Performance of the silica adsorbent from snake fruit peel for removing heavy metals of Ag, Cu, Mn, and Cr in SCW

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Article Info	ABSTRACT
	Silver crafts wastewater (SCW) typically contains environmentally harmful heavy metals, including Ag, Cu, Mn, and Cr, necessitating treatment before disposal. This study explores a promising solution using silica (SiO2) adsorbents derived from snake fruit peel through acidic activation with HCl concentrations of 2, 4, and 6 M. Qualitative analysis of the adsorbent involved Fourier-transform infrared spectrometer (FTIR) and x-ray fluorescence (XRF) techniques. XRF analysis revealed major compositions of Si (26%) and Cl (71.46%), with minor elements such as Ca (0.91%), P (0.42%), K (0.37%), Fe (0.12%), and others. FTIR analysis indicated the presence of siloxane (Si-O-Si) and silanol (Si-OH) on the adsorbent. The SiO2 adsorbent demonstrated effectiveness in removing heavy metals (Ag, Cu, Mn, and Cr) from SCW, achieving removal percentages of approximately 16.96%, 24.38%, 19.34%, and 9.82%, respectively. This research contributes to the development of an environmentally friendly approach for SCW treatment using silica adsorbents derived from agricultural waste.

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1. INTRODUCTION

Salak (Indonesian) or snake fruit is one of the local food commodities with quite a large production. Data from the Central Statistics Agency (BPS) showed the amount of snake fruit production in Indonesian reached 1,147,473 tons in 2022 [1]. Apart from being consumed directly, snake fruit can also be processed into various interesting food products such as candied snake fruit, snake fruit *dodol*, snake fruit chips, snake fruit pudding, and snake fruit brownies. Cultivated extensively for its economic value, snake fruit plants are ubiquitous across Indonesia. The fruit, prized for its delectable flesh, presents a challenge due to the substantial waste generated by its peel, which is inedible [2]. Hence, there is a pressing need for a solution to address the issue of snake fruit waste.

Currently, some research on snake fruit peel waste (SFPW) reduction is available in the literature. For instance, the SFPW can be reduced by a processing approach to produce valuable products such as activated carbon catalysts, carbon-based filter material, and carbon adsorbent. In addition, the SFPW can also be processed to produce a natural preservative of food products. However, so far, there is limited research that is associated with the use of SFPW for producing a green silica adsorbent. In addition to carbon, it is well known that snake fruit peel has also a high silica composition. Therefore, the use of SFPW for producing green silica adsorbent becomes an interesting focus to be explored.

The management and utilization of SFPW is one of the interesting topics because apart from being able to reduce negative impacts on the environment, this effort also has added value from the waste. The SFPW contains 72.45% carbon components [3], and silicon dioxide (silica, SiO2) which is quite high, namely around 65-69% [4], so it has the potential to be used as a raw material to produce SiO2 adsorbents [5] or SiO2 adsorbent. As reported in several research results. The SiO2 adsorbents can be used to reduce heavy metal pollution in wastewater [6]. The efficiency of SiO2 adsorbent against the adsorption of heavy metals in wastewater is quite good [7]. For instance, the SiO2 adsorbents can absorb the heavy metal of cadmium (Cd) by 99%, nickel (Ni) by 50%, zinc (Zn) by 69%, and lead (Pb) by 99% [8]. In other research, SiO2-based adsorbents also had an effective performance for the adsorption of heavy metals such as As (V) at 97.7%, Hg (II) at 98.5%, Cr (II) at 96.0%, Cd (II) at 96.9%, and Pb (II) was 78.7% [8].

Wastewater originating from the steel industry process, catalysts, metal plating processes, mining, and the batik industry produces wastewater that contains various types of heavy metals [10], [11]. The presence of heavy metals in wastewater is very dangerous for the environment, humans, and other living creatures. Heavy metals cannot undergo bio-degradation in the environment [12], [13], so appropriate treatment strategies need to be implemented [14], [15]. Compared to chemical processes, physical treatment is much easier, cheaper, and safer to apply in handling wastewater. One of the physical treatment processes is the adsorption process, where this process involves the use of adsorbents that can absorb heavy metals contained in wastewater.

Several methods can be used to treat hazardous waste, such as adsorption, electrodialysis, coagulation, and reverse osmosis. The adsorption method is chosen for its affordability compared to the above methods, its processing does not form sludge, and the adsorbent can be regenerated. Several studies mentioned above show that the heavy metal contaminants in wastewater can be reduced by using an adsorption method with adsorbents such as commercial silica, synthesized zeolite, and natural zeolite. Based on these references, this study tries to investigate the feasibility of green silica adsorbent for adsorbing heavy metals from the batik industry.

As explained above, silica adsorbents can be synthesized from waste such as snake fruit peel. This research aims to synthesize an adsorbent from SFPW and evaluate its adsorption ability to absorb heavy metals in silver crafts wastewater (SCW). Generally, the levels of heavy metals (i.e., Ag, Cu, Cr, and Mn) produced by SCW are quite high. Therefore, apart from synthesizing the SiO2 adsorbents from SFPW, the condition of SCW (before and after treatment) based on the metal content Ag, Cr, and Mn will also be evaluated. Based on Government Regulation No.82-2001 states that the permitted levels of heavy metals in wastewater are only around 0.10-2.00 ppm.

2.   RESEARCH METHOD
2.1. Materials and instruments
Materials such as distilled water, HCl 37%, and NaOH were purchased from the chemical shop in Yogyakarta, Indonesia. SFPW and SCW were collected from the Griyo Olahan Salak Turi Sleman and the Silver Industry in Kotagede, Yogyakarta Indonesia. Some equipment such as porcelain mortar, 100 mesh of vibrating sieve, magnetic stirrer, Erlenmeyer, pH meter, oven, heating stove, filter paper (Whatman 42 µm), furnace, and thermometer. The instruments for characterization were Fourier transform infrared (FT-IR, Shimadzu Prestige-21), gas sorption analyzer (GSA, NOVA 1200e), SEM-EDX (JSM-6510LA), and atomic absorption spectrometry (AAS, HITACHI Z2000).

2.2. Production of the silica (SiO$_2$) adsorbent from snake fruit peel waste
The silica adsorbent was produced from SFPW by using a method as described in [16]. A total of 500 g of SFPW was cleaned and washed with clean water. After that, the SFPW is dried in the oven at 100 °C for 4 hours. Then, the dried SFPW is ground by using a blender until it becomes a powder with a size of 100 mesh. Heat SFPW powder in a furnace for 60 minutes at 650 °C until SFPW turns into ash. Subsequently, the 20 gr SFPW powder was added in three different concentrations of 2, 4, and 6 M HCl solution and was stirred using a magnetic stirrer at a speed of 120 rpm for 4 hours. The stirring process was carried out for 6 hours and then filtered using Whatman 42 µm filter paper. The SFPW powder residue was then washed with distilled water until the filtrate pH was neutral (pH=7.0) and the precipitate was dried at 110 °C. Then, the SFPW powder was mixed with NaOH 6M solution, refluxed at 60 °C for 3 hours, and filtered using 42 µm Whatman filter paper. The filtrate was mixed with HCl 6M solution until a colloid was formed and left for 24 hours. Lastly, the white precipitate was separated from the solution and washed with distilled water until the filtrate pH was neutral (pH=7) and was dried in the oven at 110 °C for 2 hours. The SFPW white powder silica was used for further experiments.

2.3. Silver crafts wastewater adsorption procedure

1 gram of silica was mixed with 300 ml of SCW. The solution was stirred for 60 minutes at a stirring speed of 100 rpm and left for 24 hours. The precipitate and filtrate were separated, and the precipitate was dried at 100 °C for 2 hours. The residue was analyzed using SEM-EDX mapping to determine the adsorbed metals, while the liquid was analyzed using AAS to determine the metal content after adsorption.

2.4. Characterization of the snake fruit peel waste adsorbent

The functional group in silica and the presence functional group from MS were observed and analyzed using a Fourier-transform infrared spectrometer (FTIR) in combination with the KBr disc technique, the sample used for analysis was 2–5 mg. The pore size using GSA, NOVA 1200e, and elemental compositions were characterized by using (x-ray fluorescence (XRF) and Malvern analytical Epsilon 1), respectively. The surface morphology silica after adsorption with scanning electron microscopy (SEM-EDX JEOL JSM-65100), respectively.

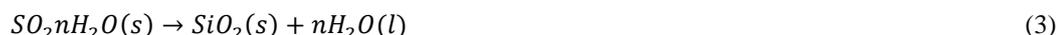## 2.5. Analyzation of heavy metals in the silver crafts wastewater

The composition of heavy metals in SCW before and after contact with the adsorbent was analyzed by using AAS, HITACHI Z 2000. Morphology of silica after adsorption was characterized using a scanning electron microscope (SEM-EDX). The composition of the main elements was used to evaluate the efficiency of the SFPW adsorbent.

3.   RESULTS AND DISCUSSION
3.1. Selection of snake fruit peel waste adsorbent

As mentioned above, the adsorbent from SFPW was produced through an acid-activated process using three different concentrations of 2, 4, and 6 M HCl. The adsorbent yields of these processes were obtained at 50.1, 62.7, and 75.1%, respectively. These results illustrate that the higher the acid concentration causes the greater the adsorbent yield. It was well known that the strong acids can erode and remove the organic material that coats and binds the SFPW powder so that the amount of silica produced will be greater. However, further experiment was required to ensure the optimal concentration of HCl that produces the best yield of silica.

The presence of silica ($SiO2$) in the residue and strong base (NaOH) causes a colloid formation during the reflux process. The high amount of colloid formation indicates a fairly high silica composition. The reaction for the formation of silica colloids during the reflux process is shown in (1), (2), and (3) [17]. Based on the adsorbent yields, the SFPW powder with an acid-activated process of 6M HCl has the highest yield of 75%. Then, this SFPW powder was called $SiO2$ adsorbent and was used for further experiments.

$$SiO_2(s) + 2NaOH \rightarrow Na_2SiO_3 \ (aq) + H_2O \tag{1}$$

$$Na_2SiO_3(aq) + 2HCl \rightarrow SO_2nH_2O(s) + 2NaCl(l) \tag{2}$$

$$SO_2nH_2O(s) \rightarrow SiO_2(s) + nH_2O(l) \tag{3}$$

3.2. Characterization of the snake fruit peel waste adsorbent using Fourier-transform infrared spectrometer

The characteristic of the SFPW adsorbent was analyzed based on the functional groups contained in the material. Figure 1 shows the FTIR spectra of three acid activation processes in making silica adsorbents from SFPW. In general, the spectral shapes of the three samples have the same spectral patterns. Peaks appear in four-wave number areas, namely at 700-800 cm^{-1}, 1000-1250 cm^{-1}, 1500-1750 cm^{-1}, and 3250-3500 cm^{-1}. The spectra of the adsorbent with 4 and 6 M HCl acid activation showed peaks at 793 and 798 cm^{-1}. The appearance of the peak in this area is caused by the symmetric stretching vibration of the Si-O-Si functional group. This spectrum also shows the presence of silica groups in the adsorbent. Although the SFPW adsorbent with 2 M acid activated was not shown the peak of symmetric stretching vibration of Si-O-Si at 793 and 798 cm^{-1}, the peak of asymmetric stretching vibration of Si-O-Si appears in the wave number region of 1106 cm^{-1}. These facts indicate that the three adsorbents contain silica (SiO_2) [18], [9], [20].

In addition, the appearance of peaks in the wavenumber areas of 1635 cm^{-1} and 1639 cm^{-1} indicates the vibration of the silanol group (Si-OH). The presence of the Si-OH group indicates that the adsorbent contains silica and water molecules. The Si-OH group is one of the active sites in the adsorption process. The existence of the Si-OH group is also proven by the appearance of peaks in the wavenumber areas 3447 cm^{-1} and 3471 cm^{-1}. The FTIR spectra of the three types of adsorbents show that the samples contain silica. Thus, SFPW adsorbent could be used as representative silica adsorbent to adsorb the heavy metals in SCW.
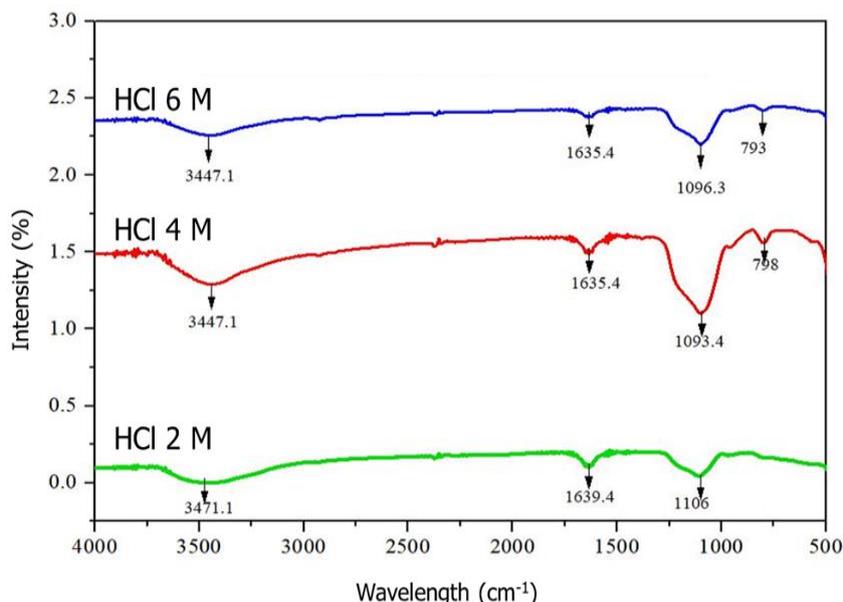
Figure 1. FTIR spectrum of the SFPW adsorbents with three different acid concentrations of 2, 4, and 6 M HCl

3.3. Type and composition of elements

Furthermore, the presence of elements in the adsorbent can be analyzed by using the XRF method. It is well known that silica (SiO_2) contains the elements silicon (Si) and oxygen (O), consequently, when the x-rays are exposed to the adsorbent, the elements will fluoresce. Each element gives a different fluorescence pattern depending on their type and composition in the sample. Generally, this XRF method can detect the elements that have relatively high fluorescence energy (>1000 eV). Whereas, elements with low levels of fluorescence energy such as O (fluorescence energy=538 eV) cannot be detected.

Table 1 shows the types and composition of the elements in the SFPW adsorbent. There were two main elements, namely chlorine (Cl) and Si, the composition of which were 71.46 and 26.59%, respectively. The high composition of Cl may be caused by the HCl acid activation process during SFPW adsorbent production. The chlorine content in the SFPW adsorbent is much higher than that of silica sand (40%) [18]. The excessively high concentration of HCl also leads to increased viscosity, which binds with NaOH, reducing the ion activity in the solution. As a result, the amount of bonds formed between NaOH and SiO_2 in the SFPW cannot react. Whereas, the high composition of Si shows the SFPW adsorbent contains quite high levels of silica. Parallel with the FTIR spectra, the XRF data also shows the presence of Si elements in the SFPW adsorbent. In addition, the other elements such as calcium (Ca), phosphorus (P), potassium (K), iron (Fe), zinc (Zn), chrome (Cr), silver (Ag), and rhenium (Re) were present in very low compositions. The composition of minor elements was observed in the range of 0.005-0.91%. It is well known that the presence of minor elements in a high composition might reduce the performance of adsorbent due to them acting as impurities. Fortunately, this work shows that the total amount of minor elements in the sample was observed in low composition (less than 2.0%). These results indicated that the purity of the SFPW adsorbent was quite good.

Table 1. Type and composition of elements in the SFPW powder

| Elements	Composition (%)
Cl	71.46
Si	26.59
Ca	0.91
P	0.42
K	0.37
Fe	0.12
Ag	0.09937
Zn	0.06044
Cr	0.00660
Re	0.00461

3.4. Surface morphology of the snake fruit peel waste adsorbent

The morphology and composition of elements on the surface of the SFPW adsorbent can be analyzed using SEM-EDX. In this research, the image magnification obtained was 5000 times with an energy of 15 kV. In detail, the SEM image of the SFPW adsorbent before and after contact with SCW is shown in Figure 2. Figure 2(a) shows the surface morphology of the adsorbent before being applied with SCW. The surface microscopic structure was still relatively clean and this indicates that silica can be used as an adsorbent. Meanwhile, Figure 2(b) shows the surface structure of the adsorbent coated with Ag, Mn, and Cr metal after being contacted with SCW. The presence of Ag, Mn, and Cr metals is indicated by blue, green, and red color mapping.

The presence of Ag, Mn, and Cr metals on the surface of SFPW adsorbent indicates the ability of silica to adsorb heavy metals in wastewater. Consistent with the research conducted by [8]-[9], [19] in which the heavy metals in wastewater could be reduced by silica adsorbent. This result described that the performance of the SFPW adsorbent was relatively good. This fact might be due to the purity of SFPW being relatively high (see Table 1). However, expanded research must be conducted in the future so the performance of SFPW adsorbent is much better. Increased purity and Si composition in SFPW adsorbent were the important approaches to explore.
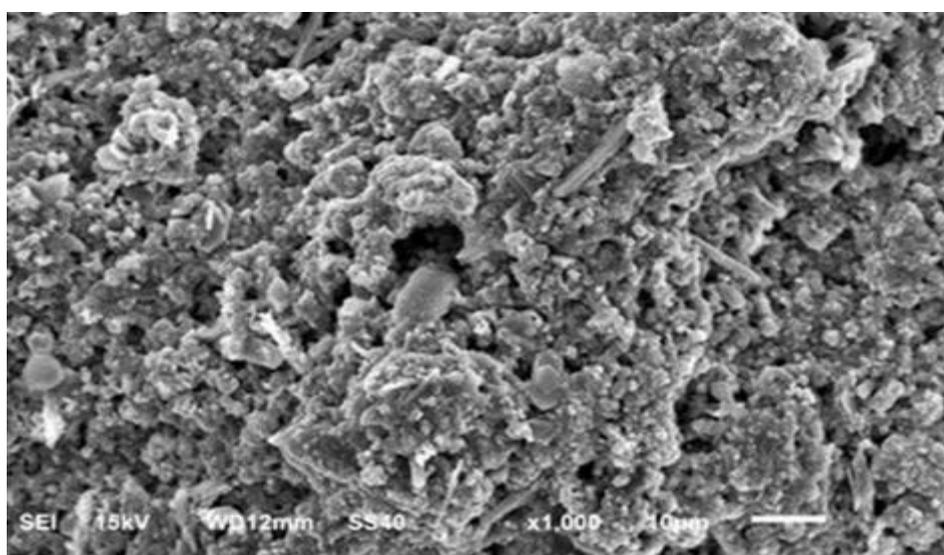
(a)
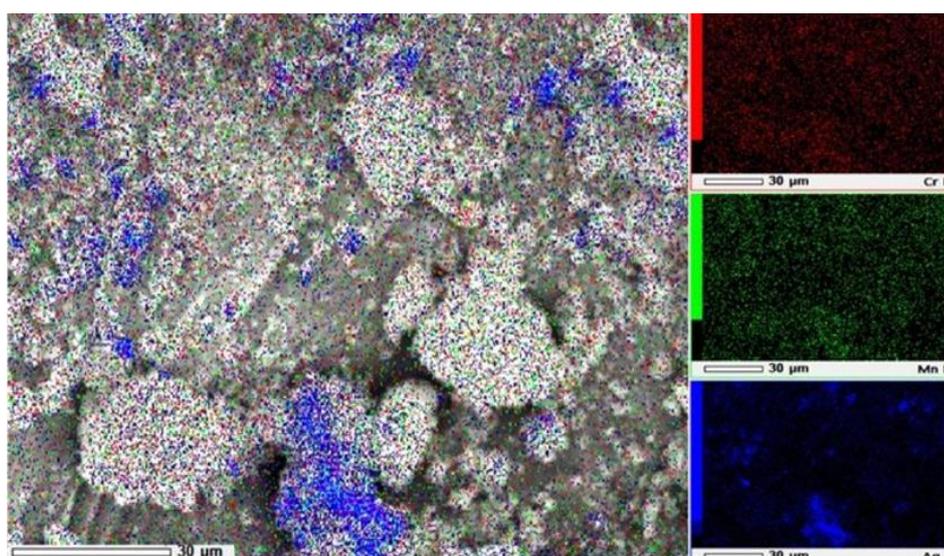
(b)

Figure 2. SEM image of the adsorbent surface (a) before and (b) after contact with SCW

3.5. Surface area and pore of the snake fruit peel waste adsorbent

Based on the GSA analysis, it was found that the surface area of silica was around 56.35 m^2/g. Whereas, the pore volume was around 1.93×10^{-4} cc/g with a pore diameter of around 26.67 mm. Based on several references, it is stated that a good silica adsorbent has a surface area of around 300-1164 m^2/g [21], [22], and [23]. The larger the surface area of the adsorbent, the better the adsorption process. In terms of surface area, the SFPW powder was less suitable for adsorbent material [24]. The total pore volume describes the adsorption and storage capacity of an adsorbent on heavy metal components. Even though the pore diameter was quite large, the total pore volume was quite small so the adsorption and storage capacity of the adsorbent was low [25]. As mentioned above, the performance of SFPW adsorbents is affected by their purity. Therefore, the increased purity and pore size might increase the adsorption and storage capacity of the adsorbent.

3.6. Silver crafts wastewater heavy metals removal

The composition of heavy metals such as Ag, Cu, Mn, and Cr in SCW samples was analyzed using the AAS method. The characteristics of the SCW samples before and after the adsorption process are shown in Table 2. The efficiency of the adsorbent in adsorbing heavy metals was obtained around 9.82-24.38%. Based on the removal percentage, the SFPW adsorbent shows the best performance to adsorb Ag metal compared to other heavy metals. This phenomenon may be attributed to the presence of -OH groups in the silanol (Si-OH) on the adsorbent, which act as ligands capable of donating a pair of free electrons to the empty s-orbitals of Ag. Additionally, it is likely influenced by numerous active sites on the adsorbent surface, thus optimizing the adsorption capacity of Ag metal. According to some references, the SFPW adsorbent has a good performance in adsorbing various types of heavy metals such as Cr (III) [9], Cu (II) [16], and Pb [26]. In addition to the Cr, Cu, and Pb, the SPWF adsorbent can also adsorb the metals Ag and Mn (see Table 2). Overall, the performance of SFPW adsorbent to adsorb Ag, Mn, and Cr was relatively low compared to the references. This fact might be due to the purity and pore size of SFPW adsorbents being relatively low. Nevertheless, this work successfully synthesized the green silica adsorbent from SFPW by using a simple and inexpensive method. Interestingly, this work successfully converted the SFPW to a valuable material.

Table 2. Characterization of SCW before and after contact with adsorbent

Type of heavy metals in SCW	Composition (mg/L)		Removal (%)
	Before	After	
Cl	122.95±2.05	102.10±4.65	16.96
Ag	0.34±0.09	0.25±0.02	24.38
Mn	0.47±0.01	0.38±0.02	19.34
Cr	0.95±0.04	0.86±0.06	9.82

4.   CONCLUSION

This research successfully synthesized the silica adsorbents (SiO$_2$) from SFPW through the HCl acid activation and reflux process. In addition to major elements of Si and Cl, minor elements such as Cl, K, and Ca, P, were observed on the SFPW powder. The presence of Si-O-Si and silanol Si-OH functional groups emphasizes the presence of silica elements in the SFPW powder. The SiO$_2$ adsorbent from the SFPW could reduce the levels of heavy metals of Cu, Ag, Mn, and Cr in SCW by around 16.96, 24.38, 19.34, and 9.82%, respectively. This result indicated that the SiO$_2$ adsorbent has a good performance in reducing some heavy metals in SCW. The feasibility of SFPW to adsorb some heavy metals from wastewater was quite good. This research can be considered an interesting approach to solving the SFPW issue at the same time producing valuable materials.

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