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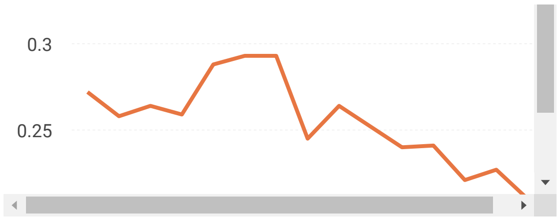

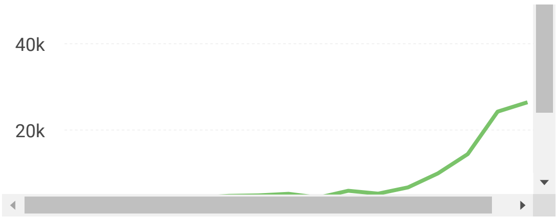

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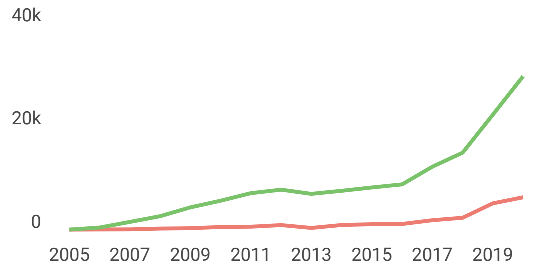

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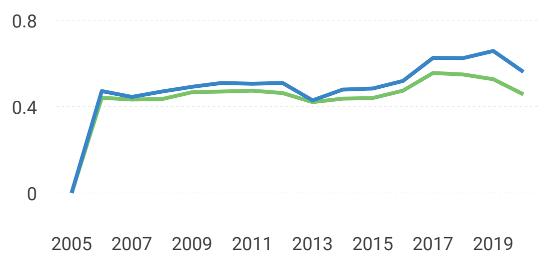

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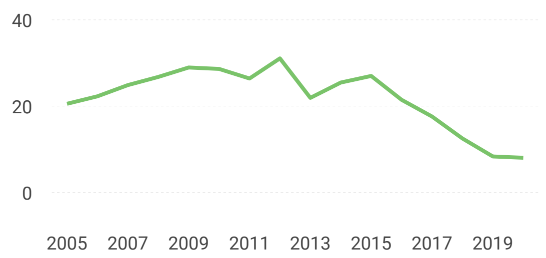

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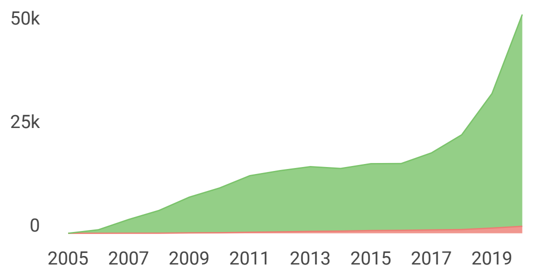

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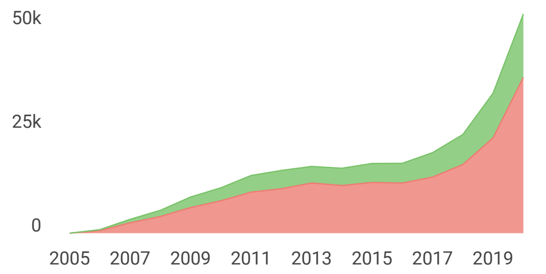

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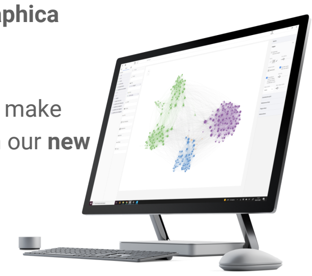

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Facile synthesis of high purity silica xerogel from rice straw

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Facile synthesis of high purity silica xerogel from rice straw

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Abstract. This research aims to synthesize a silica xerogel from rice straw that is a residue biomass generating from agriculture. Purity and morphological structure of synthesized silica xerogel are also studied. The first step of the synthesis is the preparation of sodium silicate from rice straw ash that is then used as silica source. To prepare a silica source, pretreated rice straw was burnt at 700 °C for 2 h to obtain a rice straw ash. After that resulted rice straw ash is washed and reacted with 1.0 M HCl and 2.0 M NaOH aqueous solution at 80 °C for 1 h, respectively. The reacted solution is then filtrated two times by a no.41 filter paper and ion exchange resin, respectively. The obtained sodium silicate is mixed with 1.0 M HCl under stirring for 6 h to produce the nano-silica. To increase the purity of nano-silica xerogels, as-synthesized silica was washed with deionized water for 3 times. Washed silica is dried in electric oven at 85 °C overnight and calcined at 500 °C for 5 h. Scanning electron microscopy/energy dispersive x-ray spectrometry (SEM/EDS) and % whiteness are employed to evaluate the morphology and purity of particles. Experimental results showed that nano-silica with purity up to 99.0 wt% was completely synthesized. Different morphological structure of silica synthesized under pH of 7, 8 and 9 were obtained.

1. Introduction

Nowadays, farmers in Thailand produce large amount of agricultural biomass residue such as sugarcane straw, maize straw and rice straw. Some of these residues are used as fuel in the power plant and as a raw materials in organic fertilizer production. However, much of the rest biomass residues are eliminated by burning in the fields. This burning of biomass residues is one of the most important cause of PM. 2.5 issue and others air pollution. One of the most important ways to overcome this problem is the reduction of inefficient biomass residues burning. Utilization of biomass residues to produce a high value added products likes activated carbon and silica nanoparticles, is the other ways to mitigate this problem. From literatures, silica is one of the composition of ash generating from biomass such as rice husk, rice straw and maize straw. In case of rice straw, it was found that a silica content is about 14 and 82 wt% for rice straw and rice straw ash (RSA) [1,2], respectively. In addition, content of silica increase up to 91.0 wt% by pretreatment of rice straw with acid solution [3]. Many research groups pay attention to synthesis the silica gels due to their show promising applications in a new range of new technologies such as functional nanomaterials, nanoelectronics, environmental treatment and energy storage [4]. Generally, the produced silica gel is classified into three types depending on the amount of water in the silica gel structure that is (1) Aqua-gel (rich of water gel) (2) Xerogel (drying gel) (3) Aerogel (super-critical drying gel) [5]. The preparation of silica xerogel from the ash of wheat requires the preparation of sodium silicate as the starting material of silica, and then the preparation of gel by reducing the state of acidity to the surface of the silica. Before being neutralized by salt titrate, the results of this process were found to be highly effective in improving the purity of gel. In this study, we focused on the easily, low energy and low cost method to synthesize a silica xerogel from rice straw.

In this work, experimental results from sintering of silica xerogel extracted from rice straw ash was presented. Influence of gelation pH on the morphological structure of the produced silica xerogel was studied through the micrograph from Scanning Electron Microscope (SEM). The study hope to increase the value of agricultural waste and expecting to extract silica from rice straw ash with purity up to 95%. This research is also interested to studying the physical structure and purity of silica xerogels by physical techniques.

2. Materials and methods

Hydrochloric acid (HCl, 37%) and sodium hydroxide in the pellet forms (NaOH, 98.0 % purity) were purchased from RCI LabScan and Sigma-Aldrich, respectively. Ashless filter papers no. 41 (Whatman™) were supplied from GE Healthcare Life Sciences.

In this research, rice straw was divided into two groups. The first group was just washed by tap water and RO-water, respectively while the second group of rice straw was washed by pure water for three times and treated with 0.1 M HCl at 80 °C for 1 h, respectively. After treated with HCl, 2nd group of rice straw was washed again for three times with pure water before drying at 90 °C for 15 h in electric oven. To obtain a rice straw ash (RSA), pretreated rice straw was then burnt at 700 °C for 2 h.

To prepare sodium silicate to be used as silica source from RSA, 20 g of RSA was treated with 100 ml of 1.0 M HCl and reacted with 60 ml of 2.0 M NaOH at 80 °C for 1 h. Resultant mixture was filtrated two times by ashless filter papers (No.41) and strong acid ion exchange resin, respectively. Afterwards, sodium silicate was tritrated with 1.0 M HCl to adjust a pH to 7, 8 and 9. After the adjusted pH mixtures were placed at room temperature for 18 h, phase of mixture change from liquid form to gel form. Gel was then exploded and washed with pure water for 3 times and dried at 85 °C for 12 h, respectively. These processes can increase the purity of produced-gel from biomass residue [3,6]. Steps of preparation of silica xerogel from RSA was illustrated in figure 1. Morphological structure and purity in term of % whiteness of synthesized silica gels were characterized by SEM and spectrophotometer, respectively.

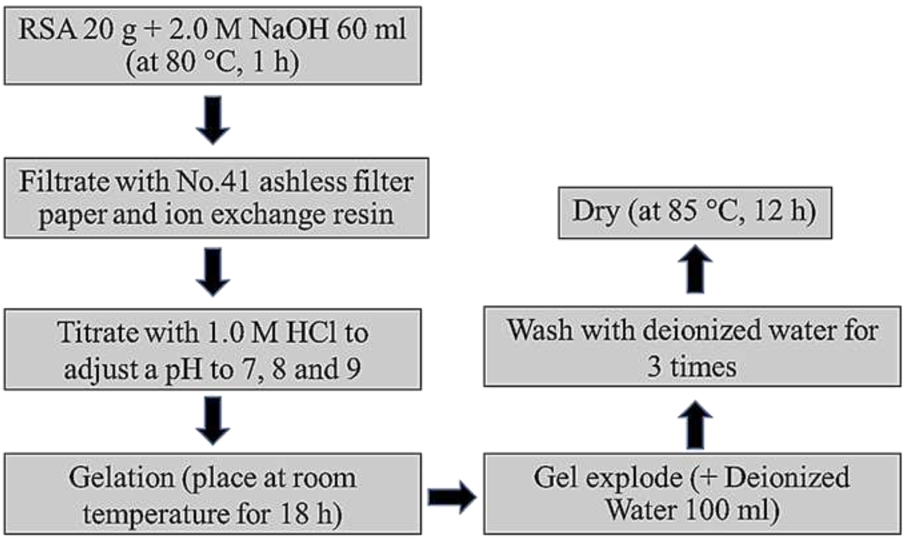

Figure 1. Synthesis process of silica xerogels from RSA.

3. Results and discussion

Figure 2 illustrates the difference of rice straw ash obtained from rice straw. It was showed that the color of ash influenced from the acid pre-treatment process of rice straw. The color of ash from rice straw washed with 0.1 M HCl is quite white. The study of SEM micrograph of silica xerogel that is synthesized from sodium silicate preparing from RSA by controlling the level of pH at 7, 8 and 9 as shown in figure 3 found that silica xerogels have the characteristics of a mixture of porous and dense gels or amorphous. A porous and dense gels were indicated by a red and yellow arrows, respectively. SEM micrographs at the magnification of 10,000x and 20,000x, it clearly show the difference of morphological structure of silica xerogels synthesized at pH 7, 8 and 9. Porous structure more appeared with increase of pH.

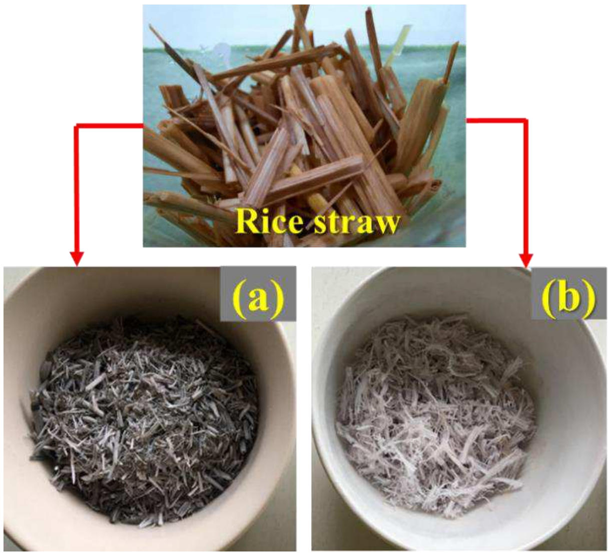

Figure 2. Color of rice straw ash from rice straw, (a) un-treated and (b) treated, by 0.1 M HCl.

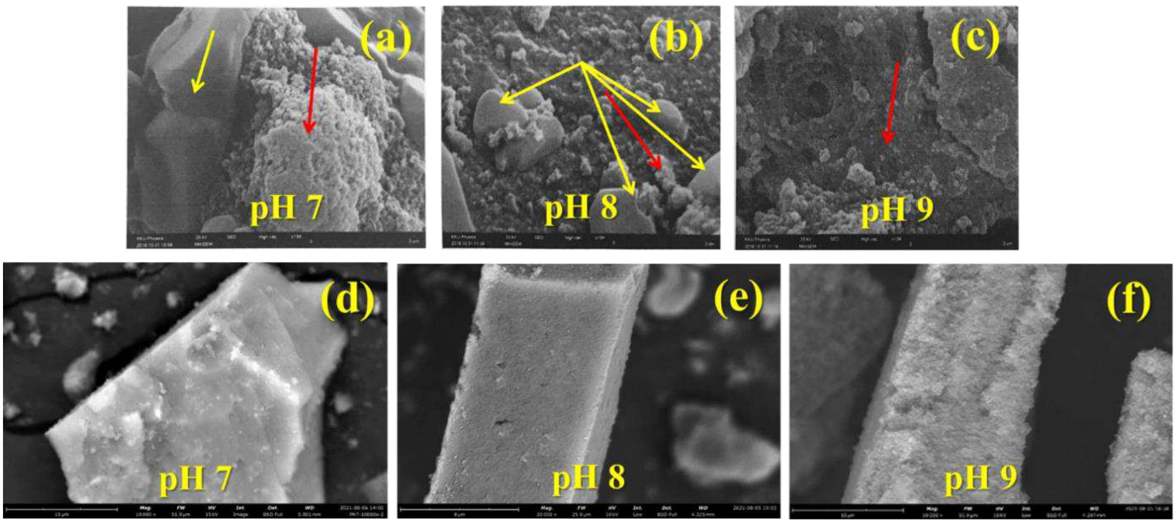

Figure 3. SEM micrograph of silica xerogels synthesized at (a,d) pH 7, (b,e) pH 8 and (c,f) pH 9.

As shown in figure 3, particle sizes of produced gel is varied from nanometers to micrometers. Silica xerogel which is prepared at pH 7 have more amorphous than silica prepared at pH of 8 and 9 due to the high solubility properties of amorphous silica in basic conditions. Silica prepared at pH 9 are almost all particles. It can be said that when pH is increased the resulting silica xerogel has a lower amorphousness. Additionally, SEM micrograph confirmed that particle size of silica xerogels smaller than 0.3 μm and the particle size distribution is fairly uniform. In this research, the synthesized silica was also subjected to color measuring to estimate the purity. Generally, if the color of the synthesized material has a high percentage of white this implies that the material is high purity [7]. From the color measuring as shown in table 1, it was found that the % whiteness of silica xerogel is quite a lot; 76.69, 81.25 and 79.69 for silica xerogel synthesized at pH of 7, 8 and 9, respectively. Comparing the silica xerogels prepared at pH 7, 8 and 9, found silica xerogel which prepared at pH 8 have higher % whiteness than the xerogel prepared at pH 7 and 9. It can be said that silica xerogels prepared in this research. relatively high purity. From literature, percent of silicon dioxide in the rice husk ash that produced from acid treated rice husk and have a % whiteness in the range of 70-75 is about 95.0-99.5 [7]. Additionally, results from EDS confirmed the purity of resulted xerogels.

Table 1. % whiteness of silica xerogels measured by ColorFlex EZ.

Name of substance	L* (% whiteness)
Silica xerogel (pH 7)	76.69
Silica xerogel (pH 8)	81.25
Silica xerogel (pH 9)	79.69

4. Conclusion

In summary, RSA prepared from acid treated rice straw is whiter than RSA prepared from untreated rice straw. Generally if the color of the ashes from the straw is very white, it means that straw ash contains a lot of silica. Amorphous silica was completely synthesized in this research. Amorphousness of produced silica xerogels decrease with increase of pH level. Percent whiteness of produced gels is quite high and it can conclude that silica xerogels synthesized in this research relatively high purity.

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