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## Effect of N-carboxybutyl chitosan on the flotation separation of apatite from dolomite

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**Abstract:** With high-grade apatite resources exhausted and economic development, enhancing the apatite quality from calcium gangue such as dolomite has a great significance for production. However, it is difficult to separate apatite from dolomite effectively due to the similar surface properties. In this study, the N-carboxybutyl chitosan (CBC) was tested as a potential selective depressant to separate apatite from dolomite in the sodium oleate (NaOL). Flotation results of single mineral and artificially mixed mineral confirmed the selective depression effect of CBC. The depression mechanism of CBC was investigated using wettability analysis, Fourier Transform Infrared (FTIR), and X-ray Photoelectron Spectroscopy (XPS) analyses. The results indicated that the CBC adsorption quantity and intensity on the dolomite surface more than that on the apatite surface, which was due to CBC absorbed on apatite surface by hydrogen bonding, while absorbed on dolomite surface mainly through chemical chelating between Ca on the mineral surface and -COO<sup>-</sup> on the depressant. These adsorption differences led to the flotation separation of the two minerals.

**Keywords:** apatite, dolomite, flotation, N-carboxybutyl chitosan (CBC), mechanism analysis

### 1. Introduction

Phosphate rock is an essential mineral resource, which is widely applied to environmental protection materials, medicine, and agricultural phosphate fertilizer (Sokolovic et al., 2012; Liu et al., 2019). Due to the metallogenic characteristics, the paragenetic and associated composition in phosphate rock results in low separation efficiency (Botero et al., 2007; Aslani et al., 2010; Luo et al., 2016), this phenomenon makes the phosphate resources high impurity content and incapable of meeting the demand of industrial manufacturing. Therefore, phosphate rock resources must be improved with further processing. Flotation is the most effective method, which produces 60% of the world's phosphate ores (Gence et al., 2006). However, it is difficult to separate apatite from dolomite because apatite and dolomite minerals are generally located together (Sis and Chander, 2003; Lv et al., 2017; Luo et al., 2017).

Depressants play a key role in flotation. An effective depressant should selectively attach to certain minerals and make the mineral surfaces hydrophilic (Liu et al., 2018; Feng et al., 2020). Over the years, several depressants have been selected as selective flotation depressants for dolomite. Wang et al. (2008) found that Ca-chelator BAPTA could depress the flotation of dolomite due to adsorption on the dolomite surface.  $\beta$ -naphthyl sulfonate formaldehyde condensate also showed a good selective inhibition on dolomite (Yu, 2016). Nevertheless, the flotation of apatite-containing calcium-bearing

minerals remains a problem in practical production. Therefore, there needs to research more selective and high-performance depressants for the separation of apatite from dolomite.

N-carboxybutyl chitosan (CBC) is a biodegradable and natural-based polymer that contains hydroxyl and carboxyl chelating groups (Katia, 2005), and has the potential to chelate with a metal ion (Muzzarelli, 1989). Therefore, this study aimed to investigate the flotation separation behavior of apatite from dolomite by CBC. Also, the mechanisms of CBC in the flotation were investigated.

## 2. Materials and methods

### 2.1. Materials

The pure samples of apatite and dolomite were purchased from Hubei Chemical Group Co., Ltd, China. The samples were crushed and sieved in the range  $-74+37\ \mu\text{m}$  for the flotation and adsorption experiments, and XPS analyses. The mineral samples of  $-5\ \mu\text{m}$  were used for FTIR spectroscopy analysis. The X-ray diffraction (XRD) analysis and chemical composition analysis of apatite and dolomite are shown in Fig. 1 and Table 1, respectively. It can be seen from Table 1 that the apatite and dolomite contained 39.28%  $\text{P}_2\text{O}_5$  and 21.32%  $\text{MgO}$ , respectively, indicated that both samples were pure enough for experiment and analyses.

The N-carboxybutyl chitosan (CBC) used as a depressant for the flotation experiments was derived from Shanghai Macklin Biochemical Co., Ltd, China, and its structure is shown in Fig. 2.

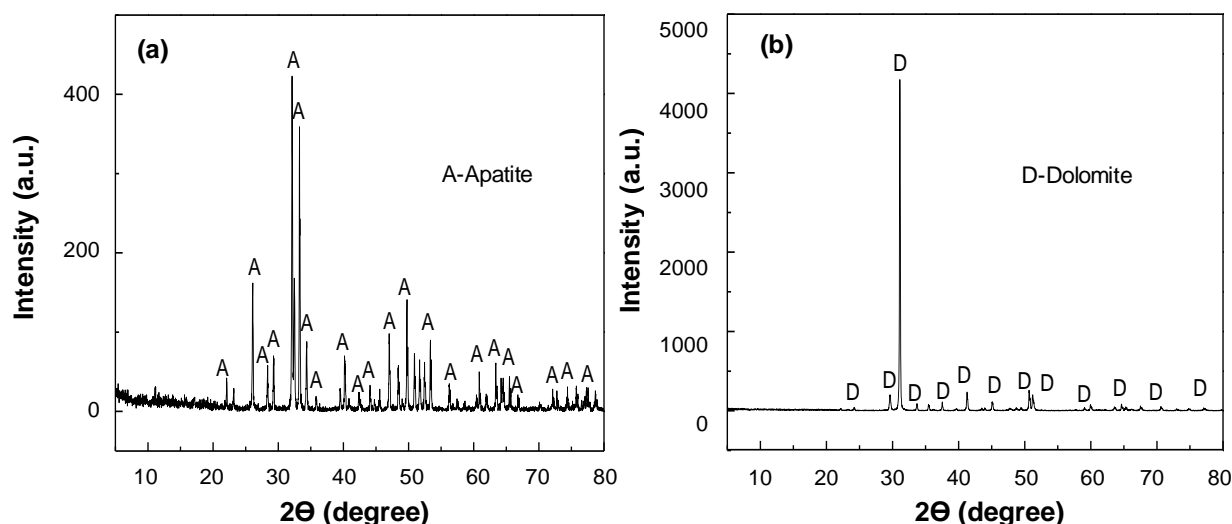


Fig. 1. XRD diagrams of (a) the apatite and (b) dolomite samples

Table 1. Chemical composition of the apatite and dolomite samples (%)

Sample	$\text{P}_2\text{O}_5$	$\text{MgO}$
Apatite	39.28	-
Dolomite	-	21.32

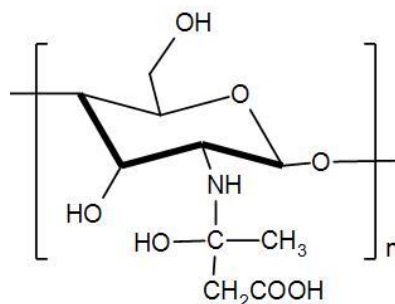


Fig. 2. The structure of CBC

Besides, sodium oleate (*NaOL*) was used as a collector (Xining Scientific Co., Ltd.), the pulp pH was regulated using potassium hydroxide and hydrochloric acid (*KOH* and *HCl*, Xining Scientific Co., Ltd.). All reagents were pure analytical grade and distilled water with a resistivity of 18.2 M $\Omega$ cm (Canrex Analytic Instrument Ltd., China) was used for the entire study.

## 2.2. Methods

### 2.2.1. Flotation experiments

The flotation experiments were conducted in an *XFGC* flotation machine (Zhengzhou Mining Machinery Group Co., Ltd, China). The stirring rate was adjusted to 1900 r/min. For each flotation test, a 2 g sample and 40 cm<sup>3</sup> distilled water were added into the flotation cell, the mixture was stirred for 2 min until a homogeneous liquid formed, and the pH was adjusted to the desired value in the meantime. Then, the depressant *CBC* and the collector *NaOL* were added to the pulp in proper order and reacted for 3 min, respectively. After the foam was taken for 3.5 min, the collected products were filtered, dried, and used to calculate the recovery. In mixed minerals experiments, the sink products were used to detect P<sub>2</sub>O<sub>5</sub> and to calculate the apatite recovery. The flowsheet of the flotation experiments is shown in Fig. 3.

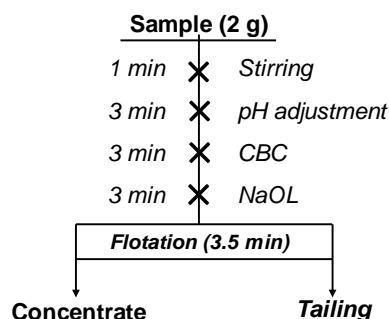


Fig. 3. Flowsheet of the flotation experiments

### 2.2.2. Wettability tests

The contact angle measurements were conducted to characterize the surface wettability of minerals. Pure block samples of apatite and dolomite were selected and polished, then the polished surfaces were treated with the related agents for 6 min. Subsequently, the samples were vacuum-dried at 50°C. The contact angle measurements were performed by a JY-82C contact angle measurement device (Dongsheng Tester Co., Ltd., China). The contact angle measurements were carried out with the sessile drop method and measured three times at different locations.

### 2.2.3. FTIR spectroscopy analysis

The *FTIR* analyses for the samples were performed using an ALPHA *FTIR* spectrometer (made by Bruker, Germany). -5  $\mu$ m samples (1 g) were mixed with 40 cm<sup>3</sup> distilled water followed by the addition of the depressant *CBC* and *NaOL* into the suspension. Then, the pulp pH was adjusted sequentially, and after stirring for 6 min, the solid particles and solution were separated. After the minerals were dried in a vacuum oven, the dried samples were mixed with *KBr* that the minerals accounted for 2% by weight. A small concentration of the mixture was placed in a tableting machine and pressed into a thin transparent film, then the spectra was scanned and analyzed. The spectra of samples were recorded at 25°C in the range of 4000-500 cm<sup>-1</sup>.

### 2.2.4. XPS spectroscopy analysis

The chemical environment of surface elements of apatite and dolomite before and after the reaction of corresponding flotation reagents was revealed by *XPS* analysis. To prepare the sample, 2 g of apatite or dolomite (-74  $\mu$ m + 34  $\mu$ m) samples and the reagents were added into 150 cm<sup>3</sup> distilled water in the absence and presence of *CBC*, after the pH adjustment, stirring, centrifuging, and vacuum drying, then dried sample was conducted for the *XPS* measurement. All high-resolution spectra were processed by

the Thermo Scientific Avantage software and referred to the correlated with literature and National Institute of Standards and Technology XPS Databases.

### 3. Results and discussion

#### 3.1. Flotation experiments

The effect of *NaOL* concentration on apatite and dolomite is given in Fig. 4. It can be seen that the recovery of apatite and dolomite increased with *NaOL* concentration up to  $3.0 \times 10^{-4}$  mol/dm<sup>3</sup>. When *NaOL* concentration arrived at  $3.0 \times 10^{-4}$  mol/dm<sup>3</sup>, the recovery of two minerals reached their maximum values. After that, the increasing concentration of *NaOL* showed a little effect on the mineral flotation recovery. The result indicated that the flotation behavior of apatite and dolomite was similar in the presence of *NaOL* and it was impossible to separate them in the absence of depressants.

The effect of pH value (5.5-10.5) on the floatability of apatite and dolomite in the absence and presence of the depressant *CBC* is shown in Fig. 5. The concentrations of *NaOL* and depressant *CBC* were  $3.0 \times 10^{-4}$  mol/dm<sup>3</sup> and 10 mg/dm<sup>3</sup>, respectively. It was seen From Fig. 5 that both apatite and dolomite were floatable in the absence of *CBC*. After 10 mg/dm<sup>3</sup> *CBC* was added, the depressive effect on dolomite was significant in the entire range of pH, the recovery of it was approximately 0. However, for apatite, although the *CBC* showed a certain depressive effect in pH 5.5-8.5, when the pH was above 8.5, the depressive effect was minor. Notably, the maximum flotation of apatite occurred at pH 8.5 exceed 90% while the recovery of dolomite was below 10%. The huge floatability difference suggested that *CBC* could be used as a depressant for separating apatite from dolomite.

Fig. 6 revealed the performance of *CBC* concentration on the flotation of apatite and dolomite at pH 8.5 when using  $3.0 \times 10^{-4}$  mol/dm<sup>3</sup> *NaOL*. It could be observed that the effect of *CBC* concentration on the recovery of apatite was low, but its influence on the recovery of dolomite was dramatic. When the *CBC* concentration was larger than 10 mg/dm<sup>3</sup>, the recovery of dolomite was only about 8%. The above results demonstrated that the selective flotation of apatite from dolomite could be realized under the reagent system of 10 mg/dm<sup>3</sup> *CBC* followed by  $3.0 \times 10^{-4}$  mol/dm<sup>3</sup> *NaOL* in pulp pH 8.5.

Single mineral flotation manifested that it was possible to separate apatite and dolomite using *CBC* as the depressant. Therefore, the mixtures flotation experiments of apatite and dolomite (mixing ratio 1:1) were further performed with and without the *CBC*. According to the results presented in Table 2, in the absence of *CBC*, the grade of P<sub>2</sub>O<sub>5</sub> in concentrate was near the same as that of the original mixture, suggesting separation was not realized. Under the condition of 10 mg/dm<sup>3</sup> *CBC*, the concentrate with a P<sub>2</sub>O<sub>5</sub> grade of 33.21% was achieved at a recovery of 70.46%, indicating that *CBC* showed an excellent depression performance for dolomite in apatite flotation.

#### 3.2. Wettability analysis

Wettability is a manifestation of hydrophilic and hydrophobic properties, which directly affect the floatability of minerals. Fig. 7 shows the changing situation of the contact angle measurements of

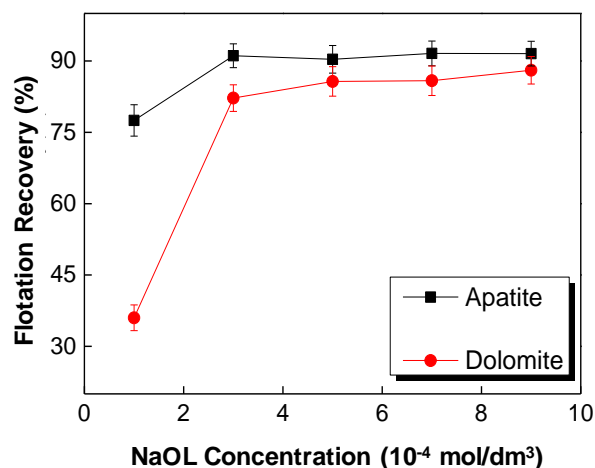


Fig. 4. Effect of *NaOL* concentration on the flotation of apatite and dolomite (pH=8)

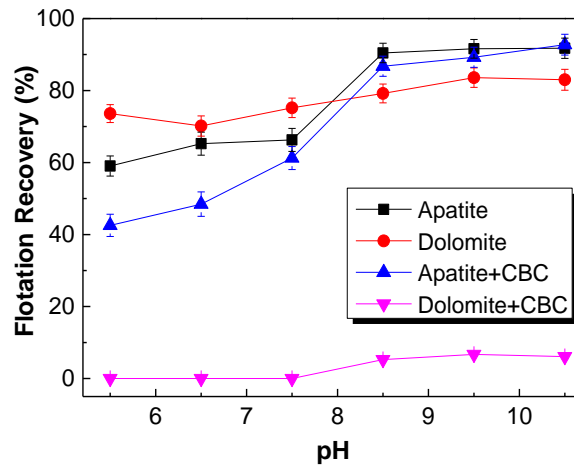


Fig. 5. Effect of pH on the flotation of apatite and dolomite in the absence and presence of depressant ( $NaOL = 3.0 \times 10^{-4} \text{ mol/dm}^3$ ,  $CBC = 10 \text{ mg/dm}^3$ )

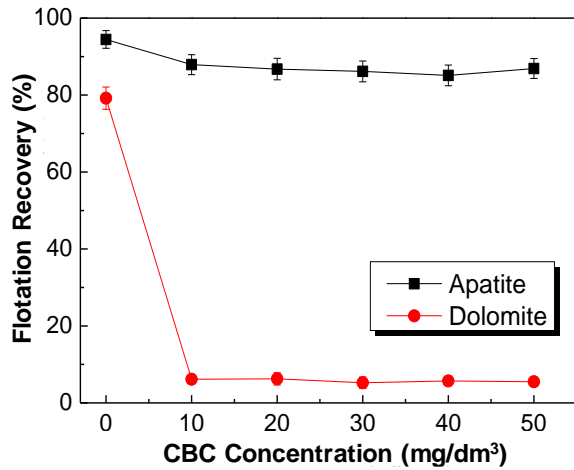


Fig. 6. Effect of  $CBC$  concentration on the flotation of apatite and dolomite ( $NaOL = 3.0 \times 10^{-4} \text{ mol/dm}^3$ ,  $pH = 8.5$ )

Table 2. Flotation results of mixed apatite and dolomite ( $NaOL = 3.0 \times 10^{-4} \text{ mol/dm}^3$ ,  $pH = 8.5$ )

Reagents	Products	Yield (%)	$P_2O_5$ Grade (%)	$P_2O_5$ Recovery (%)
$CBC: 0 \text{ mg/dm}^3$	Concentrate	87.45	19.79	86.56
	Tailings	12.55	22.41	14.44
	Initial state	100	20.12	100
$CBC: 10 \text{ mg/dm}^3$	Concentrate	45.12	33.21	70.46
	Tailings	55.88	9.43	29.54
	Initial state	100	20.16	100

apatite and dolomite in the presence of  $CBC$  as a function of  $NaOL$  concentration. The concentration of  $CBC$  was kept as  $10 \text{ mg/dm}^3$ , and pulp pH was adjusted to 8.5. Before treated by the  $CBC$ , the contact angles of two minerals increased sharply with  $3.0 \times 10^{-4} \text{ mol/dm}^3 NaOL$ , indicating the active groups of  $NaOL$  absorbed on the mineral surface increased its hydrophobicity (Merma, 2013). At the apatite-water and dolomite-water interfaces, the  $NaOL$  reactive groups have similar binding properties with the mineral surface species, which lead to very similar wettability.

After the *CBC* reacted with two minerals, a decrease occurred in the contact angle measurements on the dolomite surface and appeared a significant change when the concentration *NaOL* was  $3.0 \times 10^{-4}$  mol/dm<sup>3</sup> while the contact angle of apatite was almost unchanged at the whole concentration. Moreover, the stronger hydrophobic property increased on the apatite surface (approximately 95°) compared with dolomite (approximately 57°). The change of minerals contact angle after treated with *CBC* may ascribe to the *CBC* absorbed on the mineral surface, which reduced the landing of the collector. Likewise, the contact angle results indicated that the depressant *CBC* could adsorb on the dolomite surface more easily.

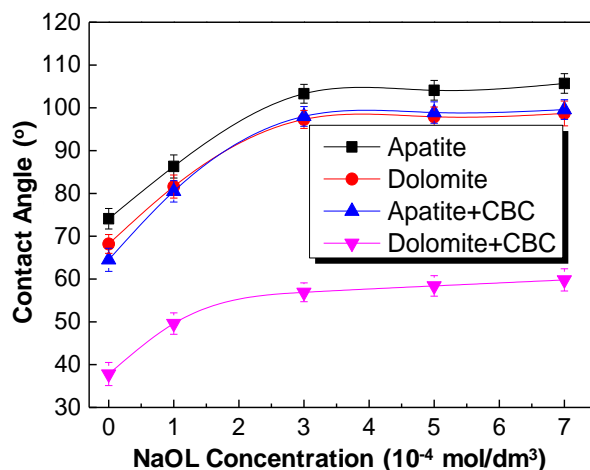


Fig. 7. Results for contact angle of mineral in the absence and presence of the *CBC* as a function of *NaOL* concentration (pH=8.5)

### 3.3. FTIR spectroscopy analysis

The FTIR spectra analysis of the samples was conducted to elucidate the mechanism of *CBC* reacted with the apatite and dolomite using *NaOL* as the collector. As shown in Fig. 8(a), in the *CBC* spectrum, the stretching bands at 1435 cm<sup>-1</sup> and 1567 cm<sup>-1</sup> are attributed to the symmetric adsorption band of C=O. The band at 1031 cm<sup>-1</sup> is assigned to the stretching vibration of C-O-C (Cao, 2017). For apatite, the hydroxyl group peak in 3428 cm<sup>-1</sup>, was present obviously in bare apatite, it reflected that the main composition of apatite was hydroxyapatite. After depressant *CBC* added, a new peak of C=O appeared at 1656 cm<sup>-1</sup> with a small shift, but this adsorption strength of C=O much weaker compared with the C=O in *CBC* spectrum indicated that very weak adsorption had occurred. Moreover, both apatite and *CBC* are negatively charged, strong electrostatic repulsive force and steric hindrance between *CBC* and apatite also make the *CBC* adsorb on apatite surface more difficult (Chen et al., 2017).

Fig. 8(b) represents the FTIR spectra of dolomite interacted with the *CBC*. The peak at 2894 cm<sup>-1</sup>

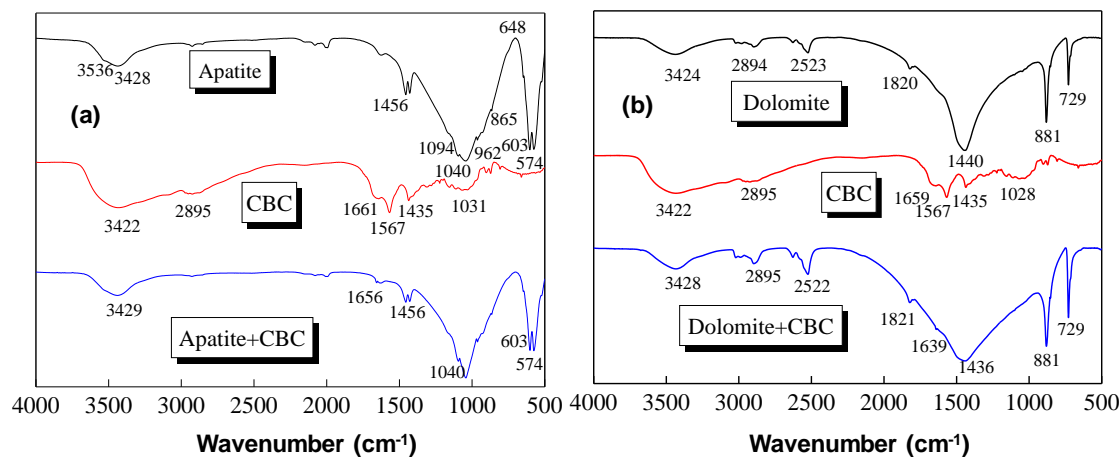


Fig. 8. FTIR spectra of minerals in the absence and presence of *CBC* (10 mg/dm<sup>3</sup>)

occurred in bare dolomite, after added *CBC* depressant, the shape and size of the band had changed significantly. Moreover, a new absorption peak appeared at  $1639\text{ cm}^{-1}$ , which was assigned to the stretching vibration peaks of carbonyl ( $-\text{COO}^-$ ) in *CBC* molecule and these absorption peaks were shifted from  $1659\text{ cm}^{-1}$ . At the same time, it's worth noting that the peaks at  $1435\text{ cm}^{-1}$  and  $1567\text{ cm}^{-1}$  derived from a symmetric adsorption band of  $\text{C}=\text{O}$  and  $1028\text{ cm}^{-1}$  attributed to the  $\text{C}-\text{O}-\text{C}$  in *CBC* disappeared, which was covered by the absorption peak of  $\text{C}-\text{O}$  in dolomite at  $1436\text{ cm}^{-1}$ . These may suggest that the presence of  $-\text{COO}^-$  groups in *CBC* molecule adsorption on dolomite through chemical chelating, indicated that *CBC* showed a strong depression effect on the dolomite surface.

### 3.4. XPS analysis

The high-resolution XPS spectra of Ca 2p, C 1s were performed to reveal the changes in the chemical environment of mineral surface elements treated or untreated with *CBC*. The results are shown in Figs. 9 and 10, respectively.

The XPS spectra of Ca 2p are shown in Fig. 9. It shows that fitting peaks of Ca 2p of bare apatite appeared at 347.21 eV and 350.77 eV which derived from the Ca 2p<sub>3/2</sub> and Ca 2p<sub>1/2</sub> in  $\text{Ca}_5(\text{PO}_4)_3\text{OH}$  (Liu., 2017). After reacted with *CBC*, no new peak appeared, which showed no direct reaction between Ca and *CBC*. For dolomite in Fig. 9(b), the fitting peak of Ca 2p of bare dolomite contained a double-peak at 347.06 eV and 350.66 eV, the peak derived from Ca 2p<sub>3/2</sub> and Ca 2p<sub>1/2</sub> in dolomite (Dong, 2019). After dolomite reacted to *CBC*, the fitting peaks had moved to 347.24 eV and 350.84 eV, respectively, with an increase of 0.18 eV. Indicating that the *CBC* reacted with Ca of the dolomite surface.

Fig. 10 shows the fitting peak of C1s of apatite and dolomite treated or untreated with *CBC*. According to Fig. 10(a), the peaks of C 1s of bare apatite appeared at 284.80 eV, 286.31 eV, and 288.91 eV, which could be attributed to adventitious carbon contamination of  $\text{C}-\text{C}$ ,  $\text{C}-\text{O}-\text{C}$ , and  $\text{O}-\text{C}=\text{O}$  on the apatite surface (Pan, 2020). After treated by *CBC*, four well-fitted separated peaks were observed. In addition to the three originally existed peaks of adventitious carbon contamination peaks at 284.80 eV, 286.34 eV, and 288.97 eV, respectively, a new peak was observed at 288.03 eV derived from the  $\text{C}=\text{O}$  in the *CBC* molecule. This also implied that the *CBC* had adsorption on the apatite surface. Fig. 10(b) showed the C 1s of bare dolomite contained three peaks at 284.80 eV, 286.07 eV, and 289.66 eV, contribu-

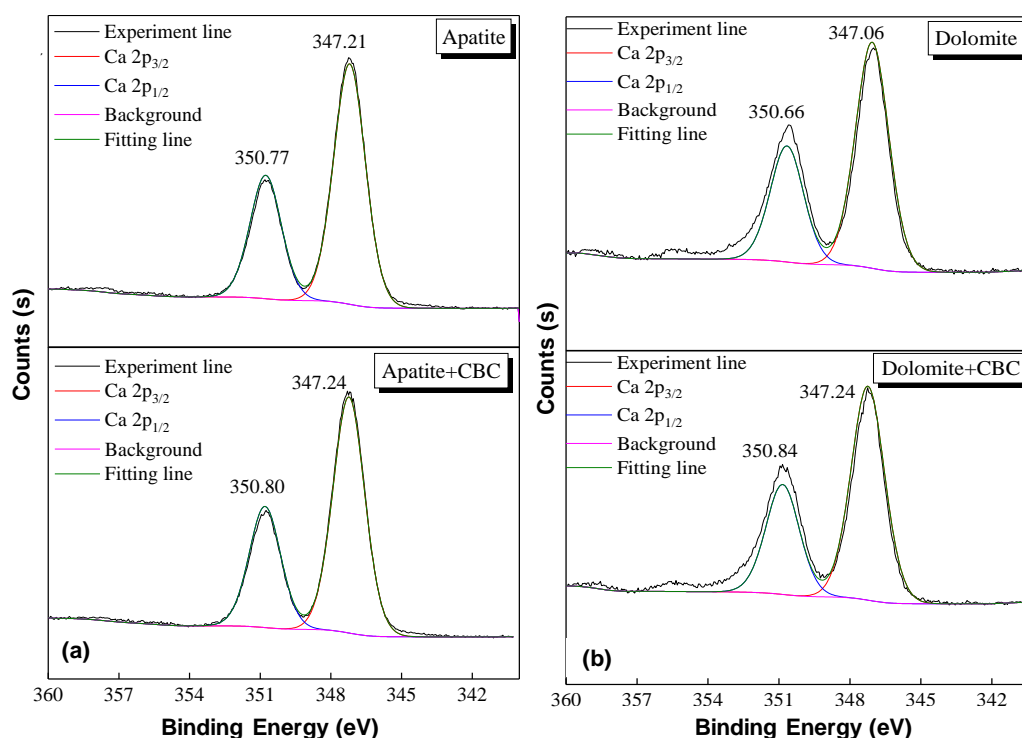


Fig. 9. Fitting peaks of Ca 2p of apatite (a) and dolomite (b) in the absence and presence of *CBC* ( $10\text{ mg}/\text{dm}^3$ )

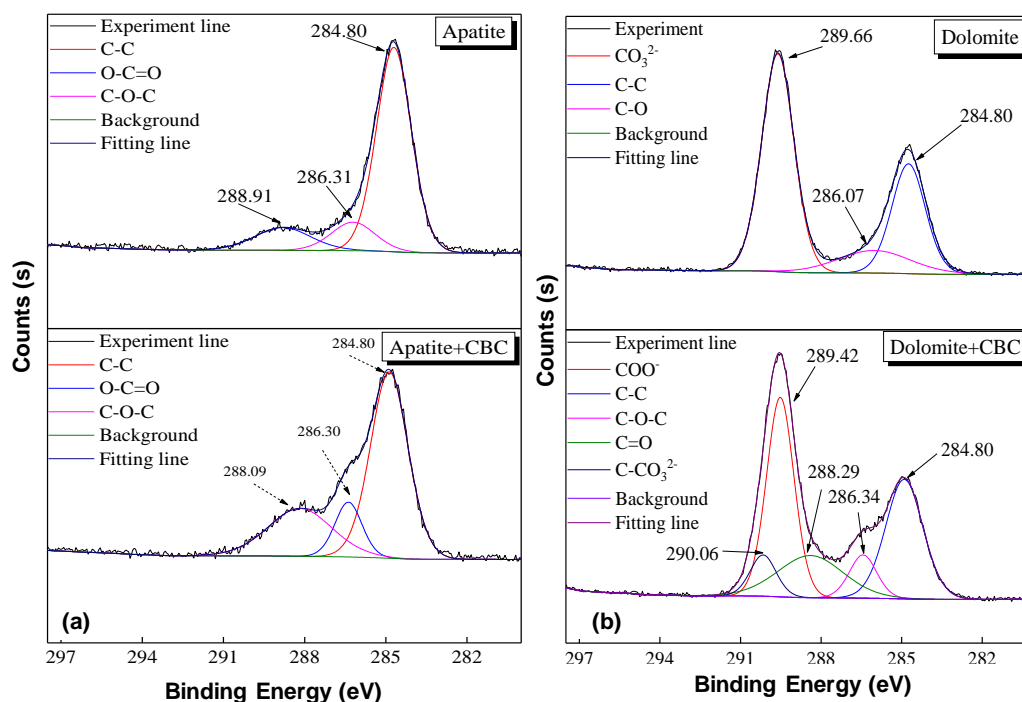


Fig. 10. Fitting peaks of C 1s of apatite (a) and dolomite (b) in the absence and presence of CBC (10 mg/dm<sup>3</sup>)

ted to the adventitious carbon contamination of C-C, C-O-C, and CO<sub>3</sub><sup>2-</sup> of dolomite, after the addition of depressant CBC, not only the shape, size and position of the three peaks above changed significantly, but also two new peaks appeared at 288.29 eV and 288.42 eV, these two peaks were attributed to the C=O and carboxyl derived from the CBC polymer. Combined with the binding energy of Ca 2p<sub>3/2</sub> and Ca 2p<sub>1/2</sub> with an increase of 0.18 eV, it could be deduced that the carboxyl group in CBC and the calcium species of dolomite were chelated.

#### 4. Conclusions

N-carboxybutyl chitosan (CBC) as an environmentally friendly and efficient depressant, manifested a high selective depression on dolomite in the apatite-dolomite flotation system when NaOL as the collector. In mixed mineral flotation experiments, the recovery rate and grade of apatite with 70.46% and 33.21% were obtained under the conditions of 10 mg/dm<sup>3</sup> CBC and 3.0×10<sup>-4</sup> mol/dm<sup>3</sup> NaOL. The wettability tests indicated that CBC could reduce the hydrophobicity of dolomite severely but showed a little effect on apatite. The FTIR and XPS analyses suggested that CBC interfered with the adsorption of NaOL on dolomite while showed no effect on the adsorption of NaOL on the apatite surface considerably. These adsorption differences were probably attributed to the direct chemical chelation between Ca on dolomite and -COO- in CBC while the CBC absorbed on apatite surface through hydrogen bonding, the chemical chelating is much stronger than hydrogen bonding. Moreover, the chemical chelating consumes the adsorption sites of NaOL on the dolomite surface heavily and the plentiful hydroxy groups in CBC make dolomite good hydrophilicity, which leads to selective depression on dolomite.

#### Acknowledgements

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