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Potential Use of Corn Cob Waste as the Base Material of Silica Thin Films for Anti-Reflective Coatings

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Abstract: This research involves silica extraction from corn cob waste with the sol-gel method using qualitative and ashless filter paper as a comparison. Anti-reflective coatings, as silica thin films, were synthesized from the extracted silica using dip-coating and spin-coating methods. Extracted silicas were characterized with FTIR, XRF, and XRD, while thin films were characterized with UV-Visible spectroscopy. The highest purity of extracted silica obtained was 66.5% using ashless filter paper. Silica thin films synthesized with spin-coating had a better refractive index compared to dip-coating, at 3.6.

Keywords: silica extraction, sol-gel method, dip coating, spin coating, anti-reflective coating

1. Introduction

Corn is a staple food in Indonesia, with a broad range of varieties and uses such as for human and animal food. According to data from Indonesia's Central Bureau of Statistics in 2019, 30 tons of corn is produced in Indonesia every year. With the large amount of corn produced, comes a large amount of waste generated as well. One part of corns considered as waste is the corn cob, which encompasses 30% of the weight of a corn. Based on that, we can assume that the amount of corn cob waste generated in 2019 amounts to about 9 tons. Usage of corn cobs are still very limited, including as cattle feed, fuel, and an alternative source of gas. If corn cobs were to be processed further, it could be used to extract silica, hence potentially becoming an alternative source for silica.

The chemical composition of a corn cob is generally 10.9% water, 36.48% cellulose, 28.86% hemicellulose, 3.16% lignin, and 20.6% silica[1)]. After combustion, the compositional percentage of silica in a corn cob will increase. Further processing based on silica extraction from corn cobs have not yet been applied on an industrial level. This is due to the limitation that current method of extraction silica form corn cobs has, in which the resulted purity of silica extracted does not reach the levels of pure silica, as reported by previous research[2)]. This still becomes one such challenge in extracting certain compounds from organic waste. As such, most silica extraction processes from organic waste are still conducted on a lab scale.

Silicon is one of the most-found elements on earth[3)], where it could be found in sources such as sand and biomass. Silica extracted from corn cobs are usually amorphous, as found in most biomasses[4)]. Processed silica in powder form is white, has no smell nor taste, and is inert in water and acids except fluoride acid[5)]. Even though the amount of natural silicon on earth is abundant, the cost of producing silicon and silica is still high, especially for high purity silicon[5)] . The production of silicon and silica from silica sand requires high energy for the conversion processes. Besides the need for high energy, this process also produces toxic gases such as carbon dioxide (CO2) in large quantities, hence endangering the environment. Due to this, researchers are searching for alternatives of silica synthesis with that have lower cost and are environmentally friendly[6)].

Silicon, its derivatives, and silicon-based compounds are used in a broad range of applications including as a structural material, a pollutant absorbent, in electronics, optoelectronics, photonics. and the energy field due to its characteristics which match the specifications needed for applications in those fields. The oxidized form of silicon is silicon dioxide, otherwise known as silica (SiO_2). This form of silicon can be extracted from various ashes from organic wastes, such as corn cobs[2)], groundnut shells[7)], and rice husks[8)]. Silica itself is an optically transparent material[9)]. Based on silica's high electric resistivity characteristic it is used as insulators in semiconductor and electronic devices[10)]. It also has a low refractive index, so it could also be used in anti-reflective coating

applications[11]. Silica gels are widely used as moisture absorbents and are often categorized as a traditional adsorbent[12]. This is because silica gel has excellent affinity with water[13]. Many researchers propose that it could be used to absorb pollutants, such as exhaust gases[14].

Focusing on anti-reflective coatings, it used to reduce reflection on a reflective surface[15]. As such, it has evolved into highly effective reflective and glare-reducing components for a variety of optical and optoelectrical applications[16], such as lenses, glasses, and telescopes[15]. One such use for in the optoelectrical field is in solar cells. A solar cell is an energy conversion device based on photonic energy from the sun. Its mechanism entails the capturing of photon energy from the sun by the solar cell which is then converted into electricity through various processes inside the solar cell. It is categorized as a renewable energy technology[17]. In conditions where sources of fuel, such as coal and oil, for energy are in limited amount, breakthroughs in the renewable energy fields are required to maximize the generation of a sustainable source of energy for the global society. One of the main parameters that could be optimized for solar cells is the conversion efficiency. One method of increasing the conversion efficiency of a solar cell is by utilizing anti-reflective coatings.

Anti-reflective coatings could increase the conversion efficiency of solar cells due to the Fresnel phenomena of interference of light in a thin film. An anti-reflective coating of a suitable thickness causes the front surface reflectivity of a solar cell to be mitigated significantly[18]. This solves the problem of the decrease of solar cell conversion efficiency caused by light reflecting from the solar cell[19]. This type of coating hinges on the refractive index of the thin films used for its application. As such, silica could be used as anti-reflective coatings for solar cells due to its low refractive index, stability at high temperatures, and scratch resistance properties[11]. The effectivity could be increased further if the silica thin films are made from nanoparticles, since the optical emission would increase due to decrease of particle size[20].

This research aims to find the efficiency of silica extraction from sweet corn cob waste, understand the properties of the silica extracted, as well as explore the potential and possibility of fabricating anti-reflective coating based on silica thin films synthesized from silica extracted from corn cobs. The method used in this research to extract silica from corn cobs is based on Okoronkwo's method[2], which is the sol-gel method, with modifications. The sol-gel is used in this study because it is reported to be a low-cost alternative to optical device fabrication[21] and an excellent method to produce a layer with a good property for coating applications[22].The sol-gel method process includes combustion on corn cob into corn cob ash (CCA), alkaline extraction, and acid precipitation. Alkaline extraction and acid precipitation is conducted to remove impurities from the silica, such as magnesium, potassium, sulphate, and calcium[23]. Sodium and carbon are also impurities that are removed in this process[24]. Silica obtained from this extraction method yields a purity of above 60%[25]. This research compares the type of filter paper used to filter sodium silicate produced from the alkaline extraction step, as well as compare the results of thin film synthesis using spin coating and dip coating methods. As such, the results of this research would adhere to a greater understanding of the better filter paper used to extract silica and the more superior method used to synthesize silica thin films for anti-reflective coating applications will be obtained.

2. Experimental

The method to extract silica from corn cobs was based on Okoronkwo's method[2], the sol-gel method, with a few modifications. Materials used in this research include sweet corn cob waste, Whatman Grade No.1 qualitative filter paper, Whatman Grade No.42 ashless filter paper, NaOH 1M, HCl 3M, distilled water, ethanol, and ITO glass. Characterization was carried out using Panalytical X'Pert Pro MPD, Panalytical Epsilon 1, PerkinElmer FTIR, and Shimadzu UV – 2450.

2.1 Extraction of Silica from Corn Cobs

Sweet corn cobs used in this research were sourced from a local farm in Cianjur, West Java. The corn cobs were first dried in the sun until fully dry, then cut into 2-3 pieces. The cut corn cobs were then burned in a furnace at 650°C for 3 hours with a heating rate of 10°C/min. This is done to ensure amorphous silica was obtained since amorphous silica could be obtained up to a combustion temperature of 750°C[26]. This combustion process resulted in corn cob ash (CCA). The efficiency of the combustion process was 1.8% based on the weight of CCA obtained.

Extraction of silica from CCA was done in two batches, in which one used Whatman Grade No.1 qualitative filter paper for the filtration process, while the other used Whatman Grade No.42 ashless filter paper. Ten grams of CCA was mixed with 50mL of 1M NaOH for 1 hour at boiling temperature. The resulting solution was then filtered using the before-specified filter papers. The residue left was thrown away after ensuring all traces of the solution were filtered out. The filtered solution, namely sodium silicate, was then titrated with 3M HCl until its pH was neutral, then incubated to form sol-gel. The addition of HCl to the sodium silicate caused silica to break apart from sodium, which further bonded with chloride, then precipitated to form gels. The created sol-gel was aged further for 18 hours before being gently broken down into slurry. The slurry was centrifuged at 2500 rpm for 5 minutes, which resulted in the separation of the gel and supernatant. This process was repeated a couple of times, in which the resulting supernatant was discarded each process until only the gel remained. The gel was stored in a beaker and dried in an oven at 80°C for

24 hours, which resulted in xerogel. The xerogel was then washed using distilled water, then dried again at 130°C for 1 hour. Washing silica xerogel with distilled water is more effective than washing the silica in its sol-gel form[8]. The results yielded silica powder, which was then ground with a mortar and pestle to a much finer form. The silica powder produced from the first batch was a very light red, while the silica powder produced from the second batch was white. Extraction from CCA had an efficiency of 10%, hence a total of 0.18% for the conversion of corn cob into silica. Both batches of silica powder were characterized using FTIR, XRF, and XRD tests.

2.2 Synthesis of Silica Thin Films

The synthesis of silica thin films was conducted by spin coating and dip coating methods. The precursor solution used was made from the two batches of silica powder extracted, with 0.5 grams each mixed with 10mL ethanol in different beakers at 50°C for 1 hour. The substrate used for this research was ITO glass that was cleaned using ethanol in an ultrasonic cleaner beforehand for 8 minutes. The first coating method conducted was spin-coating, in which the substrate was set on the spin coater and the rotational speed was set to 200 rpm. 4 drops (2mL) of the precursor solution were applied on the spinning substrate and left for 60 seconds. Afterwards, the rotational speed was increased to 1000 rpm for 120 seconds, then to 2000 rpm for 10 seconds, which finally it was turned off. The resulting thin films were left to dry before annealing inside a furnace at 400°C for 60 minutes.

The second coating method conducted was dip-coating, which was done manually. The substrate was held with pliers, while the precursor solution was set below it. The dipping-raising process was conducted for 1 minute before leaving it to dry. Three cycles of the dipping-raising process were conducted with a drying time of 10 minutes between cycles. The formed thin films were also subjected to annealing inside a furnace at 400°C for 60 minutes. The thin films synthesized from both the spin coating and dip coating methods were then characterized using UV-Visible Spectroscopy tests.

3. Results and Discussion

Analysis on the characterizations of extracted silica powder and silica thin films are done based on the results from FTIR spectroscopy, XRD, XRF, and UV-Visible spectroscopy tests.

3.1 Characterization of Silica Powder

3.1.1 FTIR Analysis

FTIR testing and analysis were conducted to identify the functional groups of a material. The presence of siloxane and silanol function groups in the material could identify silica. Fig. 1 and Fig. 2 shows the results of FTIR testing for batch one and two silica powder.

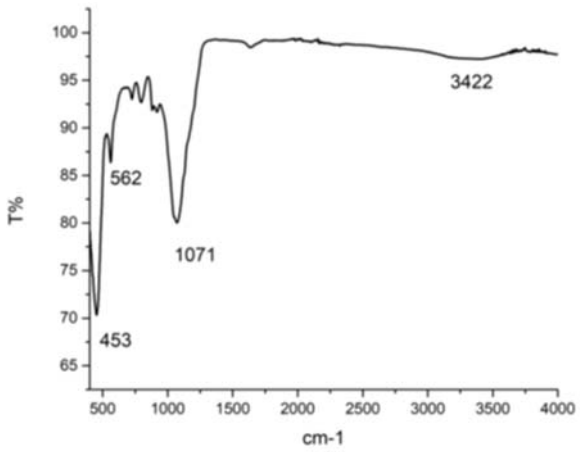

Fig. 1. FTIR of batch 1 silica powder

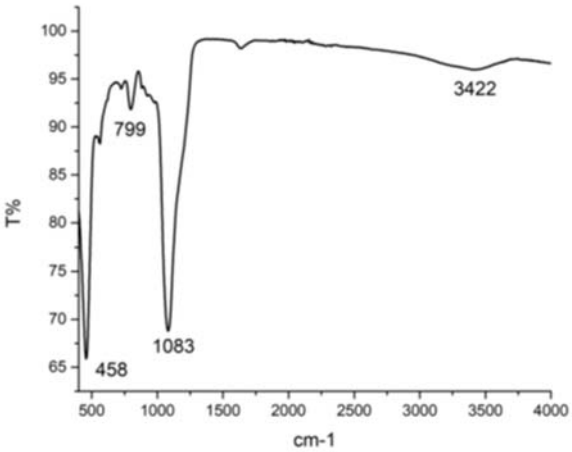

Fig. 2. FTIR of batch 2 silica powder

Based on the FTIR results shown above, FTIR of silica batch one indicates peaks at 453 cm-1, 562 cm-1, 1071 cm-1, and 3422 cm-1, whereas FTIR of silica batch two indicates peaks at 458 cm-1, 799 cm-1, 1083 cm-1, and 3422 cm-1. Peaks at 453-458 cm-1 indicate the presence of vibration mode O-Si-O bonds. The peak at 799 cm-1 found in the FTIR results of silica batch two indicates an asymmetrical bond of Si-O-Si, or siloxane, whereas the peaks at 1071-1083 cm-1 in both samples indicates symmetrical bonds of Si-O-Si. The broad peak at 3422 cm-1 in both samples indicate the low presence of silanol bonds, which are formed due to stretching vibrations of O-H from absorption of water. The important part to observe and compare from both graphs is the fingerprint region (600-1400 cm-1). Batch two silica powder shows a peak of asymmetrical siloxane at 799 cm-1, whereas batch 1 silica does not. The FTIR results of batch two silica powder correspond to FTIR results obtained by Okonronkwo[2] and Velmurugan[25], which have similar peaks indicating the presence of the same functional groups for the silica sample. This indicates that the forming of siloxane function groups in batch one silica did not occur as much as batch two silica, due to the presence of other impurities forming other bonds with oxygen, disallowing siloxane to be formed. This will be confirmed with the XRF test results.

3.1.2 XRF Analysis

The XRF results are represented by tables of composition of each batch of silica powder, based on the concentration of each element and compound in the sample. The result of batch one is in shown in Table 1, while the result of batch two is shown in Table 2. The concentration of silica of batch one silica powder in Table 1 is 59.608%, whereas the concentration of silica in batch two silica powder is 66.52%.

Table 1. Composition of batch 1 silica powder

Element	Conc.	Unit	Element	Conc.	Unit
Si	44.071	%	SiO_2	59.608	%
P	14.069	%	P_2O_5	17.017	%
Cl	2.653	%	Cl	1.313	%
K	26.435	%	K_2O	14.903	%
Ca	8.587	%	CaO	5.003	%
Mn	1.218	%	MnO	0.624	%
Fe	1.174	%	Fe_2O_3	0.664	%
Zn	1.793	%	ZnO	0.867	%
Total	100.00	%	Total	100.00	%

Table 2. Composition of batch 2 silica powder

Element	Conc.	Unit	Element	Conc.	Unit
Si	47.74	%	SiO_2	66.52	%
P	7.22	%	P_2O_5	8.95	%
Cl	15.92	%	Cl	8.09	%
K	28.73	%	K_2O	16.23	%
Zn	0.39	%	ZnO	0.20	%
Total	100.00	%	Total	100.00	%

Based on the XRF results, higher contents of silica are found in silica batch two compared to silica batch one. The composition of silica batch one shows a couple of impurities not found in silica batch 2, such as Ca, Fe, and Mn, as well as the oxides CaO, MnO, and Fe2O3. This proves and strengthens the reasoning as to why not as many siloxane groups were formed in the FTIR results. The presence of iron in the composition of batch one silica is the cause of the light red color of the silica powder observed before. Whatman Grade No.1 qualitative filter paper was used to filter sodium silicate to produce batch one silica powder and Whatman Grade No.42 ashless filter paper was used to produce batch two silica powder. Previous research involving the extraction of silica from corn cob ash also used ashless filter papers in their processes, producing high purity silica[25]. Hence, it could be concluded that ashless, or qualitative, filter papers are better to be used in the extraction of silica from corn cobs to obtain a higher concentration of silica.

3.1.3 XRD Analysis

XRD testing and analysis was conducted to identify the phases inside of the samples. It was also done to identify the structure of the silica formed from the extraction process. The XRD results are shown in Fig. 3

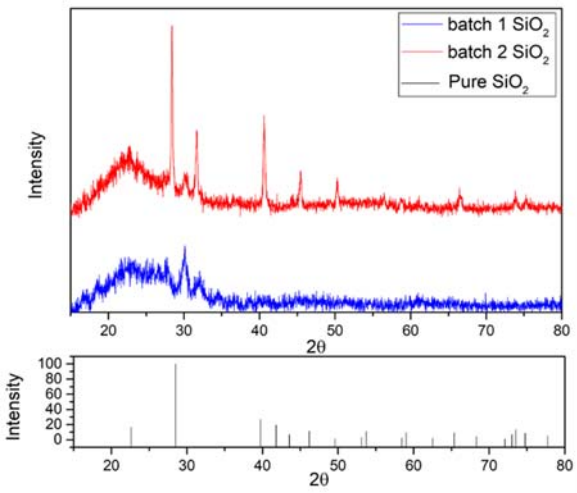

Fig. 3. Comparison of XRD pattern of batch 1 silica powder, batch 2 silica powder, and pure silica

Based on the XRD results, we can see that the diffraction patterns of silica batch one and two hold a similarity where both have a broad diffraction peak at the 2θ = 20-24° area, specifically at 23°. This indicates the presence of an amorphous silica phase. These results correspond with the XRD results of the silica samples in research conducted by Okoronkwo[2] and Velmurugan[25]. The difference between the results of this research compared to the before-mentioned literature are the presence of diffraction peaks of both samples, in which a diffraction peak at 2θ = 30° for silica batch one is observed, whereas diffraction peaks at 28°, 31°, 40°, 45°, 50°, 66°, dan 73° are observed in silica batch two. Based on the reference diffraction pattern of pure silica in Figure 3, we can observe a similarity with the diffraction pattern of silica batch two at 2θ = 28°. Thus, it shows that batch two silica also has a crystalline silica phase, alongside its amorphous phase. After further analysis utilizing the software, Highscore Plus, it is concluded that other diffraction peaks observed in the sample patterns indicate the presence of K_2O and Cl phases. This supports the findings from the XRF test before, in which both compounds are present in the compositional data of the samples.

3.2 Characterization of Silica Thin Films

The spectrum observed from UV-Visible testing is the reflectance of the samples, as shown in Fig. 4. The reflectance data could then be used to find the refractive index of the samples. Based on the graph, we can see that

the sample with the lowest reflectance is the thin film synthesized from batch two silica powder with the spin coating method, while the sample with the highest reflectance is the thin film synthesized from batch one silica powder with the dip coating method. The graph infers that the thin film synthesized from batch two silica powder results in a lower reflectance than batch one silica powder, and thin films synthesized using the spin coating method results in a lower reflectance than those using the dip-coating method.

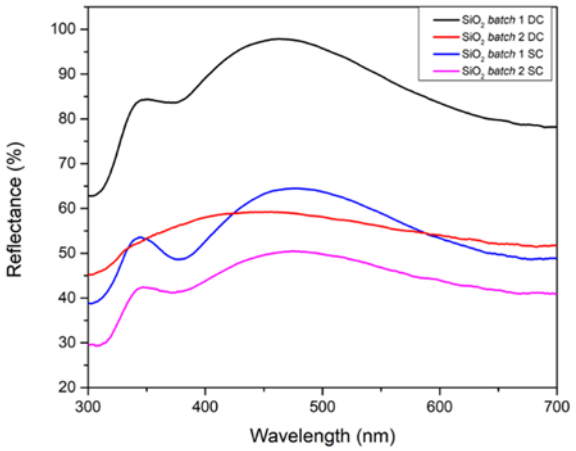

Fig.4. Comparison of silica thin films reflectance

If we consider the fact that batch one silica powder has a higher presence of impurities than batch two, we can conclude that the presence of impurities influences the reflectance, in which the more impurities are present in silica, the higher the reflectance. It can also be said that the purer the silica used to synthesize the thin film, the lower the reflectance generated. When we consider the reason behind the results in which the spin coating-synthesized thin films have a better reflectance than the ones synthesized via dip coating, the thickness and uniformity of the layer created becomes a prevalent factor. According to Oloomi, the thinner the non-metal layer formed, the smaller the reflectance created, while the thinner the metal layer formed, the larger the reflectance created[27]. From the XRF test conducted before towards the silica powders, we see that batch one silica powder has a composition of iron. This strengthens the reason why thin films synthesized from batch one silica powder has a higher reflectance than those synthesized from batch two.

The refractive index becomes an important parameter in understanding whether a certain material is suitable to be used as an anti-reflective coating. As mentioned before, the refractive index of the samples can be calculated from the reflectance data gained from the UV-Vis tests. We used the Fresnel Equation to calculate the refractive index of the samples while considering the refractive index of air, which is 1.

$$R = \left(\frac{n_{air}-n_{film}}{n_{air}+n_{film}}\right)^2 \qquad (1)$$

$$n_{film} = \frac{1+R+\sqrt{R}}{1-R} \qquad (2)$$

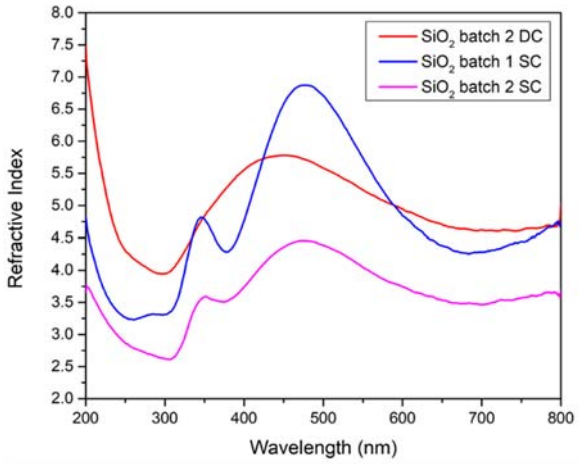

Fig. 5. Comparison of silica thin film refractive index

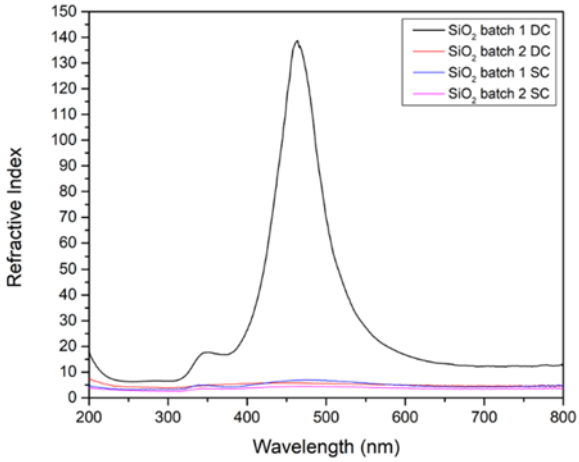

Fig. 6. Comparison of silica thin film refractive index except batch 2 silica thin film synthesized via dip coating sample

After calculations with the Fresnel Equation, the refractive index comparison graphs are obtained as shown in Fig. 5 and Fig. 6. The optimal refractive index for anti-reflective coating is 1.23 since it would eliminate all reflection on the glass[28]. The graph shows that all samples have refractive indices above 2. From the top, the sample with the highest refractive index at 632.8 nm is batch one silica thin film synthesized via dip coating, followed by batch two silica thin film synthesized via dip coating, then batch one silica thin film synthesized via spin coating, and the lowest being batch two silica thin film synthesized via spin coating. The refractive index of the samples in that order are 14, 4.8, 4.5, 3.6. Since the refractive index is related to the reflectance of the samples, the underlying reason why this occurred is due to the presence of impurities in the sample as well as the thickness and uniformity and samples not reaching their optimal state. The optimal thickness of a single layer anti-reflective coating can be calculated from the wavelength of light

used as a parameter divided by 4. Because the wavelength used as the parameter is 632.8 nm, the optimal thickness for the anti-reflective coating becomes 158.2 nm. The refractive index of a material increases with the increase of layer thickness[29]. As such, it is inferred that none of the thin films created in this research is optimal and could be used as an anti-reflective coating (ARC). Although that is the case, we can still understand which method creates thin films with better-suited properties for ARC application and the requirement of silica material needed for such thin films. Based on the graph from Fig. 6, we can see that the thin film with the nearest refractive index to the optimal value is the thin film synthesized from batch two silica powder with the spin-coating method. Whereas the thin film with the least optimal refractive index is the thin film synthesized from batch one silica powder with the dip-coating method. Hence, it could be concluded that spin coating is a better thin film fabrication method than dip-coating in terms of ARC application, and that the purer the silica used as the base material, the better refractive index it will have.

4. Conclusion

This research found out that the efficiency of silica extraction from sweet corn cob waste was 0.18% based on the weight gained. Thus, for each ton of sweet corn cob waste, we can obtain 1.8 kilograms of silica. The substantially low yield percentage from the corn cobs becomes one such limitation of silica extraction using this method. Characterizations conducted on the extracted silica showed that both batches of silica powder exhibited characteristics of silica as found in literature, in which they contain siloxane and silanol function groups. The structure of both silica sample batches is mainly amorphous as indicated by the broad diffraction peak at $2\theta = 20\text{-}24°$, while traces of crystalline silica are indicated in the batch two silica powder sample as indicated by the high-intensity diffraction peak at $2\theta = 28°$. Furthermore, batch two silica powder had a higher purity than batch one silica powder at 66.52%, with the latter at 59.608%. This shows that ashless, or quantitative, filter papers are a better option to be used in the silica extraction process than qualitative filter papers. Based on the methods used in this research to synthesize silica thin films, the method which approaches the optimum refractive index is the spin coating method using higher purity silica powder (batch two), with a refractive index of 3.6, while the method that resulted in thin films with properties furthest from the optimal state was dip-coating using lower purity silica (batch one), with a refractive index of 14.

Acknowledgements

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Nomenclature

R	reflectance
n_{air}	refractive index of air
n_{film}	refractive index of film

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