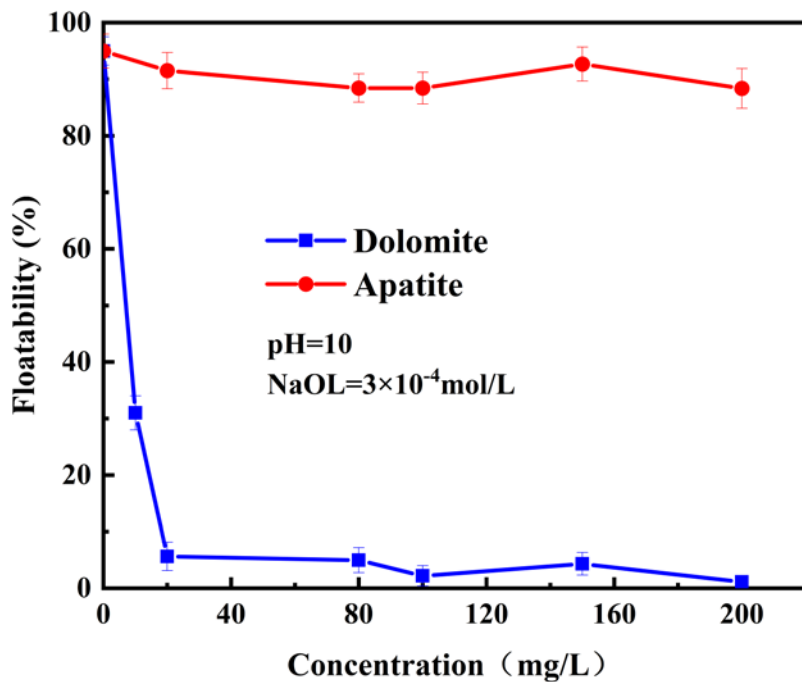
141 **2.5 XPS analysis**

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

147 **3. Results and discussions**

148 **3.1 Single mineral flotation**

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

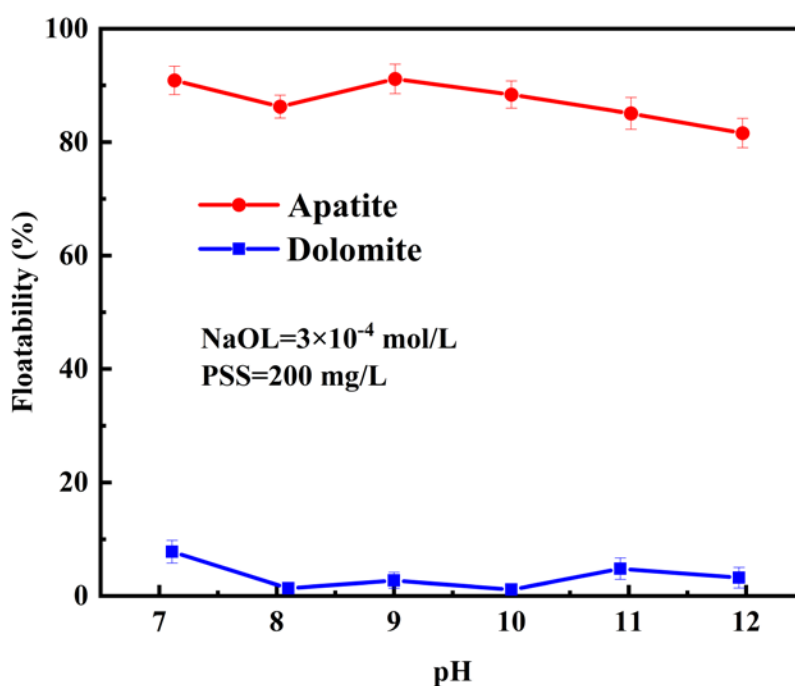


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158 **Fig. 3.** The floatability of dolomite and apatite at various PSS concentration.

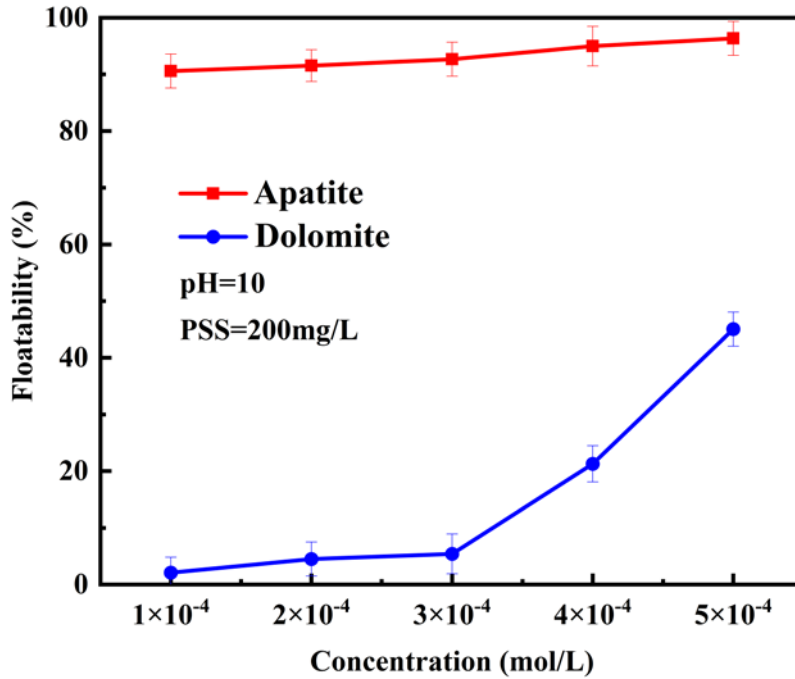
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160 Fig. 4 shows the floatability of dolomite and apatite at various pH. PSS depressed
161 dolomite powerfully at broad range (pH 7-12), but barely affected apatite. The
162 floatability difference was enlarged to approximately 80%, which reconfirmed 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 **Fig. 4.** The floatability of dolomite and apatite at various pH.
172
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.

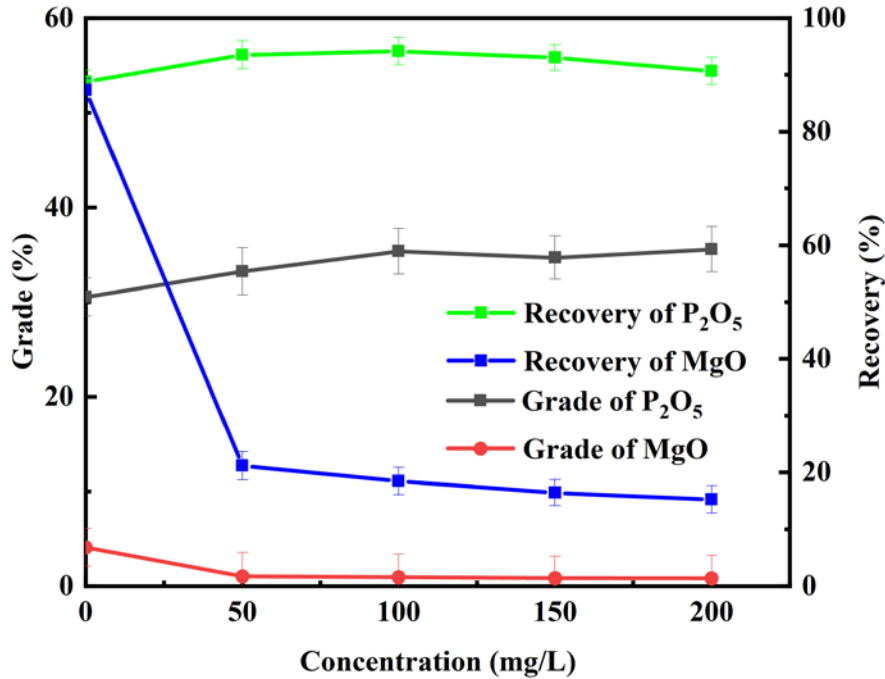


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179 **Fig. 5.** The floatability of dolomite and apatite as a function of NaOL concentration.

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 P₂O₅ kept around 90%. Moreover,
 185 MgO grade dropped from 4.07% to 0.81% in apatite concentrate, while the P₂O₅ 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.



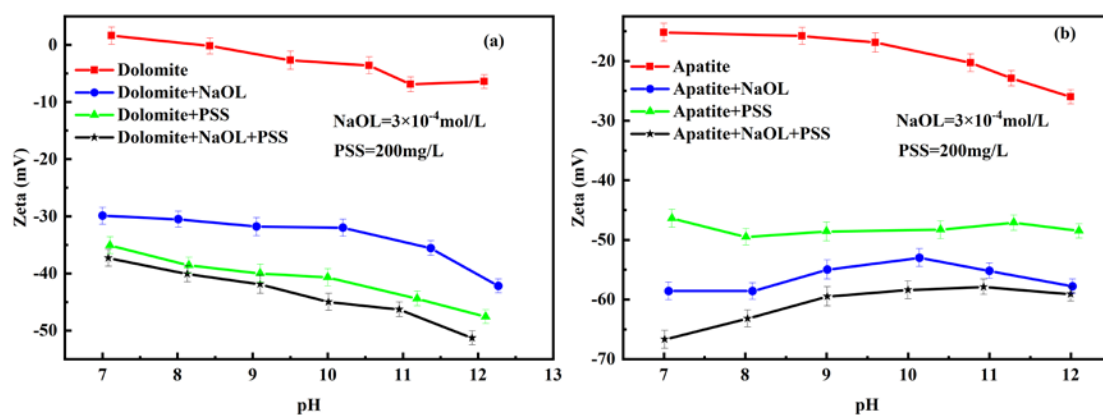
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190 **Fig. 6.** The recovery and grade results of artificially-mixed minerals as a function of
 191 PSS concentration (NaOL 3×10^{-4} mol/L and pH 10).

192 **3.3 Zeta potential results**

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

200 For apatite, zeta potential dropped obviously when PSS or NaOL was added alone,
 201 which demonstrated that both reagents could adsorb on apatite surface separately (Fig.
 202 7 (b)). When apatite was treated with both NaOL and PSS, zeta potential results were
 203 similar to that of NaOL-treated, illustrating that pre-adsorbed PSS barely influenced the
 204 adsorption of NaOL on apatite. In other words, these results suggested that sodium
 205 oleate could substitute pre-adsorbed PSS on apatite while it couldn't replace that from
 206 dolomite surface, which provided a good explanation for the selective depression effect
 207 on dolomite.



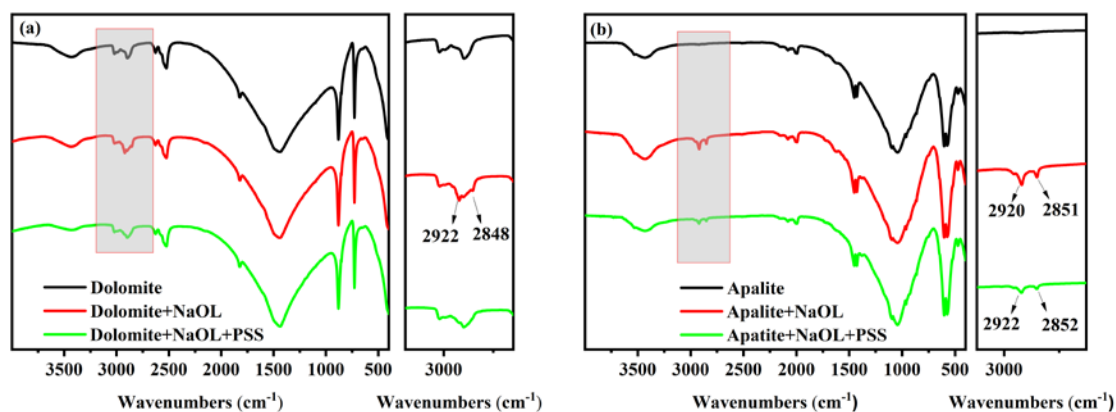
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Fig. 7. Zeta potential results of dolomite (a) and apatite (b) at various pH.

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 $-\text{CH}_2-$ and $-\text{CH}_3$ 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.



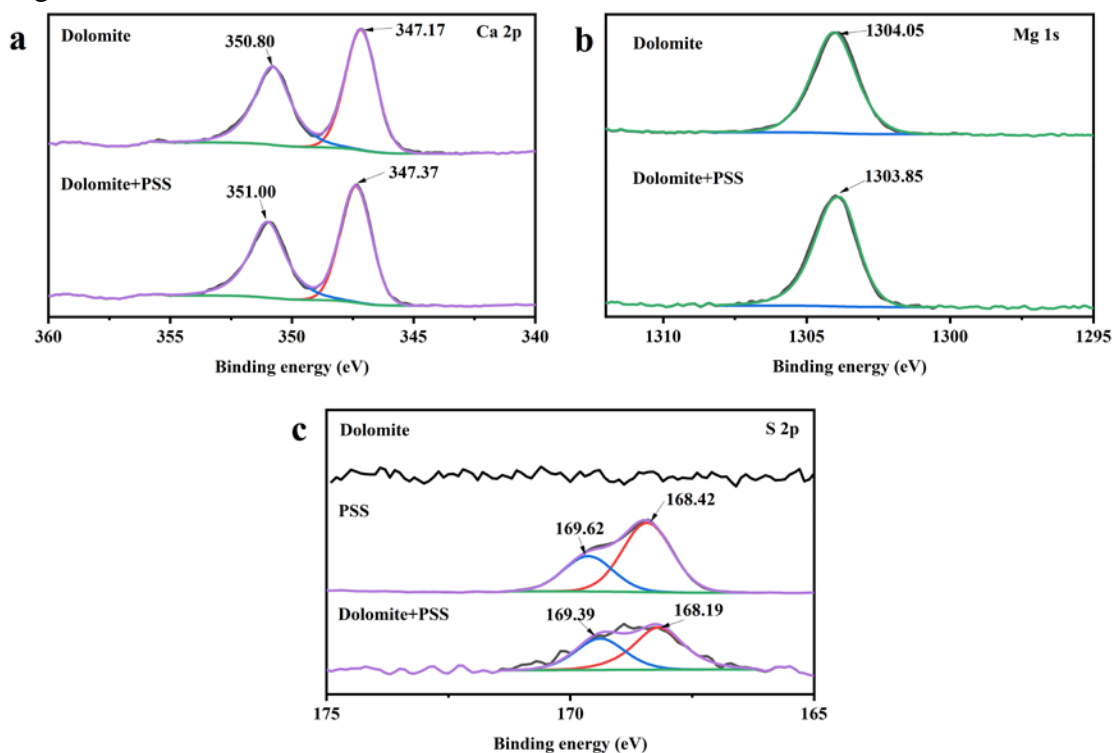
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Fig. 8 IR spectrum of dolomite (a) and apatite (b) under various conditions (PSS=200 mg/L, NaOL= 3×10^{-4} mol/L).

225 3.5 XPS results

226 Fig. 9 describes the Mg 1s, Ca 2p and S 2p XPS spectra of dolomite surface. The
227 peaks located at 350.80 eV and 347.17 eV were described as Ca 2p_{1/2} and Ca 2p_{3/2} in
228 dolomite (Fig. 9 (a)). After being treated with PSS, the bimodal peaks shifted by 0.20
229 eV to 351.00 eV and 347.37 eV, which indicated that Ca sites on dolomite might involve
230 in the chemical interaction with PSS [24]. In Fig. 9 (b), the peak belonged to Mg 1s
231 decreased from 1304.05 eV to 1303.85 eV after the introduction of PSS, which meant
232 that Mg sites on the dolomite also underwent a stronger chemical reaction with PSS
233 [25].

234 For pure PSS, the peaks appeared at 169.62 eV and 168.42 eV were assigned to -
235 SO₃⁻ groups in PSS (Fig. 9 (c)). When pure dolomite was treated with PSS, the
236 characteristic peaks of SO₃⁻ groups reappeared at 169.39 eV and 168.19 eV, which
237 indicated that SO₃⁻ groups in PSS may involve in the chemical reaction with both Ca²⁺,
238 Mg²⁺ sites on dolomite surface [26].

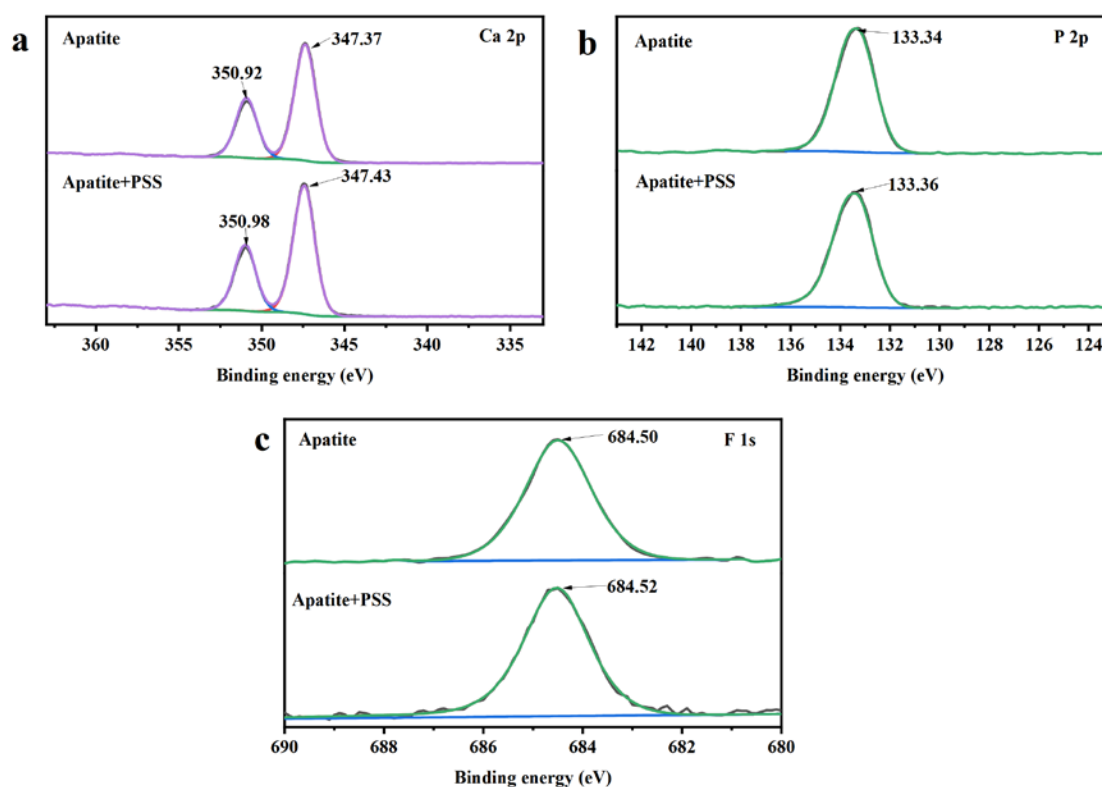


239
240 **Fig. 9.** XPS results of dolomite treated or untreated with PSS: Ca 2p (a), Mg 1s (b), S
241 2p (c).

242 Fig. 10 shows the P 2p, Ca 2p, and F 1p XPS spectra on apatite. Fig. 10 (a) displays
243 the Ca spectra of apatite. The characteristic peaks appeared at 350.92 eV and 347.37
244 eV were considered as Ca 2p_{1/2} and Ca 2p_{3/2} on apatite, respectively [27, 28]. For PSS-
245 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

266 between the PO_4^{3-} and $-\text{SO}_3^-$ groups can't be ignored. Due to the larger volume and
267 higher charge of PO_4^{3-} compared with CO_3^{2-} , PO_4^{3-} exhibited high steric hindrance on
268 the adsorption of PSS on apatite surface [33, 34]. Thus, PSS preferentially adsorbed on
269 dolomite surface through the complexation between Ca^{2+} , Mg^{2+} and $-\text{SO}_3^-$ groups,
270 which impede the adsorption of NaOL and make the dolomite surface hydrophilic, as
271 proved by XPS and zeta potential results. While PSS weakly adsorbed on apatite
272 surface, which could be replaced by NaOL and restored hydrophobicity.

273 **4. Conclusions**

- 274 (1) PSS selectively depressed dolomite but hardly affected the flotation of apatite in
275 NaOL system in the pH range of 7-12.
- 276 (2) The adsorption of PSS on dolomite was much stronger than that on apatite, which
277 prevented the adsorption of NaOL on dolomite and made its surface hydrophilic.
- 278 (3) PSS chemically adsorbed on dolomite surface through the complexation of $-\text{SO}_3^-$
279 groups with Ca, Mg sites. In contrast, PSS weakly adsorbed on apatite surface-.
- 280 (4) PSS was an efficient and potential dolomite depressant in the upgrading of apatite
281 ores, which may be a good alternative for apatite depressants in future.

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