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The Effect of Varying the HCl Solution on the Purity, Morphological, and Electrical Properties of Silicon Dioxide Extracted from Rice Straw

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ABSTRACT

Rice straw is a waste product from rice manufacturing that contains cellulose (32–47%), hemicellulose (19-27%), lignin (5–24%), and ash (13–20%). The ash form consists of a large percentage of silicon dioxide (SiO_2) with widespread use in the industrial field. The extraction of silicon dioxide has been conducted using the sol-gel and ashing method combined with the leaching process using an acid solution such as hydrochloric acid (HCl) at a concentration of 3%. In using HCl with a concentration of 3%, impurities are often found in the SiO2 sample. Therefore, this study uses the leaching method with HCl of several variations (3%, 5%, and 7%). By raising the concentration of HCl in this process, the quality of SiO_2 without impurities is increased. The results indicate that increasing the concentration of HCl can significantly lower the sample's impurity content. In the 3% treatment, impurities were found in the form of Carbon and Calcium. The treatment obtained no impurities using 5% and 7% HCl concentrations. As a result, the highest purity of SiO_2 obtained was 89.31% in the 5% HCl treatment. The sample treated with 5% HCl was in the semiconductor region and exhibited an amorphous structure.

Keywords: Electrical, morphological, purity, rice straw, silicon dioxide

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INTRODUCTION

The availability of biomass in Indonesia is significant, including sources from agriculture and plantations such as oil palm, rice, maize, and others (Kurniawan et. al, 2019). Generally, biomass is used as fuel (biofuel) (Yusof et al., 2018) and raw material for various alternative products such as biomaterial (Rohaeti et al., 2010; Aminullah et al., 2015), biochemistry (Davis et al., 2018), bioadsorbent (Aichour et al., 2018; Zainal et al., 2018), and biosurfactant (Ebrahimi et al., 2019).

Biomass from rice farming in rice straw has not been maximally utilized in Indonesia. Rice straw ash contains a high percentage of silicon dioxide (SiO_2), ranging from 61.39% to 84.60% (Nandiyanto et al., 2016; Jenkins et al., 1998; Khorsand et al., 2013). SiO_2 is a material with widespread use in the industrial field, and one of its applications is in silica gel (Sabara et al., 2022a), which reduces humidity. It is used in glass, cement, electronics, ceramics, and other industries (Abdurrahman et al., 2019). However, utilizing SiO_2 in natural minerals is highly inefficient and might result in environmental problems when stored indefinitely (Abdurrahman et al., 2020). Therefore, SiO_2 from plant materials is in great demand for natural mineral

Several studies related to the extraction of SiO_2 from biomass using rice husks (Sintha et al., 2017; Irzaman et al., 2020; Casnan et al., 2019), rice straw (Nazopatul et al., 2018; Hapsari et al., 2020), bamboo leaves (Irzaman et al., 2018; Aminullah et al., 2018), bagasse (Adli et al., 2018), and coir coconut (Anuar et al., 2020) have been reported. The sol-gel method extracted SiO_2 in those studies, followed by hydrolysis and subsequent condensation operations. By employing this method (Nandiyanto et al., 2016), SiO_2 of purity of 68% can be extracted from rice straw. Several studies reported the extraction of SiO_2 by adopting a simpler method consisting of combustion of biomass (forming charcoal) (Sabara et al., 2022b), leaching (using acid), and ashing (Rohaeti et al., 2010; Aminullah et al., 2015; Adli, 2018). This method has been used to extract SiO_2 from rice husk biomass, with a purity of 99% (Sintha*,* 2017; Irzaman et al., 2020). The test results revealed the minor presence of many other elements such as carbon, calcium, and others as impurities (Palupi et al., 2020). Then, in another study by (Hapsari et al., 2020), SiO_2 was extracted using rice straw biomass with variations in the combustion temperature and a purity value approaching 99%. However, SiO_2 contained many other potassium, calcium, chlorine, and sodium elements, significantly affecting the sample's electrical characteristics. To reduce the impurity in the extracted SiO_2, the study (Nazopatul et al., 2019) modified the extraction method (Husna et al., 2020) by washing rice straw before the combustion process. Treatment of gray temperature variations was implemented to obtain purer SiO_2. The results show that this method extracts SiO_2 with reduced impurity content compared to other methods (Palupi et al., 2019a; Palupi et al., 2019b). The purity value reaches 85.41% at a combustion temperature of 800°C. It turns out that by modifying the system, the effects of contaminants can be mitigated (Sabara et al., 2022b). However, the calculated purity value is lower than the previous method; therefore, further studies are still needed.

Based on some references, this study aims to modify the extraction method by leaching rice straw biomass before burning and measuring the samples' purity, electrical, morphological, and structural properties. The treatment is different from previous studies by taking the variations in the concentration of acid solutions in the leaching process. The concentrations of HCl utilized vary between 3%, 5%, and 7%. Based on the treatment, it is reasonable to assume that using a more significant concentration of HCl increases SiO_2 purity.

EXPERIMENTAL PROCEDURES

The extraction process included leaching the rice straw, making charcoal, and ashing the charcoal of the rice straw. First, the rice straw is cut into small pieces with a size of ± 5cm, then weighed as much as 100 g. Next, rice straw is soaked with technical grade HCl concentrations of 3%, 5%, and 7% heat for 2 hours. Samples were washed using distilled water until the pH reached 6.5–7 and then dried. After that, the samples were burned in an open space without adding fuel. The rice straw charcoal was burned in a furnace initially at 400 °C for 2 hours and subsequently at 800°C for 1 hour to obtain the ash. The sample structure was then determined by using X-Ray Diffraction (XRD) technique. Spectroscopy Scanning Electron Microscopy (SEM) technique was employed to determine the surface morphology of the sample using a magnification of 5000 and 15000. Energy Dispersive X-Ray Spectroscopy (EDS) was used to determine chemical constituents such as silicon, oxygen, carbon, and others contained in a sample. The purity of SiO_2 was determined based on the percentage of Si (silicon) atoms and calculated by Equation 1:

$$SiO_2(\%atom) = 3 \times (\%atom\ Si) \quad (1)$$

An Inductance, Capacitance & Resistance (LCR) meter was used to determine some electrical properties of a material, such as electrical conductance, dielectric constant, and impedance. One of the parameters detected in the LCR meter characterization results is the conductance to calculate the material's electrical conductivity. The conductivity values of materials can be categorized into three parts: insulators, semiconductors, and conductors. This study is expected to obtain a semiconductor sample because the results from SiO_2 will be reduced to Si and can be used as a base material for sensor manufacture.

RESULTS AND DISCUSSION

Composition Analysis of SiO_2

The composition analysis of SiO_2 determines the elements contained in the sample. The effect of HCl concentration on the purity of SiO_2 was conducted by testing the elemental composition at 800^{o}C with HCl concentrations of 3%, 5%, and 7%. Table 1 shows the value of the purity of SiO_2 calculated based on the results of EDS.

Table 1
The value of the purity of SiO_2 calculated based on EDS results

Element	Atom (%)		
	3%	5%	7%
Oxygen (O)	63.83	69.31	71.44
Silicon (Si)	28.47	29.77	27.62
Carbon I	7.51	–	–
Calcium (Ca)	0.19	–	–
Gold/Aurum (Au)	–	0.92	0.94
Purity of SiO_2	85.41	89.31	82.86

The data shows that the sample in the 3% HCl treatment has a purity of 85.41% but still contains calcium and carbon impurities. Meanwhile, the sample obtained with the treatment of 5% HCl has no impurities, and the purity is 89.31%. HCl solution is an effective acid to remove metal impurities such as Ca. The C^{l-} ions from HCl will bind with Ca^{2+} ions from the sample and form $CaCl_2$ salt. Since salt is soluble in water, it will be lost during infiltration. The purity result of treatment with 7% HCl is 82.86% (which is lower compared to 3% and 5% of HCl), even though no impurities were found. The effect of the sample coating caused the gold element in these results during the characterization process. Therefore, leaching rice straw with HCl concentrations above 5% is not recommended for extracting the desired SiO_2. The same method applied to rice husk material (Sintha et al., 2017) also shows that HCl concentrations above 5% yield decreased purity of SiO_2. The study (Lu & Hsieh, 2012) indicated a difference in purity of approximately 1.49% more than the results from this investigation. In addition, the acid concentration is increased to 10% H2SO4.

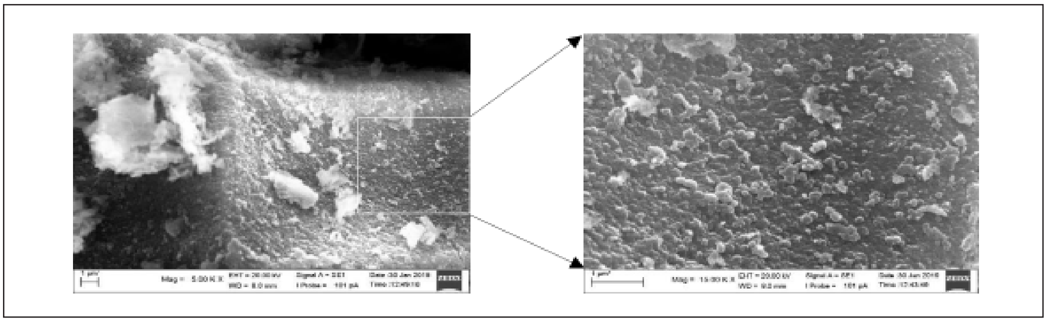
Morphological Analysis of SiO_2

Surface morphological analysis of SiO_2 samples extracted from rice straw illustrated in Figure 1 was observed at a magnification of 5.000 and 15.000. The surface morphology obtained with the treatment of 3%, 5%, and 7% HCl are shown in Figures 1 (a), (b), and (c), respectively. The surface structure of the 3% HCl sample has a distribution of fairly homogeneous grain shapes with a relatively larger grain density.

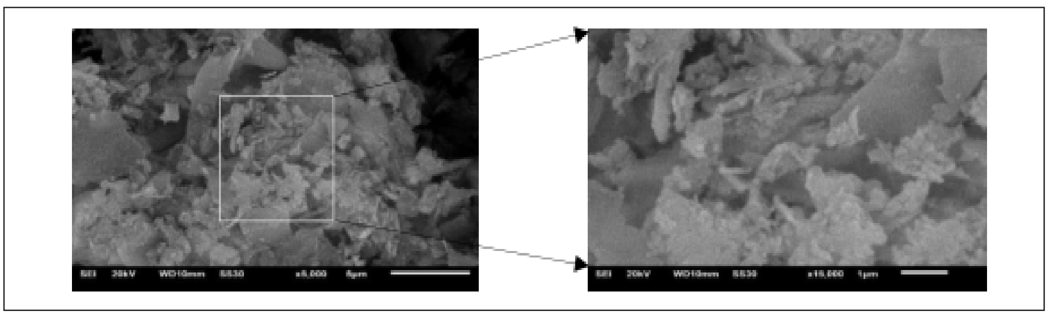
The treatment of 5% HCl produces morphology with a more acceptable surface shape composed of a distribution of heterogeneous grain shapes. The ideal surface shape may be connected to the highest purity of SiO_2 extracted with the treatment of 5% HCl. Meanwhile, the concentration treatment of HCl 7% in Figure 1 (c) has a surface morphology with more uniform and larger grain shapes.

Structure Analysis of SiO_2

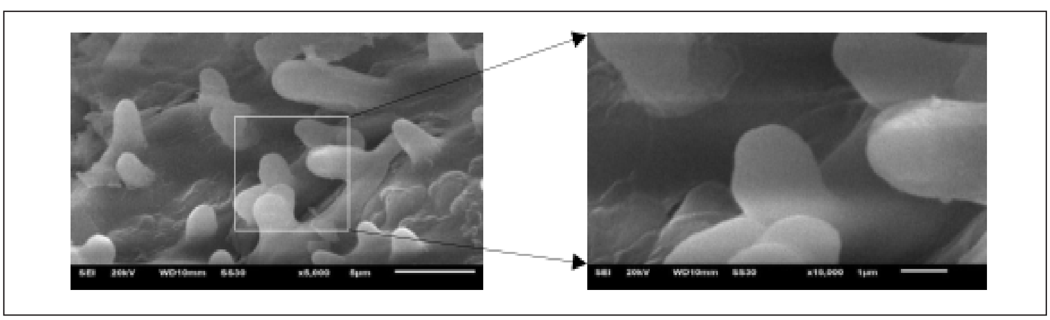
The structure of SiO_2 extracted from rice straw was analyzed at an angle of 10^o to 80^o, as shown in Figure 2. The figure shows the structural phase of SiO_2 samples extracted upon leaching with variable (3%, 5%, and 7%) HCl concentrations. The diffraction pattern



(a)



(b)



(c)

Figure 1. Results of SiO_2 morphological analysis of rice straw using SEM at variations of: (a) HCl 3%; (b) HCl 5%; and (c) HCl 7%.

obtained with the treatment of 3% HCl gives the highest peak at an angle of 21.96°. Compared with JCPDS data, the diffraction pattern gives the highest peak at an angle of 21.98°. Meanwhile, SiO_2 samples obtained from 5% and 7% HCl treatment give the diffraction patterns with the highest peak value at an angle of 21.80°. Based on the results in Figure 2 (Khorsand et al., 2013), SiO_2 from rice straw has an amorphous structure compared with the results of previous studies. Note that the amorphous form of SiO_2 is more utilized than SiO_2 in crystals form. For example, amorphous SiO_2 ash is useful as a substitute for cement additives (Oyekan & Kamiyo, 2011) in zeolite production (Cheng et al., 2012) and other ceramic applications (Sembiring et al., 2014).

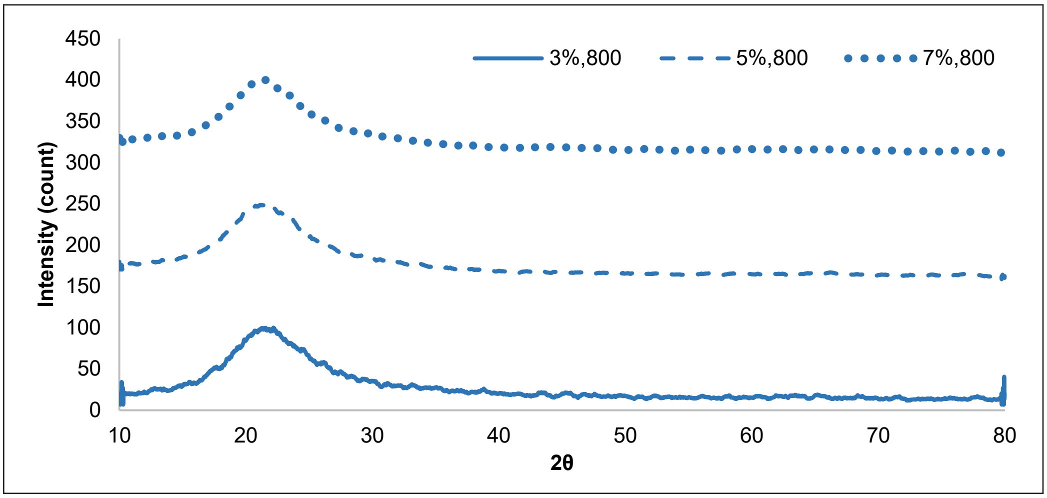


Figure 2. XRD analysis of SiO_2 on variations of HCl 3%, 5%, and 7%

Electrical Properties of SiO_2

The electrical properties' measurements aim to determine the electrical characteristics of extracted SiO_2 sample. Furthermore, the electrical conductivities of the SiO_2 were recorded at different frequencies. This study collected data at 200 points within a frequency range of 50 Hz to 5 MHz to cover both the low and high-frequency regimes. Based on electrical conductivity value, a material can be categorized into insulator, semiconductor, and conductor. This study obtains semiconductor properties with conductivity values ranging from 10^{-8} S/cm to 10^3 S/cm (Kwok, 1995).

The graph of the relationship between electrical conductivity and frequency is shown in Figure 3. In the figure, the electrical conductivity of each sample increases steadily with increasing frequency. It is entirely consistent with the fact that increasing the frequency increases the kinetic energy of the particles in the sample, resulting in a significant amount of charge transfer between the capacitor plates. The increase in HCl leaching concentration value also causes the value of the electrical conductivity to increase. At frequencies of 50 Hz and below, the electric conductivity value is relatively the same for all the samples. However, at frequencies above 50 Hz, the highest electrical conductivity values were obtained for the sample upon leaching with 3% HCl. It is because, at higher frequencies, the content of the minority atoms also vibrates and impacts the conductance values. The EDS data in Table 2 shows that the treatment of 3% HCl has the highest impurity than the other samples due to the most significant conductivity values compared to the treatments of 5% and 7% HCl. Interestingly, the 5% HCl-leached SiO_2 sample gave the lowest conductivity values with a maximum value of 5.28×10^{-6} S/cm, a typical semiconducting material value.

An analysis of the capacitance also shows the ability of a material to store electric charge. Based on the capacitance value, the dielectric constant of the sample can be

Table 2
Electrical conductivity values at 800°C with HCl concentrations of 3%, 5%, and 7%

Frequency (Hz)	Electrical conductivity (S/cm)		
	HCl 3%	HCl 5%	HCl 7%
(100-800)	7.46×10^{-10}-2.90×10^{-8}	6.72×10^{-10}-2.03×10^{-8}	5.17×10^{-10}-2.51×10^{-8}
(10000-40000)	8.77×10^{-8}-2.45×10^{-7}	2.16×10^{-8}-5.58×10^{-8}	3.04×10^{-8}-9.33×10^{-8}
(>1000000)	2.51×10^{-6}-5.82×10^{-6}	4.99×10^{-8}-1.67×10^{-6}	4.46×10^{-7}-2.87×10^{-6}

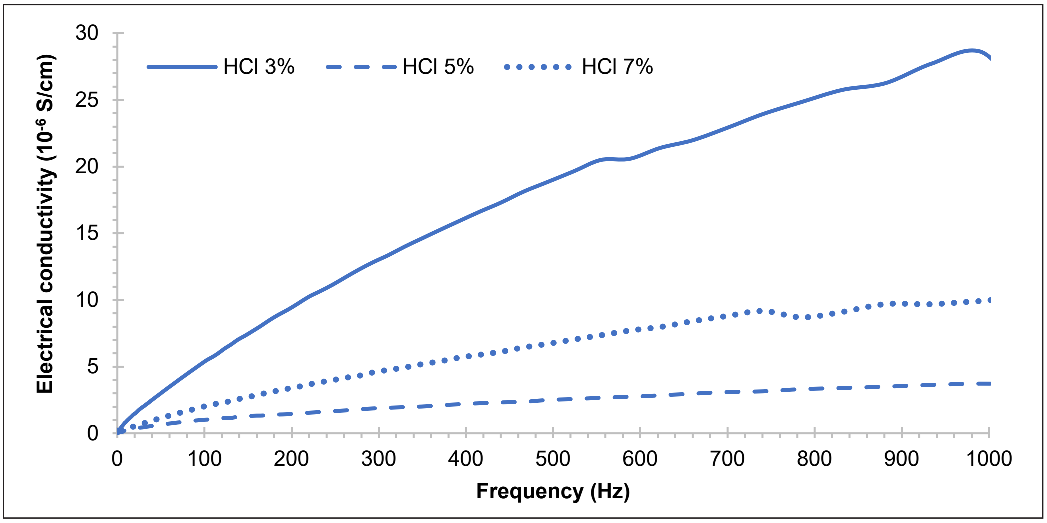


Figure 3. Results of electrical conductivity to the frequency of SiO_2 from rice straw in the treatment of HCl 3%, 5%, and 7%

determined. The results of data analysis of the dielectric constant values are shown in Figure 4. The frequency dependence shows that the frequency is inversely proportional to the dielectric constant value. All the SiO_2 samples showed a decreasing dielectric constant with increasing frequency but with different values. Each sample exhibits a high dielectric constant at frequencies less than 50 Hz and a low dielectric constant above 50 Hz. Therefore, SiO_2 obtained with treatment of 3% HCl gave the highest dielectric constant value compared to 5% and 7% HCl.

The effect of frequency on the electrical impedance value of SiO_2 samples from rice straw is shown in Figure 5. Electrical impedance is a measure of resistance at an alternating current source. It is the electrical resistance of an electronic component to the flow of current in a series at a specific frequency (Giancoli, 2020). The amount of electrical resistance that yields the impedance of a capacitive component is frequency-dependent. Therefore, the resistance of such a component will vary depending on the signal's frequency. The effect on the impedance of the capacitive electrical component SiO_2 samples from rice straw is shown in Figure 5. The graph displays the impedance-to-frequency relationship

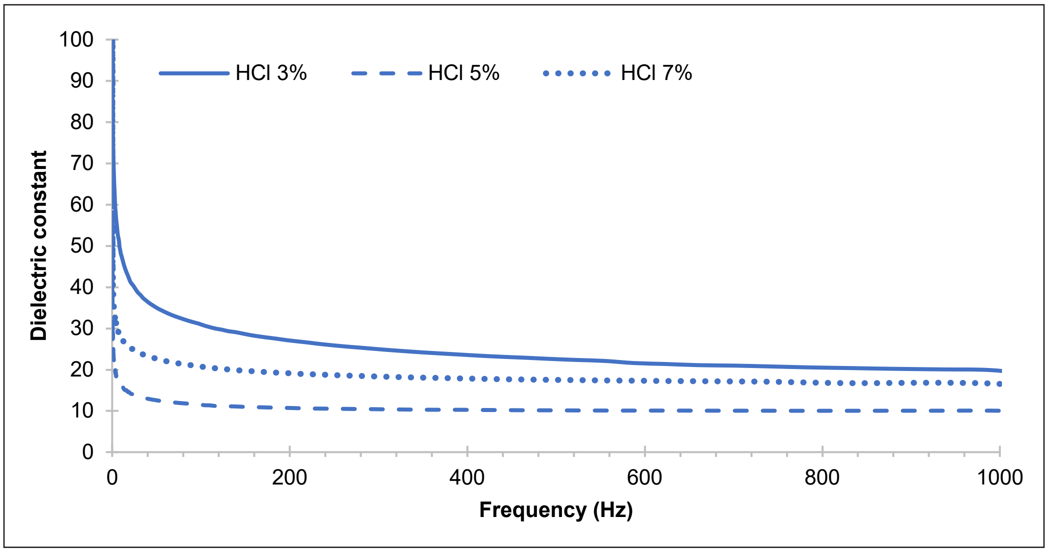


Figure 4. Dielectric constant values of samples at treatments of HCl 3%, 5%, and 7%

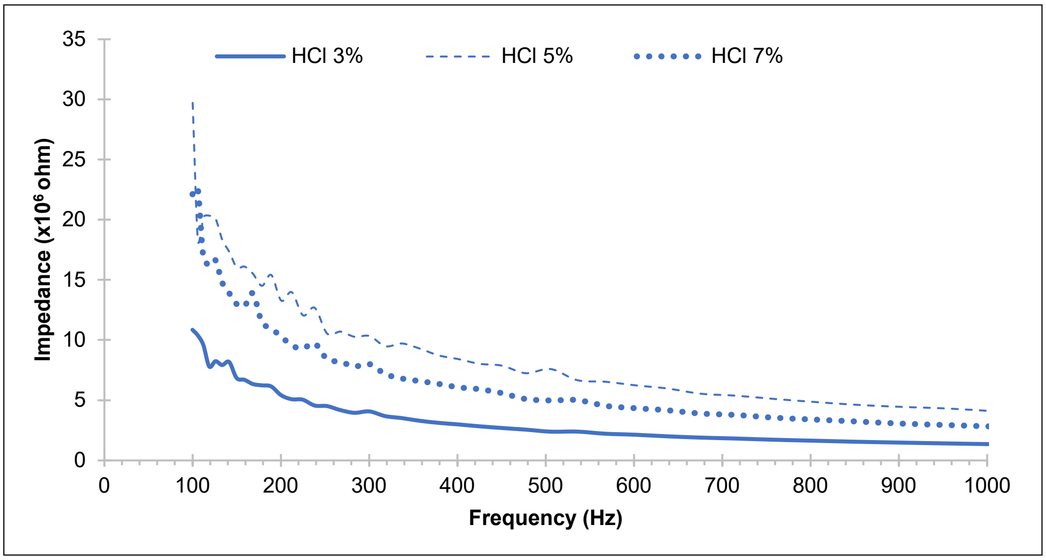


Figure 5. Impedance values of samples at treatments of HCl 3%, 5%, and 7%

in the frequency range 50 Hz to 1000 Hz. The frequency is inversely proportional to the impedance value. Therefore, sample impedance values below 100 Hz frequency are still volatile. Above 100 Hz, the impedance value decreases exponentially for all the SiO_2 samples. SiO_2 extracted with 3% HCl treatment has lower impedance values. The SiO_2 samples at 5% HCl treatment have higher values, while the sample extracted with 7% HCl showed impedance between the 3% and 5% samples. This value is supported by the electrical conductivity data of the sample. 5% HCl treatment samples gave the lowest electrical conductivity values. It means that such samples can inhibit the most significant electric current. The value of the electrical impedance is higher than the sample in the HCl treatment of 3% and 7%.

CONCLUSION

Based on the results obtained, it can be concluded that a higher concentration of HCl can increase the value of SiO_2 purity and can remove impurities. This result was supported by structural characteristics, which indicate the amorphous phase of SiO_2. Furthermore, based on electrical analysis, SiO_2 extracted from straw was in the range of semiconductor materials.

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