Preparation and Effectiveness of Slow-release Silicon Fertilizer by Sintering with Iron Ore Tailings

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Recycling of iron ore tailings (IOT) which are by-products of iron ore processing has attracted attention but there have been few studies on their use as fertilizers. In this study, slow-release silicon fertilizers are prepared from iron tailings by solid-phase sintering and characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The solubility results acquired from the optimal sample (Si-IOT) indicate that the available SiO2 is 20.77% and that of available K2O is 18.43% in 0.50 mol L\(^{-1}\) HCl. For 10 g of Si-IOT in 200 mL of distilled water at 25°C, the cumulative release of K2O is 4.32% in the first day and 19.4% in 28 days and that of SiO2 is 2.12% in the first day and 16.30% in 28 days. The performance satisfies the Chinese national standard for slow-release fertilizers and Chinese agricultural standard for silicon fertilizers. The results obtained from Si-IOT pertaining to release of heavy metals indicate that heavy metals will not affect the quality of the crops and pakchoi growth is improved. Hence, they are potential raw materials for slow-release silicon fertilizers. © 2017 American Institute of Chemical Engineers

KEYWORDS: iron tailings, silicon fertilizers, potash fertilizers, slow release

INTRODUCTION

Silicon constitutes the fourth major ingredients of fertilizers besides N, P, and K and is also a major component of soil that is also present in plants. The yield is increased by introducing Si to crops such as rice [1], sugar cane [2], and cucumber [3]. Moreover, the use of silicon fertilizers is known to increase the soil pH, decrease nitrogen leaching by increasing the efficiency of nitrogen use, enhance pollination, and increase the disease resistance [4–6]. Soluble silicates such as Na\(_2\)SiO\(_3\) and K\(_2\)SiO\(_3\) are common industrial chemicals but their applications in agriculture are limited due to cost and other concerns.

Industrial wastes containing SiO\(_2\) are often used as the raw materials to manufacture silicon fertilizers. Although industrial wastes such as steel slags have been investigated as starting materials for production of silicon fertilizers [7–9], the effective silicon content is quite small and subsequent crop yield is not improved obviously. Hence, more improvement is necessary to make it practical. Yoon et al. [10] reported economical synthesis of complex silicon fertilizers using loess with loess processed materials (LPM). LPM was produced by adding 30% NaOH at 1200°C for 1 h. Treatment with a 200-fold diluted LPM solution resulted in increases of 7.8 and 8.3% in the weight and quantity of tomatoes, respectively, and 31.7% in the quantity of cucumbers.

Iron ore tailings (IOT) are a mining and mineral waste produced by the iron ore mining industry [11]. As a by-product of iron ore, the iron tailings production ratio is 1:2.5–3.0 with respect to iron ore [12]. Being the largest crude steel producer in the world, the annual IOT production in China increased from 189 million tons to 839 million tons from 2004 to 2013 [13,14] and the number is increasing thus posing severe environmental threat. The allowable disposal method for iron tailings is outdoor stacking after solidification with curing agents and may cause soil contamination, river and underground water pollution, and other hazards [15]. To satisfy environmental protection and sustainable development, proper utilization of iron tailings is a big issue. The use of iron tailings to produce green and sustainable products such as concrete stabilizers [16], road materials [17], concrete aggregate [18,19], ceramic tiles [20], fired blocks [21], glass-ceramic tiles [22], and geo-polymer bricks [23] has been explored. Moreover, IOTs have been used to treat heavy metal waste water [24,25]. However, there have been few studies on the use of IOTs as fertilizers and soil supplements. IOT contain silicon, magnesium,
calcium, and iron are potential raw fertilizer materials, even though these elements in the form of undissolved silicate minerals cannot be readily absorbed by plants directly. In this study, in order to develop slow-release silicon fertilizers and enhances utilization of IOT; a slow-release silicon fertilizer is prepared with IOT by sintering. The solubility of the slow-release silicon fertilizer based on IOT in 0.50 M HCl and distilled water is also investigated and the nutrient release properties and mechanism are studied. In addition, pot experiments are performed with pakchoi to verify whether the silicon fertilizer promote crop growth.

EXPERIMENTAL DETAILS

Materials

The iron ore tailings were provided by Chende in Hebei Province, China and as shown in Table 1, they contained 39.21% SiO₂, 16.73% CaO, 12.98% MgO, 11.2% Fe₂O₃, 11.11% Al₂O₃ and other ingredients. If the SiO₂, CaO, and MgO in the iron tailings could be converted into soluble substances, they would provide nutrients to plants.

Preparation of Silicon Fertilizer

Appropriate amounts of IOT, K₂CO₃, and KOH were mixed and calcined at a certain temperature and the SiO₂ contents in the samples were determined by ICP-OES. To determine the optimal preparation conditions for the silicon fertilizer, the raw materials content, calcination temperature, and calcination time were studied in details. The optimal sample was labeled Si-IOT.

Characterization

The mineral components and chemical composition were investigated by X-ray fluorescence (XRF, Advant XP) and X-ray diffraction (XRD, Rigaku D/max-rA). Thermal decomposition of the starting materials in the silicon fertilizer was studied by thermo-gravimetric differential thermal analysis (TG, Netzsch TG-209C). Scanning electron microscopy (SEM, JSM-6301F), and micromeritic analysis (ASIQM0002-4, USA) were performed to characterize the components, structure, morphology, charges, and surface area of the samples. The available SiO₂ and available K₂O were determined by inductively-coupled plasma optical emission spectrometry (ICP-OES, ICAP7000SERIES, USA).

Available SiO₂ Test and Release Properties

The available SiO₂ was determined in accordance with the NY/T 2272-2012 soil amendment (Determination of calcium, magnesium and silicon content in China) as follows. 1.0 g of the sample and 150 mL of 0.5 mol L⁻¹ HCl (28–30°C) were added into a 250-mL volumetric flask which was shaken in a digital water bath oscillator for 80 min at 180 rpm. The sample was filtered and measured by ICP-OES.

The nutrients released from the samples were investigated according to the Chinese national standard of slow-release fertilizers (GB 23348-2009). About 10 g of the sample were put into a nylon mesh bag and added to 200 mL of distilled water in a 250-mL volumetric flask. The mixture was kept at 25°C in a biochemical incubator and then sampled and filtered after 24 h, 3 days, 5 days, 7 days, 10 days, 14 days, and 28 days. They were then transferred and diluted with distilled water to 250 mL and the amounts of K₂O and SiO₂ in the filtrate were determined by ICP-OES.

Pot Experiments

“Sijihuagjin” pakchoi seeds obtained from Hefei Feng Seed Industry were planted in flower pots with a diameter of 20 cm and height of 19 cm. The soil obtained from the Beijing Olympic park and its available SiO₂, K₂O, and MgO were 3.50, 4.5, and 0.01%, respectively. About 3 kg of the soil, 0.6435 g urea (CO(NH₂)₂), 0.5735 g KH₂PO₄, and different amounts of the Si-IOT were added to every pot and thoroughly mixed. The dosages of Si-IOT were 0 g kg⁻¹ soil, 1 g kg⁻¹ soil, 2 g kg⁻¹ soil, 4 g kg⁻¹ soil, and 8 g kg⁻¹ soil, respectively, and the flower pots were labeled 0 #, 1 #, 2 #, 3 #, 4 #, 5 #, respectively. The flower pots were kept in a greenhouse at a relative humidity of 60%. Each pot was evenly planted with 15 optimized pakchoi seeds and after 10 days, the germination rate was recorded. During the experiment, they were watered once a week and ventilated. After 40 days, the plant height, root weight, and fresh weight were recorded.

RESULTS AND DISCUSSION

Improvement of Available SiO₂

According to Available SiO₂ Test and Release Properties section, the available SiO₂ in the iron tailings is determined to be only 0.699% and improvement is necessary. This is accomplished by adding alkaline substances at a certain temperature to convert them into soluble silicates. Because K and Ca are essential elements and abundant, K₂CO₃, KOH, Ca(OH)₂, and CaO are used as activators.

The TG-DTG of the mixture of IOT, KOH, and K₂CO₃ shown in Figure 1 reveals three weight losses of 4.5, 2.9, and 12.3% at 141, 515, and 898°C, respectively. The first stage arises from elimination of physically adsorbed water and the second stage represents the melt reaction between KOH and IOT. The third stage stems from decomposition of K₂CO₃ and the melt reaction between K₂CO₃ and IOT. According to TG-DTG, the sintering temperature of IOT; KOH; and K₂CO₃ is between 600 and 1000°C and the sintering temperature in subsequent experiments is set to be 900°C.

Effects of K₂CO₃ Content

Figure 2 shows the available SiO₂ for different mass ratios of iron tailings to K₂CO₃ (4.0:1, 4.0:25, 4.0:5, 4.0:75, 4.1:0, and 4.1:5) at 900°C for 4 h. The available SiO₂ increases from 0.70% to 11.57% when the mass ratios of iron tailings to K₂CO₃ increase from 0 to 4.0:75. The XRD results in Figure 3 show that the increase in the available SiO₂ is due to formation of soluble KAlSiO₄. KAlSiO₄ (PDF, NO 11-0579) is known as a natural or synthetic kalsilite or kaliophilite and crystallized in a hexagonal unit cell with the space group of P63 with cell parameters of a = b = 5.159 A, c = 8.709 A. The strong diffraction lines of kalsilite are at 25.1.27, 2.47, 4.35, 2.22, and 1.57 [26]. It is because the amorphous SiO₂ and Al₂O₃ in IOT reacted with K₂CO₃ at 900°C. The possible chemical reaction equation is shown in Eq. (1):

<table>
<thead>
<tr>
<th>Chemical composition</th>
<th>SiO₂</th>
<th>CaO</th>
<th>MgO</th>
<th>Fe₂O₃</th>
<th>Al₂O₃</th>
<th>TiO₂</th>
<th>P₂O₅</th>
<th>Na₂O</th>
<th>K₂O</th>
<th>LOI</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt %</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>Mineral components</td>
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<td></td>
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<tr>
<td>wt %</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Amphibole</td>
<td>39.21</td>
<td>16.73</td>
<td>12.98</td>
<td>11.2</td>
<td>11.11</td>
<td>5.97</td>
<td>5.97</td>
<td>5.97</td>
<td>0.535</td>
<td>1.46</td>
</tr>
<tr>
<td>Augite</td>
<td>46</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Clinohlore</td>
<td>38</td>
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<td></td>
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</tr>
<tr>
<td>Muscovite</td>
<td>9</td>
<td></td>
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</tr>
</tbody>
</table>

Table 1. Main constituents in the iron tailings.
\[ 2\text{SiO}_2 \text{(amorphous)} + \text{Al}_2\text{O}_3 \text{(amorphous)} + \text{K}_2\text{CO}_3 = 2\text{KAlSiO}_4 + \text{CO}_2 \uparrow. \] (1)

\( \text{KAlSiO}_4 \) has a structure similar to \( \text{K}_2\text{MgSi}_3\text{O}_8 \) which is a conventional slow-release potassium fertilizer [27–30]. Because of the slower dissolution rates, the mineral potassium fertilizers are more efficient than chemical fertilizers for crops. Meanwhile, the available \( \text{SiO}_2 \) content increases because of formation of \( \text{KAlSiO}_4 \). However, when the mass ratio of iron tailings to \( \text{K}_2\text{CO}_3 \) is larger than 4:0.75, the available \( \text{SiO}_2 \) content increases slowly. It is because amorphous \( \text{SiO}_2 \) or amorphous \( \text{Al}_2\text{O}_3 \) reacts completely with \( \text{K}_2\text{CO}_3 \) or other minerals such as amphibole cannot react with \( \text{K}_2\text{CO}_3 \) at 900°C. Therefore, in subsequent experiments, 4:0.75 is chosen as the optimal mass ratio and the available \( \text{SiO}_2 \) reaches 11.57%.

**Effects of Other Activators**

Because the available \( \text{SiO}_2 \) of 11.57% does not meet the Chinese Agricultural standard (NY/T 2272-2012 stating available \( \text{SiO}_2 \geq 20\% \)), improvement is needed. Certain amounts of KOH, Ca(OH)\(_2\), and CaO are added to improve the available \( \text{SiO}_2 \). As shown in Figure 4, the effects of KOH are more pronounced than those of Ca(OH)\(_2\) and CaO and the available \( \text{SiO}_2 \) reaches 20.89% when the mass ratio of iron tailings to KOH is 4:0.4 thus satisfying NY/T 2272-2012. With respect to CaO and Ca(OH)\(_2\), the maximum available \( \text{SiO}_2 \) values are 18.00 and 18.32% when the mass ratio is 4:0.75. The mass ratios continue to increase but the improvement is not obvious. The XRD results in Figure 5 for the IOT modified by KOH, Ca(OH)\(_2\), and CaO at 900°C for 4 h show characteristic peaks of \( \text{KAlSiO}_4 \) being consistent with the changes in the available \( \text{SiO}_2 \). Considering the economic effects and problems in practice, the mass ratio of iron tailings to KOH is chosen to be 4:0.4.

**Effects of Calcination Temperature and Time**

To determine the optimal preparation conditions and minimize cost, the calcination temperature and time are studied. Figure 6a shows the effects of the calcination temperature. The available \( \text{SiO}_2 \) increases from 3.37 to 22.02% as the calcination temperature goes up from 300 to 950°C because a
higher temperature promotes the reaction between $\text{SiO}_2$ and activators. When the calcination temperature is higher than 950°C, the available $\text{SiO}_2$ increase is slower. Figure 7 shows that the sintered sample contains mainly $\text{KAlSiO}_4$ and $\text{CaMgSiO}_4$ (monticellite, PDF NO 35–0590) [31]. Monticellite is slightly soluble in water and releases Ca and Mg to crops. In addition, the shorten calcination time reduces the production cost and so this factor is also studied. As shown in Figure 6b, when the calcination time is 2 h, the available $\text{SiO}_2$ reaches 20.77% meeting NY/T 2272-2012 and 950°C and 2 h are chosen in subsequent experiments. The characteristic peaks of amphibole (PDF 73–1135, $\text{Al}_2\text{Ca}_3\text{Fe}_{4.02}\text{K}_{0.6}\text{Mg}_6\text{Na}_{1.8}\text{O}_{44}(\text{OH})_4$) and augite ((CaMg$_{0.74}\text{Fe}_{0.25}$)Si$_2$O$_6$) become significantly weaker indicating that some amphibole and augite have decomposed and generated the amorphous $\text{SiO}_2$ and $\text{Al}_2\text{O}_3$ [32,33]. The amorphous $\text{SiO}_2$ and $\text{Al}_2\text{O}_3$ react with KOH and the possible chemical reaction is shown in Eq. (2):

$$2\text{SiO}_2\text{(amorphous)} + \text{Al}_2\text{O}_3\text{(amorphous)} + 2\text{KOH} = 2\text{KAlSiO}_4 + \text{H}_2\text{O}. \quad (2)$$

### CHARACTERIZATION

#### XRD

The XRD results of Si-IOT and Si-IOT after 84 days in water are presented in Figure 8. The diffraction peaks of $\text{KAlSiO}_4$ and $\text{CaMgSiO}_4$ after 28 days in water are significantly weaker indicating that $\text{KAlSiO}_4$ and $\text{CaMgSiO}_4$ dissolve or partly dissolve. After 28 days in water, the diffraction peaks corresponding to augite shows that mineral augite cannot dissolve in distilled water in such a short period.

#### SEM

Figure 9a,b disclose that the IOT has an irregular morphology and the micrographs of Si-IOT (Figure 10c,d) show larger particles and smoother granular edge than IOT. Figure 9e,f show the micrographs of Si-IOT after 28 days in distilled water. There are many holes on the surface of particles indicating parts of the particles dissolve into effective nutrients of K, Si, Ca, and Mg.

#### FT-IR

As shown in Figure 10, IOT and Si-IOT exhibit absorption bands at 970, 672, and 460 cm$^{-1}$ assigned to asymmetrical stretching of Si(Al)-O, symmetrical stretching of Si(Al)-O, and...
bending vibration of Si(Al)-O, respectively, as consistent with the FT-IR spectra of KAlSiO$_4$ reported by Su et al. [34] and ABW-type KAlSiO$_4$ reported by Becerro et al. [35]. After 28 days in distilled water, the adsorption peaks weaken indicating the crystal structure of KAlSiO$_4$ decomposes. It further verifies the slow release from Si-IOT enabling prolonged supply of nutrients to plants.

**BET**

The surface area and porosity of IOT, Si-IOT, and Si-IOT after 28 days in water are measured by N$_2$ adsorption isotherm on the micrometric instrument (ASIQM0002–4, USA) and the results are displayed in Table 2. The surface area of Si-IOT is larger than that of IOT and that of Si-IOT after 28 days in water increases further to 99.3 m$^2$ g$^{-1}$. Meanwhile, the porosity of Si-IOT after 28 days in water increases from 7 to 24% corresponding to the SEM results in Figure 9.

**Release Properties and Mechanism**

The chemical composition of the Si-IOT prepared at 900°C for 2 h is listed in Table 3 and the concentrations of SiO$_2$, K$_2$O, CaO, and MgO are 32.04%, 19.56, 13.91, and 10.55%, respectively.

In accordance with the Chinese national standard for slow-release fertilizers (GB 23348-2009), 10 g of Si-IOT are put in a nylon mesh bag and added to 200 mL of distilled water in a 250 mL volumetric flask. After sampling and filtration at time points of 24 h as well as 3, 5, 7, 10, 14, and 28 days, the filtrates are analyzed by ICP-OES and the results are shown in Figure 11. The cumulative release (%) of SiO$_2$ and K$_2$O is calculated based on Eq. (3):

![Figure 8. XRD patterns of (a) Si-IOT and (b) Si-IOT after 28 days in water.](wileyonline-library.com)]

![Figure 9. SEM images: (a, b) Iron ore tailings, (c, d) Si-IOT, and (e, f) Si-IOT after 28 days in water.](wileyonline-library.com)
where i stands for K₂O or SiO₂, \(c_i (\text{g mL}^{-1})\) is the concentration of component i in the filtrate, \(V = 250 \text{ mL}\), the optimal sample is \(m (\text{g})\), and \(w_i (%)\) is the content of component i in the optimal sample.

Figure 11 shows that the cumulative release of K₂O, SiO₂, MgO, and CaO is 1.44%, 2.99%, 0.01%, and 0.02%, respectively in the first day and increases with time reaching 47.18% SiO₂, 61.11% K₂O, 1.7% MgO, and 0.5% CaO, respectively, in 28 days. In 84 days, the cumulative release can reach 52.1% SiO₂, 67.5% K₂O, 2.0% MgO, and 0.7% CaO. As the extension of testing days, the cumulative release of K₂O and SiO₂ increases more slowly than that of K₂O. Conversely, the cumulative release of MgO and CaO is close to equilibrium with little release of MgO and CaO. The reason is that the aqueous solution of Si-IOT is alkaline thus producing Mg and Ca in the form of precipitation. The SiO₂ and K₂O in Si-IOT can dissolve in distilled water and the cumulative release is <80% in 28 days satisfying the Chinese national standard for slow-release fertilizer.

According to the FT-IR results of Si-IOT after 28 days in water, the absorption peaks weaken, especially disappearance of the band at 680 cm⁻¹ related to Si―O―Mg symmetrical stretching, suggesting that the tetrahedral framework Si―O―Al of KAlSiO₄ is broken in the dissolution process. The reaction is Eq. (4):

\[
4\text{KAlSiO}_4 + (n+2) \text{H}_2\text{O} = 4\text{K}^+ + 2\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot n\text{H}_2\text{O} + \text{OH}^-.
\]  

**Heavy Metal Test**

The heavy metal content in solid wastes is an important parameter in practice. Table 4 shows the heavy metal contents determined by ICP-OES. The heavy metal contents of Si-IOT are considerably below the current Chinese fertilizer standard for heavy metals. The heavy metals after 28 days in water and in 0.5 mol L⁻¹ HCl are also measured. Compared with Si-IOT, the heavy metal contents of Si-IOT after 28 days in water and in 0.5 mol L⁻¹ HCl decrease slightly indicate release of small amounts of heavy metals to the solution. Under acidic conditions, the heavy metals are released more easily. Nonetheless, the magnitude of release is very small and should not affect crops.

**Pot Experiments Result**

According to Pot Experiments section, the pH of 0#, 1#, 2#, 3#, 4#, and 5# is measured with CLEAN water quality analyzers (pH 500 pH/TEMP Meter) and shown in Table 5. The initial soil sample is weakly alkaline. After adding Si-IOT, the pH increases to 8.05 when the dose of Si-IOT is 8 g kg⁻¹.
soil. Although the added Si-IOT increases the pH, the change is fairly minor and should not affect the growth of crops. Thus, from the perspective of pH, its application is safe.

The germination rate, plant height, root weight and fresh weight are recorded and presented in Figure 12. The germination rate after applying Si-IOT is larger than those without Si-IOT. The germination rate is 80% for 4 g kg$^{-1}$ Soil of Si-IOT. Compared to the blank, the germination rate increases by 30%. However, the germination rate decreases sharply for a dose larger than 4 g kg$^{-1}$ Soil of Si-IOT. It may create soil salinity and the pakchoi seeds cannot grow well in the soil. The plant height after applying Si-IOT is significantly larger than that without but the increase is not obvious with increasing Si-IOT usage. Meanwhile, the root weight also increases indicating that Si-IOT make the roots stronger and enhances absorption of nutrients from the soil. The fresh weight results show that Si-IOT increases production. When the dosage is 4 g kg$^{-1}$ Soil of Si-IOT, the fresh weight reaches 10.32 g which is about two times that of the unused Si-IOT fresh weight confirming the improved plant growth.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Hg (mg kg$^{-1}$)</th>
<th>As (mg kg$^{-1}$)</th>
<th>Cd (mg kg$^{-1}$)</th>
<th>Pb (mg kg$^{-1}$)</th>
<th>Cr (mg kg$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si-IOT</td>
<td>0.3</td>
<td>10</td>
<td>0.38</td>
<td>7.94</td>
<td>24.6</td>
</tr>
<tr>
<td>GB/T 23349-2009</td>
<td>≤5</td>
<td>≤50</td>
<td>≤10</td>
<td>≤200</td>
<td>≤500</td>
</tr>
<tr>
<td>GB/T 18877-2009</td>
<td>≤5</td>
<td>≤50</td>
<td>≤10</td>
<td>≤150</td>
<td>≤500</td>
</tr>
<tr>
<td>NY/T 525-2012</td>
<td>≤2</td>
<td>≤15</td>
<td>≤3</td>
<td>≤50</td>
<td>≤150</td>
</tr>
<tr>
<td>Si-IOT after 28 days in water</td>
<td>0.28</td>
<td>9.1</td>
<td>0.323</td>
<td>7.3412</td>
<td>23.13</td>
</tr>
<tr>
<td>Si-IOT after 28 days in 0.5 mol/L HCl</td>
<td>0.201</td>
<td>8.2</td>
<td>0.2732</td>
<td>7.01</td>
<td>15.45</td>
</tr>
</tbody>
</table>

Table 5. Effects of Si-IOT on pH of soil samples.

<table>
<thead>
<tr>
<th>Samples</th>
<th>0* (0 g/1 kg soil)</th>
<th>1* (1 g/1 kg soil)</th>
<th>2* (2 g/1 kg soil)</th>
<th>3* (4 g/1 kg soil)</th>
<th>4* (8 g/1 kg soil)</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>7.79</td>
<td>7.80</td>
<td>7.83</td>
<td>7.87</td>
<td>8.05</td>
</tr>
</tbody>
</table>

Figure 12. Results of pot test of pakchoi after applying Si-IOT.
CONCLUSION
The mixture of IOT, K2CO3, and KOH subjected after calcination at 950°C has a kalsilite (KAlSiO4) mineral structure and the available SiO2 increases from 0.70 to 20.77%. The performance satisfies Chinese agricultural standard for silicon fertilizers (available SiO2 ≥ 20%). The slow release experiment in water confirms significant sustained release of IOT and pakchoi grows better as well. The results indicate that IOT has large potential in slow-release fertilizers.

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