

Synthesis and Characterization of Silica and Silica Cellulose from Natural Materials as Matrix for Various Sensor Applications: A Mini Review

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Abstract. Sensors play a crucial role in various fields by enabling the detection and analysis of a wide range of substances, including hazardous substance detection, environmental and food safety monitoring, pharmaceutical industry, gas analysis, and others. Research continues to identify and develop sensor matrix materials that can increase the sensitivity, selectivity and responsiveness of sensors. Silica, an oxide mineral is a potential matrix material for sensor applications because of its unique characteristics. It has a large pore structure and modifiable pore size distribution. Silica's stable chemical properties, high-temperature resistance and corrosion resistance make it an ideal matrix material for a wide range of sensor applications. In recent years, silica cellulose also become a potential material for sensor applications. Silica cellulose is produced by combining silica with cellulose components from natural materials, such as rice husk ash, bamboo leaf ash, rice straw ash, and other plant fibers. This article provides a comprehensive exploration of various methods of synthesis and characterization of silica and silica cellulose materials. The methods include sol-gel, acid leaching, alkaline extraction, and other techniques for extracting cellulose from natural sources. In addition, sensor applications that have been tested using this material are also discussed, including its use in detecting molecular compounds, food and environmental applications. The development of silica and silica cellulose materials based on natural materials is considered because of their sustainability. By continuing to explore the potential of these materials, it is hoped that it can make a significant contribution in the development of sensor technology that is more innovative, environmentally friendly and sustainable.

1 Introduction

Sensor fields appears to be in an endless expansion and this discussion become an integral part of modern technology, influencing industries, environmental monitoring, food safety, healthcare, and more [1]. A sensor is an analytical device that utilizes a certain reagent as a detection agent which will read the presence of a certain analyte as a target [2]. According to the material used as a specific reagent, sensors can be divided into three categories including chemical sensors, physical sensors and biosensors. A physical sensor measures physical properties or phenomena such as temperature, pressure, light intensity, motion, etc. They detect changes in the physical state or characteristics of the environment without involving chemical reactions or interactions.

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While chemical sensors have reactions or interactions between the target substance and the sensor material called reagents. Then a biosensor is a specialized type of sensor that combines a biological component such as enzymes, antibodies, or cells as a bioreagent with a transducer, a device that converts a biological response into an electrical signal [3].

Sensor development continues to be carried out to analyze a particular analyte in the hope of achieving high sensitivity and selectivity as well as the fastest possible response time. One of the components in the sensor is a reagent that is embedded in a certain matrix. The reagent is adsorbed into a matrix and integrated with a transducer to produce a measurable output [4]. Generally, there are several types of matrices that can be used as reagent immobilization media, including using Whatman paper [5] however, the use of Whatman paper has low chemical resistance, gold-coated electrodes [6], Ag/AgCl electrodes [2], molecularly imprinted polymers [7], and other solid supports which are economically expensive. Selecting the appropriate matrix for a chemical sensor is a crucial decision that significantly influences the sensor's performance and accuracy [8]. The development of silica and silica cellulose materials as sensor matrices is considered because both have the ability as receptor storage and are easier to obtain than other support media which are relatively expensive, and some have low chemical stability [9]. Both also have several advantages compared to other matrices, including ease of obtaining, chemical resistance, thermal stability, porosity, compatibility with various methods and ease of modification [10]. In addition, sources of silica and silica cellulose can be extracted from several natural materials such as rice husk ash [11–13], bamboo leaf ash [14–16], rice straw ash [17–19], and other plant fibers. So it can support sustainability and renewable resources. The matrix that reagents are trapped should have several criteria, including being able to store the reagents without destroying their activity and being easy to apply [20]. This study delves into the significance of carefully choosing the matrix for chemical sensors and its implications for achieving reliable and meaningful analytical results.

Silica can be manufactured synthetically and extracted from natural sources. This compound is also a part of the prospective material selected for development and application in various sensor applications [21]. The synthesis method significantly influences the structure, morphology, and surface chemistry of silica and silica-cellulose materials. For sensor applications, precise control over these parameters is vital, as they directly impact the material's sensitivity, selectivity, and overall performance. By adjusting parameters such as precursor concentration, reaction time, and temperature, it shows the material's properties to achieve optimal sensor behavior [22].

Characterization techniques provide insights into the structural and surface properties of the synthesized materials, which are crucial for sensor design and optimization. By characterizing the material's porosity, surface area, particle size, and crystallinity using methods like BET analysis, XRD, and electron microscopy, these guide to enhance analyte adsorption, binding, and overall sensing efficiency [23]. Additionally, techniques like FTIR and XPS (X-ray photoelectron spectroscopy) can help identify surface functional groups, aiding in the design of specific interactions with target molecules. Understanding the synthesis and characterization methods of silica and silica-cellulose as sensor matrices is of paramount importance to harness their unique properties and tailor them for specific sensing applications.

The development of sensor matrices based on silica and silica-cellulose holds paramount significance in the realm of analytical chemistry. These matrices, derived from natural sources, exemplify the integration of cutting-edge technology with renewable resources. Silica, a versatile inorganic compound, and silica-cellulose, a hybrid material, have emerged as promising candidates for sensor matrices due to their unique properties. The utilization of natural materials not only contributes to sustainable practices but also aligns with the global

pursuit of renewable resources. This discourse delves into the pivotal importance of harnessing silica and silica-cellulose as sensor matrices, elucidating their intrinsic attributes and their profound link to the concept of renewable resources.

2 Results and Discussion

2.1 Sensor Matrix Based on Silica and Silica-cellulose

Silica (SiO_2) is a versatile compound with a variety of applications in various fields, including sensors as its matrix. Their synthesis from natural resources has received great attention because of their abundant availability and potential environmental benefits. Additionally, combining silica with other natural materials, such as cellulose, can lead to the development of hybrid materials with better properties. Silica, also known as *silicon dioxide* (SiO_2), is a chemical compound consisting of silanol and siloxane groups [24]. In the context of sensor matrices, silica can be used as a base material or matrix which allows the integration of reagents and analytes in the development of various types of sensors [25].

Silica has several properties that make it suitable as a sensor matrix, including thermal resistance, porosity, chemical stability, and optical transparency [8]. Also in recent years, silica-cellulose has been used as a sensor matrix that combines the properties of silica and cellulose for a variety of sensor applications. Silica-cellulose has surface interactions in the presence of Van der Waals forces and hydrogen bonds. The presence of $-\text{OH}$ groups in silica-cellulose causes these groups to hydrogen bond with other molecules to be used as sensor matrix as an adsorbent of reagent. Adsorption can occur due to the withdrawal of particles on the surface which involves the adsorption process. The adsorption process occurs due to the presence of pores so that the molecules are attracted to the porous surface which causes the molecules to be retained through hydrogen bonds, Van der Waals forces, or dipole-dipole [26].

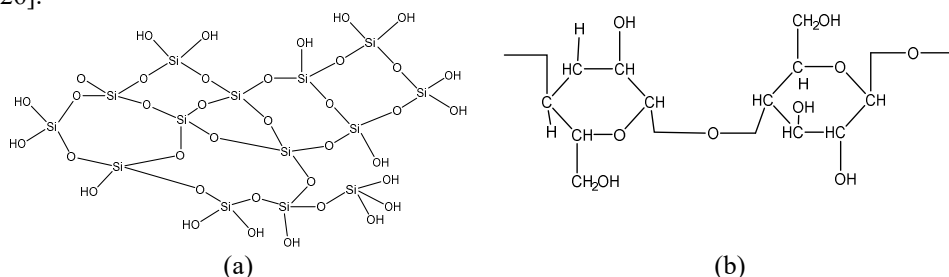


Fig. 1. Silica and cellulose structure (a) structure of bonded SiO_4 to bond SiO_2 , (b) structure of cellulose.

Silica-cellulose based membranes have been used in sensor applications in recent years and have played a very important role in various fields. As a new natural material, with many advantages it can function for all types of sensors as a reagent embedding medium. Cellulose is a type of polysaccharide consisting of several glucose which form β -1,4-glycosidic bonds consisting of hydroxyl ($-\text{OH}$) and carboxyl ($-\text{CO}$) functional groups [27]. The cellulose matrix can be obtained from the hydrolysis of nata de coco which is the result of fermenting coconut water with the microorganism *Acetobacter xylinum*. Nata de coco is basically cellulose, when viewed under a microscope it will appear as a mass of irregular fibrils resembling thread or cotton. Bacterial cellulose as a membrane has several advantages including high purity, has a density between 300 and 900 kg/m^3 , high tensile strength, and elasticity and biodegradable [26]. The chemical structure of silica and silica-cellulose can be shown as follows:

2.2 Synthesis of Silica and Silica Cellulose from Natural Materials

Silica can be synthesized from natural sources of waste from agriculture which are usually burned or disposed of without prior treatment so these can cause pollution such as rice husk ash, bamboo leaf ash, rice straw ash, and other plant fibers which have sufficient abundance and easy to obtain [9]. Silica is the raw material for making sensor matrices, both in the form of silica itself and silica cellulose matrices. While the cellulose material is obtained from the hydrolysis of nata de coco combined with silica [28]. Rice husk ash is one of the sources for obtaining silica because it contains a material whose composition consists of a very high silica content so this material has the potential to be used as a source for obtaining silica. Silica sourced from rice husk has several advantages compared to mineral silica, where rice husk silica has finer grains, is more reactive, can be obtained in an easy way at a relatively low cost, and is supported by the availability of abundant and renewable raw materials, in addition, other content in rice husk ash does not significantly affect the process of making silica [29].

Silica can be obtained or manufactured synthetically by extracting from the abundant amounts found in nature. The synthesis of silica and cellulose-silica from natural resources involves diverse methods such as sol-gel [30,31], acid leaching [32,33], and alkaline extraction [34,35]. and other techniques, each offering unique advantages in material design and application [9]. The sol-gel process is a versatile technique for synthesizing silica-based materials. In this method, a precursor, often tetraethyl orthosilicate (TEOS), undergoes hydrolysis and condensation reactions in the presence of water and a catalyst. The resulting sol evolves into a gel, which can then be dried and calcined to obtain the desired silica structure. Sol-gel enables precise control over particle size, morphology, and porosity, making it suitable for various applications, including catalysts, coatings, and sensors [36]. The acid leaching method involves treating silica-rich natural sources such as rice husks, with strong acids. This process dissolves other minerals and organic matter, leaving behind purified silica [32]. The extracted silica can then be processed into different forms. Acid leaching is especially effective for obtaining high-purity silica from complex matrices and is commonly used for industrial applications [33]. Also alkaline extraction using alkali solutions, such as sodium hydroxide (NaOH), to extract silica from raw materials. This method is often employed with plant-based materials like rice husks or sugarcane bagasse. Alkaline treatment breaks down cellulose and other organic components, facilitating the release of silica. The extracted silica can be further processed through precipitation and drying [37]. Table 1 shows the difference in yield percentage and particle size in the extraction of silica from top three natural materials containing silica.

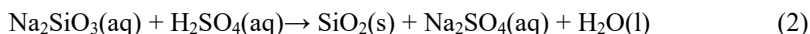
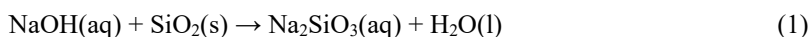
Table 1. Different of yield percentage and particle size in the extraction of silica from several natural materials containing silica.

Starting Material	Method	Product	Yield Average (%)	Pore Size (nm)	References
Rice Husk	Sol-gel	Silica	< 95	22.7	[30]
	Acid-leaching	Silica	< 90	6	[32]
	Alkaline	Nanoparticle silica	86.17	49	[34]
Bamboo Leaves	Acid-leaching	Amorphous nano-silica	34.70	43	[14]
	Sol-gel	Silica	61.2	-	[15]
Rice Straw Husk	Alkaline	Silica	74.11	-	[18]
	Acid-leaching	Silica	35	15.85	[17]

Coconut Cobs Husk	Green Synthesis	Silica	11	200-275	[38]
Corn Cobs Husk	Acid-leaching	Silica	35.3	40-70	[39]
Palm Ash	Sol-gel	Silica	90	-	[40]

Rice husk ash is the best source for obtaining silica because it contains a very high silica content so this material has the potential to be used as a source for obtaining silica [41]. Silica sourced from rice husk has several advantages compared to mineral silica, where rice husk silica has finer grains, is more reactive, can be obtained in an easy way at a relatively low cost, and is supported by the availability of abundant and renewable raw materials.

Silica or silicon dioxide compounds (SiO_2) are widely used as adsorbents, desiccants, filter media, and catalyst components. Silica is obtained by burning rice husks and extraction using NaOH solvent. The reactions that occur are as follows:



From the reaction equation, silica which has polar properties can dissolve with NaOH which also has polar properties to produce sodium silicate (1). Next, the sodium silicate will precipitate silica (2) by adding a strong acid in the form of sulfuric acid (H_2SO_4) to obtain pure silica. The formation of silica from the addition of acid produces silica with a brownish-white color [10]

2.3 Characterization of Silica and Silica Cellulose from Natural Materials

Characterizing silica and cellulose-silica materials before their application as sensor matrices is crucial due to several key reasons such as: performance optimization because silica and silica-cellulose matrices directly influence the sensor's sensitivity, selectivity, and response time [42]. Thorough characterization helps identify the material's structural, chemical, and physical properties that impact these aspects. By understanding these properties, can modify the material composition and structure to optimize sensor performance for specific target analytes [43]. Secondly to achieve selectivity enhancement because silica-based sensors are often used in detecting specific analytes in complex environments. Characterization allows to study how the material interacts with different substances. This knowledge aids in tailoring the sensor's selectivity, enabling it to distinguish between multiple compounds and reducing false positives or negatives [44]. In addition, this also shows surface functionalization or the ability to functionalize the material's surface is critical in sensor design. Characterization helps identify the presence of functional groups on the material's surface, which can be modified to enhance analyte adsorption or interaction. This enables the sensor to achieve higher sensitivity and accuracy [45].

There were several physical characterizations of the silica and silica cellulose matrices which were carried out including determining the water content, ash content, density, absorption, analysis using Brunauer-Emmett-Teller (BET), Scanning Electron Microscopy (SEM), and Fourier Transform Infrared (FTIR) analysis [46]. Determination of water content aims to determine the amount of water content trapped in the matrix. Determination of ash content aims to determine the content of mineral salts and inorganic substances in the matrix. Density measurements express the mass of silica and silica cellulose matrix per volume. measurement using a device called a pycnometer. Testing the absorption capacity of the silica matrix and silica cellulose aims to provide information on the absorption capacity of the matrix. The solution used is iodine solution because iodine has a strong affinity for the surface of silica

and silica cellulose. The percentage absorption of iodine obtained is directly proportional to the specific surface area of the matrix.

FTIR analysis aims to determine the functional groups in the silica and cellulose matrices before and after immobilizing the reagents. This is done to ensure no binding between the matrix and the reagent which could interfere with the analysis results. meanwhile, the SEM analysis aims to determine the surface typology of the silica and silica cellulose matrices both before and after immobilization with reagents. Characterization techniques such as BET analysis (Brunauer-Emmett-Teller) also provide insight into the distribution of pore sizes and surface area of materials, helping to understand adsorption mechanisms and optimize sensor designs accordingly [47].

The surface structure of the silica and silica-cellulose matrices was characterized using SEM to determine the surface topology of the matrix in the form of indentations, protrusions, surface holes, and the size of the surface hole diameter. SEM image results of silica and silica-cellulose can be seen in the fig. 2 below:

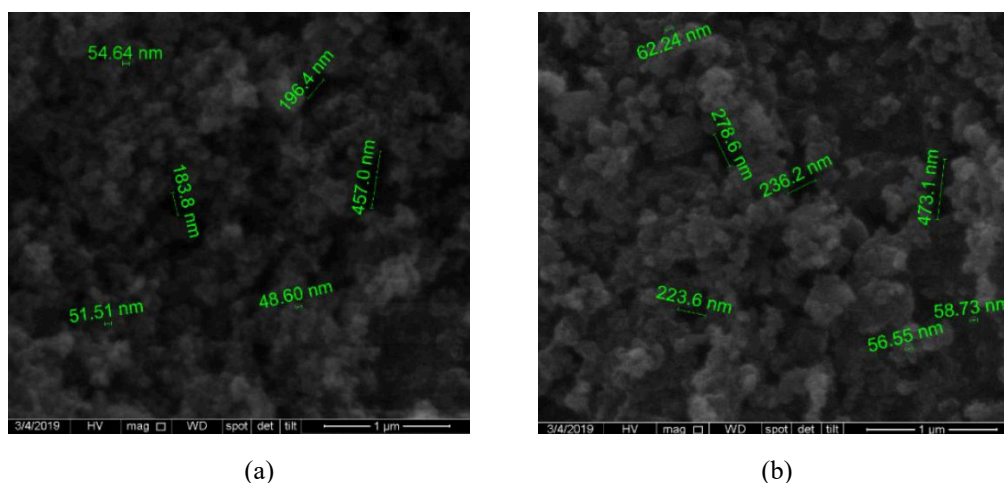


Fig. 2. (a) SEM analysis results of silica matrix 80,000x magnification, (b) SEM analysis results of silica-cellulose matrix 80,000x magnification. adapted from [48].

The uneven surface indicates the presence of pores on the surface of silica and silica-cellulose. The presence of surface pores can increase the surface area of the two matrices to be used as adsorbents. Surface topology analysis was also carried out on immobilized silica and silica cellulose matrices. It can be seen that there are reagents adsorbed on the surface of the matrix (spherical in the picture). This indicates that the pores in the matrix of silica and silica cellulose can be used as an adsorbent as a place to embed reagents in the sensor.

2.4 Sensor Matrix Based on Silica and Silica-Cellulose for Various Sensor Applications

A sensor matrix based on silica and silica-cellulose materials holds significant promise for various sensor applications. These matrices provide a versatile platform for designing sensors that can cater to a wide range of needs across different industries and applications including their use for matrices for gas sensing, chemical sensing, biosensing, environmental monitoring, optical sensing, food quality and safety and other industries [49]. Silica and silica-cellulose matrices can be functionalized to selectively adsorb specific gases. By modifying the surface chemistry of these materials, sensors can be developed to detect gases such as carbon dioxide, methane, volatile organic compounds (VOCs), and even toxic gases

[8]. Table 2 shows the application of silica and silica-cellulose for various sensor applications.

Table 2. The application of silica and silica-cellulose matrix for various sensor applications

Kind of Sensor	Matrix	Target (Analyte)	References
Gas Sensor	Silica-based	Toxic gas	[50]
	Silica-based	Ammonia (NH ₃)	[51]
Humidity Sensor	Silica-cellulose based	Soil moisture	[51]
Temperature sensor	Silica-based	High-temperature monitoring	[52]
pH Sensor	Silica-based	Acidity level	[53]
Biosensor	Silica-based	Glucose	[54]
	Silica-cellulose based	Polyphenol	[54]
	Silica-cellulose based	Allergens in food	[55]
Metal Sensor	Silica-based	Lead (Pb ²⁺)	[56]
	Silica-based	Copper (Cu ²⁺)	[56]

The high surface area and tunable properties of silica-based matrices contribute to their sensitivity and selectivity in gas sensing. These matrices are well-suited for detecting various chemicals and analytes [1]. By tailoring the surface properties through functionalization, sensors can be designed to identify specific chemicals in liquids or gases. This makes them valuable tools for environmental monitoring, industrial processes, and even medical diagnostics. Silica and silica-cellulose matrices offer a biocompatible platform for biosensors. By immobilizing biomolecules like enzymes, antibodies, or DNA probes onto the surface, these sensors can detect biological markers, pathogens, or specific DNA sequences [57]. This is particularly useful in medical diagnostics, food safety testing, and biotechnology applications. Sensors based on these matrices can contribute to monitoring environmental parameters such as humidity, temperature, and pollution levels. Silica's stability and robustness make it suitable for outdoor and harsh environments, enabling long-term monitoring of conditions [58].

3 Conclusion

Silica and silica-cellulose can be synthesized from the natural materials such as agricultural waste including rice husk ash, bamboo leaf ash, rice straw ash and other plants fibers by several methods such as sol-gel method, acid-leaching method and alkaline extraction or the other extraction techniques while silica-cellulose can be obtained from nata de coco hydrolysis then combine with silica material formed a composite. The matrix based on silica and silica cellulose can be applied to a variety of sensors as a medium that can adsorb reagents as specific agents that will interact with the target analyte because their properties are in accordance with the requirements of good sensor matrices.

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