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含钾硅酸盐肥料在盐酸与柠檬酸中的溶解行为

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摘 要：为研究高温煅烧法制备的含钾硅酸盐肥料在酸性条件下的溶解行为，选择盐酸与柠檬酸作为浸提液，在室温下研究营养元素溶出与矿物组成的变化，探讨硅酸盐肥料包含矿物遇酸发生的化学反应。结果表明：浸提液 pH 值越低，元素溶出越多；pH 值为 2.1 的柠檬酸的溶解作用强于 pH 值为 2.1 的盐酸。pH 值为 0.3 的盐酸对硅酸盐肥料矿物的溶解率最高，滤渣中仅检测到了白榴石的衍射峰。钾霞石和钙铝黄长石两种矿物遇酸反应后生成的高岭石、三水铝石进一步遇酸反应是铝溶出的直接原因。

关键词：pH 值；盐酸；柠檬酸；高岭石；铝毒
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Dissolution Behaviors of K-containing Silicate Fertilizer in Hydrochloric and Citric Acids

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Abstract: To clarify the dissolution behavior of K-containing silicate fertilizers produced by high-temperature sintering under acidic condition, the chemical element release and mineral evolution were investigated at ambient temperature in the extractants of hydrochloric and citric acids based on the chemical reactions of minerals contained in silicate fertilizer with acids. The results show that the more elements are released at a lower pH value of extractant. 2% citric acid with pH 2.1 is significantly more effective in dissolving the silicate fertilizer than hydrochloric acid with the same pH value. The dissolution rate of silicate fertilizer in hydrochloric acid at pH value of 0.3 is maximum, and only a mineral detected in the filter residue is leucite. Kaolinite and gibbsite are formed in the reactions of kalsilite and gehlenite with acid. Aluminum release is due to the further reactions of kaolinite and gibbsite with acid.

Keywords: pH value; hydrochloric acid; citric acid; kaolinite; aluminum toxicity

1 Introduction

The beneficial effects of silicate fertilizers for improving crop growth and increasing plant tolerance to both abiotic and biotic stresses[1–2] have been reported for a wide variety of plant species. Meanwhile, the positive effects of silicate fertilizers on the soil environment[3–5] (including soil acidity neutralization, aluminum toxicity alleviation, and others) have shown great promise in sustainable agriculture. Among the major silicate fertilizers, soluble potassium or sodium silicates can be applied as foliar fertilizers, but are usually too expensive for soil applications[6]. Slag-based silicate fertilizers may pose a potential environmental risk because of heavy metals contained in them. Fortunately, citrate-soluble K-containing silicate fertilizers have shown the advantages of low cost and low heavy metal contents.

There are two approaches for producing citrate-soluble silicate fertilizer from K-rich silicate minerals (such as K-feldspar) and calcareous additives (such as limestone, dolomite, lime or phosphogypsum): (1) high-temperature and (2) hydrothermal treatments[7]. Previous studies have focused on the production process,

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chemical reactions, and mineralogical phases[8–12]. The mineral composition of the silicate fertilizer, produced by the high-temperature sintering in the range of 1 200–1 300 ℃ using K-feldspar, limestone, and dolomite as raw materials, mainly incorporates pseudo-wollastonite ($Ca_3Si_3O_9$), kalsilite ($KAlSiO_4$), akermanite ($Ca_2MgSi_2O_7$), gehlenite ($Ca_2Al_2SiO_7$), and leucite ($KAlSi_2O_6$)[9–10]. However, details about the dissolution process, nutrient release, and the residue after nutrient absorption remain unknown.

When it comes to the determination of plant-available elements in silicate fertilizers, the previously selected extractant was 2% citric acid (CA, pH=2.1) but currently is 0.5 mol/L hydrochloric acid (HCl, pH=0.3) according to NY/T 2272-2012 and NY/T 2273-2012. As a low-molecular-weight organic acid, CA can not only promote lattice destruction of primary minerals (including potash feldspar, muscovite, and others) in the soil by transforming the contained elements into plant-available nutrients[13], but also affect element dissolution and mineral structure of secondary minerals (kaolinite, montmorillonite, and others)[14]. In order to understand the dissolution behaviors of the K-containing silicate fertilizers under acidic conditions and to clarify the relationship between nutrient release and mineral dissolution, HCl at different concentrations (pH=0.3, 2.1, and 4.0) and 2% CA (pH=2.1) were selected as extractants. The research should be helpful in unraveling the origin of so-called “plant-available elements” and the chemical reactions in the determination.

2 Experimental

2.1 Preparation of K-containing silicate fertilizer

The K-containing silicate fertilizer was prepared *via* high-temperature sintering at 1 260 ℃ for 60 min, using K-feldspar, limestone, and dolomite as raw materials. The elemental analysis results are shown in Table 1. The content of heavy metal elements is below the limits regulated by the fertilizer standards in China (GB/T 23349–2009). The pH value of the silicate fertilizer was 10.98 (pH value was tested according to NY/T 1973–2010), which meets the standard GB/T 36207–2018.

Table 1 Elemental analysis results of the K-containing silicate fertilizer

Mass fraction *w*/%							Mass ratio/(mg·kg^{-1})				
Si	Ca	K	Mg	Na	Al	Fe	As	Pb	Cd	Cr	Hg
20.12	22.04	5.74	3.02	0.83	5.77	0.65	9.1	42.6	4.2	8.5	0.0

2.2 Dissolution experiments

Considering the acidity, the volume of HCl with pH 0.3 and CA with pH 2.1 in the dissolution experiments ranged from 10 mL to 200 mL while the volume of HCl with pH=2.1 and 4.0 changed from 50 mL to 2 000 mL.

One gram of sample was weighed and transferred to a beaker. A certain volume of extractant was then added. The beaker with pulp was placed on a magnetic stirrer and stirred for 30 min at ambient temperature. After leaching, the solution was filtered off after settling for at least 10 min. The filtrate was used as the test solution. The residue on the filter paper was washed and dried at 110–150 ℃. It was then ready for mineral composition and morphology analyses.

2.3 Analytical methods

Sample powders (0.6 g) were dissolved with $Li_2B_4O_7$ (6 g) in a TR-1000S automatic bead fusion furnace at 1 100 ℃ for 10 min. The elemental contents were determined by XRF-1500 sequential X-ray fluorescence (XRF) spectroscopy. Powder X-ray diffraction (XRD) patterns were obtained with a Bruker D8 Advance powder diffractometer at 40 kV and 40 mA using monochromatized Cu K_α radiation (λ=0.154 056 nm). The specimen’s morphology was observed with a cold-cathode field-emission scanning electron microscope (FESEM, S-4800, HITACHI, Japan) operated at a 20 kV acceleration voltage and a 20 μA beam current after gold-coating of the specimen surface. The ion concentration in the filtrate was measured by inductively-coupled plasma-optical emission spectrometry (ICP-OES) and then converted into the mass fraction of dissolved element in the silicate fertilizer, as shown in formula (1).

$$X(\mathrm{M}) = \frac{a \times b}{c \times 10^4} \tag{1}$$

M denotes one of the Si, Ca, K, Mg and Al elements; X(M) is the mass fraction (%) of dissolved M element by a certain volume of extractant; a is the resulting ion concentration in mg/L; b is the the volume of constant volume in mL; and c is the mass of the silicate fertilizer in g.

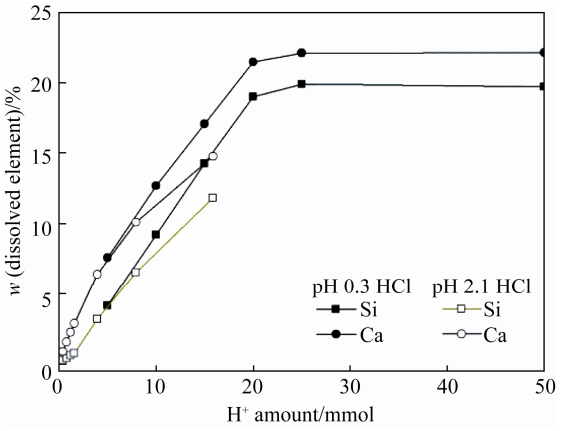
3 Results and discussion

3.1 Elemental release of K-containing silicate fertilizer in HCl and CA

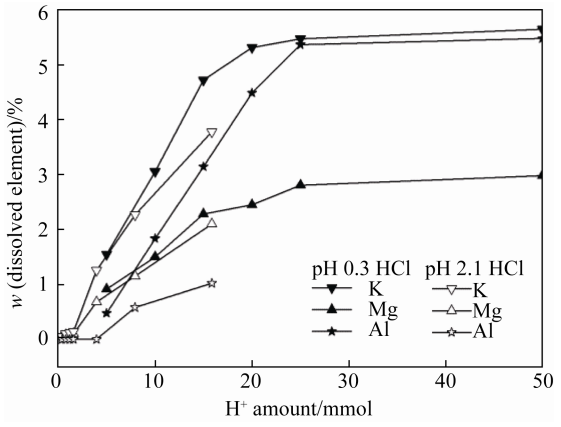
Changes in the content of dissolved elements with hydrogen ion under the action of HCl with pH 0.3 and pH 2.1 are shown in Fig. 1. It can be seen that not only Si, Ca, K, and Mg were dissolved, but Al was also dissolved. With the increase of H^+ amount, the contents of dissolved elements also showed an increase. When the H^+ amount was >25 mmol (50 mL HCl with pH 0.3), the contents of dissolved elements showed minimal changes, indicating the reaction was nearly complete. Compared with Table 1, the final dissolution rates of elements, Si, Ca, K, Mg (as a group), and Al by HCl with pH 0.3 were 98% and 95%, respectively.

With addition of 30 mL HCl with pH 0.3 and

2 000 mL HCl with pH 2.1, H^+ amounts were 15 and 15.88 mmol, respectively. The latter was marginally more but comparable on the whole. Comparing the two groups of data in Fig. 1(a) and (b), the content of dissolved element by 15 mmol H^+ from HCl with pH 0.3 was higher than that of 15.88 mmol H^+ from HCl with pH 2.1. It can be concluded that elemental release is not only related to the H^+ amount but also is affected by the strength of the acid. Considering the dissolution performance induced by comparable H^+ amounts, Si, Ca, K, or Mg dissolved by HCl with pH 2.1 accounts for 80.1% to 91.9% of that by HCl with pH 0.3, whereas Al dissolved by HCl with pH 2.1 was only 27.3% of that by HCl with pH 0.3.



(a) Si and Ca



(b) K, Mg, and Al

Fig. 1　Content of the dissolved element as a function of H^+ amount introduced by HCl with pH 0.3 and pH 2.1

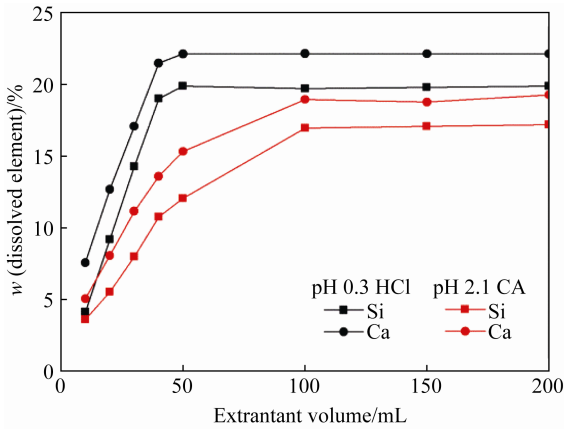
Given that CA is subject to different degrees of protonation depending on the pH and the difficulties in the calculation of the hydrogen ion concentration, the extractant volume is treated as the variable in the following section. The contents of dissolved elements by CA with pH 2.1 are displayed in Table 2; meanwhile, the results with HCl with the same pH are also listed for comparison. It can clearly be seen that the content of dissolved elements is significantly higher in CA with pH 2.1 than that by HCl with pH 2.1 with the same volume of extractants. When the volume was 500 mL, Si, Ca, K or Mg dissolved in CA with pH 2.1 was 3.1–5.5 times of that by HCl with pH 2.1. With the extractant volume fixed at 100 mL, contents of elements dissolved in HCl with pH of 0.3, 2.1, and 4.0 can be observed in Table 2. It is obvious that the extractant pH has a great impact on the mineral dissolution and element release. Under the action of strong acids (such as pH 0.3), almost all elements in the silicate fertilizer are dissolved, while under the weak acid conditions (such as pH 2.1, pH 4.0), the dissolved element content was relatively low. In order to release more elements, the volume of extractant should be increased.

Fig. 2 compares the contents of dissolved elements under the action of CA with pH 2.1 and HCl with pH 0.3. When the CA volume was less than 100 mL, the contents of dissolved elements obviously increased after increasing the CA volume. When the CA volume was more than 100 mL, the contents of dissolved elements barely changed. Compared with Table 1, CA with pH 2.1 finally dissolved about 85% to 90% of elements (Si, Ca, K, and Mg) and about 74% of Al in the silicate fertilizer. The final dissolved element in CA with pH 2.1 was less than that of HCl with pH 0.3, some elements were not finally dissolved.

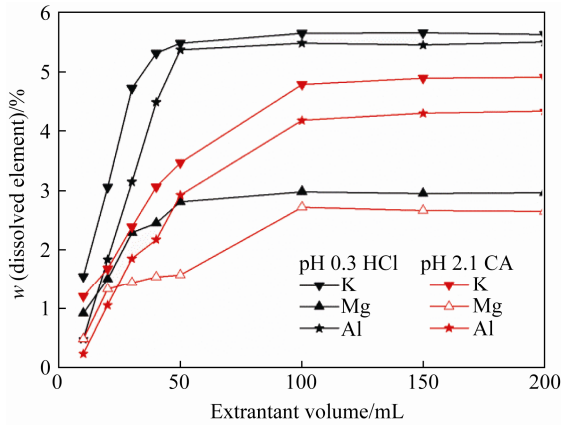
Table 2　Content of the dissolved element by CA and HCl

Extractant	Volume/mL	Mass fraction/%					pH value of filtrate
		Si	Ca	K	Mg	Al	
Citric acid (pH 2.1)	50	12.05	15.32	3.47	1.57	2.92	4.49
	100	16.96	18.96	4.79	2.72	4.18	3.73
	150	17.07	18.77	4.89	2.66	4.30	3.14
	200	17.20	19.28	4.91	2.65	4.34	2.97
	500	17.56	19.93	4.90	2.69	4.29	2.62
HCl (pH 2.1)	50	0.25	0.87	0.05	0.06	0	10.49
	100	0.40	1.55	0.10	0.08	0	10.14
	150	0.61	2.24	0.12	0.07	0	9.55
	200	0.77	2.86	0.14	0.10	0	9.22
	500	3.19	6.38	1.26	0.68	0	4.40
HCl (pH 4.0)	100	0.14	0.72	0.03	0.06	0	10.90
HCl (pH 0.3)	100	19.72	22.15	5.65	2.98	5.48	1.40

Table 2 shows that with the same pH of 2.1, the dissolution amount of one particular element by CA was significantly more than that by HCl. Fig. 2 shows the result of pH 2.1 CA is similar level to pH 0.3 HCl. As an inorganic acid, HCl can release H^+, which exchanges with cations (Ca^{2+}, Mg^{2+}, K^+) on the surface of silicate minerals, promoting the dissolution of silicate minerals. The reaction in the HCl solution is mainly due to proton-promoted dissolution. However,



(a) Si and Ca



(b) K, Mg, and Al

Fig. 2 Mass fraction of the dissolved element by citric acid (CA) with pH 2.1 and by HCl with pH 0.3

as an organic acid, CA has three ionizable hydrogen ions in the molecule. The released H^+ can exchange with the surface cations of silicate minerals. Moreover, CA also affects the dissolution of silicate minerals by releasing the organic ligand from CA. Citrate ions can react with metal ions (mainly Al ions) to form a complex with Al at the mineral surface[15], which improves the dissolution rate of minerals and promotes element release. Namely, the dissolution reaction in CA is a combination of proton- and ligand-promoted dissolution. Because of third-order ionization and influence of organic ligands, the solubility of the silicate fertilizer in CA with the same pH is significantly higher than that in HCl.

Fig. 3 shows the relationship between Al release and filtrate pH after the action of HCl and CA. The content of dissolved aluminum is negatively related to filtrate pH values. After the actions of HCl with pH 0.3 and 2.1, Al ions could be detected when the filtrate pH was below 4.05. However, the Al ion could be detected when the filtrate pH was below 7.24 after the application of CA with pH 2.1.

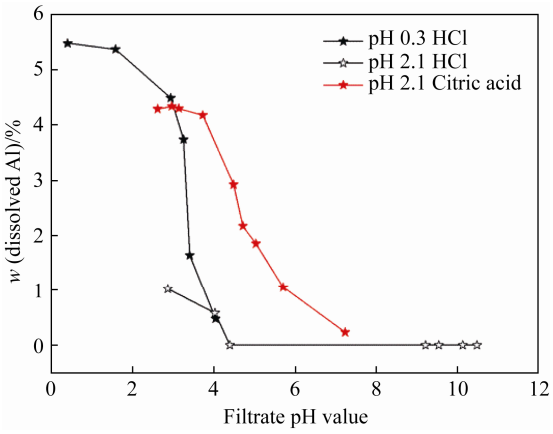


Fig. 3 Relationship between the content of dissolved aluminum and filtrate pH

3.2 Composition and microstructure of filter residue

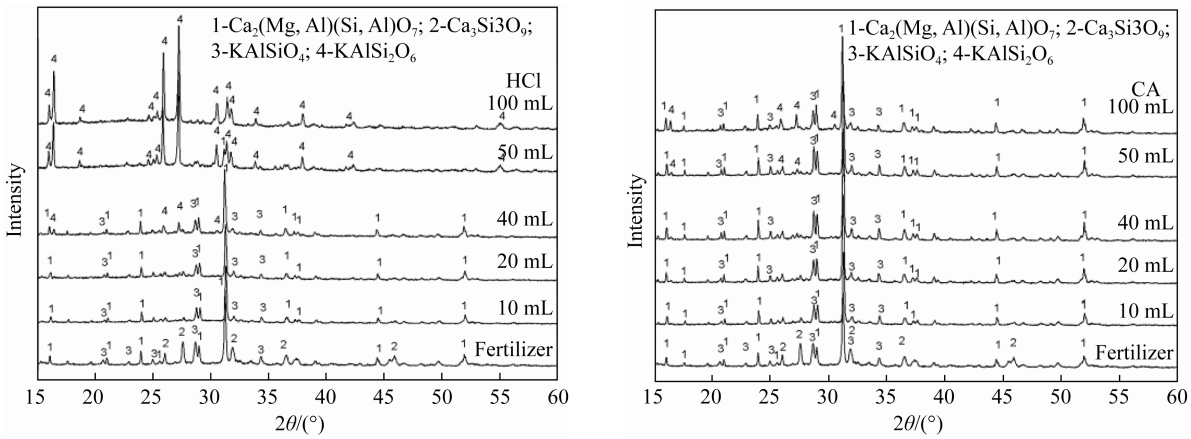
The filter residue became visibly less after increasing the volume of pH 0.3 HCl and pH 2.1 CA. When the volume of pH 0.3 HCl exceeded 40 mL, little filter residue was left. Fig. 4 shows the XRD patterns of the filter residues after being dissolved in different volumes of extractant. Compared with XRD patterns from the silicate fertilizer in Fig. 4a, the diffraction peak intensity of pseudo-wollastonite ($Ca_3Si_3O_9$, ICDD 74-874) decreased significantly after being subject to 10 mL HCl. When the volume of HCl reached 40 mL, the diffraction peaks of leucite ($KAlSi_2O_6$, ICDD 38-1423) appeared. Furthermore, when it came to 50 mL, the diffraction peaks of Ca_2(Mg, Al)(Si, Al)O_7 (ICDD 79-2474) and $KAlSi_2O_6$ were obvious and the diffraction peaks of kalsilite ($KAlSiO_4$, ICDD 33-988) were blurry. As for 100 mL HCl, only the diffraction peaks of $KAlSi_2O_6$ were detected. Among the four main minerals, pseud-wollastonite ($Ca_3Si_3O_9$) seemed to be the most active to react with acid, while leucite ($KAlSi_2O_6$) was the most inert to react with acid. The reaction progress of gehlenite/akermanite (Ca_2(Mg, Al)(Si, Al)O_7) and kalsilite ($KAlSiO_4$) were modest. In Fig. 4b, the diffraction peaks of Ca_2(Mg, Al)(Si, Al)O_7, $KAlSi_2O_6$, and $KAlSiO_4$ were obvious when the CA volume was increased to 100 mL. Considering that only $KAlSi_2O_6$ was detected after the reaction of 100 mL HCl in Fig. 4a, it can be concluded that further dissolution of Ca_2(Mg, Al)(Si, Al)O_7 and $KAlSiO_4$ could be the reason why more element release was achieved in the case of pH 0.3 HCl.

Fig. 5 displays SEM micrographs of silicate fertilizer and filter residues. The morphology changed greatly after the leaching of pH 0.3 HCl and pH 2.1 CA. It can be seen from Fig. 5(a), the K-containing fertilizer exhibited grains from 5 μm to 20 μm, including plate-like gehlenite grains, hexagonal kalsilite grains and strip-like pseud-wollastonite grains. After the

reaction with 20 mL of pH 2.1 CA or pH 0.3 HCl, the crystal structures were destroyed, leaving some colloidal and flocculating residues as shown in Fig. 5(b) and Fig. 5(d). The microstructure seemed more uniform after the leaching of CA than HCl. The breakdown of original minerals released nutrients into the soil and produced more micropores. After the reaction with 100 mL extractant, some lumps were clearly observed due to aggregation of insoluble grains after the complete dissolution of dissolvable grains. The EDS results showed the big lump denoted by the red spot was leucite in Fig. 5(e). During the dissolution, synthesis of clay minerals occurs.

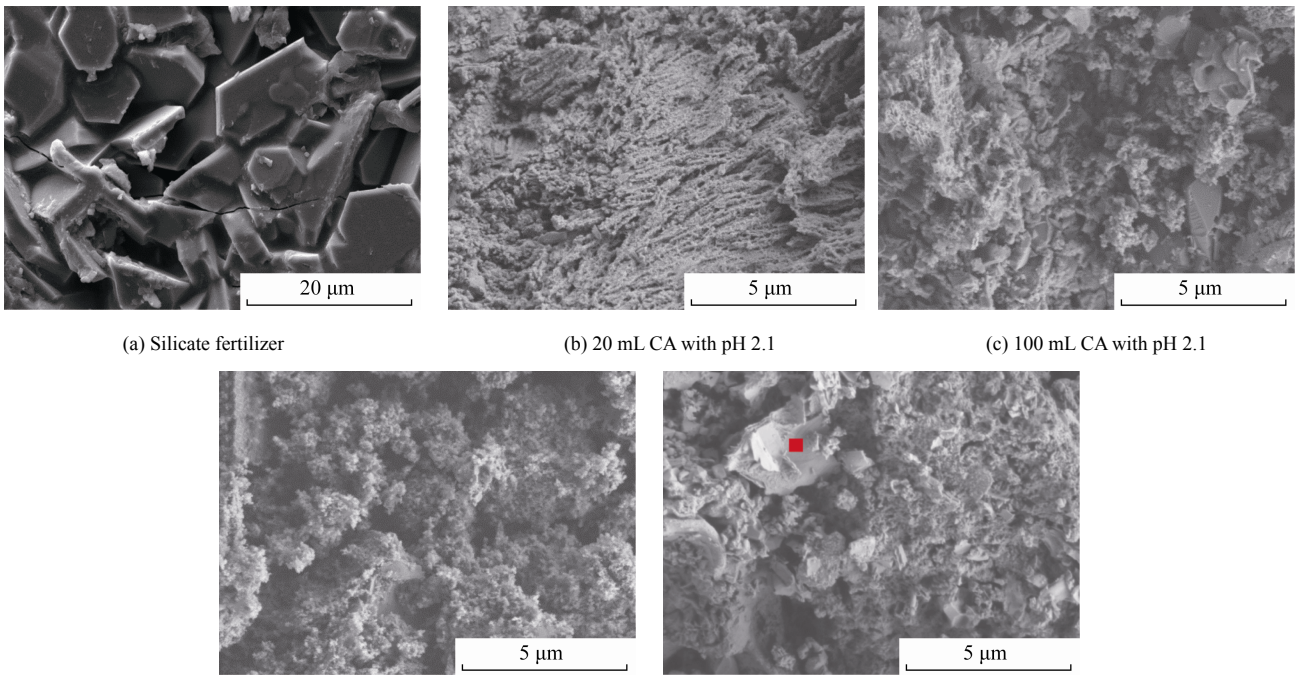
3.3 Dissolution behaviors of K-containing silicate fertilizer induced by the acid

The weathering reactions for leucite ($KAlSi_2O_6$), kalsilite ($KAlSiO_4$) and pseud-wollastonite ($Ca_3Si_3O_9$) are expressed by equations (2) to (4)[16] in Table 3. For the weathering of gehlenite ($Ca_2Al_2SiO_7$) and akermanite ($Ca_2MgSi_2O_7$), it would be reasonable to infer reactions (5) and (6) according to the characteristics of weathering reactions. As shown by the reaction products, kaolinite ($Al_2Si_2O_5(OH)_4$) and gibbsite ($Al_2O_3{\cdot}3H_2O$) appear during the weathering process. Gibbsite is regarded as the stable polymorph at earth surface conditions. With reference to the above reactions, when the K-containing silicate fertilizer discussed here comes into contact with acid, reactions (2)–(6) will occur. In this process, base ions (Ca, Mg and K) are released and monosilicic acid (H_4SiO_4) is formed, which provides beneficial effects in the agronomical practice.



(a) HCl with pH 0.3　　(b) CA with pH 2.1

Fig. 4　XRD patterns of filter residues after the silicate fertilizer dissolved by different volumes of extractant



(a) Silicate fertilizer　　(b) 20 mL CA with pH 2.1　　(c) 100 mL CA with pH 2.1

(d) 20 mL HCl with pH 0.3　　(e) 100 mL HCl with pH 0.3

Fig. 5　SEM micrographs of silicate fertilizer and filter residues

Kaolinite and gibbsite are generally considered to be relatively stable. However, when the system pH is below 5.5, layered aluminosilicate clay minerals (including kaolinite) and aluminum hydroxides begin to dissolve and release hydroxyl aluminum and Al^{3+}[17]. The overall reaction equations of kaolinite and gibbsite dissolution under strong acid condition are shown in reactions (7) and (8)[17–19].

The dissolution rates of kaolinite and gibbsite are strongly related to the solution pH. The research on the kaolinite dissolution in HAc-NH_4Ac buffer solution[20] shows that the release of Al and Si increases with the acidity increase of the reaction solution; Al concentrations slightly increases in the solution at pH 5.5 but significantly increases in the solutions at pH 4.5 and 3.5. The flux of Al from gibbsite into the solution strongly depends on pH in the range of 2 to 6[18]. It has been confirmed that low-molecular-mass organic acids (such as citric and oxalic acids.) have a certain dissolving capacity for kaolinite[21]. When CA was added to HAc-NH_4Ac buffer solution, the concentrations of Al and Si in the reaction solution showed a significant increase[22]. The presence of chloride, silicate, and citrate also affects the dissolution of gibbsite. CA can significantly promote the dissolution of gibbsite, with a large amount of aluminum dissolved at pH value≤6. Dissolved Al was dominated by strong complexation of Al^{3+} with citrate at pH value>3.5 [18].

Table 3 Reaction equations for mineral weathering or mineral reaction with acid

No.	Mineral	Reaction equation
2	$KAlSi_2O_6$	$KAlSi_2O_6+2H^++H_2O\rightarrow 2K^++Al_2Si_2O_5(OH)_4+2SiO_2$
3	$KAlSiO_4$	$KAlSiO_4+2H^++H_2O\rightarrow 2K^++Al_2Si_2O_5(OH)_4$
4	$Ca_3Si_3O_9$	$Ca_3Si_3O_9+6H^+\rightarrow 3Ca^{2+}+3H_2O+3SiO_2$
5	$Ca_2MgSi_2O_7$	$Ca_2MgSi_2O_7+6H^+\rightarrow 2Ca^{2+}+Mg^{2+}+3H_2O+2SiO_2$
6	$Ca_2Al_2SiO_7$	$2Ca_2Al_2SiO_7+8H^++H_2O\rightarrow 4Ca^{2+}+Al_2Si_2O_5(OH)_4+Al_2O_3\cdot 3H_2O$
7	$Al_2Si_2O_5(OH)_4$	$Al_2Si_2O_5(OH)_4+6H^+\rightarrow 2Al^{3+}+2H_4SiO_4+H_2O$
8	$Al_2O_3\cdot 3H_2O$	$Al_2O_3\cdot 3H_2O+6H^+\rightarrow 2Al^{3+}+6H_2O$

It is worth mentioning that the nutrient release of the K-containing silicate fertilizer in the soil is a far more complicated process. Besides the influence of hydrogen ion and citrate ion, the presence of various anions and soil environmental factors (including moisture, temperature, soil texture and so forth) may affect the dissolution behavior of K-containing silicate fertilizers. The present study only focused on the performance under acidic condition, results of which could help the understanding of dissolution behaviors in soils. Moreover, Al-induced toxicity has been a concern since the invention of this silicate fertilizer due to the Al-bearing minerals contained. It is well known that Al toxicity is highly related to the activity of Al^{3+} and hydroxy aluminum in soil solution. Organic acids in soil can promote the dissolution of Al-bearing minerals and the release of Al, which have influences from two aspects. First, the total Al concentration in the soil solution increased. Second, the highly active Al^{3+} and hydroxy aluminum in the solution can react with organic anions and form Al-organic complex with low activity, thus reducing the phytoavailability of Al[23]. Although the role of organic acids is still not fully understood, the increase in pH alleviates Al toxicity, and Si- or Ca-induced amelioration of Al toxicity has been reported [4, 24] after the application of similar products. In addition, Ca^{2+}, Mg^{2+}, and other cations within this silicate fertilizer entering the soil solution also have an impact on Al^{3+}; thus, the toxicity change of Al needs further study.

4 Conclusions

Because of the alkalinity of the K-containing silicate fertilizer, the mineral dissolution and element release appear to be dominated by the hydrogen ion content, the acid strength and organic ligand. The order is as follows: HCl with pH 0.3>CA with pH 2.1>HCl with pH 2.1>HCl with pH 4.0. In the silicate fertilizer reaction with HCl with pH 0.3 and CA with pH 2.1, pseud-wollastonite ($Ca_3Si_3O_9$) is easiest to react with acid, followed by kalsilite ($KAlSiO_4$), gehlenite ($Ca_2Al_2SiO_7$) and akermanite ($Ca_2MgSi_2O_7$), while leucite ($KAlSi_2O_6$) is the most inactive one. The data difference about plant-available elements during the determination, deriving from 2% CA and 0.5 mol/L HCl results from dissolution of gehlenite/akermanite and kalsilite. Although Al ions are released during the fertilizer dissolution in the acidic solution, Al-induced toxicity due to the Al-bearing minerals is a far more complex progress. The stability of kaolinite and gibbsite in the soil solution is the key.

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