Research Article

Characterisation of silica derived from Bamboo Leaves Ash (*Gigantochloa albociliata*); preliminary results for moisture content test on rubber wood

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ABSTRACT

Bamboo leaves (*Gigantochloa albociliata*) constitute substantial amounts of agro-waste from farming and planting activities, offering valuable biomass resources rich in silica. Employing the sol-gel method facilitates silica extraction from bamboo leaves ash (BLA), opening avenues for various applications. Characterisation of the obtained silica involves FTIR analyses providing insights into both chemical and physical properties. FTIR analysis highlights a significant chemical group in SiO_2 at 3381.25 cm^{-1}, corresponding to –OH groups of -Si-OH, indicating strong and intense OH-stretching vibrations. This research explores the potential of silica from BLA for practical use. Utilizing silica as a moisture absorbent reveals promising properties. Selection of bamboos as a source of silica is grounded in scientific rationale. Its abundant silica content, inherent in its anatomical structure, offers a readily available and prolific natural source. The rapid growth and sustainability of bamboos further align with eco-friendly practices, showcasing its minimal ecological footprint and swift regeneration compared to alternatives. Opting for bamboo as a silica source not only taps into its mineral richness but also promotes a sustainable and environmentally conscious approach. This strategic choice supports the overarching goal of encouraging green and renewable resources across various industries

Keywords: *gigantochloa albociliata;* silica; sol gel

1. Introduction

Gigantochloa albociliata, commonly known as "buluh madu," is a type of bamboo plant originally native to Burma and Thailand. It was later introduced to India and can now be found in many Asian countries, recognized as a tropical Asian and Papuasian genus of giant clumping bamboos in the grass family. In China, bamboo leaves have a long history of medicinal use due to chemical groups such as flavonoids, coumarins, lignans, anthraquinones, and polysaccharides (Weiping *et al.,* 2023). All these components can be easily identified in the leaves after extraction. The extracted bamboo leaves are beneficial, with shoots of the bamboo and part of the leaves, having positive impacts on the human body, including anticancer, antibacterial, and antiviral activities. Bamboo shoots also possess antioxidant capacity due to the presence of phenolic compounds (Chongtham *et al.*, 2011), which are essential for the quality of plants, especially food-based plants, influencing the color of fruits and juices and contributing to flavor properties. For decades, the application of antioxidants in the food and pharmaceutical industry has been widespread. In the past, antioxidants were previously used to control oxidation and retard spoilage but today, many are used because of putative health benefits. With increasing health consciousness in consumers, there is much demand of food for health and wellbeing (Chongtham *et al*., 2018).

Bamboo species are abundant in Malaysia, totaling around 70 species across Peninsular Malaysia, Sabah, and Sarawak, playing a significant role in the forest products of the country and contributing to its vital income (Aminullah *et. al.,* 2015). In Malaysia, where vast forest areas are prevalent, bamboo stems are extensively used, particularly in construction, underscoring their pivotal role. However, bamboo leaves, though utilized, often go unnoticed in terms of their importance, rendering them potential agro wastes. Despite the varied uses, including turning leaves into consumables or medicines, demand remains relatively low. Unfortunately, post-harvest, excessive bamboo leaves often end up being burned, contributing to environmental issues and the release of harmful gases (Chin *et al*., 2017).

The research on extractions encompass various methods such as solid-phase extraction, liquid-liquid extraction, and acid-base extraction. However, this study focuses on the sol-gel method, a unique technique where nanoparticles in a solid state disperse in a liquid, forming a gel through agglomeration. This method is particularly suitable for molecular separation using membranes, offering an energy-efficient alternative to conventional industrial separation techniques (Robert *et al.,* 2008). In the sol-gel process, a stable colloidal solution called sol is obtained through hydrolysis and partial condensation of precursors. Gel materials, created by the condensation of sol particles into three-dimensional networks, can result in solid products known as xerogel and aerogel through evaporative drying or extraction processes.

The silica obtained in this research is called BLA silica, present in the form of white powder, also known as xerogel. Xerogels are derived from sodium silicate by the sol-gel method followed by the drying process. To produce silica gel, the sodium silicate solution needs to be neutralized with hydrochloric acid (Ameram *et. al.,* 2019). The addition of acid to the sodium silicate can cause rapid gelation. The results will be compared to silica from other sources, particularly from other studies such as those using ash from rice husks. Many people are unaware of the uses and importance of silica in life. Bamboo leaves, in abundance, are often overlooked, leading to uncontrolled burning and contributing to global warming. Specifically, there is a lack of understanding regarding the uses of SiO_2 in the field of forest resources technology. The expected outcome of this research is in obtaining silica, contributing to addressing this knowledge gap.

Furthermore, previous research in this field has neglected to conduct a comparative analysis of silica derived from various sources, a focus that distinguishes the present study. In a previous investigation, the identification of organic compounds within rice husks was limited to NaOH treatment, with no subsequent advancements post-identification (Fernandes *et al.,* 2017). This study aims to compare the characteristics of silica obtained from the calcination process of bamboo leaves, labeled as bamboo leaves ash (BLA), with that from rice husk ash (RHA), as explored in previous research.

The primary objective of this research is the extraction and characterisation of BLA silica. The choice of bamboo leaves is motivated by their relatively high silica content, as opposed to other plants like rice husks, although the latter has a higher silica content. The comparison in this study focuses on forest products with substantial silica content. The bamboo leaves used were sourced from Agropark Universiti Malaysia Kelantan Jeli campus, Kelantan. To extract silica from BLA, the research used the sol-gel extraction method, as it offers distinct advantages. Among the advantages of using the sol-gel process in the synthesis is that it can be carried out at room temperature, it allows us to produce a wide range of novel and functional materials, with potential applications in different areas; and finally, it is more attractive compared to other methods, due to its low production costs.

Sol-gel samples can be designed with a wide variety of morphologies, such as monoliths, films, fibers, and powders (Kalapathy *et al.,* 2002). In particular, films are the most important from the technological point of view. This method, creating solid materials from small molecules, operates at comparatively low temperatures and produces a fine powder. The results of this extraction method highlight the potential applications of bamboo leaves, particularly in moisture absorption and as an anti-sticking agent. The current study includes a simple experiment to assess the moisture-absorbing capabilities of silica produced from agro-wastes. Four woods of specific and similar weights were placed in separate Petri dishes under different conditions: one with silica from RHA, another with silica from RHA along with commercial silica, and a control without any silica. Changes in moisture content were measured by weighing the pieces of wood after a designated period.

2. Materials and Methods

2.1 Preparation of bamboo leaves ash (BLA)

BLA was chosen as the source of amorphous silica, which is abundant. The silica was extracted from bamboo leaves using the methods as described by Ameram *et al.* (2019). Bamboo leaves were collected from Agropark UMK. Next, bamboo leaves were washed with a considerable amount of water and then rinsed with distilled water. After drying, the bamboo leaves were crushed into small sizes. They were then leached and stirred with 1 M hydrochloric acid (HCl) at room temperature (27°C) for about 2 hours (Chanade *et al.*, 2016). The bamboo leaves were then rinsed and washed with distilled water until the leaves were neutralized to pH 7.0. After the leaves were left to dry, the bamboo leaves were calcined in a muffle furnace at 700 °C for a period of 3 hours. The white ashes are used as the source of silica in this study.

To analyse bamboo leaves ash using FTIR spectroscopy, the process involved collecting and grinding the ash into a fine powder (Hamizah *et al.,* 2016). A small amount of the powder was placed on the FTIR sample holder, ensuring a uniform pellet. Background spectrum was recorded and subtracted for baseline correction. The instrument was set to a suitable resolution and scans were accumulated. The resulting spectrum was analysed identifying characteristic peaks corresponding to functional groups in the bamboo leaves ash. The composition was qualitatively assessed based on peak identification, and if needed, quantitative analysis was performed using calibration curves or standards. Instrument settings, sample details, and spectral interpretation were documented for reporting. This method provided insights into the chemical composition of bamboo leaves ash, aiding in its characterisation.

2.2 Extraction of the Silica from BLA (Sol-gel method)

Silica was extracted from bamboo leaves ash (BLA) by using sol-gel method to produce the white powder silica, also known as xerogel. A total of 5.0 g of silica extracted from BLA was taken and added with 30 mL of NaOH and boiled for a duration of 1 hour with constant stirring to extract the silica. While silica was extracted, the sodium silicate solution produced was filtered through Whatman No.41 filter (Norsuraya *et al.,* 2016). The filtrate sodium silicate solution was cooled to room temperature and the pH solution was reduced with 1N HCl to 7.0 with constant stirring until the silica gels were produced. The aging process took 18 hours after the solution gel was formed. After aging, the solution gel was filtered and washed by using distilled water then the supernatant was discarded and the gel was dried at 80°C for 12 hours. After the drying process, the white powder silica, also known as xerogel was produced.

2.3 The study of moisture content

A simple experiment was done to measure the changes in moisture content of the pieces of wood. Four small pieces of wood of similar weight were taken as the materials in this experiment. They were put in four different Petri dishes. Three of the Petri dishes contained various types of silica including silica from BLA, silica from RHA sources and common silica that is commercially available. One Petri dish was set as the control without any silica. The weight of silica was also set as a controlled factor, which was 0.2 g for every Petri dish with silica. Moisture content of the pieces of wood were measured by weighing the changes in weight after 2 hours, 5 hours, 24 hours and 48 hours, respectively. The initial weight of all rubber woods was 1.29 g.

3. Results and Discussion

Extracted silica from RHA and BLA is shown in Figure 1. From the naked eye, we can see similar structure of white powder of porous silica for both derivation. The extraction of silica from both biomasses can be understood by studying this process (Okoronkwo *et al.*, 2013). The initial stage of this research was the process of silica extraction from bamboo leaves. This process is based on the principle that silica dissolves in an alkaline solution and easily precipitates when acid solution is added until it reaches neutral pH of 7. The reaction that occurs between SiO_2 contained in bamboo leaves when dissolved in NaOH solution is shown in equation (1):

$$SiO_2(s) + 2NaOH(aq) \rightarrow Na_2SiO_3(aq) + H_2O(l) \quad (1)$$

The reaction produces a solution of sodium silicate. The sodium silicate solution was reacted with H_2SO_4 solution until SiO_2 gel was obtained. The reaction is shown in equation (2):

$$Na_2SiO_3(aq) + H_2SO_4(aq) \rightarrow SiO_2(s) + Na_2SO_4(aq) + H_2O(l) \quad (2)$$

The H_2SO_4 solution serves to provide an acidic condition, removing the impurities of inorganic compounds present in bamboo leaves ash and reacts with Na_2SiO_3 producing Na_2SO_4, SiO_2, and water in neutral pH in gel form. In this process, the formation of siloxane functional groups (Si-O-Si) and silanol (SiOH) groups occur to form silica gel. The sodium sodium salt ions formed and attached to the silica gel can be removed by washing it using the hot DM water repeatedly. The silica gel is heated in the oven at a temperature of 110°C resulting in dehydration of the silica gel and forming an amorphous and hard solid.

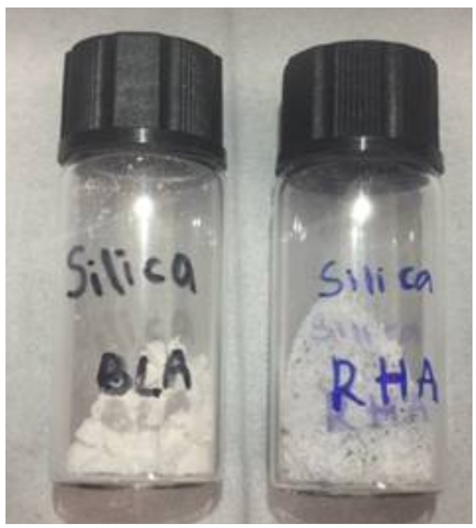

Figure 1 Silica BLA and silica RHA.

3.1 Fourier Transforms Infrared (FTIR) Spectroscopy analysis

Fourier Transforms Infrared (FTIR) Spectroscopy is a technique commonly used in characterizing silica. FTIR uses interferometry to record information about materials placed in the infrared spectroscopy beam. The results that can be analysed in spectra can be used to determine or to quantify the material. Some inorganic, organic and also polymeric materials can be identified by using this method. The FTIR test uses infrared light to observe some chemical characteristics and to scan the samples.

FTIR was used as a technique to analyse the key functional groups along with the organic and polymeric materials as well as to identify the inorganic materials that can be found in the silica derived from BLA in this current research and from RHA in another related study. The data have been interpreted as in Figure 2.

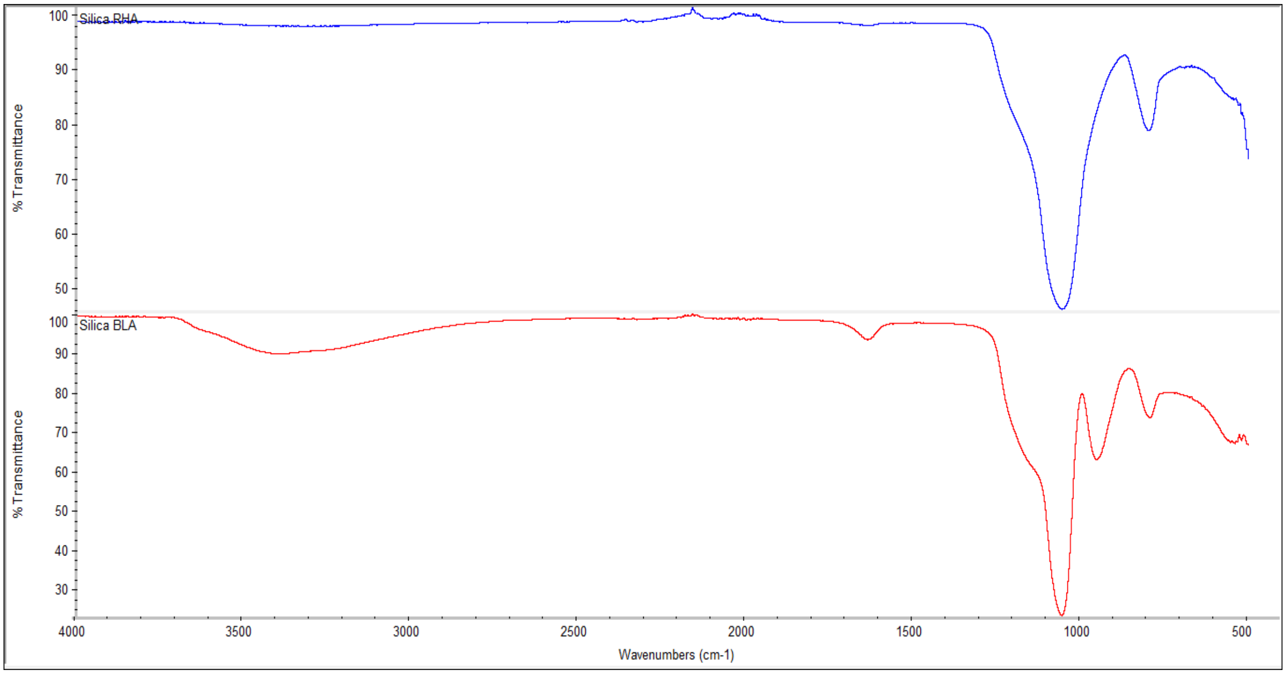

Figure 2 FTIR results for silica from rice husk ash (RHA) and silica from bamboo leaves ash (BLA).

From Figure 2, one of the major chemical groups that can be found in SiO_2 (BLA) is shown at band 3381.25 cm^{-1} which is the peak and responsible for the functional –OH and -Si-OH groups. The band shows that the absorption characteristics of the silica are strong and intense by the OH- stretching vibrations of free surface silanols (Pallavi Deshmukh *et al.,* 2012). Band 1635.37 cm^{-1} is assigned in the range of bonds that have strong C=O stretch, indicating the presence of aliphatic amide groups. Silica as in amide groups are proven as chemicals with quite high boiling point because they are capable of strong intermolecular interactions. Aliphatic or aromatic groups do not just have high boiling point but they also have generally high melting point relative to their size. In one study, the production of sugarcane silica from sugarcane bagasse was studied. FTIR spectra of sugarcane silica were documented on an instrument (Shimadzu FTIR 8400) ranging from 400–4000 cm^{-1} with a resolution of 4 cm^{-1}. Moreover, sugarcane bagasse showed bands for carboxylate (COO^-) and hydroxyl (OH^-) groups during the determination of its surface functional groups. Negative charged groups of sugarcane bagasse that were available on the surface somehow showed its potential to be used as an additive in membrane fabrication.

Figure 3 shows the important peaks for commercial silica. The FTIR spectra of silica nanoparticles typically exhibit prominent absorption bands in the 400-1000 cm $^{-1}$ range due to the stretching vibrations of Si-O-Si and in the 1400-1600 cm $^{-1}$ range due to the bending vibrations of Si-O-Si. Other than existing as minerals in sand and rock, silica can also be attained from organic materials such as bamboo leaf, rice husk, wheat husk and other agro-wastes in the form of ashes (Silviana *et al.,* 2018). Silica is present in several phases in the form of ash including amorphous, and crystalline. The molecules in amorphous form silica are arranged in the random three-dimensional repeating arrangement while the structure of crystalline silica explains about the molecules that must be in the three-dimensional repeating arrangement by repetition of a basic unit. The forms of silica are very essential in order to determine the effective applications of the ash (Irzaman *et al.,* 2018). Figure 4 shows the difference in structure of crystalline and amorphous silica.

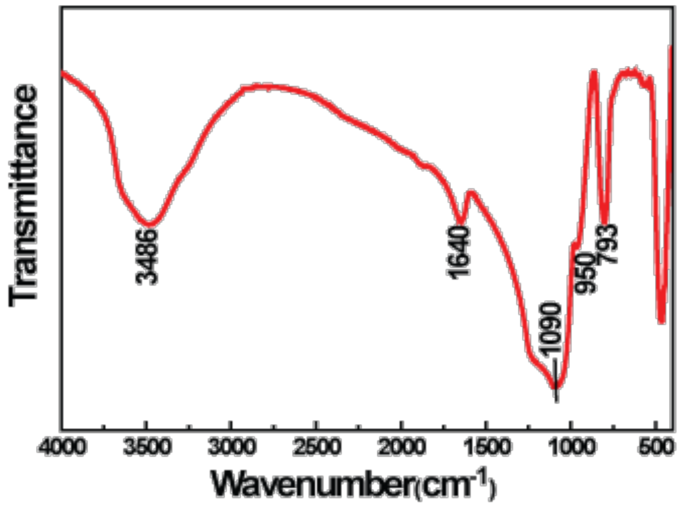

Figure 3 FTIR peaks for commercial silica.

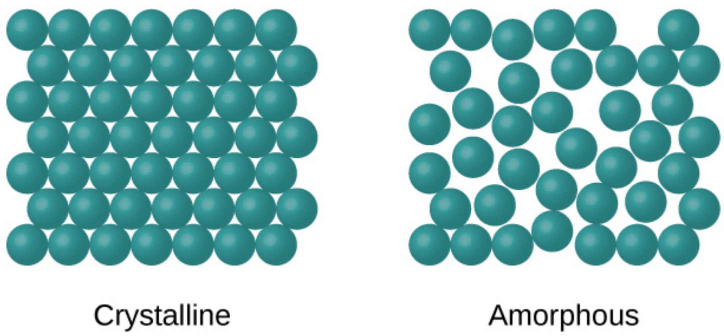

Figure 4 The differences in structure of crystalline and amorphous silica.

Silicon is an element that can be present in both metal and non-metals, or can be known as metalloids (Fatimah *et al.,* 2017). Silicon exists in two allotropic forms which makes silicon an element that differs in both physical and chemical properties. One form of the allotropes form shiny, needle-like crystals or flat plates grayish-black in color while another form of allotrope is commonly found as brown powder and does not exist in crystal structure (Fatimah *et al.*, 2018).

3.2 The study of moisture content

A controlled experiment was conducted to quantify alterations in the moisture content of wood specimens. Four small rubber wood samples, each with comparable weights, were employed as the experimental materials. Subsequently, these samples were placed in separate Petri dishes, with three of the dishes containing various types of silica—namely, silica from bamboo leaves ash (BLA), silica from rice husk ash (RHA), and commercially available silica. The fourth Petri dish served as a control without any silica. The quantity of silica in each Petri dish was maintained at a controlled factor of 0.2 g. The moisture content of the wood specimens was assessed by measuring changes in weight after 2 hours, 5 hours, 24 hours, and 48 hours, respectively. The initial weight of all wood specimens was recorded as 1.29 g.

Based on results shown in Figure 5, silica derived from RHA gives the best result as moisture absorbent and silica from BLA comes in second. This can be seen through decreasing weight value of tested rubber wood from 1.2897 g to 1.2411 g compared to Silica from BLA which is 1.293 g decreasing slightly to 1.2604 g. The moisture absorption of silica extracted from Rice Husk Ash (RHA) was observed to be higher in comparison to silica obtained from Bamboo Leaves Ash (BLA). This significant difference in moisture absorption properties underscores the distinct characteristics of silica derived from these two agricultural residues. The enhanced moisture absorption capability of RHA-derived silica could be attributed to several factors, such as variations in the silica structure, surface area, or the presence of specific functional groups. The specific chemical composition and physical properties inherent in silica sourced from rice husk ash contribute to its superior moisture absorption performance.

This information suggests that, when considering moisture absorption properties, silica from Rice Husk Ash may be a more effective option than silica extracted from Bamboo Leaves Ash. These findings can be valuable in applications where moisture management is a critical factor, influencing material selection based on the desired performance characteristics. This may be due to the bigger surface area of RHA and BLA compared to the surface area of the commercial silica. Silica from both RHA and BLA may also have good porosity level compared to commercial silica which allows the process of moisture absorption be faster. The bigger size of surface area gives it wider space to absorb moisture. This somewhat proves that silica derived from agro-waste materials can be applied for moisture absorbence (Ameram *et al.,* 2019). The sol-gel method using BLA as a precursor can result in silica materials with enhanced purity and homogeneity, showcasing a cleaner extraction process and reduced impurities (Adam *et al.,* 2010).

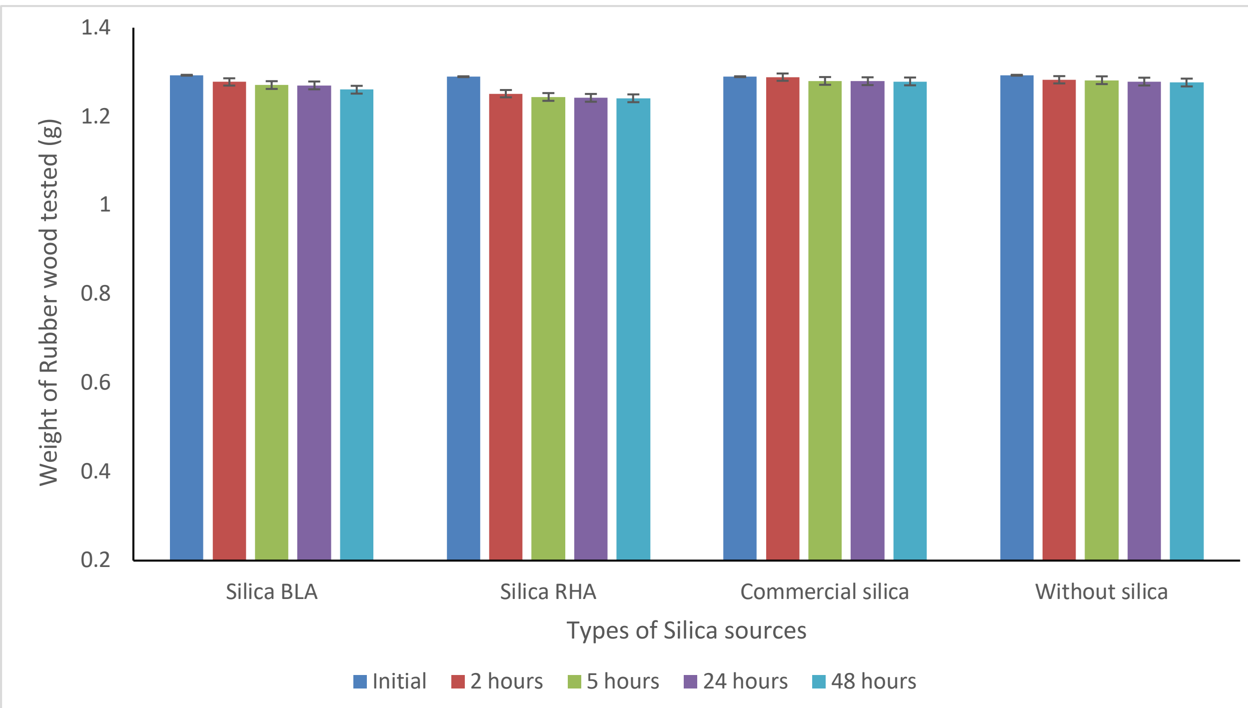

Figure 5 Graph of the changes of moisture content in rubber woods with time taken as the parameter.

BLA silica exhibits biocompatibility, making it suitable for biomedical applications and aligns with sustainability practices due to its sourcing from rapidly renewable bamboo with minimal environmental impact. Its unique mechanical strength and structural integrity, depending on processing methods, cater to applications requiring robustness in materials. The versatility of BLA silica spans diverse industries, from agriculture to healthcare, while its reduced environmental footprint and potential economic viability further position it as a promising alternative to silica derived from other sources like rice husks (Imyim *et al.,* 2010). However, the remaining two tests on moisture content, involving both commercial silica and the absence of silica, still yield measurable values, indicating ongoing absorption activities within the petri dish. It is recommended to position the samples, like pieces of wood, in environments where air circulation is restricted, such as within desiccators. This ensures a more accurate and precise assessment of moisture absorption. Furthermore, broadening the range of parameters applied to silica during the experiment is suggested. This expanded set of parameters may encompass considerations such as mass, temperature, and speed, aiming to enhance the validity and accuracy of the obtained results.

4. Conclusion

The FTIR analysis of bamboo leaf ash (BLA) silica indicates the presence of –OH bonds at 3381.25 $cm^{-1,}$ responsible for -Si-OH functional groups, and a strong C=O stretch at 1635.37 $cm^{-1,}$ suggesting the existence of aliphatic amide groups with a high boiling point due to strong intermolecular interactions. These characteristics deem silica derived from agro-waste materials like BLA and rice husk ash (RHA) suitable for various applications, particularly in wood manufacturing industries as a moisture absorbent. However, the effectiveness of BLA silica as a moisture absorbent may not be as pronounced as other alternatives. Repurposing bamboo leaves into silica provides a sustainable solution, preventing environmental pollution caused by burning and contributing to beneficial applications in different industries.

Recommendation

The characterisation of silica can be analysed using a few instruments. With characterisation of silica, knowledge on the chemical and physical properties of the silica can be useful to assist the process of turning the silica into other useful products. A study on the synthesis of hybrid organic and inorganic materials involving SiO_2 and other inorganic materials can be a good incentive for future research. One good inorganic material that can be synthesised with SiO_2 is urea. The synthesis of SiO_2-urea can be done by functionalising both organic and inorganic materials using 3-chloropropyltriethoxysiline (CPTES). For example, functionalised BLA silica and CPTES will produce BLACCl. The BLACCl is used as the support to immobilise the urea onto the silica surface. The catalyst that can be gained by the end of this research is called BLAC-urea. CPTES contain Cl which is a good leaving group that can be easily removed from the BLACCl and this is where the compound in urea will take place and the BLAC-urea catalyst will be produced. This green catalyst can be used for the production of biodiesel. These good results show that rice husk could contribute well in making many useful things such as fillers in paper making and rubber products. Silica can also act as anti-sticking agents. The catalyst obtained is also important in chemical industry and also serves as raw material for silicon production (Bakar *et al.,* 2016). For the application of silica as moisture absorbents, there are a lot of improvements that need to be done such as the pieces of wood need to be put in a place that will not allow any air circulation such as in a desiccator so that the results obtained will be more precise. In addition, more parameters can be tested on the silica in the experiment and these may include effects of mass, temperature and speed for more informative results.

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