**The Journal of Applied Science**
วารสารวิทยาศาสตร์ประยุกต์
ISSN 1513-7805 Printed in Thailand

**Vol. 20 No. 2: 43-51 [2021]**
doi: 10.14416/j.appsci.2021.02.005
ISSN 2586-9663 (Online)

**Research Article**

# The Controlled-release Phosphorus Fertilizer with Silica from Rice Husk

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Received: 09/09/2020; Revised: 04/07/2021; Accepted: 05/10/2021

## Abstract

The current study was conducted with objectives to study the adsorption process of silica from rice husk, to study the phosphorus content before and after its adsorption in silica, to prepare the phosphorus fertilizer pellets, and to study the release of phosphorus of the prepared fertilizers. The product is used as a controlled-release fertilizer. The rice husk after calcined at 900 °C for 6 h. got 12% of the silica, which qualified as absorbent material by the XRD and FT-IR techniques. The porous silica has phosphorus contents at 15.20 g/kg; 1.52% in the raw material. After soaking with 20 ppm $P_2O_5$, the phosphorus was increased to 38.13 g/kg (3.81%). The phosphorous content of the sample was over the organic fertilizer standard. The pellet fertilizers from silica were prepared by compressing the mixture of silica and binder, which can be maintained as the fertilizer granules for up to 30 days in distilled water. The phosphorus in fertilizer powder released up to 79.35% in 648 h while tablets released 48.72% at the same time. The powder phosphorus fertilizer was found to be a standardized controlled-release fertilizer while the pallet fertilizer can be considered an excellent fertilizer as it has high phosphorus content and slow-release capability. It indicates that the mesoporous silica from the rice husk is one of the alternative components to prepare the controlled-release fertilizer.

**Keywords:** Controlled-release fertilizer, Mesoporous, Phosphorus, $SiO_2$, Rice husk

## Introduction

The rapid expansion in world population brings about higher demand in agricultural yield. Therefore, agriculturist needs to increase their productivity. In addition to the requirements for more agrarian produce, technological advancement is also bringing about an increase in health consciousness. Many chemical fertilizer products are inorganic material of wholly or partially synthetic origin that is added to the soil to sustain plant growth. The chemical fertilizers increase the acidity of the soil, reduce the soil's beneficial organism population, and interfere with plant growth because they contain properties such as sulfuric and hydrochloric acids. To solve these environmental problems, organic and non-chemical fertilizers have been widely used as green industrial and agricultural practices get more attention. Organic fertilizer is one of the alternatives to chemical fertilizers for adding nutrients to the soil. It increases soil organic matter, improves

 doi: 10.14416/j.appsci.2021.02.005

soil structure, and reduces soil crusting problems. The growth of plants and their quality is mainly related to the quality of the used fertilizers (Wu and Liu, 2007; Zhang et al., 2013). Many researchers are interested in organic agriculture (Bengtsson et al., 2005; Muller et al*.,* 2017) to achieve sustainable food systems. Organic fertilizers have low crop yields, dissolve rapidly, and are expensive. However, the disadvantages of organic fertilizers were low stability and low nutrient, it could be beneficial when used in an appropriate amount. New technologies to improve fertilizer quality and protect the environments are being developed by such methods as encapsulating the fertilizer by graphene (Zhang et al*.,* 2014), coating them with inorganic materials (Tian et al*.,* 2017; Teixeira et al., 2016; Wu et al*.,* 2008) and/or adsorping them into Zeolite or Silica ($SiO_2$) (Notario del Pino et al., 1995; Cui et al., 2015). These would make controlled-release fertilizer. The controlled-release fertilizer consists of water-soluble fertilizer that was encased within a polymer coating-forming a round pill. The polymer coating has small holes in which water can pass into the pill, solubilizes the fertilizer, and slowly release the fertilizer from the pill. However, these processes may cause polymer residue in the soil.

Phosphorus (P) is an essential nutrient required for plant growth. Although the substance is needed in a very low concentration in soil, it greatly affects the growth processes of plants when there is not enough phosphorus in the planting area. To explain, phosphorus (P) deficiency can adversely affect the growth and development of crops. The leaf and root can be damaged, and a break in overall growth could be caused due to a low concentration of phosphorus (Zhang et al., 2017). A low concentration of phosphorus can be explained by the reactivity of phosphorus and the environment. This results in numerous phosphate ions in the soil which is not beneficial for the growth of plants (Zhang et al., 2017; Zhang et al., 2019; Redel et al., 2019).

Silica ($SiO_2$) is a material used widely in many industries as a catalyst, a polymer filler, an optical device, an absorbent, and a drug deliverer (Cui et al., 2015; Pijarn et al., 2010; Li et al., 2011a, Li et al., 2011b) because it has a lightweight and high surface area. The $SiO_2$ can be used as adsorptive material for supporting and/or improving the stability of the adsorbate. Moreover, the $SiO_2$ is easy to extract by different agricultural materials such as rice husk, corn cob, or sugar cane, etc. (Okoronkwo et al., 2016; Affandi et al., 2009; Deshmukh et al., 2012). Because of the cost, high surface area, and non-toxicity, the capability to decrease the soil acidity (Tavakkoli et al., 2011) and reduce the residues in soil, silica can be considered a promising material for fertilizers.

In this study, we prepared the controlled-release phosphorus fertilizer supported by $SiO_2$, that obtained from rice husk to increase phosphorus and improve fertilizer efficiency with low toxic residues. The $SiO_2$ composite fertilizer can be a promising alternative because it can control phosphorus release and extend the dynamics of the nutrient uptake during the growing season as well as increasing productivity or reducing chemical fertilizer consumption by replacing it with controlled-release fertilizers.

## Experimental Materials

Phosphorus pentoxide ($P_2O_5$) was obtained by Riedel -de Haën. Hydrochloric acid (HCl), Ammonium fluoride ($NH_4F$), Ammonium molybdate ($(NH_4)_6Mo_7O_{24}.4H_2O$) and potassium antimony tartarated ($K_2Sb_2(C_4H_2O_6)_2$) were purchased by Carlo Erba Co., Inc. Sulfuric acid ($H_2SO_4$) was obtained by RCI Labscan limited Co., Inc, and boric acid ($H_3BO_3$) was purchased from Fisher Scientific. All the reagents were analytical grade and were used without further purification. Mesoporous silica was prepared from rice husk collected in the northeastern area of Thailand.

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## Preparation of Silica from the Rice Husk

Mesoporous silica from rice husk was prepared by a modified method from Devarpanah and Kiasat (Davarpanah et al., 2013). The sticky rice husk was washed with distilled water several times and dried at 100 °C for 12 h, then refluxed with 1.0 M sulfuric acid ($H_2SO_4$) for 4 h. The product was washed with distilled water until the pH of the rinse reached 7.0 and dried at 100 °C for 24 h. The product was calcined in air at 900 °C for 6 h. (Muhammad et al., 2016) The product was mesoporous silica.

## Preparation of Phosphorus-Silica Fertilizer

The phosphorus-silica fertilizer was performed by adsorbing $P_2O_5$ into mesoporous silica. The 5 g of mesoporous silica was stirred in 60 mL of 20 ppm $P_2O_5$ solution for 24 h. The sample was filtrated and dried at 60°C for 24 h. The $P_2O_5$ contents before and after adsorption were investigated by a colorimetric method with Brey II reagent extraction (0.03 N HCl and 0.1 N $NH_4F$) (Murphy & Riley, 1962). The phosphorus-silica fertilizer was pelleted by adding the powder sample 1 g in 10 mL of 10% tapioca flour solution boiling for approximately 30 minutes with continuous heat to make the solution volatile. The samples were passed to 8 pellets by a hand mechanic. The fertilizer tablets were dried at 50°C for 6 h.

## Determination of the Fertilizer Release

The controlled-release behaviors of phosphorus-silica fertilizer were investigated by the following methods. In the powder fertilizer release, 1 g of powder was added into round bottles containing 60 ml of distilled water. The bottles were kept and incubated at room temperature, then 2 ml of sampling was collected at 3, 12, 18, and 24 h, and every 3 days to 30 days. The 2 mL of water was carefully injected into the bottles to maintain a constant amount of solvent (Zhang et al., 2014). The tablet fertilizer release applied a similar method. However, 8 tablets of 1 g were used as the sample. Phosphorus content was determined in terms of $P_2O_5$ by the Murphy-Riley method (Murphy & Riley, 1962) in both groups. In detail, the samples at 2 mL were added to the 25 ml volumetric flask. 5 mL of 20% $H_3BO_3$, 2 mL of Murphy's reagent, and 1 mL of 2.5% ascorbic acid were then added and diluted to volume. After 10 minutes, the absorbance was measured by a spectrophotometer at 870 nm. The experiment was repeated three times and the percentage of phosphorus released by the fertilizer is calculated using the following equation where release P is the concentrate of phosphorus that release at the time points (g/Kg of $SiO_2$) and total P is phosphorus content after absorbed P2O5 (38.1 g/Kg of $SiO_2$).

$$\text{percentage P release} = \frac{release\ P}{total\ P}\ x\ 100$$

## Characterization

Powder X-ray diffraction patterns (XRD) were obtained on a Bruker D8 ADVANCE diffractometer using monochromatic CuKα radiation. Diffuse reflectance spectra were recorded on a Perkin-Elmer double beam spectrophotometer. Infrared spectra of the samples were performed on a Perkin-Elmer Spectrum One FT-IR spectrophotometer by the KBr disk method. The scanning electron microscopy (SEM) was obtained from Coax Group Corporation Ltd. (Model TM3030 S/N 145045-05).

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## Results and Discussion

After the rice husk was refluxed with $H_2SO_4$ and calcined in the air at 900 °C, the pinkish-white powder (Figure 1a) with 6.62 percentage of the ash and 12 percentage of silica was obtained. Generally, the white colour of the ash was received in the amorphous silica structure while the punkish tinge was established in a higher crystalline content (tridymite or cristobalite) (Deshmukh et al., 2012). The colour of the product suggests high crystalline and high porous silica were formed. Moreover, the crystalline was relative to the solubility of the silica, and the high crystalline was low solubility. Therefore, the non-reactive $SiO_2$ prepared from the rice husk can be applied to control the fertilizer released. The crystalline $SiO_2$ might be confirmed by XRD. To improve the potential of the phosphorus-silica fertilizer by using the tapioca flour to coordinate the powder sample to form the fertilizer tablet, the powder sample 1 g was formed to 8 pellets showed in Figure 1b. The phosphorus-silica fertilizer tablets were rod-shaped at 1 cm in length and 0.13 g in weight per tablet.

**Figure 1** The powder (a), Fertilizer tablets (b), and Fertilizer tablets after related (c)

The XRD pattern of the sample is shown in Figure 2. The XRD pattern of the $SiO_2$ from the rice husk revealed the three main peaks at 2Ө 20.8°, 21.8°, and 26.6°, which corresponding to the tridymite, cristobalite, and tridymite crystals characteristic, respectively (Deshmukh et al., 2012; Adams et al., 2014). From the XRD pattern of the product indicating the mixture between tridymite and cristobalite, crystals of silica were formed. Usually, the $SiO_2$ can be transformed between the tridymite and cristobalite by heating at over 900 ºC under suitable purity, especially the phosphorous content in the$SiO_2$. From the characterization, it was found that the mesoporous silica that mixes tridymite and cristobalite crystals can be prepared from the sticky rice husk collected in the northeastern area of Thailand. It had been found that the phosphorous content of the fresh rice husk (RH) was 0.7-2.2 g/kg and rice husk ash (RHA) at 0.4-1.9 g/kg (Okon et al., 2005). The material in the current had a high phosphorous content (15.20 g/kg; 1.52%) that consistent with the phosphorous fertilizer application.

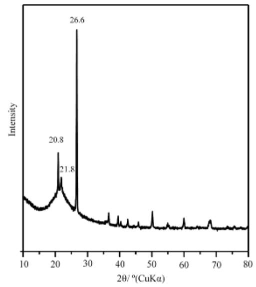
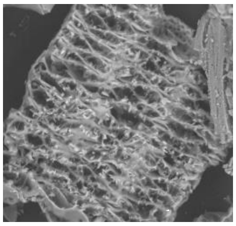



**Figure 2** The XRD pattern of the silica sample

 doi: 10.14416/j.appsci.2021.02.005

The SEM image of silica powder is revealed in Figure 3 The SEM image of silica powder corresponding to the colour of the sample, silica from rice husk revealed the microporous with a diameter at 6–7 μm and length at around 20–25 μm, which leads to an excellent absorbent. The porous structures were highly distributed throughout the rice husks. The material can be applied



to phosphorus adsorption for slow or controlled-release fertilizer.
**Figure 3** The SEM image of silica sample

The functional groups of the extracted silica were identified by FT-IR spectrum as exposed in Figure 4 The FT-IR spectrum of the product revealed the strong absorption peak at 1090 cm$^{-1}$ corresponding to the asymmetric stretching vibration of tetrahedra $SiO_4$ (Cui et al., 2015; Okoronkwo et al., 2016; Attia et al., 2015). The peak at 750 cm$^{-1}$ was designated to the asymmetric stretching vibration of $SiO_4$. It may be associated with structures having a Si/Al ratio close to one (Okoronkwo et al., 2016; Adams et al., 2014). Additionally, the signal at 659 cm$^{-1}$ was associated with a network Si-O-Si symmetric stretching that is characteristic of the crystalline cristobalite, and the band at 480 cm$^{-1}$ was due to network Si-O-Si bending vibration modes (Okoronkwo et al., 2016; Adams et al., 2014). The characterizations of the silica from the rice husk exhibited in the functional groups of silinol and silicates confirmed the formation of crystalline of silica.

The phosphorus-silica fertilizer was performed by absorbing $P_2O_5$ into mesoporous silica. Table 1 indicated that the silica from rice husk had phosphorus in high content (15.20 g/kg; 1.52%). It has been reported that the amount of phosphorus in rice husk ash (RHA) was 0.4-1.9 g/kg (Okon et al., 2005), which acts as reflux for tridymite formation of silica (Shinohara et al., 2004; Haslinawati et al., 2009). The silica in raw material presented the high content of phosphorus that favours tridymite crystallization was formed corresponding to the XRD pattern of the sample. After absorbing $P_2O_5$, the sample presented the phosphorus content at 38.13 g/kg (3.81%). The phosphorous content of silica from the rice husk was over the organic fertilizer standard, which was 0.1-0.4% by weight (Riwandi, 2018). Consequently, phosphorus-silica can be considered as the alternative fertilizer.

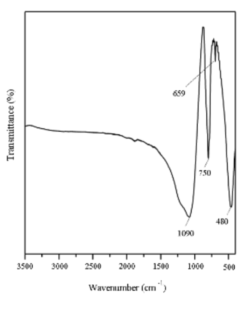



**Figure 4** The FT-IR spectrum of the silica sample

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**Table 1** The phosphorus content in silica from rice husk

| Silica from rice husk | Before absorbed $P_2O_5$ | After absorbed $P_2O_5$ |
|---|---|---|
| phosphorus content ($g/100$ g of $SiO_2$) | 1.52±0.04 | 3.81±0.15 |

The phosphorus release behaviors of fertilizer powder and fertilizer tablets in distilled water at room temperature were revealed in Figure 5. The release of phosphorus in fertilizer powder and tablets were increased with time. The phosphorus in fertilizer powder was released at about 2.03 g/kg (5.31%) after soaking for 3 h and the maximum discharge was 79.35% for 648 h (Table 2). After forming the tablets by using the tapioca flour, the phosphorus fertilizer pellets were still kept in a good shape as they were at the beginning, and some pellets drifted on the distilled water without structural collapse (Figure 1c). The phosphorus was not released from the initial stage to the 18 h; then it was slowly released to 0.85 g/kg (2.20%) at 24 h and maximum expulsion was 18.49 g/kg (48.72%) for 648 h.

It has been reported that the release of potassium was 93.8% after soaking in water for 8 h to encapsulate $KNO_3$ pellets with graphene oxide (Zhang et al., 2014) and was 60% in the soil for potassium silicate (Wu & Liu 2007). The release of phosphorus was 96% after soaking in water for 7 h for phosphorus superabsorbent composite (Liang et al., 2007). The standard of slow-release fertilizers of the Committee of European Normalization (CEN) was the release of nutrients at lower than 15% on the third day and not above 75% on the $30^{th}$ day. The release of phosphorus in fertilizer powder and tablet revealed at 32.50% and 5.45%, respectively on the third day and at 79.35% and 48.72%, respectively on the $30^{th}$ day. It can be claimed that the phosphorus fertilizer powder agreed with the standard, while the phosphorus fertilizer tablet had an excellent slow-release property (Wu & Liu 2007). Although Phosphorus is an indispensable nutrient, it has a low requirement (2.5-5 g/kg), on the other hand, it needs to be released with consistency (Riwandi, 2018). However, phosphorus in general organic or chemical fertilizer was 100% released and decomposed which is not appropriate for plants. Findings show that trend of phosphorus release were decreased at 72 h and increased again after that. It indicates that the phosphorus was adsorbed and desorbed into the mesoporous of the silicas. This observation suggested that phosphorus can be absorbed into the mesoporous silica to collect and decrease solubility as well as increase stability. From this behavior, the fertilizer can be adjusted to the equilibrium of phosphorous after adsorbed by plants to decrease the decomposition of phosphorous and continuously release it for plants. The release of phosphorus fertilizer powder and tablets were slowly consistent; this was good behavior for controlled-release fertilizer. Both phosphorus fertilizer powder and tablet were suitable for application in controlled-release phosphorus fertilizer, which is a non-toxic, low-cost, and locally available product. Moreover, silica can be used to improve the soil by reducing soil salinity. The phosphorus-silica fertilizer samples were a new alternative to eco-friendly fertilizer.

**Table 2** The phosphorus release behaviors of fertilizer powder and fertilizer tablets in water

| **time (hour)** | **The release of phosphorus in water** | | | |
|---|---|---|---|---|
| | **fertilizer powder** | | **fertilizer tablets** | |
| | **g P/kg** | **%** | **g P/kg** | **%** |
| 0 | 0.00 | 0.00 | 0.00 | 0.00 |
| 3 | 2.03±0.19 | 5.31 | 0.00±0.00 | 0.00 |
| 12 | 4.80±0.20 | 12.60 | 0.00±0.00 | 0.00 |
| 18 | 7.53±0.50 | 19.88 | 0.00±0.00 | 0.00 |
| 24 | 9.97±0.50 | 26.18 | 0.85±0.35 | 2.20 |
| 72 | 12.37±1.21 | 32.50 | 2.07±0.37 | 5.45 |
| 144 | 14.90±0.36 | 39.31 | 4.21±0.71 | 11.04 |
| 216 | 17.10±0.85 | 45.06 | 6.70±0.79 | 17.68 |
| 288 | 19.20±0.80 | 50.45 | 8.10±0.90 | 21.24 |
| 360 | 21.53±1.14 | 56.42 | 10.27±1.25 | 26.85 |
| 432 | 23.93±1.30 | 62.81 | 12.08±1.10 | 31.59 |
| 504 | 26.33±1.30 | 69.11 | 14.93±1.70 | 39.35 |
| 576 | 28.57±0.85 | 75.02 | 16.39±1.30 | 42.93 |
| 648 | 30.13±0.35 | 79.09 | 18.49±1.50 | 48.46 |
| 720 | 30.20±0.72 | 79.35 | 18.59±1.40 | 48.72 |

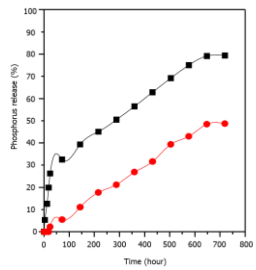


**Figure 5** The phosphorus release behaviors of fertilizer powder (■) and fertilizer tablets (●) in water

## Conclusions

The phosphorus-silica fertilizer was successfully prepared by absorbing $P_2O_5$ into mesoporous silica from the rice husk. The phosphorous content of the sample was 38.13 g/kg (3.81%) which is over the organic fertilizer standard. The phosphorus in fertilizer powder and tablets were maximum discharge at 79.35% and 48.72% for 648 h, respectively. The pellets were kept in a good shape for up to 30 days. The releases of phosphorus fertilizer powder and tablets were slowly consistent. The phosphorus fertilizer powder was standardized, while the tablet was excellent as controlled-release fertilizers. Both phosphorus-silica fertilizer powder and tablet were suitable for application in controlled-release phosphorus fertilizer, which is a non-toxic, low-cost, and locally available product.

## Acknowledgments

This research was supported by the Research and Development Institute, Rajabhat Maha Sarakham University, and thank you for the Faculty of Science and Technology at Rajabhat Maha Sarakham University.

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