Extraction of Silica from Rice Straw Using Alkaline Hydrolysis Pretreatment

Ayu Haslija Abu Bakar*,1, Chong Jia Ni Carey1

1Department of Chemical and Petroleum Engineering, Faculty of Engineering, Technology and Built Environment, UCSI University, 56000 Cheras, Kuala Lumpur, Malaysia

ayuhaslija@gmail.com

Abstract. Rice straw is an abundant resource in Asian countries, for instance, Malaysia and India. Rice straw is a major agricultural waste product, which caused disposal issue in areas. Residue burning crops was adopted for the farmers to dispose of rice straw after harvest in order to prepare seedbed preparation for next season. An alarming air pollution report impacted by open burning of rice straw contributes to global warming. Commercialization of silica is an alternative method to dispose of agro-waste residues. The main objective of this research is to extract silica from rice straw. The amorphous silica was derived by first digesting rice straw with Soda AQ solution by varying NaOH concentration from 5 wt% - 25 wt% and 0.1 wt% anthraquinone (AQ) to dissolve silica content and acid precipitation at pH 7 to obtain silica gel. Other parameters such as the effect of digestion temperature (60 °C to 90 °C), digestion time (1 hour to 3 hours), and Soda AQ to rice straw impregnation phase ratio (150:5 to 500:3). The optimum effects of parameter had yielded 74.11 % of silica and it was demonstrated by Soda AQ with NaOH concentration 20 wt% and 0.1 wt% AQ, temperature of 75 °C, digestion time of 3 hours and the ratio 200:5 of Soda AQ to rice straw. Characterization of rice straw was performed by using Fourier Transforms Infrared Spectroscopy (FTIR). The commercialization of silica had the potential to be utilized as construction materials and silica gel that aids to absorb moisture.

1. Introduction
Residue burning crops is incomplete combustion process that liberates greenhouse gases such as carbon dioxide (CO2), methane (CH4), nitrous oxide (N2O), air pollutants (NOx, CO, fine dust) and toxic pollutants which have been reported for air pollution [1]. Particularly in Asian countries, current practice to overcome the agricultural residues is field burning of rice straw. Nevertheless, releasing a large amount of NOx to the atmosphere can lead to photochemical smog and PM2.5 (fine dust). Smoke generated from biomass combustion is which released into the air is toxic. These pollutants dissipate to the atmosphere may undergo the chemical and physical transformation and eventually adversely affect human health, for example, acute respiratory infection [2]. Rice straw burning has been linked to increased asthma attacks in children in a prefecture in Japan [3]. Rice straw burning also dwindles a large portion of soil nutrients, around 80 % of nitrogen, 25 % of phosphorus, 21 % of potassium and organic matter [4]. Not only that, the soil fertility would be affected by this practice when most of the beneficial insects and microorganisms in the soil are killed.

Rice straw is a biological waste with abundant content (10 to 15 %) of silica that can be applied as an alternative source of bio-nano-silica. Accumulation of silica in rice straw via polymerization of the water-soluble silicic acid (H4SiO4) absorbed from the soil into insoluble polysilicic acids, followed by precipitation as amorphous silica and deposition in exterior plant cell walls [5]. Hence, there is a large amount of silica can be extracted from rice straw by various types of extraction process for delignification of rice straw are sodium chloride, alkali, alkaline peroxide, and aqueous ammonia under optimized condition [6]. According to the report of a researcher, to extract silica successfully, the mole ratios of extraction chemical and silica, extraction time, and process temperature affect the operation [7].
Silica (SiO₂) is an element that polymerized in the epidermal cell wall. Rice straw contains a high amount of silica compared to other crops like barley and wheat straw [8]. Silica is one of the essential components that promote silica production by nanotechnology to achieve sustainable development goals because it consists of substantial applications. For instance, silica gel as absorbents, construction materials, polymer nanocomposites, food additives [9] and pharmaceutical products [10]. Silica has great performances in terms of chemically, physically and thermally stable, and compatible in a variety of materials. Therefore, silica is used to a great extent due to its properties and relatively low cost.

Apart from silica, rice straw also contains cellulose (32 % to 47 %), hemicellulose (19 % to 27 %), and lignin (5 % to 24 %). These components are known as lignocellulose biomass. Rice straw requires an efficient pretreatment to improve the extraction of silica. There are three types of main processes that are classified into biological, physical and chemical. Biological pretreatment is an environmental friendly method and cost-saving with the use of bacteria and fungi to decompose lignin and produced products with high yield without generating toxic products, whereas physical pretreatment helps to improve the surface area and enzyme digestibility. For biological pretreatment, it would consume longer time and stringent control of the microbe’s growth conditions compared to other pretreatments. Chemical pretreatment is used extensively to remove the lignin from plant and reduce the polymerization and crystallinity structure of cellulose, so that enzyme can pass through cellulose to hydrolyse them into monomers [11]. Numerous works have been published on the pretreatment of rice straw for silica extraction, such as microbial hydrolysis pretreatment for rice straw [12], alkaline pretreatment with hydrogen peroxide solution for rice straw [13], and alkaline hydrolysis pretreatment of rice straw with potassium hydroxide solution [7]. In the present study, chemical pretreatment was adopted with the use of the alkaline solution, Soda Anthraquinone, which sodium hydroxide solution was added with anthraquinone to accelerate the delignification process of lignin and solubilize hemicellulose. Chemical pretreatment can help to improve the pore volume and surface area of the substrate. In the literature study, Kiryushina et al. had studied the effect of anthraquinone (AQ) in alkaline wood delignification [14]. Anthraquinone is known as a catalyst to speed up the delignification rate and increase the pulp yield.

Commercialization of silica production is challenging due to technical and economic barriers. It involves four major steps, first is the size reduction of rice straw by a grinder, second is the alkaline pretreatment for delignification, third is the acid neutralization to prepare suitable medium for silica precipitation, and fourth is the purification of silica. Alkaline pretreatment took place in the untreated rice straw that plays an important role in the formation of silica gel. Most of the reactions in alkaline pretreatment involved removal of lignin and hemicellulose and de-esterification of intermolecular ester bonds [15]. Removal of lignin binding with hemicellulose can be achieved by adding alkaline solution, it helps in strengthening the digestibility of hemicellulose as well as promoting the access of enzyme to cellulose. Apart from that, alkaline pretreatment can increase the internal surface and enhance dissolution of the binding wax between cellulose, hemicellulose and lignin. Basically, for the digestion of lignin depends on the utilization of alkali and conditions of pretreatment as well as the lignin content in lignocellulosic material. Nevertheless, the degree of polymerization can be altered during the alkaline pretreatment, eventually altering the physical properties of treated solids. This involved changes in the crystallinity, porosity, and surface area. The removal of lignin and hemicellulose from amorphous regions caused the change in the gross crystallinity index, instead of alteration in the structure of cellulose fibers.

During acid neutralization, sulfuric acid acts as the precipitator. Sulfuric acid has been used to remove the metal oxides including sodium, calcium, magnesium, ferum, manganese in the rice straw from the final product to achieve up to 99.8 % of silica content. Many researchers focused on studies of rice straw with some findings been patented. The production of high content pure amorphous silica from rice straw was efficient by using acid leaching of rice straw to boil in strong acid solutions like sulfuric acid (H₂SO₄), nitric acid (HNO₃) and Hydrochloric acid (HCl) under pressure before burning process to remove the metal oxides and impurities involved in the rice straw. The formation of metallic sulfate is insoluble in water, so sulfuric acid is commonly used in the acid neutralization. Nitric acid is not as efficient and low in price as hydrochloric acid. Normally possible with the use of sulfuric acid solution during the acid neutralization of the rice straw. It is necessary to remove alkali metal impurities from the rice straw due to the eutectic reaction with silica. Mild acid solutions like citric acid (C₆H₈O₇) were
seldom used during the leaching process [16]. Apart from that, during the acid neutralization, sodium silicate is titrated with sulfuric acid solution at pH 7 to precipitate of silica gel aging for 72 hours.

In this research study, the pretreatment conditions such as reaction temperature, reaction time, ratio of Soda AQ solution to rice straw and alkaline concentration are closely affected to the performance of hydrolysis that related with the structural features to the solid residues. The present work aims to analyze the silica content in rice straw and effect of parameters towards silica yield by developing a method of alkaline hydrolysis for treating the rice straw to extract silica.

2. Methodology and Experimental Setup

2.1. Materials and Chemicals
The commercialized of silica with rice straw was collected from Goat 2 Go Farm at Seri Kembangan, Malaysia. Anthraquinone was purchased from Sigma-Aldrich and both sodium hydroxide (NaOH, 97 %) and sulfuric acid (H$_2$SO$_4$, 95-98%) were purchased from Chemiz and as received without purification.

2.2. Preparation of rice straw
The rice straw was washed with de-ionized water until the washing water turned clear. This is to remove adhering soil, dust and other substances on the rice straw, which might cause inaccuracy of the result. Then, the washed rice straw was spread evenly on the aluminium foil and put into the oven at a temperature of 100 °C for 8 hours until the constant weight. Oven treatment is to remove excess moisture on the rice straw. The moisture content was measured by the weight loss on drying due to evaporation by applying Eq. (1). Rice straw was ground into 50 mm in length.

$$\text{Moisture content (\%) = \frac{m_1 - m_2}{m_1} \times 100}$$

where $m_1$ is the initial weight of rice straw (g), $m_2$ is the final weight of rice straw (g), and $m_1 - m_2$ is the loss of drying weight (g).

2.3. Preparation of Soda Anthraquinone solution
Soda Anthraquinone (AQ) was prepared by mixing both sodium hydroxide solution (NaOH) and anthraquinone powder. Firstly, a specified amount of sodium hydroxide pellets were dissolved in 200 mL of distilled water to achieve 5 wt% to 25 wt% of NaOH solution. The 200 mL of NaOH solution was performed 24 hours earlier, for heat dissipated and cool down to room temperature before use. Meanwhile, 0.1 wt% of anthraquinone (20 g of AQ) was added into 200 mL of NaOH solution during the experiment. Hence, Soda AQ with alkali of 5 wt% to 25 wt% and 0.1 wt% Anthraquinone (AQ) was prepared.

2.4. Preparation of sulfuric acid solution
Sulfuric acid (H$_2$SO$_4$) with 90 wt% was prepared by dissolving 95 - 98% of sulfuric acid (H$_2$SO$_4$) into 1000 mL of distilled water. The preparation of sulfuric acid solution must be handled in the ventilated fume hood to prevent inhalation exposure. It is an exothermic reaction. Sulfuric acid should be prepared 24 hours earlier, for heat dissipated and cool down to room temperature before use.

2.5. Silica extraction from rice straw
Firstly, 5 grams of rice straw was immersed into a beaker with 200 mL of Soda Anthraquinone (AQ) with NaOH concentration of 5 wt% and 0.1 wt% of anthraquinone (AQ) and the ratio of 200:5 of Soda AQ solution to rice straw. The mixture was boiled at 60 °C for a proper digestion time of 1 hour with constant stirring in order to dissolve the silica contained in rice straw and produce of sodium silicate (Na$_2$SiO$_3$) solution. Then, the obtained slurry was known as black liquor, which was cooled down for 30 minutes at room temperature [7]. Secondly, the rice straw pulp was filtered with cloth strainer and the pulp was dried with oven. Thirdly, acid-base neutralization took place in the remaining Na$_2$SiO$_3$ solution with 90 wt% sulfuric acid (H$_2$SO$_4$) solution as a neutralizing agent. Sulfuric acid solution was added into the solution until it reached pH 7. When the pH was decreased to pH 7, vigorous stirring process on the filtrate of Na$_2$SiO$_3$ solution was proceeded for consecutive 24 hours after neutralization to avoid the formation of large agglomerates. It was kept over a period of another 48 hours to promote
the precipitation of silica gel. Eventually, the sediment formed in the Na₂SiO₃ solution, a vacuum pump was used to separate the silica gel and Na₂SiO₃ solution. Silica gel was kept in room temperature overnight for removal of water until it dried. Lastly, the dried silica which was a brownish-white fine powder was stored in a vacuum desiccator for chemical analysis [7]. The yield of extracted silica from rice straw was estimated according to Eq. 2.

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\text{Percent yield of silica (\%)} = \frac{\text{Weight of extracted silica (g)}}{\text{Weight of rice straw (g)}} \times 100\% \tag{2}
\]

2.5.1. Effect of Soda Anthraquinone concentration. The effect of Soda AQ concentration with NaOH concentration 5 wt% to 25 wt% and 0.1 wt% anthraquinone was investigated, while the temperature of the process was constant at 75 °C, digestion time of 3 hours, and ratio of 200:5 of Soda AQ solution to rice straw. The similar extraction method was carried out as described in the Section 2.5. The silica yield attained was compared for all concentration of Soda AQ solution based on percent yield of silica.

2.5.2. Effect of digestion temperature. The effect digestion temperature was investigated in the range of 60 °C to 90 °C, while the concentration of Soda AQ was adjusted to the optimum Soda AQ concentration obtained from Section 2.5.1, with 3 hours of digestion time and soda AQ to rice straw impregnation phase ratio of 200:5. The final temperature will be monitored to study the effect temperature on the yield of silica extracted from rice straw. The similar extraction method will be performed as described in the Section 2.5.

2.5.3. Effect of digestion time. The effect of digestion time was investigated by using the optimum Soda AQ concentration and digestion temperature based on the results obtained from Section 2.5.1 and 2.5.2 respectively. The digestion time in this present study was investigated in the range of 1 hour to 3 hours. The final digestion time was monitored by extending the time behind of 3 hours to examine the effect digestion time on the yield of silica extracted from rice straw. The similar extraction method will be performed as described in the Section 2.5.

2.5.4. Effect of Soda Anthraquinone to rice straw impregnation phase ratio. The effect of Soda AQ to rice straw impregnation phase ratio was investigated in the range of 150:5 to 500:5, by fixing the optimum Soda AQ concentration, digestion temperature, and digestion time based on the results obtained from Section 2.5.1, 2.5.2 and 2.5.3 respectively. The optimum Soda AQ solution to rice straw ratio was determined based on the yield of silica extracted from rice straw under working conditions.

3. Result and Discussion

3.1. Effect of Soda Anthraquinone concentration

Fig. 1 shows that the yield of silica increased with the concentration of Soda AQ solution in pretreatment process. At 60 °C, the highest yield of silica was 45.8 %. Besides, the highest yield of silica at 75 °C was 76.7 %. Not only that, the highest yields of silica at 90 °C was 71.7 %. The best yield of silica was obtained when the Soda AQ concentration reached at 20 wt%. According to the report of researcher, the yield of silica increased with the concentration of sodium hydroxide until an optimum condition [17]. Higher concentration of Soda AQ solution has higher compressive strength, hence precipitation of silica exhibited in higher strength and more yield of silica produced [18]. The concentration of Soda AQ solution that used in the alkaline pretreatment process plays an important role in the formation of silica gel. As the concentration of Soda AQ solution increased, the surface area of silica composition including planar surfaces increased [19], resulting in higher yield of silica [20]. The efficiency of lignin separation increased when the concentration of Soda AQ solution increased. Once the physical barrier was broken down, it allowed carbohydrates to undergo hydrolysis process. This is because hydroxide ions accelerated the formation of silica gel when the solution is enough of alkaline. During the alkaline pretreatment, hydroxide ions that helps to accelerate the formation of silica gel when the solution is enough of alkaline. According to the report of a researcher, formation of amorphous silica occurred in concentrated sodium hydroxide solutions [21]. Silica is known as polycondensation polymer of silicic acid, hence hydrolysis took place in the depolymerization reaction. The reaction is catalyzed by
hydroxide ions (OH\(^-\)), whereas highly alkaline solution indicated higher concentration of Soda AQ solution is necessary for completion of process.

![Fig.1: Effect of Soda Anthraquinone concentration and digestion temperature on silica yield.](image)

### 3.2. Effect of digestion temperature

Fig. 1 shows that the digestion temperature is directly proportional to the yield of silica. At digestion temperature of 60 °C, the plant cell might not fully decompose in order to destroy the osmotic barrier, so the yield of silica attained was slightly lower compared to the yield of silica attained at 75 °C and 90 °C. Besides, digestion temperature at 90 °C might be overheating to the rice straw to cause the protein structures to denature. This caused the overall structure of molecules to change and no longer function efficiently in the plant. Not only that, increasing in cooking temperature caused an increase in pressure level, hence the appearance of silica might be fractured, so that the water molecules could trapped in the silica matrix. Meanwhile, the optimum digestion temperature was observed at 75 °C, because the yield of silica precipitated from 15 wt%, 20 wt%, and 25 wt% NaOH solution were higher compared to the digestion temperature at 90 °C. The silanol group (Si-OH) was consumed at high temperature, that eventually leads to the formation of siloxane (Si-O-Si) bonds formation. Siloxane bonds formation helps to enhance the crystallization of silica, hence the yield of silica content increases [22].

Majority of the components from the plant cell are impermeable through the plant cell wall. Therefore, the plant tissue was required to heat at high temperature in order to destroy its osmotic control. Thus, the water-soluble components were able to diffuse out of the cell. Although osmotic barrier was eliminated, but larger molecules such as proteins still remained as slow to diffuse from tissues. Hence, tissues would break down first while the other compounds would diffuse from the cell sap to the solvent. According to the report of a researcher, the digestion temperature of 100 °C was preferred as optimum condition for the research. It was found out that the silica yield increased until 100 °C and remained constant behind of 120 °C [17]. The selection of digestion temperature began a 60 °C in silica extraction process from rice straw is because rice straw is considered as food grade components, it probably started to decompose at this temperature [23]. The extraction of silica is not only depending on the alkaline concentration, whereas temperature and pH both played vital role in the process. The gelation process is stimulated with increasing temperature and ionic strength. Smaller gel fragments can be due to an increase of energy dissipation. Crystallization of silica gel usually occurs in high temperature and high pressure [24].

### 3.3. Effect of digestion time

Fig. 2 shows that the relationship of yield of silica is directly proportional to the digestion time. Meanwhile, the digestion temperature was observed from the graph to show that reaction at 75 °C was able to yield higher silica content compared to the reaction at 90 °C. At 75 °C, it observed that the silica yield increased from 23.60 % to 74.11 % with increasing the reaction time from 1 hour to 3 hours. At 90 °C, by increasing the reaction time from 1 hour to 3 hours, the yield of silica increased from 24.76 % to 71.72 %, which it did not show better efficiency in the silica yield when reaction time at 3 hours. As a result, the optimum digestion time is 3 hours, since higher silica yield attained at this period of time. According to the report of a researcher, the optimum time obtained was 2.5 hours, because there is no significant extraction at time behind of 2.5 hours [17]. At longer heating treatment, more lignin
and cellulose were ruptured due to more heat generated in longer period of time. Whereas, moderate digestion time was required to prevent the rice straw from overcook and increment of burnt silica yield [25].

3.4. Effect of Soda Anthraquinone to rice straw impregnation phase ratio

The silica yield depends on the total liquid volume per dry mass of solid. As shown Fig.3, the yield of silica increased, as the ratio of Soda AQ solution to rice straw increased until it reached an optimum point. By increasing the liquid-to-solid ratio from 150:5 to 200:5, the silica yield increased from 34.62 % to 74.11 %. Besides, increasing the liquid-to-solid ratio from 200:5 to 500:5, the silica yield decreased from 74.11 % to 68.76 %. Since there is no significant difference of silica yield, which is 5.35 %, it is considered as small difference in yield. Therefore, the optimum ratio of Soda AQ to rice straw is 200:5. When the volume of Soda AQ solution is higher, it has higher permeability to the pore structure on the fibre of rice straw to precipitate more silica yield. When the volume of Soda AQ is lower, the contact area for liquid to permeate to the fibre of rice straw is lesser. Hence, the fibre of rice straw would compact together in low volume of Soda AQ solution, while the liquid was not fully contacted.

3.5. Characterization of silica

3.5.1. FTIR Spectra of commercial silica, extracted silica and raw rice straw. According to Fig.4 below, three of the samples of commercial silica, extracted silica powder and raw rice straw showed the sharp peak of IR bands at 1063.05 cm\(^{-1}\), 1036.33 cm\(^{-1}\), and 1090.33 cm\(^{-1}\) respectively due to Si-O-Si asymmetric stretching. The IR bands at 795.79 cm\(^{-1}\), 783.99 cm\(^{-1}\), and 858.48 cm\(^{-1}\) respectively were due to Si-O-Si symmetric stretching. The IR spectrum was agreed with the study by Musić et al. [26]. These results indicated the presence of silica component in the samples. The extracted silica powder product had achieved the similar IR spectrum as compared to the commercial silica gel which is the benchmark of the project and slightly different when comparing to raw rice straw which is the raw material of the experiment. In order to confirm on the existence of silica gel, strong peaks around 1090.33 cm\(^{-1}\), 858.48 cm\(^{-1}\), and 3479.38 cm\(^{-1}\) were determined from the FTIR spectrum, which corresponded to the silica absorptions of Si-O-Si and O-H functional groups [27]. Not only that, the weak peak at 3479.38 cm\(^{-1}\) was an alcohol group (-OH), which due to the water content in the filtrate,
whereas the variable strong peak at 1634.79 cm\(^{-1}\) was due to the content of cellulose in the rice straw. Cellulose in the rice straw might not fully remove at during the alkaline pretreatment at shown in IR band 1634.79 cm\(^{-1}\).

According to the report of researchers, the extracted silica powder product was verified to present with silica component, at the same time to achieve the second objective of the project [9]. However, there is a peak of C=S stretching absent in raw rice straw, but present in commercial silica at IR band 1120.5 cm\(^{-1}\) and extracted silica powder product at IR band 1121.3 cm\(^{-1}\). One of the possible reasons is because sulfuric acid was added into sodium silicate solution (Na\(_2\)SiO\(_3\)) during the acid neutralization to promote the precipitation of silica at pH 7 or below. pH is one of the factors that affects the precipitation of silica. This is because pH could affect the structure and firmness of silica gel. Both Wilhelm and Kind had been studied that acidic reactant condition able to form stronger silica gel instead of basic reactant condition [18]. Therefore, extracted silica powder product and commercial silica gel contain of sulfur compound, whereas raw rice straw does not. This caused sulfur component to be contaminant in the sample.

![Fig.4: FTIR spectra of commercial silica, extracted silica powder and raw rice straw.](image)

### Table 1 FTIR Spectra of experimental samples.

| No. | Functional Group            | Wavenumber (cm\(^{-1}\)) |
|-----|------------------------------|--------------------------|
|     | Commercial Silica Gel        | Raw Rice Straw            | Extracted Silica Powder | Extracted Brown Pulp | Sodium Silicate Filtrate | Sodium Sulfate Crystal |
| 1   | Si-O-Si asymmetric stretching| 1063.05                  | 1036.33                 | 1090.33               | 1064.8                   | 1054.8                   | 1040.6                   |
| 2   | Si-O-Si symmetric stretching | 795.79                    | 783.99                  | 858.48                | 965.2                    | -                        | -                        |
| 3   | OH stretching                | 3280.86                   | 3344.38                 | 3479.38               | 3284.2                   | 3354.42                  | 3330.85                  |
| 4   | OH bending                   | 1635.11                   | 1621.92                 | 1634.79               | 1634.43                  | 1644.04                  | 1667.80                  |
| 5   | C=S stretching               | 1120.5                    | -                       | 1121.3                | 1123.9                   | 1127.1                   | 1121.3                   |

3.5.2. **FTIR Spectra of extracted silica, extracted brown pulp and raw rice straw.** According to Fig.5, by comparing three of the samples which were extracted silica powder, extracted brown pulp and raw rice straw. The extracted brown pulp has similar IR spectrum as raw rice straw. Although extracted brown pulp is the undesired product in the project, but it still consists the functional groups of Si-O-Si asymmetric stretching and Si-O-Si symmetric stretching around the peaks 1064.8 cm\(^{-1}\) and 965.3 cm\(^{-1}\).
that attributed to the presence of silica component. The extracted brown pulp which formed during the precipitation of silica powder and it was then removed from the silica powder with spatula in order to get the weight measurement of silica powder. In some of the literatures, the pulp obtained from the experiment could undergo further pulp and paper manufacturing process. Meanwhile, the presence of silanol group in the brown pulp was due to silica bonded to the fibre.

Apart from that, the hydroxyl bending peak around $1634.43 \text{ cm}^{-1}$ in the extracted brown pulp had stronger absorption peak compared to extracted silica powder and raw rice straw at peaks around. The cellulose in the rice straw that made up $\beta - 1, 4$ bonds linked D - glucose. The glucose chains are held strongly by hydrogen form to form greater stability of microfibrils [28]. Therefore, both extracted brown pulp and raw rice straw had an obvious absorption peak of hydroxyl peak, because the brown pulp could be the residues from the alkaline pretreatment.

![FTIR Spectra of extracted silica, extracted brown pulp and raw rice straw.](image)

3.5.3. FTIR spectra of sodium silicate filtrate solution and sodium sulfate crystal. Sodium silicate filtrate solution is the solution that required to undergo neutralization with acid to pH 7, in order to promote silica aging for 48 hours. The crystal was obtained from the sodium silicate filtrate solution after aging of 48 hours. As shown in Fig.6, both sodium silicate filtrate solution and sodium sulfate crystal had broad and strong IR band around $1054.8 \text{ cm}^{-1}$ and $1040.6 \text{ cm}^{-1}$ respectively that indicated the presence of Si-O-Si asymmetric stretching group. This explained that silica component might not be fully filtered during the purification process. Meanwhile, Si-O-Si symmetric stretching functional group was absent, but present with Si-O-Si asymmetric stretching functional group. The weak band around $3354.42 \text{ cm}^{-1}$ and $3330.85 \text{ cm}^{-1}$ for both sodium silicate filtrate solution and sodium sulfate crystal respectively, it indicated the presence of hydroxyl stretching in the silanol group and some absorbed moisture. The strong IR peaks around $1644.04 \text{ cm}^{-1}$ and $1667.80 \text{ cm}^{-1}$ for both sodium silicate filtrate solution and sodium sulfate crystal respectively, showed the hydroxyl bending vibration of water trapped in the anhydrous salt, Na$_2$SO$_4$ crystal as well as Na$_2$SiO$_3$ solution. Since both sodium silicate filtrate solution and sodium sulfate crystal were the by-products of the experiment weak peak around $1127.1 \text{ cm}^{-1}$ and $1121.3 \text{ cm}^{-1}$ respectively to indicate the presence of C=S stretching, while the sulfur component might be formed from the sulfuric acid neutralization step.
Fig. 6: FTIR spectra of sodium silicate filtrate solution and sodium sulfate crystal.

4. Conclusion

In a nutshell, the precipitated amorphous silica was successfully extracted at 74.11 % of silica yield with the optimum conditions of Soda AQ solution with NaOH concentration 20 wt% and 0.1 wt% of anthraquinone and ratio 200:5 of Soda AQ solution to rice straw, at temperature of 75 °C for 3 hours of digestion time. Higher concentration of Soda AQ solution promotes higher precipitation of silica. The ratio of Soda AQ solution to rice straw is important, because higher liquid-to-solid ratio improves the contact area of fibre to the solution, thus more lignin in the rice straw can be decomposed by Soda AQ solution. At longer heating treatment, more lignin and cellulose were ruptured due to more heat generated in longer period of time. Furthermore, the amorphous silica was affirmed by FTIR analysis with the presence of silica content and it matched with the spectrum of commercial silica gel available in the market.

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