Synthesis of Potassium Silicate Nanoparticles from Rice Straw Ash Using a Flame-assisted Spray-pyrolysis Method

A B D Nandiyanto1,2, N Permatasari1, T N Sucahya1, A G Abdullah2 and L Hasanah3

1Departemen Kimia, Universitas Pendidikan Indonesia, Jl. Dr. Setiabudi no 229, Bandung 40154, Jawa Barat, Indonesia
2Departemen Pendidikan Teknik Elektro, Universitas Pendidikan Indonesia, Jl. Dr. Setiabudi no 229, Bandung 40154, Jawa Barat, Indonesia
3Departemen Fisika, Universitas Pendidikan Indonesia, Jl. Dr. Setiabudi no 229, Bandung 40154, Jawa Barat, Indonesia

*nandiyanto@upi.edu

Abstract. The purpose of this study was to synthesize potassium silicate nanoparticles from rice straw ash using a flame-assisted spray-pyrolysis method. Rice straw, as one of the agricultural wastes, was used as a source of silica. In the experimental procedure, rice straw was burned at 700°C for 3 hours to produce rice straw ash. Then, the rice straw ash was extracted using an alkaline method. We used potassium hydroxide (KOH) as an alkaline chemical agent as well as a source of potassium. The solution was then put into the flame-assisted spray-pyrolysis apparatus to produce potassium silicate nanoparticles. The results showed that the spray method can assist the production of spherical potassium silicate nanoparticles with sizes of about 50 nm.

1. Introduction
Potassium silicate has been used in agriculture as additional nutrients for plants [1, 2, 3]. Utilization of potassium silicate in agriculture as a fertilizer is potential for increasing amounts of sugars and amino acids in plants. Thus, plants have a strong resistance to insect pests and diseases, as well as increased quality in terms of taste, shape, and color [1]. This is due to nature of potassium silicate as fertilizer has proven to high stability, chemical flexibility, and biocompatibility, which are crucial for a wide scope [4].

To obtained potassium silicate material, many researchers used commercial precursors, such as fume silica, TEOS (Tetraethylorthosilicate), and colloidal silica [5]. However, the use of these commercial precursors as a source of silica is impractical since the price of these chemicals is expensive and it is harmful to health and environment [6, 7]. Thus, finding a new source of silica that is cheaper, safer, and environmentally friendly is crucial [4, 7].

Rice straw waste is one of the abundant agricultural wastes, while some researchers found that the silica content in the ash of rice straw is quite high [8, 9, 10, 11]. This makes it potential as a source of silica. Further, silica from rice straw waste is abundant and relatively cheap, compared with commercial precursor. Therefore, rice straw waste can be used as an alternative raw material for fertilizer, which is cheap, environmentally friendly, and its availability is abundant in Indonesia.

Silica from rice straw waste is not fully utilized until now. Most of the existing researches applied the silica derived from agricultural waste as a catalyst [12, 13, 14, 15, 16]. Therefore, this study aims to synthesize potassium silicate nanoparticles from rice straw waste using a flame-assisted spray-pyrolysis
method. The result showed that the present method is effective to assist the synthesis of spherical potassium silicate nanoparticles with sizes of 50 nm, which is potential to be used for fertilizer.

2. Experimental Method

2.1. Raw material
The following raw materials were used: rice straw waste (rice field in Cimahi, Indonesia), potassium hydroxide (KOH, PT. Bratachem, Indonesia), and phosphoric acid (H₃PO₄, PT. Bratachem, Indonesia). Rice straw was washed with ion-exchanged water, dried naturally for 7 days, and then cut to get sizes of about 1 cm.

2.2. Synthesis of potassium silicate particles
Rice straw was heated in furnace at 973 K to produce rice straw ash. Then, the rice straw ash was grinded and put into the potassium hydroxide solution. The mixture was then stirred at 900 rpm and heated at 338K for 2 hours. Next, the extraction solution was put into the flame-assisted spray-pyrolysis apparatus (equipped with commercial liquid petroleum gas (LPG; Pertamina) and flow of air to introduce droplet into flame apparatus).

2.3. Characterization
Functional groups, morphology, and particle size of potassium silicate particles were analyzed using a Transmission Electron Microscopy (TEM) and a Fourier Transform Infrared (FTIR).

3. Results and Discussion
Figure 1a shows the mechanism illustration of particle formation during the flame-assisted spray pyrolysis process. As shown in this figure, the droplets were dried into the flame apparatus and transform into particle via nucleation and growth process. Figure 1b shows the photograph image of the flame profile during the process. This figure confirmed that during the process, the particle was dried by the assistance of yellow flame. Figure 1c is the profile of temperature inside the reactor. From Figure 1c, the maximum temperature of the flame using an LPG gas flow rate of 0.90 L/minute, gas carrier flow rate of 0.60 L/min, and air gas flow rate of 1.50 L/minute is more than 700 °C.
Figure 1. Schematic illustration of particle formation mechanism (a), appearance of flame burner (b), and temperature profile (c).

Figure 2a shows the TEM analysis results of the particles prepared by the flame-assisted spray pyrolysis method. The image showed that the potassium silicate particles have a spherical morphology with sizes of less than 100 nm. The high magnification image (displayed in the attached figure) shows that the particle contained smaller dots, in which this is possible due to embedded kalium component in the particle.

To confirm the chemical composition of particles prepared by the spray method, FTIR analysis was conducted (Figure 2b). The FTIR spectra showed absorption peaks at 453.27 cm\(^{-1}\), denoting the Si-O-Si symmetric bending. Peak at 669.30 cm\(^{-1}\) was detected, informing Si-H functional group. The peaks at 806.25, 883.40, 1058.92, and 3194 cm\(^{-1}\) ascribed to the existence of Si-O-Si symmetric stretching, Si-OH, Si-O-Si asymmetric stretching, and SiO-H, respectively. The band located at 1128.36 and 1662.64 cm\(^{-1}\) belonged to Si-O-X (X = K, Si, or H) stretching and \(K_2SiO_3\), respectively.
4. Conclusions
Potassium silicate particles have been synthesized from rice straw using the flame-assisted spray-pyrolysis method. The results showed that the spray method can assist the production of spherical potassium silicate nanoparticles with sizes of about 50 nm.

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