

Full Paper | <http://dx.doi.org/10.17807/orbital.v15i3.17915>

Extraction of Silica from Bamboo Leaves Ash (*Bambusoideae*) Using Hydrochloric Acid and Nitric Acid

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Bamboo leaves, a readily available agricultural waste globally, serve as a valuable source of organic silica, often referred to as biosilica. Despite containing a substantial silicon dioxide (SiO_2) content, bamboo leaves are frequently discarded by communities. Remarkably, the silica content in bamboo leaves exceeds 70%. Acid leaching is employed to extract the silica, with various factors and types of acids being compared in the process. The resulting silica is highly porous and firmly bonded. The extracted silica's purity, amorphous nature, and the presence of functional groups have been verified through XRD and FTIR studies. Additionally, TGA is utilized to monitor the material's weight changes with increasing temperature.

Graphical abstract



Keywords

Extraction
Silica
Rice husk

Article history

Received 18 Jan 2023
Revised 13 Jun 2023
Accepted 11 Sept 2023
Available online 10 Oct 2023

Handling Editor: Adilson Beatriz

1. Introduction

One of the most common plant species found in tropical and subtropical countries between 46 degrees north and 47 degrees south, bamboo is a member of the *Poaceae* family and the *Bambusoideae* subfamily. Bamboo has the potential to become the single most valuable economic resource for the people who live in this area. One of the native plants in Asia plays a major economic function in the livelihoods of local people living in this area. These woody-stemmed grass species are known as some of the fastest growing plants in the world [5]. More than 0.8% of the world's forest area is occupied by bamboo, and more than 31 million hectares of that is in China, Brazil, and India. It is also common in other

countries on three continents that were Asia, Latin America, and Africa [6].

Bamboo stands out as the optimal supplier of cellulose-based nanofibers, primarily because it is a rapidly growing plant with attributes that make it an excellent source of cellulose fibers. These fibers possess relatively short microfibrillar angles and boast a high cellulose content [2]. Malaysia boasts an impressive array of over fifty different bamboo species, with approximately 14 of them being commercially utilized. These specific species are favored due to their consistent size, thick culm walls, and ease of

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cultivation, making them some of the most employed bamboos in Malaysia. The versatility of this bamboo genus makes it a highly suitable choice for various industrial applications [3]. Conversely, as the forest resources, both in terms of quantity and quality of timber, continue to decline, there is a growing urgency to explore alternative construction materials instead of their traditional use. Wood is favored for its environmental benefits, such as renewability, biodegradability, and a favorable carbon footprint. Bamboo, with its comparable mechanical properties to wood and a faster growth rate, presents a promising alternative. Additionally, bamboo aids in carbon sequestration, further enhancing its potential as a viable substitute for wood as a raw material [4]. Numerous factors hold significance when seeking an alternative source to produce silica nanoparticles, including affordability, elevated silica content, equivalence in silica quantity, substantial energy content, as well as feasibility for cost-effective, sustainable, and large-scale industrial production [7]. With the growing demand for silica and its derivatives, there is a need for a synthesis approach that utilizes cost-effective and abundantly available resources efficiently. Furthermore, assessing the environmental impact and long-term sustainability plays a crucial role in selecting a suitable silica precursor of adequate quality. Solid waste generated by different sectors of society, such as agricultural residue, industrial byproducts, and consumer waste, all serve as examples [8].

The cultivation of economically significant crops including rice, wheat, bamboo leaves, sugarcane, corn, and others results in a daily influx of agricultural waste. Therefore, it is advantageous that most of this trash has higher silica content due to their absorption of silicic acid from soil, even though this disposal of this solid waste causes environmental problems. Investigation of this garbage's viability as a raw material for silica synthesis follows. Silica precursors can be made from a variety of waste products, including those derived mostly from the agricultural sector, such as rice husk, wheat straw and husk, bamboo leaves ash, sugarcane bagasse, and many more.

Bamboo leaves are one of the main ingredients in making bamboo charcoal and are also commonly used to wrap food in the Asian region. Since bamboo leaves are typically discarded after being used for building materials or pulp manufacture, they are a good source of biodegradable material waste [26-27]. Research on bamboo leaves first centered on their potential as a pozzolanic material in cement manufacturing. The presence of a higher silica level, prompting further investigation into this potential silica source) [16-19]. It has been stated that the proportion of silica in bamboo varies depending on factors including the leaf maturity, soil, and country of origin. Chemically processing residual bamboo leaf from thermal combustion to create amorphous silica could be profitable, as described in a 2017 study by Rangaraj and Venkatachalam. In conclusion, their research was a success. 99% pure amorphous silica nanoparticles (average size 25 nm) are extracted at a yield of 50% [7].

2. Results and Discussion

Fourier Transform and Raman (FTIR)

In **Figure 1**, which illustrates the FTIR results of silica BLA treated with hydrochloric acid, the observed functional group corresponds to carboxylic acids. In this experimental analysis, the absorption bands detected in the range of 1050-1040 cm⁻¹

are indicative of the CO=O group, which is a characteristic feature of anhydrides. The peak observed at 1042.02 cm⁻¹ can be attributed to CO-O-O stretching vibrations, and the sharpness of this peak indicates asymmetric deformations of O-CO bonds.

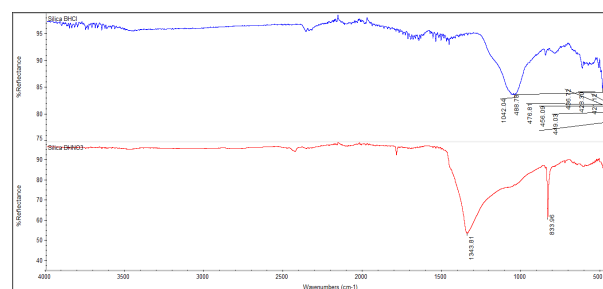


Fig. 1. FTIR spectrum of silica BLA using a) hydrochloric acid and b) nitric acid.

Similarly, in the figure displaying the FTIR results of silica BLA treated with nitric acid, the functional group identified belongs to carboxylic acids. In this experiment, the absorption bands in the range of 1350-1342 cm⁻¹ correspond to the C=O group, which is characteristic of sulfuric acid. The peak observed at 1343.81 cm⁻¹ is attributed to S=O stretching vibrations, and the sharpness of this peak suggests asymmetric deformations of -S-O bonds.

When silica is subjected to acidic extraction, its FTIR spectrum can undergo alterations due to its interaction with the acid. For instance, the utilization of a potent acid like hydrochloric acid (HCl) can trigger a reaction with silica, resulting in the formation of silicic acid (H₄SiO₄) or other soluble silica compounds. This chemical reaction may lead to the weakening or complete disappearance of the Si-O-Si stretching band in the FTIR spectrum, indicating the dissolution or transformation of silica. Furthermore, depending on the specific acid used and the reaction conditions, various other spectral changes may be observed.

For example, when sulfuric acid (HNO₃) is employed, it can react with silica to create nitrate species. This reaction can manifest as the appearance of new bands in the FTIR spectrum that are associated with sulfate groups.

Moreover, the acidic extraction process can occasionally result in surface modifications or etching of silica particles, which can be discerned through FTIR analysis. The emergence of new peaks or shifts in existing peaks within the spectrum may signify the presence of surface functional groups or alterations in the silica's structural composition.

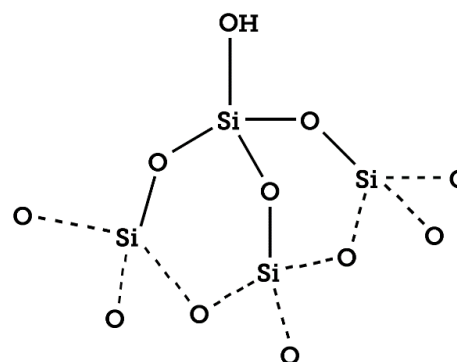
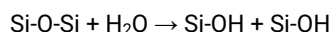


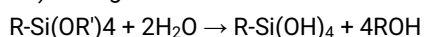
Fig. 2. Chemical structure showing silanol (Si-OH) and siloxane bond (Si-O-Si).

Acids can be used as solvents themselves in certain applications, and their solvent properties can vary depending on the specific acid and the substances being dissolved or reacted with. In the context of HCl (hydrochloric acid) extraction, the interaction between silica and HCl can result in the formation of silanol groups on the silica surface. As shown in Figure 2, silanol groups (-Si-OH) are formed when the hydroxyl (-OH) group of water reacts with the surface silicon atoms of silica, which have a propensity to bond with oxygen. The reaction can be represented as follows:

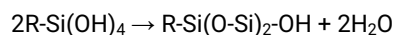


In the presence of HCl, the reaction is promoted by the acidic conditions. HCl can act as a catalyst and enhance the hydrolysis of the silica surface, resulting in an increased formation of silanol groups.

In the sol-gel method, silica-based silicate powders are typically synthesized through the hydrolysis and condensation of silicon alkoxides, such as tetraethyl orthosilicate (TEOS) or tetramethyl orthosilicate (TMOS), in the presence of a solvent and a catalyst. This process leads to the formation of a gel, which is subsequently dried and calcined to obtain the final silicate powder. During the sol-gel process, the chemical structure of silica in the resulting silicate powder undergoes several stages of transformation. Initially, the hydrolysis of the silicon alkoxide precursor leads to the formation of silanol groups (Si-OH) through the reaction with water:



Here, R represents an alkyl group (e.g., ethyl or methyl), and R' represents another alkyl or aryl group. As the condensation reaction progresses, the silanol groups can undergo further condensation reactions, resulting in the formation of siloxane bonds (Si-O-Si) and the release of water molecules:



The ongoing progression of condensation reactions persists, ultimately culminating in the generation of an

intricate, three-dimensional lattice of interconnected silicon dioxide (silica) structures. Within this network, opportunities for branching, cross-linking, and the inclusion of various elements or functional groups emerge, contingent upon the precise choice of precursors and additives employed in the sol-gel methodology. The ensuing chemical architecture of the silica present within the synthesized silicate powder via the sol-gel approach becomes profoundly contingent upon the prevailing reaction conditions, encompassing the selection of precursor substances, the composition of the solvent, the catalytic agents employed, and the temperature regimes applied. Through the deliberate manipulation of these parameters, the morphology, pore arrangement, and elemental composition of the ultimate silicate powder can be judiciously modulated. Importantly, it warrants mention that the sol-gel technique facilitates the introduction of additional metal oxides or dopants during the synthesis procedure, thus yielding composite silicate powders endowed with augmented properties tailored to specific applications [30, 31].

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis, also known as TGA, is a method of thermal analysis that measures the change in weight of a material as a function of temperature. This method can provide information about the relative thermal stabilities of these silica packing materials, which in turn gives an indication of the bond strength between the silica and the active phase. The figure below shows the different comparison of weight loss between the silica BLA of hydrochloric acid and silica BLA of nitric acid. The comparison of silica BLA using nitric acid exhibited a smaller total weight loss over the temperature range evaluated. For both different types of acid, the initial weight loss represented the loss of surface moisture, while the second weight loss corresponded to loss of active phase.

Table 1. TGA analysis of silica from BLA using different types of acid

Types Of Acid Use To Extract Silica From Bla	Heating Rate (°C/Min)	Sample Mass (Mg)	Purge Gas	Temperature Range (°C)	Weight Loss (G)
HCl	20	Approximately 2	Nitrogen	37-1200	84.71
HNO ₃	20	Approximately 2	Nitrogen	37-1200	10.82

Table 1 showed the TGA analysis of silica from BLA using different types of acid. The first type of acid that used to extract silica from BLA was HCl. For the HCl, the heating rate was 20 °C/min and sample mass that used was approximately 2 mg. Then, nitrogen gas was used as for the purge gas. The temperature range that used was 37 °C to 1200 °C. Thus, from that, weight loss of the TGA analysis for silica from BLA using HCl was about 84.7114 g.

Then, the second acid that was used for extracting silica from BLA was HNO₃. For the HNO₃, the heating rate was 20 °C/min and sample mass that used was approximately 2 mg. Then, nitrogen gas was as for the purge gas. The temperature range that used was 37 °C to 1200 °C. Thus, from that, weight loss of the TGA analysis for silica from BLA using HNO₃ was about 10.817 g.

The thermogravimetric analysis method was utilized in order to ascertain whether or not the bamboo leaves ash contained any organic components. **Figure 2** and **Figure 3** depict the thermogravimetric curves of silica BLA using hydrochloric acid and silica BLA using nitric acid. Initial weight

loss can be shown to take place anywhere between 50 °C and 150 °C, regardless of the presence of acid leaching process. This weight loss between these acids ranges differently corresponds to the loss of water and other volatile components.

The second stage demonstrates a significant and speedy weight reduction at temperatures ranging from 810 °C to 950 °C for the silica BLA using hydrochloric acid. Meanwhile, for the silica BLA using nitric acid was 640 °C to 740 °C. The reason for this is the heat degradation of hemicellulose and cellulose as the primary organic components in the bamboo leaves ash. According to Antal, 1983 and Shafizadeh, 2012, hemicellulose is primarily broken down between 150 °C to 350 °C. For the silica BLA using hydrochloric acid, hemicellulose primarily broke down between 380 °C to 810 °C. Then, the silica BLA using nitric acid, hemicellulose primarily broke down between 160 °C to 600 °C. This is because the component of that is the least stable. The bamboo leaves ash that had been acid-leached exhibited a decreased thermal stability. For the comparison, bamboo leaves ash that has not been leached, because of the acid hydrolysis of hemicellulose and cellulose

into molecules with lower weights could undergo thermal degradation with less difficulty. The weight loss between these acids shows a weight loss of approximately 10% to 86%,

which may be attributed to lignin, an aromatic compound that is more thermally stable. The ash that is left over is primarily composed of inflammable silica.

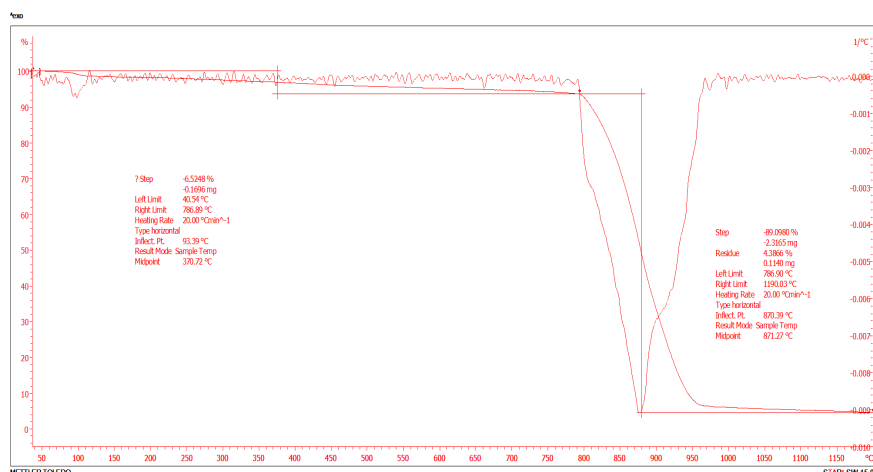


Fig. 2. Thermogravimetric curves of silica BLA using hydrochloric acid.

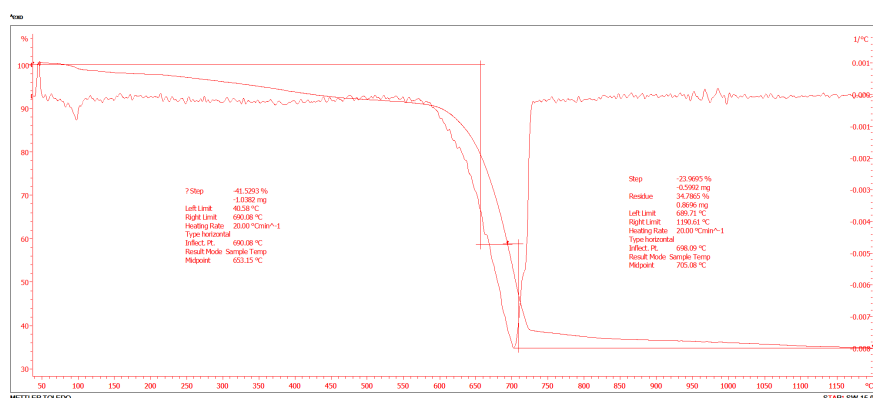


Fig. 3. Thermogravimetric curves of silica BLA using nitric acid.

Yield of silica

$$\text{Silica yield percentage (\%)} = \frac{\text{Actual Yield (g)}}{\text{Theoretical Yield (g)}} \times 100\%$$

Table 2. Percentage yield of silica from BLA using different types of acid

Types of acid	Types of samples	Weight of BLA used (g)	Weight of extracted silica (g)	Silica yield percentage (%)
HCl	HCl 1	5.0004	3.259	65.17
	HCl 2	5.0012	2.958	59.15
	HCl 3	5.0010	3.004	60.07
HNO ₃	HNO ₃ 1	5.0094	2.362	47.15
	HNO ₃ 2	5.0052	1.946	38.88
	HNO ₃ 3	5.0067	2.781	55.55

Table 2 shows the percentage yield of silica from BLA using different types of acid. First type of acid was HCl and the second type of acid was HNO₃. From the table above, we can see that the highest percentage yield of silica from BLA using HCL was from type of sample HCl 1 that was 65.17 %. Then, the lowest percentage yield of silica from BLA using HNO₃ was from type of sample HNO₃ 2 that was 38.88 %. Thus, the difference in this yield percentage were because of the acid that used in the experiment.

In addition to this, acid concentration is an important aspect that must be taken into consideration in the acid leaching procedure in order to produce desirable outcomes. In proportion to an increase in the molarity of hydrochloric acid and nitric acid, there was also an increase in the percentage of bamboo leaves ash. As can be seen in Table 2, the concentrations of the types of acid that were added to the bamboo leaves ash caused the purity of the silica (SiO₂) to drastically not static, going from 65.17 % to 38.88% for the different types of acid from BLA, respectively. The extraction technique utilizing pure solvents at the same concentration and ratio as described by Handayani et al., 2018, resulted in a lower percentage of yield. Silica production was hindered by using specialized solvents.

It has been demonstrated that different types of acid used for acid leaching were significantly useful and successful in increasing the purity of silica and removing contaminants from the leaves of bamboo [11-15]. Due to the presence of carboxyl groups in their overall chemical structure, organic acids like hydrochloric acid and nitric acid, for example, have the potential to operate as chelating agents. Because of their ability to donate protons (H⁺), these carboxyl groups have the potential to form stable complexes with a variety of metal ions. These complexes were characterized by their negative charge [16].

Comparison Mass of Silica Powder (g) Between Volume Required (mL) Using Hydrochloric Acid and Nitric Acid

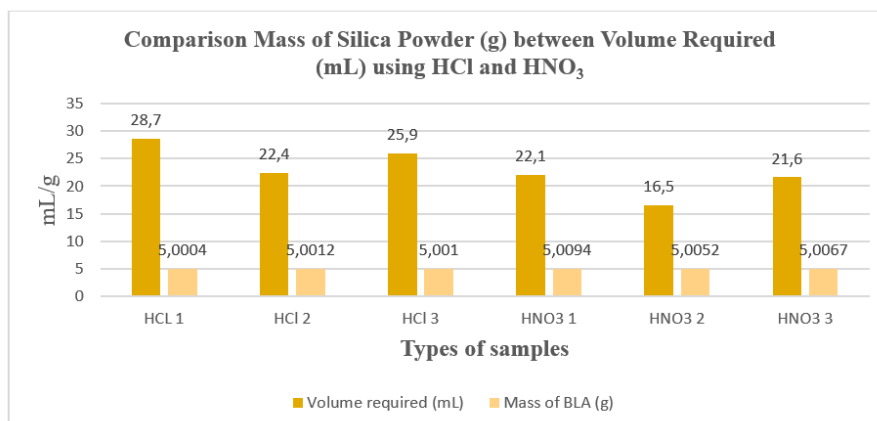


Fig. 4. Comparison mass of silica powder (g) between volume required (mL) using HCl and HNO₃.

From Figure 4, it showed the comparison mass of silica powder (g) between volume required (mL) using HCl and HNO₃. From the figure, we can see that volume of HCl (mL) required to extract silica from BLA were more than volume of HNO₃ required to extract silica from BLA. This is due to the mass of silica powder (g) that has been produced. According to the figure, HCl solution produces more of mass powder of silica (g) than HNO₃ solution. Furthermore, according to Liou et al., 2011, among the acid solutions, HCl possesses the higher performance as a BL leaching agent as compared to other types of acid solution. This was found in their research on acid solutions. Numerous researchers have demonstrated that it is the most effective leaching agent in generating high purity amorphous silica with better pozzolanic characteristics. Apart from that, through the acid solution also can make a considerable difference in both the surface area and the pore volume of the particle when there is silica present [18].

3. Material and Methods

Sample preparation of bamboo leaves ash

The BLA was decided upon as the derivation of the amorphous silica due to the amount of the material's availability. Bamboo leaves will be collected from Taman University Malaysia Kelantan, Jeli campus. A total of 5 kg of bamboo leaves will be collected. The bamboo leaves will be cleaned with tap water to remove any impurities or any soil that stuck around the bamboo leaves. After that, the bamboo leaves will be cut into smaller pieces so that it will make easier to dry [24-27] Then, the bamboo leaves will be put into aluminium foil and then dry in oven for 90°C for duration 24 hours [30]. Next, the bamboo leaves were calcined in manual furnace that have in the wood workshop in UMK in 300°C for 30 minutes to produce coal. After that, the coal will be calcined in muffle furnace for 800°C for duration 3 hours to produce white ashes. This whites ashes next will be used in the extraction of silica [31].

Extraction of silica from bamboo leaves ash (Sol-gel method)

5 g of bamboo leaves ash were put into a beaker. Then, 30 ml of NaOH (2M) will be added into the beaker. The mixture was then boiled for 1 hour with constant stirring in a hot plate.

Then, this mixture will produce sodium silicate solution. Figure 3.1 showed an overview of the types of processes that can be done with sol-gel method and the products of each process [30-31].

Acid leaching process

Thus, after the cooling process, 1 M of HCl were added to the sodium silicate solution to reduce pH until 7.0 under constant stirring. Then, silica was produced. Then, the silica will go through an aging process [20-23]. The silica was then filtered with Whatman No 41 ashes less filter paper and washed with distilled water so that any impurities would be discarded. After that, the gel was dried at 80°C for 15 hours. At last, the white powder silica will be produced. This process will be repeated by using nitric acid to replace hydrochloric acid. The results will then be compared [32-33].

4. Conclusions

According to the study's findings, silica extracted from bamboo leaves ash exhibited desirable qualities suitable for practical applications. All the analyses utilizing X-ray diffraction, Thermogravimetric analysis, and Fourier Transform Infrared and Raman have confirmed this. Thus, from the result of analysis FTIR, it demonstrated the presence of aliphatic amide group, proving them to be chemicals with a relatively high boiling point to form strong intermolecular connections. Then, for TGA, it calculates how much energy will be expended in breakdown reactions and how fast silica weight and the temperature will rise and fall. Next, quantitative composition analysis is a common use of the measurements. Moreover, TGA also can be used to determine the amount of water or residue solvents in a material. Furthermore, porous level can be determined by XRD. This low energy technique, which involves the alkaline solubilization of amorphous silica, has the potential to be less expensive than the conventional smelting process. As a result, it offers less alternative energy than the prevalent high energy approach. Multiple commercial uses of BLA have emerged after a low energy chemical process that was developed to extract silica from bamboo leaves.

The use of a combination comparison of HCl and HNO₃ for

silica extraction can offer advantages over using a single acid. The combination may provide better control over the reaction, improve silica dissolution, or enhance impurity removal. This acid combination may result in improved silica purity, yield, or other desirable characteristics compared to conventional methods using only one acid. The novelty could also lie in the specific silica source being targeted for extraction. The extraction is focused on recovering silica from unconventional or waste sources, such as industrial byproducts or agricultural waste which is bamboo leaves ash (BLA), the novelty would be in adapting the acid extraction method to BLA.

Acknowledgments

Authors would like to thank the Malaysian Government and the University Malaysia Kelantan for sponsoring this study through a grant number RACER/1/2019/STG01/UMK//2.

Author Contributions

Conceptualization; Siti Nurul Huda Binti Azman, Nadiah Binti Ameram, Data curation; Siti Nurul Huda Binti Azman, Nadiah Binti Ameram, Formal Analysis; Siti Nurul Huda Binti Azman, Nadiah Binti Ameram, Funding acquisition; Siti Nurul Huda Binti Azman, Nadiah Binti Ameram, Methodology; *, Nadiah Binti Ameram, Hidayani binti Jaafar, Mohd Hazim Mohamad Amini. Supervision, Nadiah Binti Ameram Visualization; Hidayani binti Jaafar, Mohd Hazim Mohamad Amini, Arlina Ali. Writing – original draft; Siti Nurul Huda Binti Azman, Nadiah Binti Ameram. Writing – review & editing; Hidayani binti Jaafar, Mohd Hazim Mohamad Amini, Arlina Ali. All authors have read and agreed to the published version of the manuscript.

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How to cite this article

Azman, S. N. H. B.; Ameeran, N. B.; Jaafar, H. B.; Amini, M. H. M.; Ali, A. *Orbital: Electron. J. Chem.* **2023**, *15*, 142. DOI: <http://dx.doi.org/10.17807/orbital.v15i3.17915>