Evolution of mineral phases and microstructure of high efficiency Si–Ca–K–Mg fertilizer prepared by water-insoluble K-feldspar

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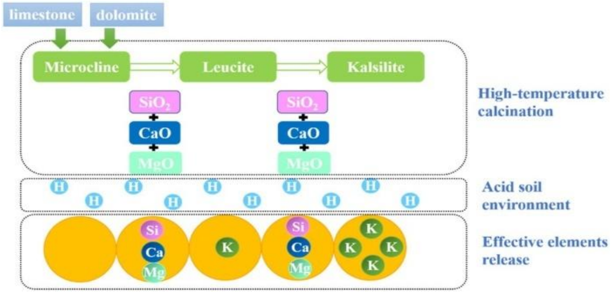
Abstract

Potassium feldspar (K-feldspar) was decomposed with limestone and dolomite as additives to prepare alkaline mineral silicon-calcium-potassium-magnesium fertilizer (abbreviated as Si–Ca–K–Mg fertilizer) via high temperature calcination route. The effects of calcination temperature on the mineral composition, extraction ratios of available effective elements, and the microstructure of powder particles of Si–Ca–K–Mg fertilizer were studied in detail. The results show that the high- efficiency potassium extraction ratio (83%) and silicon extraction ratio (96%) are obtained in the fertilizer calcined at 1250 °C. The reason is that most of the K-feldspar is decomposed into nanoscale kalsilite crystalline grains, which are easily soluble in weak acids. In addition, part of the kalsilite and most leucite melt into amorphous substances. Above 1300 °C, the extraction ratios of potassium and silicon, including calcium and magnesium, are all close to 100%, because the fertilizer particles are basically in an amorphous state. Furthermore, the elements in the Si–Ca–K–Mg fertilizer can be directly absorbed by the crops in the acidic soil. Therefore, this work can promote the comprehensive development of water- insoluble K-feldspar resources and enhance the sustainable use of soil.

Graphical Abstract

Evolution of mineral phases during calcination of potassium feldspar and release of effective elements in Si–Ca–K–Mg fertilizer under acidic environment. First of all, this graphical abstract provides a good description of the evolution of mineral phases during the high-temperature calcination of K-feldspar ore. As shown in the figure, limestone and dolomite are selected as additives and calcined with microcline (major mineral in K-feldspar ore) at high temperatures. At high temperatures, limestone is thermally decomposed into calcium oxide, and dolomite is thermally decomposed into calcium oxide and magnesium oxide. Calcium oxide reacts with microcline, destroying the stable silicate skeleton in the microcline. Thus, the microcline is converted into leucite and silica. With the increase of calcium oxide content, leucite will be further decomposed into kalsilite. At the same time, reactions occur between silica, calcium oxide, and magnesium oxide. In addition, the figure also describes the release performance of effective elements in Si–Ca–K–Mg fertilizer under acid soil environment. Microcline will not be dissolved in weakly acidic environment. Leucite is difficult to be dissolved in weak acids. However, kalsilite is easily dissolved by weak acids. Therefore, kalsilite is the main mineral source of available potassium. Other silicate minerals formed in fertilizers can release silicon, calcium, and magnesium. Therefore, this graphical abstract provides a good description of the main points of this article.



Keywords Potassium feldspar · Kalsilite · Potassium extraction ratio · Amorphous

Highlights

- An High-temperature calcination technology that comprehensively utilizes all effective elements in water-insoluble potassium feldspar.
- Soil conditioner capable of correcting soil pH and passivating heavy metals.
- The high-efficiency potassium extraction ratio (83%) and silicon extraction ratio (96%) are obtained at 1250 °C.
- All effective elements extraction ratios are close to 100% above1300 °C.
- The appearance of nanoscale kalsilite crystalline grains promotes the extraction of potassium.

1 Introduction

Potassium (K), nitrogen (N), and phosphorus (P), as the most important and indispensable elements for plant growth, have been in long-term studied and produced into multiple types of fertilizers [1–3]. It is reported that potas- sium can increase yield by at least 20% for crops in potassium-deficient soils, because it improves crop resis- tance to drought, cold, and pathogen erosion [4, 5]. More than 90% of the world's potassium fertilizer is in the form of traditional water-soluble potassium salts that account for <1% of global potassium reserves [6, 7]. It is noteworthy that the distribution of traditional water-soluble potassium salts is extremely imbalanced around the world [8]. Shel- drick et al.'s investigations show that the global potash supply is lagging far behind the growing demand [9, 10]. Therefore, the searching for alternative sources of potas- sium is critical to the sustainable development of agri- culture. Fortunately, many countries and regions are abundant in water-insoluble K-feldspar resources, which exceeds 10 billion tons [11, 12]. Therefore, many scholars have been studying how to convert it into water-soluble potash in an economical and efficient way since the nine- teenth century [11, 13, 14].

At present, the main approaches for extracting of potassium from K-feldspar can be divided into two cate- gories basically: high-temperature calcination method and static hydrothermal method. For the former method, relying solely on high temperature can't destroy the stable tetra- hedral structure in K-feldspar and extract potassium from them. Generally, it is essential to add one or several alkaline earth metal salts that can form eutectic with K-feldspar at high temperature above 600 °C [7]. Using gypsum and calcium carbonate as additives, Wang et al. achieved a maximum potassium extraction of 90% at 1050 °C [15, 16]. Replace the gypsum with sodium carbonate, Zhao et al. calcined K-feldspar at the temperature range from 1280 to 1330 °C, and then the potassium extraction rate reached 70% using the dissolution route of sodium hydroxide [17]. The static hydrothermal method mainly accelerates the decomposition of K-feldspar with acidic aqueous fluid by increasing the pressure (a few to two hundred atmospheres) at low temperature (100–300 °C) [7, 18], which has higher requirements on the hydrothermal high pressure reactor and lower product conversion rate. In addition, the common solutions that have been widely used include hydrofluoric acid [19, 20], sulfuric acid [21, 22], hydrochloric acid [23], and fluosilicic [24], resulting in environmental pollution. Uniquely, some scholars use microorganisms, mainly fungi, to interact with K-feldspar to release potassium [25–28].

Obviously, among the methods for extracting potassium above, the high-temperature calcination method is a more promising approach. Water-insoluble K-feldspar ore is not only rich in potassium but also contains silicon and essential secondary elements such as calcium and magnesium. Comprehensive utilization of all elements in K-feldspar ore that are benefit for the growth of crops, and meets the concept of sustainable development. Motivated by these ideas, limestone and dolomite as additives are mixed with K-feldspar in a certain proportion, and calcined at a high temperature to produce Si–Ca–K–Mg fertilizer. It not only provides the elements needed for crop growth, but also plays an important role to correct acidic soil and passivates harmful metal elements such as cadmium and chromium as a weakly alkaline silicate fertilizer. Thus, it can also be called as soil conditioner. In addition, Si–Ca–K–Mg ferti- lizer is a citric acid soluble slow release fertilizer that is extremely ideal for tropical soils with low cation exchange capacity [7].

In this study, limestone and dolomite as additives were used to decompose K-feldspar to produce Si–Ca–K–Mg fertilizer via high-temperature calcination route. Si–Ca–K–Mg fertilizer is mainly used in soils that are acidic and lack trace elements. The relationship between the evolution of mineral composition and microstructure and the extraction performance of available elements in Si–Ca–K–Mg fertilizers was systematically investigated by X-ray diffraction, X-ray fluorescence (XRF), and trans- mission electron microscopy. The reaction mechanism during the calcination process was proposed on the basis of experimental results.

2 Experimental section

Materials and methods

The K-feldspar, limestone, and dolomite ore raw materials used in the experiments were mined in Inner Mongolia, China. The ore was crushed and ball milled to a particle size of 10–100 µm. The elemental compositions of raw materials and Si–Ca–K–Mg fertilizer were measured by XRF and the results are listed in Table 1. The K-feldspar ore is mainly composed of microcline ($KAlSi_3O_8$), and little quartz (SiO_2) and albite ($NaAlSi_3O_8$). The main phase of limestone ore is calcite ($CaCO_3$), and the dolomite ore mainly contains dolomite ($CaMg(CO_3)_2$), calcite ($CaCO_3$), and quartz (SiO_2). According to our previous experimental research on the ratio of raw materials and the expected content value of each element in the Si–Ca–K–Mg fertilizer, the raw mate- rials were mixed uniformly in a certain proportion after calculation (45 wt% K-feldspar, 39.33 wt% limestone, and

15.67 wt% dolomite). A certain amount of the mixture was weighed and put into the alumina crucible. The thermal treatment was performed in the box furnace by heating to

desired temperature and holding at this temperature for 1 h. Then the crucible was removed quickly and the fan was used for rapid cooling. The cooled calcined products were milled afterwards to obtain Si–Ca–K–Mg fertilizers.

2.2 Characterization and analysis

XRD patterns of the Si–Ca–K–Mg fertilizers were obtained on X-ray powder diffraction (Smartlab 3 kW, Rigaku, Japan) using Cu Kα radiation (λ = 1.5418 Å) with an accelerating voltage of 30 kV and a tube current of 40 mA. The experiment was carried out at room temperature, and the scanning speed and angle range were set to 2°/min and 10–70°, respectively. The microstructure of the Si–Ca–K–Mg fertilizers was taken from field emission transmission electron microscope using an accelerating voltage of 200 kV (JEOL, JEM-2100F, Japan). The content of available elements is one of the most important criteria for evaluating fertilizer quality. Although the nutrient ele- ments in the fertilizer are released in ionic form, they are conventionally expressed as the form of equivalent weight percent of corresponding oxides when calculating their content [29]. The 0.5 mol/L hydrochloric acid solution was selected as the extraction solution to completely dissolve the fertilizer samples to extract K^+, Si^{4+}, Mg^{2+}, and Ca^{2+}. The operation steps were as follows. (a) Accurately weigh 1 g of fertilizer sample into a 250 mL conical flask. (b) Add 100 mL of 0.5 mol/L hydrochloric acid with a pipette and stir for 60 min. (c) The solution was filtered to collect the filtrate, and the residue was dried. (d) Finally, the content of available K (in terms of K_2O) in the filtrate was measured by the atomic absorption spectrometer (ZEEnit 700 P, Germany). The content of available Si (in terms of SiO_2) in the filtrate was determined by potassium fluorosilicate volumetric method. Available Ca (in terms of CaO) and Mg (in terms of MgO) were detected by EDTA titration. The heat of hydration was measured by a TAM Air thermal conductivity isothermal calorimeter.

3 Results and discussion

The XRD analysis of the unheated Si–Ca–K–Mg fertilizer sample was performed firstly, as shown in Fig. 1. The result shows that this sample is mainly composed of microcline leucite and kalsilite. Under the high temperature condition, the limestone ore is decomposed into calcium oxide (CaO), and a similar thermal reaction occurs in dolomite ore. Calcium oxide can be combined with silica (SiO_2) resolved from K-feldspar or from dolomite ore materials to form wollastonite ($CaSiO_3$, PDF 84-0655). As the calcium oxide content increases, the newly generated leucite will be fur- ther decomposed into kalsilite. Therefore, the calcination reaction process of microcline and calcium oxide can be expressed as Eqs. (1) and (2).

Table 1 Elemental compositions of raw materials and Si–Ca–K–Mg fertilizer (wt%)

Composition	L.O.I	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	Total
K-feldspar	0.42	64.62	17.74	1.18	0.67	0.42	11.86	1.82	98.73
Limestone	43.02	0.96	0.72	0.24	52.94	1.52	0.16	0.01	99.57
Dolomite	30.50	21.53	1.70	0.57	29.02	15.36	0.28	0.28	99.24
Si–Ca–K–Mg fertilizer	–	42.03	10.92	0.91	32.86	4.09	6.97	1.11	98.90

Intensity

$$KAlSi_3O_8 + CaO \rightarrow KAlSi_2O_6 + CaSiO_3 \quad (1)$$

$$KAlSi_3O_8 + 2CaO \rightarrow KAlSiO_4 + 2CaSiO_3 \qquad (2)$$

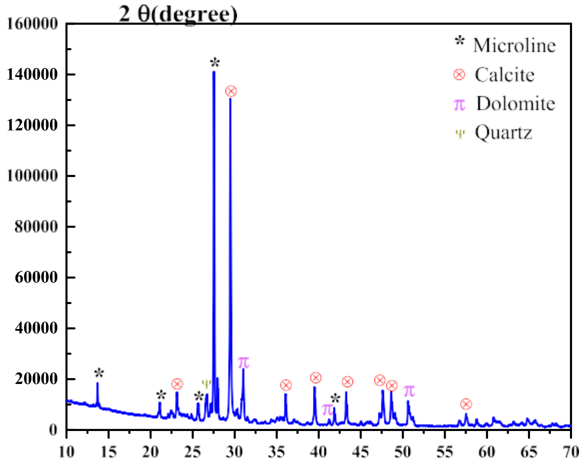


Fig. 1 XRD pattern of the unheated Si–Ca–K–Mg fertilizer

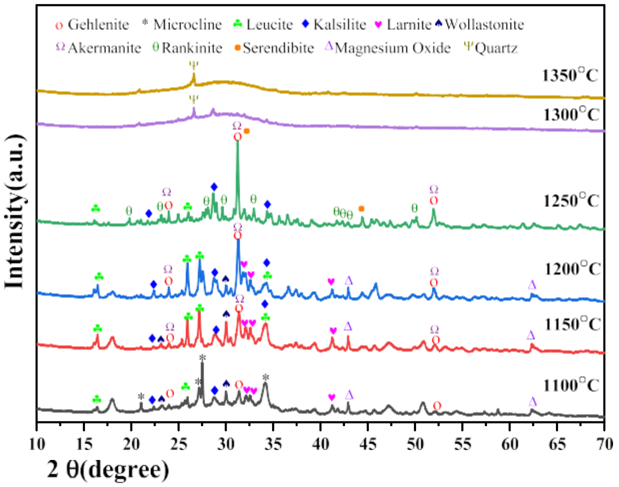


Fig. 2 XRD patterns of the Si–Ca–K–Mg fertilizers calcined at dif- ferent temperatures

($KAlSi_3O_8$, PDF 76-0918), calcite ($CaCO_3$, PDF 99-0022),
dolomite ($CaMg(CO_3)_2$, PDF 99-0046), and some quartz (SiO_2, PDF 79-1906). Therefore, the minerals in the raw materials did not react without being calcined. In order to explore the evolution of mineral composition during the calcination process, the XRD patterns of the Si–Ca–K–Mg fertilizers obtained by calcination at different temperatures range from 1100 to 1350 °C are shown in Fig. 2. XRD analysis results indicate that the main phases of Si–Ca–K–Mg fertilizers, when the calcination temperature is below 1250 °C, are leucite ($KAlSi_2O_6$, PDF 85-1626), kalsilite ($KAlSiO_4$, PDF 85-1413), gehlenite ($Ca_2Al_2SiO_7$, PDF 99-0054), and akermanite ($Ca_2MgSi_2O_7$, PDF 74-0990). The main forms of potassium are leucite and kalsi- lite. At 1100 °C, the presence of high intensity diffraction peaks of microcline ($KAlSi_3O_8$, PDF 76-0918) and weak diffraction peaks of leucite and kalsilite shows that part of silicate framework in the K-feldspar decomposed into

The diffraction peaks' intensity of leucite and kalsilite is significantly enhanced at 1150 °C, while the diffraction peaks of microcline disappear. This finding indicates that microcline is completely decomposed at 1150 °C. The intensity of the diffraction peaks of leucite decreases and the intensity of the diffraction peaks of kalsilite increases at 1200 °C, indicating that the degree of decomposition of microcline into kalsilite is getting stronger. Calcium and magnesium as the other two main elements in the Si–Ca–K–Mg fertilizers mainly exist in the form of gehle- nite and akermanite. Gehlenite has been generated at 1100 °C, while the peaks of akermanite begin to appear at 1150 °C. Therefore, the following two reactions may occur, as shown in Eqs. (3) and (4).

Al_2O_3 þ SiO_2 þ 2CaO ! $Ca_2Al_2SiO_7$ ð3Þ

MgO þ $2SiO_2$ þ 2CaO ! $Ca_2MgSi_2O_7$ ð4Þ

When the temperature rises to 1250 °C, new rnkinite ($Ca_3Si_2O_7$, PDF 76-0623) phase appears, and wollastonite disappears. The XRD pattern shows that the overall inten- sity of diffraction peaks is at a relatively low level at 1250 °C. Especially for leucite, its diffraction peaks' intensity decreases most obviously. It means that the phases begin to melt into amorphous during the calcination pro- cess, resulting the reduction in crystallinity of the fertilizer after cooling. When the temperature continues to rise above 1300 °C, it can be clearly observed that the crystallinity of the Si–Ca–K–Mg fertilizers deteriorates, almost in the form of amorphous substances.

The element content (in the form of oxides) in the Si–Ca–K–Mg fertilizer is 6.97 wt% K_2O, 42.03 wt% SiO_2, 32.86 wt% CaO, and 4.09 wt% MgO (Table 1). Figure 3a, b show the contents of available elements (K, Si, Mg, and Ca) and the corresponding extraction ratios in the Si–Ca–K–Mg fertilizers calcined at different temperatures in the range of

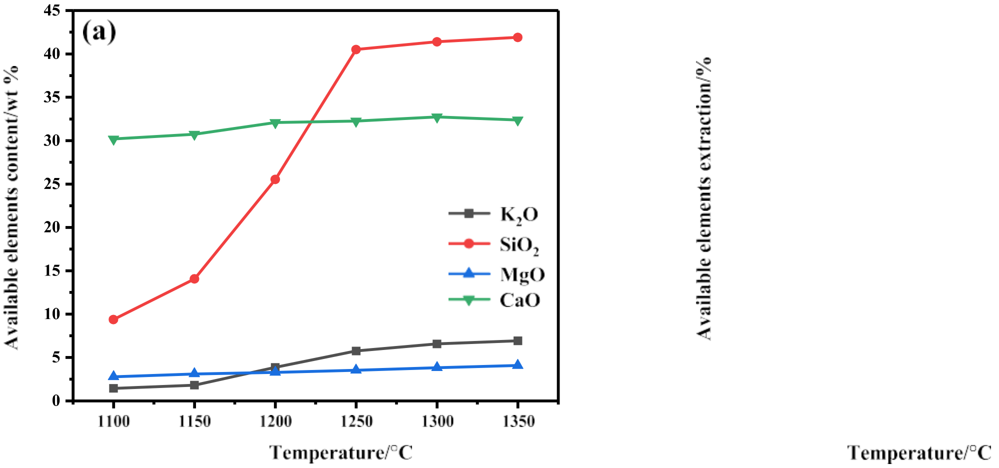


Fig. 3 Effect of calcination temperature on (a) available elements contents and (b) corresponding extraction ratios of the Si–Ca–K–Mg fertilizers

1100–1350 °C. The results suggest that the available K_2O- content increases rapidly and has an increment of 4.31 wt% from 1.43 to 5.74 wt% for the temperature of 1100–1250 °C. With the further increase of temperature, it increases much slowly and reaches 6.91 wt% at 1350 °C, and the corresponding K-extraction ratio is close to 100%. There is a similar growth trend in available SiO_2-content. At 1100 °C, the available SiO_2-content in the Si–Ca–K–Mg fertilizers is 9.37 wt% and the corresponding extraction ratio is only 22.3%. The available SiO_2-content and the corresponding extraction ratio have an increment of

40.51 wt% and 96.38% at 1250 °C, respectively. And the silicon in Si–Ca–K–Mg fertilizers is nearly completely extracted at 1350 °C. As for available magnesium and cal- cium, their extraction ratios have reached a very high level at 1100 °C because limestone and dolomite are easily decomposed into calcium oxide and magnesium oxide after being heated. It can be seen that the curves of their extraction ratios rise steadily as the calcination temperature increases until available calcium and magnesium are fully extracted.

It has been found that the extraction ratio of available K has a large leap at 1200 and 1250 °C (Fig. 3b). In order to explore the reasons, the X-ray diffractions of the residues of the Si–Ca–K–Mg fertilizers calcined at 1100, 1150, 1200, and 1250 °C after 0.5 mol/L hydrochloric acid leaching were carried out as shown in Fig. 4. The result shows that the main phases in the residues are gehlenite, akermanite, and leucite, without the kalsilite phase. This indicates that the kalsilite present in Si–Ca–K–Mg fertilizers is easily dissolved into available potassium in weak acid environ- ment. However, the microcline and leucite are not easily dissolved by weak acid. Manning et al.'s experimental results indicate that the dissolution rate of nepheline is 100 times faster than K-feldspar in a weak acid environment [30]. The basic structural unit of microcline is a tetrahedron as shown in Fig. 5. Each silicon-oxygen tetrahedron or aluminum-oxygen tetrahedron shares an oxygen atom with another tetrahedron to form a three-dimensional skeleton. K^+ only exist in the voids in the framework, playing a role of balancing charge. Therefore, the highly ordered crystal structure of microcline makes it difficult for K to be dissolved by weak acid. Calcination with limestone and dolomite at high temperature causes its crystal lattice to be destroyed and converted into crystals or amorphous sub- stances that are readily soluble in acid. Combined with the previous XRD analysis results in Fig. 2, it can be inferred that more kalsilite is formed in Si–Ca–K–Mg fertilizers with the increase of calcination temperature. Therefore, the extraction ratio of potassium at 1200 and 1250 °C has increased significantly. Gehlenite and akermanite can be slowly dissolved by weak acid.

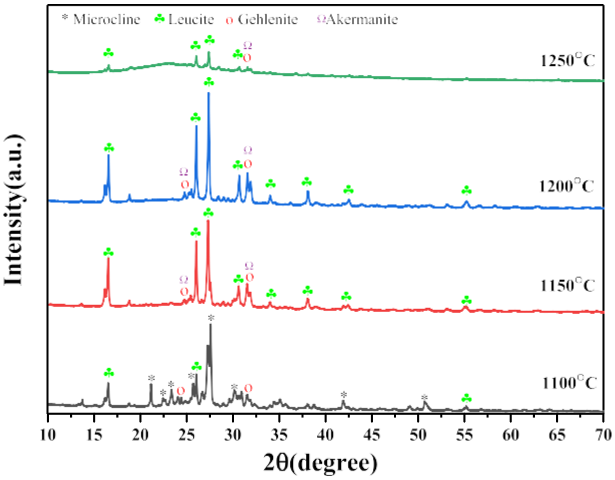
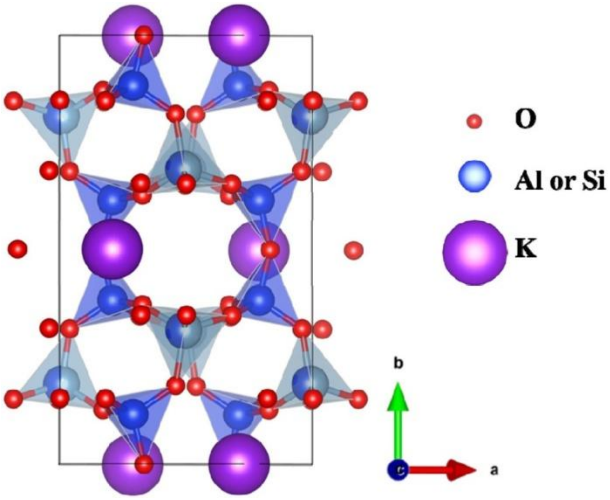


Fig. 4 XRD patterns of the residues of the Si–Ca–K–Mg fertilizers calcined at different temperatures after 0.5 mol/L hydrochloric acid leaching

According to the results of XRD analysis, kalsilite is the main mineral source of available potassium. In order to further determine the phases in the Si–Ca–K–Mg fertilizers and explore the reasons for the significant increase in potassium extraction ratio, the microstructure of fertilizers were analyzed by TEM.

Figure 6a gives the TEM image of the Si–Ca–K–Mg fertilizer calcined at 1200 °C. It indicates the presence of large particle in the fertilizer and the size is about 0.8 µm.



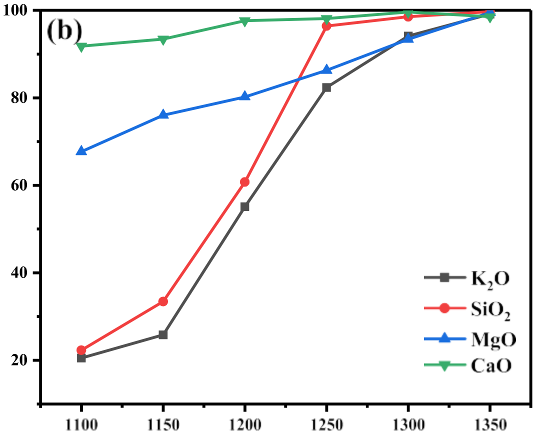


Fig. 5 The crystalline structure of microcline: the red balls represent the oxygen atoms, the blue balls represent the silicon and aluminum atoms in the $[SiO_4]^{4-}$ or $[AlO_4]^{5-}$ tetrahedral, and the purple balls in the tetrahedral void are the potassium cations that balance the charge (color figure online)

The regularity and periodicity of the SAED pattern (Fig. 6b) confirms the intrinsic single-crystalline structure of the particle, and the particle is identified as kalsilite crystal by indexing the SAED pattern. The HRTEM image (Fig. 6c) shows that the crystal structure is intact without crystal defects and lattice distortion. The Si–Ca–K–Mg fertilizer calcined at 1250 °C also contains large particle as shown in Fig. 6d. The corresponding ring diffraction pattern (Fig. 6e) from the particle which are made up of discrete sports indicates that the large particle is formed by agglomeration of nano-grains with random orientations. It is also con- firmed that the nano-grains are kalsilite by indexing the ring diffraction pattern. The microstructure of the crystal was further obtained by HRTEM image as shown in Fig. 6f. It can be observed that the size of the nano-grains is about 10 nm.

Based on the above observations, the grain size of kal- silite in the Si–Ca–K–Mg fertilizer calcined at 1250 °C is much smaller than that at 1200 °C. Nano-grains usually have faster dissolution rate than large crystals under the same weak acid environment. In addition, a small portion of kalsilite and most leucite melts to amorphous substances at 1250 °C (Fig. 2), which is also favorable for potassium extraction. This result further explains the reason why the potassium extraction ratio of the Si–Ca–K–Mg fertilizer calcined at 1250 °C is 28% higher than that at 1200 °C (Fig. 3).

Figure 7 gives the TEM images of the Si–Ca–K–Mg fertilizers calcined at 1300 and 1350 °C, and the corresponding diffraction patterns show the characteristics of

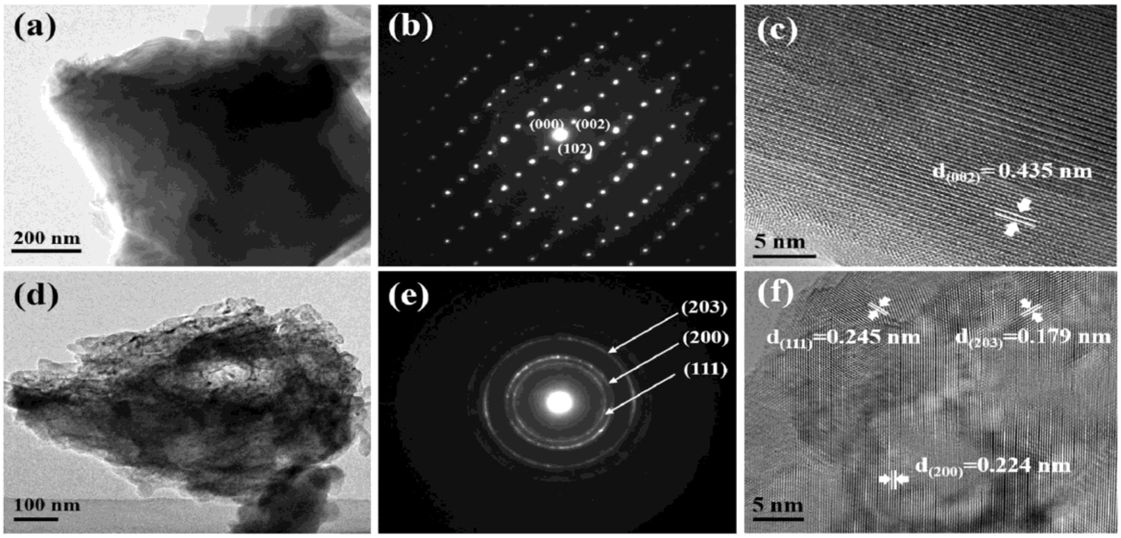


Fig. 6 TEM images of the particles in the Si–Ca–K–Mg fertilizers calcined at (a) 1200 °C and (d) 1250 °C, (b–c) and (e–f) the corresponding SAED patterns and HRTEM images, respectively

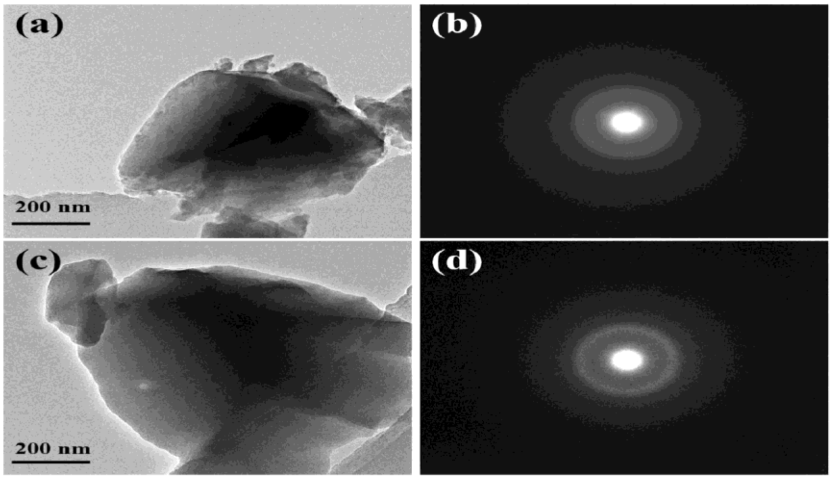


Fig. 7 TEM images of the particles in the Si–Ca–K–Mg fertilizers calcined at (a) 1300 °C and (c) 1350 °C, (b, d) the corresponding SAED pattern

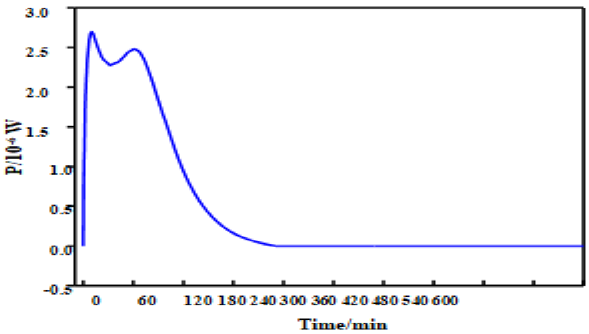


Fig. 8 Thermal power-time curve of hydration of the Si–Ca–K–Mg fertilizer calcined at 1250 °C

dispersion disk. The result indicates that the Si–Ca–K–Mg fertilizers calcined above 1300 °C are basically in the form of amorphous substances, which is consistent with the XRD thermal fluctuations. Therefore, Si–Ca–K–Mg fertilizer does not cause the risk of crops being burned due to the application to the soil.

Conclusions

The high-temperature calcined Si–Ca–K–Mg fertilizer, using water-insoluble potassium feldspar ore, limestone, and dolomite as raw materials, was systematically studied. It was found that the calcination temperature has a great influence on the mineral composition and the microstructure of the fertilizer particles, which finally determine the level of the ratios of available effective elements. Below 1250 °C, Si–Ca–K–Mg fertilizers are mainly composed of kalsilite, analysis results. And the amorphous structural feature is beneficial for the extraction of all elements including potassium.

In order to test whether the possibility of burning seed- lings occurs when Si–Ca–K–Mg fertilizer is applied to the soil, the heat of hydration of the fertilizer calcined at 1250 °C was tracked, which is shown in Fig. 8. The heat of hydration between 0 and 4 h measured by integration is around 16 mJ. After that, the sample hardly generates leucite, gehlenite, and akermanite. With the increase of temperature, the yield of water-soluble kalsilite is gradually increasing, and the size of kalsilite crystals changes from micron to nanometer. At the same time, some leucite transfer into kalsilite, and almost all of the remaining leucite has been melted into amorphous substances at 1250 °C. So the extraction ratios of potassium and silicon of the Si–Ca–K–Mg fertilizer calcined at 1250 °C reach 83% and 96%, respectively. The relatively high extraction ratios of calcium and magnesium are due to the acid solubility of gehlenite and akermanite. Above 1300 °C, the crystalline minerals in the fertilizers are basically in the form of amorphous structures, and almost all effective elements, including Si, Ca, K, Mg, can be extracted in weak acid environment.

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