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## Reverse flotation of collophanite at natural pH using isoctyl polyoxyethylene ether phosphate as a collector

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**Abstract:** Reverse flotation of collophanite at natural pH could significantly decrease the cost of pH regulators. In this study, isoctyl polyoxyethylene ether phosphate (AEP) was tested as a new surfactant in the reverse flotation of collophanite. Micro-flotation tests were conducted, and the adsorption mechanism of the new collector was analysed using X-ray photoelectron spectroscopy (XPS) and zeta potential analyses. The results of the flotation tests demonstrated that AEP could enable dolomite to float under natural pH (pH=7.2) and showed profound selectivity towards dolomite as opposed to fluorapatite. Based on the zeta potential and XPS results, the adsorption phenomena are mainly attributed to calcium active sites on both mineral surfaces. Dolomite possesses more magnesium active sites than fluorapatite, which tend to reinforce the interaction effect between AEP and dolomite. Furthermore, when compared to CO<sub>3</sub><sup>2-</sup> ions on the dolomite surface, PO<sub>4</sub><sup>3-</sup> ions on the fluorapatite surface tend to exhibit a stronger hindrance to the adsorption of AEP on the fluorapatite surface. This is attributed to their larger volumes and more charges on their surfaces, thereby causing a floatability difference between the two minerals.

**Keywords:** reverse flotation, dolomite, fluorapatite, AEP, selective adsorption

### 1. Introduction

Phosphate ores are raw materials that are important in the phosphorus industry (Liu et al., 2017b). Eighty percent of the global phosphate rock production is derived from sedimentary phosphate deposits (Oelkers et al., 2008). In China, sedimentary phosphate rock (collophanite) accounts for approximately 85% of the total reserves, most of which are medium- and low-grade ores. Collophanite ore is a type of colloidal apatite that coexists with carbonate minerals, such as dolomite (Liu et al., 2017a). Fluoroapatite is a primary mineral in this type of phosphate rock. Reverse flotation is considered as one of the most effective approaches to achieve the recovery of apatite from associated gangue minerals (Abdel-Khalek, 2000; Sis and Chander, 2003). The removal of dolomite reduces the MgO content, which enhances the P<sub>2</sub>O<sub>5</sub> grade of the recovered minerals (Abouzeid, 2008).

To date, the flotation of such minerals is increasingly facing challenges (Hoang et al., 2019) because apatite and its main gangue, such as carbonate gangue minerals (dolomite, calcite, ankerite etc.), possess similar active sites on their surfaces. The sites tend to consume large doses of a collector, thus leading to the production of low-grade concentrates (Filippova et al., 2018). Particularly, apatite and carbonate minerals exhibit similar reactivity towards conventional fatty acid collectors, and this reduces the selectivity of such collectors to a certain extent (Yang et al., 2020). Thus, the past few decades saw an increase in the study, development, and synthesis of new flotation reagents with effective selectivity for

the advantage of carbonate and phosphate minerals (Santos et al., 2015), such as gutter oil, saponified gutter oil, jojoba oil, and bio-collectors (Liu et al., 2017a; Santos et al., 2015; Merma et al., 2013).

Fatty acids (e.g., sodium oleate) are the most widely used collectors in apatite and dolomite flotation; a small amount or more than 10 kg of phosphoric acid ( $H_3PO_4$ ) was added as a depressant to produce satisfactory results for the flotation of carbonates under acidic conditions ( $pH = 5.0-5.5$ ) (Liu et al., 2017b; Cheng et al., 2018). However, the pH of an alkaline ( $pH = 9.0-9.5$ ) is required in the direct flotation of apatite when using sodium oleate as the collector. That is, after the addition of phosphoric acid, a large amount of sodium carbonate is required to adjust the pH to the desired value. Therefore, during this process, the addition of phosphoric acid can cause corrosion of the equipment, and the low pH value caused by phosphoric acid significantly increases the production cost because of the increased investment of regulators.

Recently, polyoxyethylene ether phosphate (AEP) has been widely employed in the textile chemicals industry (Zhao et al., 2020; Feng et al., 2020). It was employed as a new and effective surfactant/collector during the reverse flotation of collophanite; it showed ideal performance during the tests. In particular, we found that when AEP was selected as the collector, selective separation of fluorapatite from dolomite could be achieved under natural pH without the addition of sodium carbonate to create an alkaline environment. This reduced the cost of reagents. Additionally, sodium tripolyphosphate (STPP), rather than phosphoric acid, was used as the depressant, which alleviated the corrosion of the equipment in an acidic environment. Hence, the objective of this work was to demonstrate its selectivity and collecting ability toward the target mineral and to elucidate the adsorption mechanism. Therefore, this study selected fluorapatite and dolomite as the valuable mineral and gangue mineral, respectively. The research was conducted using various techniques, including micro-flotation tests, bench-scale flotation tests, zeta potential measurements, and XPS analysis.

## 2. Experimental

### 2.1. Materials and reagents

The fluorapatite sample used for this study was procured from Yunnan Province, China. The fluorapatite sample was ground using a porcelain ball mill, screened using a wet sieve, and a size fraction of 38–74  $\mu m$  was obtained that was used for micro-flotation tests. The  $P_2O_5$  content of the fluorapatite sample was measured using the chemical method with a grade of 37.86%. Additionally, the XRF analysis (Table 1) shows that the chemical composition of  $P_2O_5$  was 37.29% which is approximately equal to the chemical analysis result. This further demonstrates that the purity of fluorapatite is sufficiently high for pure mineral flotation. The dolomite sample was procured from Hebei Province, China. The  $MgO$  content of the dolomite sample was 22.06%. The samples were treated similar to fluorapatite, and a size fraction of 38–74  $\mu m$  was obtained which was used for micro-flotation tests. The purities of the samples are demonstrated in the X-ray diffraction analysis presented in Figs. 1a and b. The figure shows that the fluorapatite and dolomite exhibit high purity.

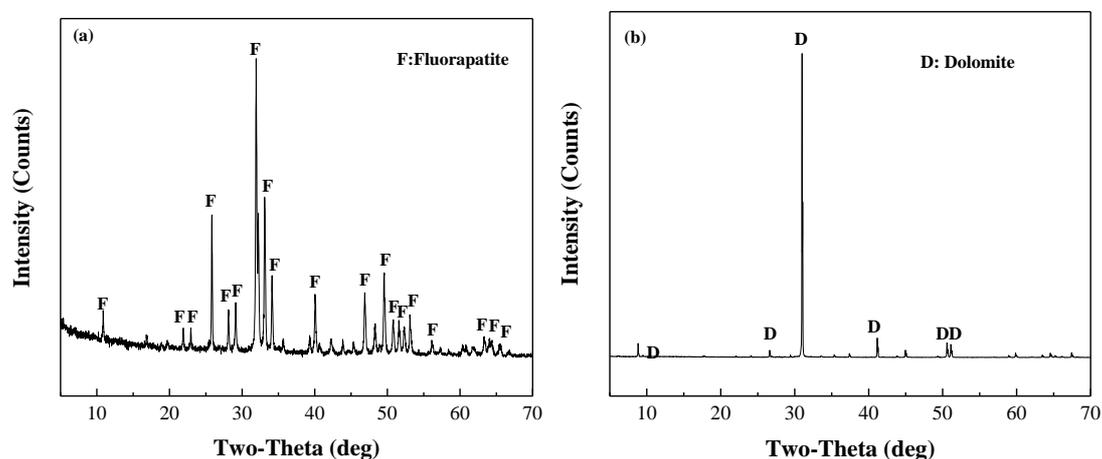


Fig. 1. XRD patterns of (a) Fluorapatite, (b) Dolomite

Table 1. Chemical compositions of fluorapatite (wt.%)

Composition	CO <sub>2</sub>	F	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	Cl
Content (%)	2.85	3.12	0.46	0.47	0.90	5.63	37.29	1.00	0.042
Composition	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	SrO	Y <sub>2</sub> O <sub>3</sub>			
Content (%)	0.42	47.38	0.099	0.22	0.11	0.009			

Table 2. Chemical compositions of collophanite ore sample (wt.%)

Composition	CO <sub>2</sub>	F	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	Cl
Content (%)	9.07	2.49	0.37	4.21	2.70	11.60	24.9	1.42	0.03
Composition	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	MnO	Fe <sub>2</sub> O <sub>3</sub>	CuO	BaCO <sub>3</sub>	SrO	BaO
Content (%)	1.12	39.83	0.17	0.02	1.66	0.02	0.16	0.07	0.16

The collophanite ore sample was obtained from the Yichang district, Hubei Province, China. Its chemical composition, shown in Table 2, was determined using X-ray fluorescence method. The amount of P<sub>2</sub>O<sub>5</sub> and MgO content were 24.9% and 4.21%, respectively, indicating that this sample belongs to medium-grade sedimentary ores (Mohammadkhani et al., 2011).

The chemical reagent, AEP, was procured from Green Forest Chemical Co., Ltd, Shandong province, China. Dilute solutions of hydrochloric acid (HCl) and sodium hydroxide (NaOH) were employed to modify the pH of the test samples. To maintain the ionic strength in zeta potential measurements, potassium nitrate (KNO<sub>3</sub>) of an analytical grade was used (Chen et al., 2020; Chen et al., 2020). The deionised water used throughout these experiments was obtained from Milli-Q Direct 16 (Millipore Q, USA). Sodium tripolyphosphate (Na<sub>5</sub>P<sub>3</sub>O<sub>10</sub>) was obtained from Sinopharm Chemical Reagent Co., Ltd.

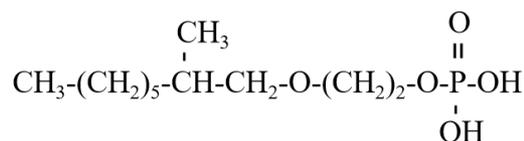


Fig. 2. Molecular structure of AEP

## 2.2. Methods

### 2.2.1. Flotation tests

To evaluate the single mineral floatability of fluorapatite and dolomite, micro-flotation tests for a single mineral of 3.0 g were conducted. These experiments were conducted as a function of AEP dosage and different pulp pH values. In artificially mixed mineral flotation tests, the total weight of the feed was 3.0 g with a fluorapatite to dolomite ratio of 2:1.

An XFG flotation machine with a 50-mL plexiglass cell was used for the micro-flotation tests; the agitation speed and airflow rate were fixed at 1700 rpm and 300 cm<sup>3</sup>/min, respectively. The pure mineral sample (3.0 g) was placed in the plexiglass cell, which was then filled with 40 mL of deionised water (Millipore deionised, resistivity of 18.25 MΩ cm). The slurry was conditioned for 2 min, and the pH was adjusted as required. Then, the pulp was conditioned again for 2 min, and the required dosage of AEP was added to the suspension and conditioned for 3 min. Finally, the minerals were floated for 3 min. The products from the flotation process were collected, dried, weighed, and analysed to calculate the single mineral floatability. The results of each micro-flotation test were measured three times, with the average reported as the final value.

Flotation tests of sedimentary collophanite samples were conducted in an XFDII (1.5 L) mechanically agitated flotation machine with an agitation speed fixed at 1750 rpm. The airflow rate was fixed at 2500 cm<sup>3</sup>/min. In the first step, the samples were ground using a ball mill to achieve the required fineness of 87% and passed through a 0.074-mm sieve. Sodium tripolyphosphate (STPP) and AEP were added to the pulp stage wise and agitated for 3 min. The flotation time was set for 4 min, and the flotation

products were schemed out as tailings. The remaining pulp in the cell formed the concentrate. Finally, the concentrates and tailings were dewatered, dried, and weighed. The  $P_2O_5$  content of the concentrates and tailings was determined via chemical analysis.

### 2.2.2. Zeta potential measurements

To investigate the interaction mechanism between minerals and the collector, zeta-potential measurements of the minerals in the absence and presence of AEP were conducted using a Zeta sizer Nano Zs90 (England). Mineral samples with a size fraction of 5  $\mu\text{m}$  were added to 45-mL ultrapure water. The pH of the mineral suspension was regulated through HCl or NaOH stock solutions, and the background electrolyte solution was  $1 \times 10^{-3}$  mol/L  $KNO_3$ . The ambient temperature was maintained at 25 °C. The mineral suspension with the desired AEP concentration was magnetically stirred for 5 min and allowed to settle for 10 min before the solution was drawn for zeta potential measurements. Three measurements were conducted for each experimental condition, and the average calculated was used as the final value.

### 2.2.3. XPS analysis

XPS was used to provide adsorption information of the flotation reagent. In this study, XPS data were obtained using a Thermo Fisher ESCALAB 250 XPS system (American Thermo Fisher Scientific) for dolomite. The C1s binding energy (284.8 eV) was employed to calibrate each specific spectrum. The procedure for the XPS sample preparation was same as that described in the flotation section.

## 3. Results and discussion

### 3.1. Single mineral flotation

Fig. 3a shows the effect of the AEP concentration on the floatability of dolomite and fluorapatite. The results show that when the concentration of AEP increased, dolomite and fluorapatite experienced an upward trend. However, the floatability of dolomite is much higher than that of fluorapatite over the entire concentration range tested; thus, the floatability of dolomite is better than that of the fluorapatite when AEP is employed as a collector. At a low collector concentration (9 mg/L), either dolomite or fluorapatite responded poorly in terms of floatability, reaching 45% and 6%, respectively. By increasing the collector concentration from 9 mg/L to 36 mg/L, the floatability difference between these two minerals reaches its maximum, assessed by 70%, and a further increase in the same concentration would weaken the selectivity of the collector.

As shown in Fig. 3a, the AEP concentration was selected as 36 mg/L to evaluate the effect of pH on the floatability of these two minerals, and the results are shown in Fig. 3b. Dolomite performed well in the pH range of 6–8, without a requirement for pH regulators, namely, natural pH conditions. In contrast, the floatability of fluorapatite was lower than 20% over the entire tested pH range. In addition, when fatty acids are selected as collectors for the reverse flotation of phosphate ore, large amounts of phosphoric acid ( $H_3PO_4$ ) are indispensable for producing satisfactory results for the flotation of carbonates under acidic conditions (pH < 5.5) (Abdel-Khalek, 2000; Liu et al., 2017b). Thus, instead of increasing capital investment and causing equipment corrosion, the superiority of our technique is highlighted.

### 3.2. Flotation separation of artificially mixed minerals

Single mineral flotation tests revealed that AEP could offer excellent collecting capabilities; therefore, an artificially mixed mineral flotation was employed to determine whether AEP exhibits better selectivity. The grade of artificially mixed minerals was 25.94%. Because reverse flotation was utilised in this study, the product floating in the froth was considered as tailings, while the concentrate in the flotation cell was regarded as concentrates. Thus, Fig. 4 shows that the  $P_2O_5$  grade tends to increase in response to increased AEP concentration, from 26.33% to 33.84%. In contrast, recovery displays an opposite variation tendency, reducing from 96.65% to 87.92%. This is mainly because when the AEP concentration increases, an increasing amount of dolomite floats along with the bubbles and decreases

the amount of dolomite remaining in the flotation cell; therefore, the  $P_2O_5$  grade of the concentrate increases. Simultaneously, when the AEP concentration increased, certain amount of the fluorapatite was mixed in the froth and floated with the dolomite. This resulted in a decrease in the fluorapatite quantity in the flotation cell and obtained a lower recovery. The results demonstrate that AEP exhibits an ideal collecting ability and functions well in mixed mineral systems.

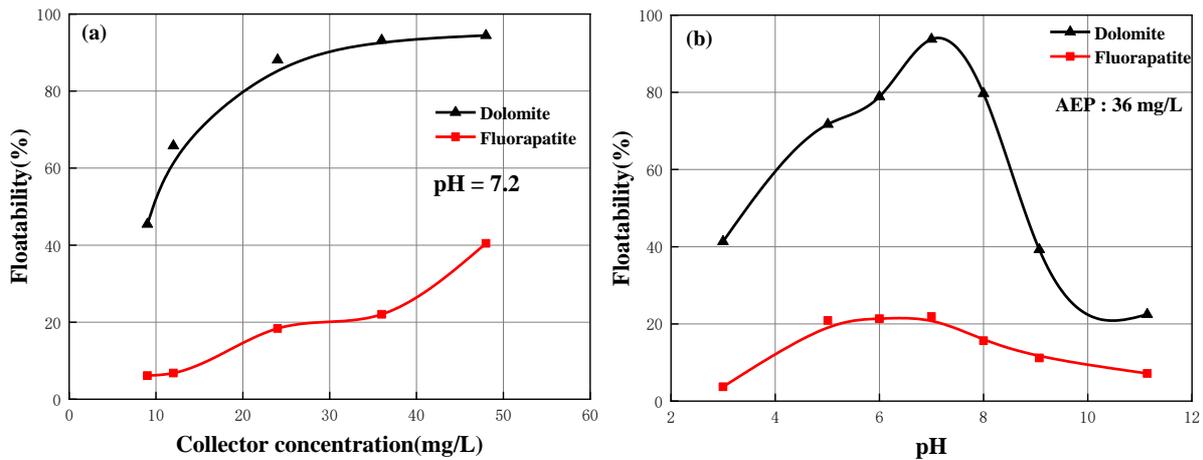


Fig. 3. Effect of (a) AEP concentration and (b) pulp pH on the floatability of fluorapatite and dolomite

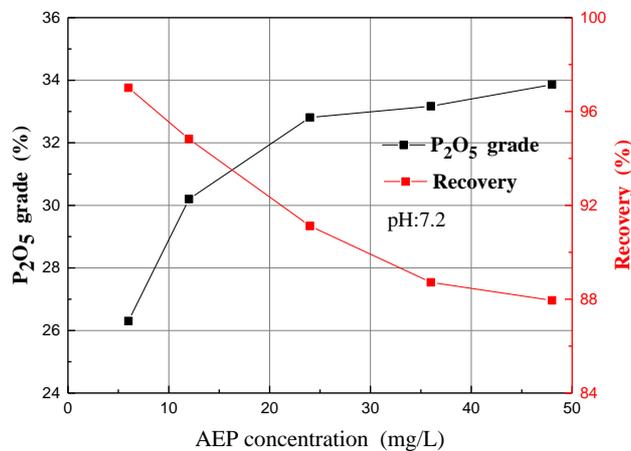


Fig. 4. Artificially mixed minerals separation as a function of AEP concentration

### 3.3 Bench-scale flotation tests of sedimentary collophanite ore

Despite the excellent flotation results obtained from pure mineral micro-flotation, further experiments are required to demonstrate the selectivity of the collector based on the actual ore. Hence, the effect of STPP and AEP dosage on the flotation performance of sedimentary collophanite ore was investigated and further evaluated.

Depressants, such as phosphoric acid and its derivatives (e.g., diphosphonic acid and sodium tripolyphosphate), should be added to the solution to consider the inhibition of valuable minerals in reverse flotation. In this study, STPP was tested as a depressant under natural pH conditions (Sis and Chander, 2003). As shown in Fig.5a, at a reagent concentration of 0.4 kg/t AEP, the  $P_2O_5$  grade shows no significant change over the entire STPP dosage tested, while the recovery experiences a slight increase from 65% to 71.5%. Thus, an STPP dosage of 0.5 kg/t was selected as the optimal dosage.

As shown in Fig. 5b, the  $P_2O_5$  grade of the feed was 24.9%; after a rougher flotation, a concentrate with the grade of 29.22% could be obtained with the addition of 0.3 kg/t AEP. This indicates that AEP is an ideal collector with high selectivity. Additionally, with an increase of AEP amount from 0.3 kg/t up to 0.6 kg/t,  $P_2O_5$  grade increased by 1.6%. In contrast, the recovery reduces by 13.8%. The results correspond well with the results proposed in artificially mixed minerals flotation, thus strongly

demonstrating the selective collecting ability of AEP towards dolomite. A scavenged flotation is required to further improve the recovery of the concentrate.

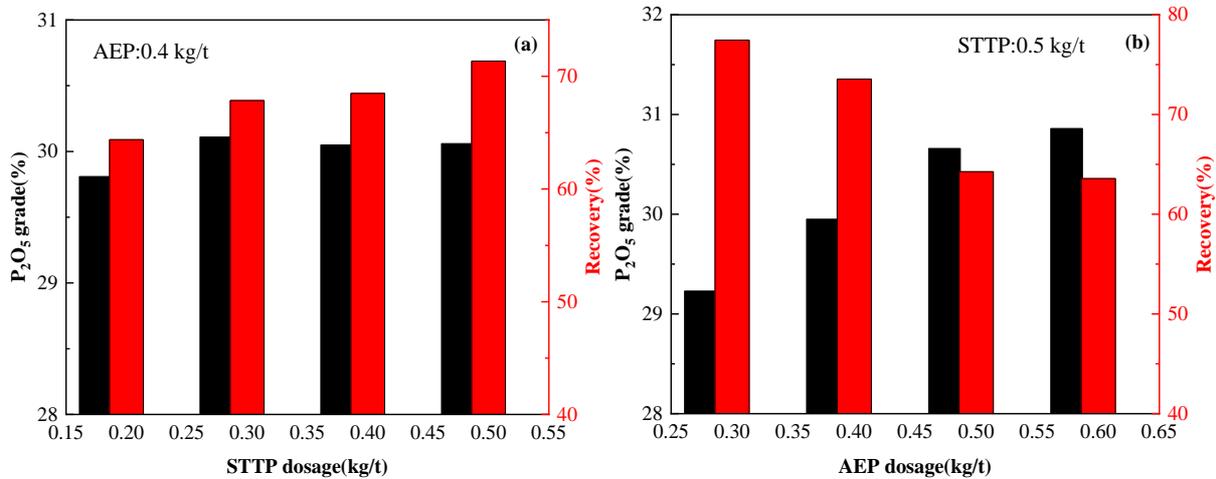


Fig. 5. Effect of (a) STPP and (b) AEP concentration on the  $P_2O_5$  grade and recovery of the concentrate

### 3.4. Effects of AEP on Zeta potentials of dolomite and fluorapatite as a function of pH

Fig.6 illustrates the zeta potential results with respect to the pH value. In the presence of the AEP collector, the zeta potentials of fluorapatite experienced negligible changes over the entire pH range. However, the zeta potential of dolomite was reduced by approximately 10 mV in the given pH range, i.e., the interaction of AEP with dolomite is stronger. Minerals with different crystal structures have different surface elements. The figure shows that the two minerals exhibit negative charge over the pH range tested, i.e., the principal ions exposed to their surfaces appear to be negatively charged ions. In other words,  $F^-$  and  $PO_4^{3-}$  may be the main anions on the fluorapatite surface and  $CO_3^{2-}$  may be the anions on the dolomite surface. Thus, it can be deduced that such anions on the mineral surface can deteriorate the adsorption of the AEP collector ( $R-O-HPO_3^-$ ) with  $Ca^{2+}$  and  $Mg^{2+}$  ions on mineral surfaces via steric hindrance and electrostatic repulsion. Furthermore, anions with larger volumes and charges are expected to weaken the adsorption, indicating that  $PO_4^{3-}$  ions exhibit a stronger inhibition effect than  $CO_3^{2-}$  ions (Yang et al., 2020). Hence, more AEP collectors are prone to interact with dolomite than with fluorapatite.

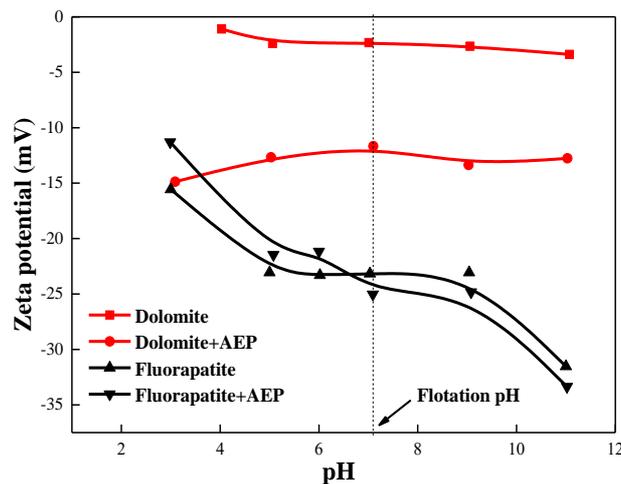


Fig. 6. Zeta potential of dolomite and fluorapatite in the presence and absence of AEP as a function of pH

### 3.5. XPS analyses

To further elucidate the adsorption mechanism of the AEP collector on the dolomite surface, XPS analysis of dolomite in the presence and absence of AEP was conducted, and the results are shown in

Fig. 7 and Table 3. Table 2 shows that the addition of AEP resulted in noticeable changes in the surface composition of dolomite. The relative content of the C atom increased from 25.99% to 35.15%, while the relative contents of Ca, Mg, and O decreased by 1.27%, 2.03%, and 4.45%, respectively. The noticeable increase in the relative concentration of carbon indicates that there was an adsorption of AEP on the dolomite surface which was very strong, and corresponded well with the zeta potential results. In addition, a P component occurred when the dolomite surface was treated with AEP, which supports the aforementioned proposal. The appropriate decrease in Mg when compared to Ca showed that AEP displays a more intensive interaction with Mg sites on the dolomite surface. Thus, the high-resolution spectra of Ca 2p, Mg 2p, O 1s, and P 2p of dolomite with and without AEP are shown in Figs. 7a, b, c, and d, respectively.

Table 3. Atomic concentration of dolomite in presence and absence of AEP

Sample	Atomic concentration (at%)					
	Ca	Mg	O	C	P	Others
Dolomite	11.64	8.85	50.21	25.99	-	3.31
Dolomite + AEP	10.37	6.82	45.76	35.15	0.90	1.00

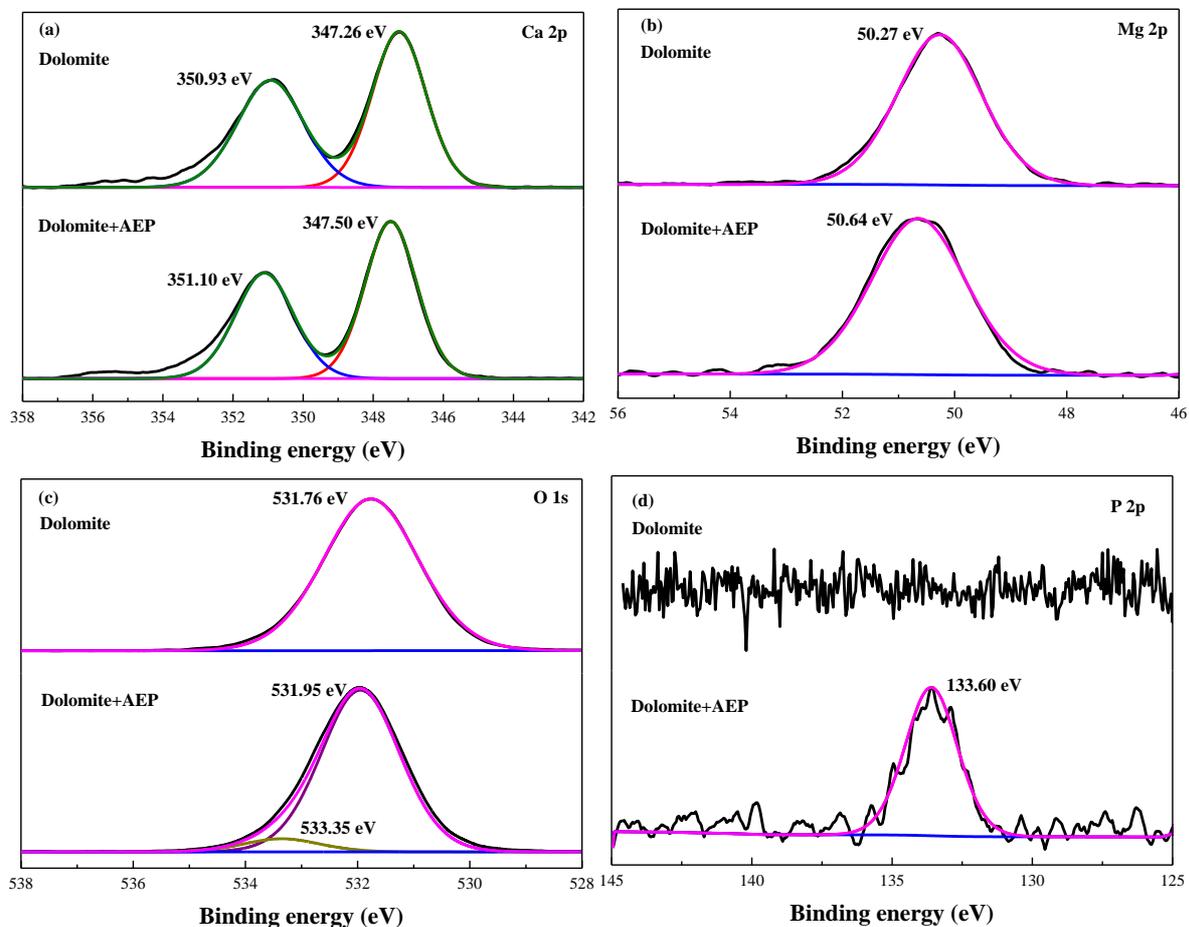


Fig. 7. XPS fitting peaks of (a) Ca 2p, (b) Mg 2p, (c) O 1s, and (d) P 2p on the dolomite surface before and after being treated with AEP

The high-resolution XPS spectra of Ca and Mg from dolomite are shown in Fig.7a and b, respectively. Considering Ca 2p, the binding energies of Ca 2p<sub>1/2</sub> and Ca 2p<sub>3/2</sub> appear at 350.93 eV and 347.26 eV, respectively, which are attributed to CaCO<sub>3</sub> in dolomite. Similarly, the peak at 50.27 eV may be attributed to MgCO<sub>3</sub> in dolomite (Fig.7b), which is consistent with previous reports (Liu et al., 2018; Yin et al., 2019). However, for the dolomite pre-treatment with AEP, the changes in the binding energies of

Ca 2p and Mg 2p are beyond the instrument error (0.2 eV). That is, electron transfer seems to occur between AEP and, Ca and Mg atoms on the dolomite surface. Particularly, the shift of Ca 2p<sub>3/2</sub> peak from 347.26 eV to 347.56 eV, Mg 2p peak from 50.27 eV to 50.64 eV maybe because Mg and Ca atoms can react with R-O-HPO<sub>3</sub><sup>-</sup> to form certain complexes. Moreover, the new O 1s and P 2p peaks (as shown in Fig.7c and d) appeared after being treated with AEP. This further demonstrates that R-O-HPO<sub>3</sub><sup>-</sup> species can interact with Ca and Mg active sites on the dolomite surface.

#### 4. Conclusions

The AEP collector used in this study shows good collecting ability towards dolomite and exhibits excellent selectivity for dolomite in actual flotation systems under natural pH. Additionally, the zeta potential measurements and XPS analysis demonstrate that R-O-HPO<sub>3</sub><sup>-</sup> species in AEP are expected to interact with calcium and magnesium active sites on the dolomite surface to form complexes, thereby rendering the dolomite surface hydrophobic. Furthermore, because of the larger volumes and charges of PO<sub>4</sub><sup>3-</sup> ions in fluorapatite than CO<sub>3</sub><sup>2-</sup> ions in dolomite, a stronger hindrance would occur on the fluorapatite surface owing to the electrostatic repulsion. This prevents further interaction between AEP and fluorapatite to a certain extent.

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