

Article

## A Review on the Extraction of Silica Nanoparticles from Poaceae Family via Sol-Gel

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**Abstract**— One of the valuable inorganic multifunctional chemical substances is silica, which can be found in crystalline, gel, and amorphous forms. On the crust of the earth, silica is the most complex and abundant family of materials, and it has been used extensively in ceramics, adhesives, detergents, dental materials, electronics, pharmaceutical items, and chromatography column packing. Several kinds of research have fascinated the recycling of agricultural wastes using extraction for useful products such as silica, lignin, adsorbents, cellulose, biofuels, silicon, carbon, and inhibitors. In this review article, we report most of the silica nanoparticles extracted from the Poaceae family since they accumulate significant amounts of silica, and most of them are easily found in Malaysia. This study investigates the significant amount of extracting silica nanoparticles from the Poaceae family via green technology sol-gel technique as pure silica can be produced with the potential to manage the size of the particle, size of distribution, and morphology via the reaction parameters in systematic monitoring. This technique can also produce pure silica using a low-energy method. Subsequently, the key chemical compounds that existed in silica nanoparticles are characterized and identified using FTIR analysis. According to this article, most of the Poaceae family species are considered to gain a high purity of above 90% of silica based on the quantification results. The Poaceae family is recognized for having high silicon concentrations. However, the concentration varies depending on the species.

**Keywords**— Silica nanoparticles, Poaceae family, sol-gel, characterization, FTIR.

### I. INTRODUCTION

Silica is known as a mineral component that represents silicon and oxygen and is categorized as the two most abundant elements in the crust of the earth, which can be found in crystalline, amorphous, and aqueous forms. In synthetic chemistry, silica is applied as a significant precursor for various organometallic and inorganic materials, such as thin films or coatings for electronic catalysts and optical materials [1]. However, instead of using natural silica

compounds, which are hydrophilic, silica nanoparticles are more in demand nowadays since they provide more crucial benefits than natural silica compounds. Some of the general benefits are ease of functionalization, biocompatibility, large surface area, site-directed delivery, high loading capacity, and in a few situations, actuated drug release in antimicrobial therapeutics in the medical fields [2].

Nanoparticles are defined as particles with a size less than 1000nm by the Food and Drug Administration (FDA).

Meanwhile, in the medical field, they are commonly described as particles with a size less than 500 nm, whereas this size suits for cells to get endocytosed [3]. Silica nanoparticles, also called nano-silica, have a major section in nanoparticle research regarding their lack of toxicity and body stability based on biomedical applications. Silica nanoparticles are commonly assigned with mesoporous silica nanoparticles (MSNs) as it has a special property like presence in mesoporous or consisting of a porous diameter 2 nm until 50 nm [3].

Significant amounts of silica are known to be found in the sand that lines our ocean's edges instead of the soil, as well as quartz crystals in granite, emeralds, and a variety of other rocks. In addition to being a common component of rock, silica can also be found in a variety of plants, and the plant silicon content is based on silicon existence in the soil, while the transpiration stream affects silicon deposition in the plant

[4-5]. It occurs when silicic acid (an uncharged molecule) is absorbed by roots, transported to the shoots, and deposited as opal, which is an amorphous silica type that cannot be re-contributed [6-7]. Various forms of silicon can be available in plants, and some research has found that polymeric silicic acid (aqueous form) is supposed to present as silicon in plants [8]. Moreover, silica gel is shown as an amorphous silica form in which silica is found in higher plants and diatoms that contributes to 90% until 95% of silica composition in rice plants based on the research on infrared absorption and dissolution rates [9-10]. Generally, we can classify silica in plants as an amorphous silica type or more, specifically as an opal [11]. In identifying the classification of types of silica forms accurately, the details are presented in Table I.

Table I. Summary of the Forms of Silica [4].

Forms of Silica	Types of Forms	Details
Crystalline	Cryptocrystalline	Contains fibrous crystallites possessing pores of submicroscopic, known as chalcedony
	Microcrystalline	Tridymite, quartz, or cristobalite
Amorphous	Gelatinous silica	Performs in solution as continuous gel or gelatinous flocs, constructed either by allowing a supersaturated solution to stand, by acidifying a concentrated solution of an alkali silicate, or by evaporation of silica solution
	Opal	Silica occurs naturally, and the silica of radiolarite and diatomite frequently has a water content of less than 12%. Some forms seem to be on the verge of becoming crystalline (cristobalite)
	Silica gel	Hard amorphous silica with a water content of 20% to 30%, used extensively as a desiccant or as a chemical reagent
	Silica glass	Obtained via the silica melt quenching method
	Silica sol or colloidal silica	Silica distributed into colloidal dimensions particles in water ( $10^{-3}$ to $10^{-6}$ mm)
Aqueous	Dissolved or colorimetric silica	Silica in true solution ( $H_4SiO_4$ ), which interacts with ammonium molybdate over 2 minutes after the solutions are combined
	Orthosilicic acid	The main form of silica with a pH less than 9 under saturated solutions is the monomer $H_4SiO_4$ . Above pH 9, orthosilicic acid dissolves ( $K_1=10^{-9.8}$ )
	Polymerized silica	Silicic acids consist of two (disilicic), three (trisilicic), or more atoms of silicon per molecule (including colloidal suspensions). Noncyclic silicic acids interact with ammonium molybdate at room temperature within 5 min, yet cyclic polysilicic acids need a few hours to interact. High-order polymers do not interact at all. Polysilicic acids depolymerize to the monomer in unsaturated solutions
	Total silica	All species of silica in the solution can be calorimetrically measured following the conversion of polymerized silica to the treatment of monomer with NaOH or by gravimetric techniques. In most natural waters, colorimetric and gravimetric silica are equivalent, indicating the prevalence of the monomer $H_4SiO_4$

Plant species differ in the quantity of silicon they take up and accumulate, as evidenced by differences in silicon content, absorption processes, morphologies, and deposition sites. In general, there are three major types of plant species due to their silicon uptake potentialities, in which the plant takes up the quantity of silicon (in the xylem) respective to the quantity of existing silicon in the soil solution [12]. When the silicon quantity of the plant is significantly greater than the quantity in the soil solution, the plant actively takes up silicon. However, when the silicon quantity in the plant is significantly lower than the silicon quantity in the soil

solution, the plant passively takes up silicon, as the plant excludes silicon if both contents are comparable [13]. In addition, silicon uptake also regards rates of transpiration, with certain species displaying passive silicon uptake (transpiration-driven) as well as active silicon uptake (transporter-governed) [12,14].

Several types of research have fascinated the recycling of agricultural wastes using extraction for useful products such as silica, lignin, adsorbents, cellulose, biofuels, silicon, carbon, and inhibitors [15]. Some examples of agricultural wastes that produce silica are palm kernel shells, rice husk ash, coffee

husk, corn cob ash, cassava periderm, sugarcane bagasse, and maize stalk [15-16]. Most of them are from the Poaceae family since they are yielded in a huge amount of silica and are easily obtained. However, there are still many agricultural wastes from the Poaceae family that researchers have not explored yet.

Poaceae family, also identified as the grass family, is worldwide distributed with more than 10,000 species and is the largest family of flowering plants. Agricultural grains (essential food and beverage sources of alcohol) such as wheat (*Triticum*), oats (*Avena*), barley (*Hordeum*), rye (*Secale*), rice (*Oryza*), corn (*Zea*), and others, including grazing plants and crucial fodder, make up the grasses. Many environments, such as grasslands and savannahs, benefit from the presence of members of this family [17].

Plants of the Poaceae family are known to be high in silicon, which is deposited in the plant body as amorphous hydrated silica ( $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ), while most plants do not accumulate silica appreciably. Silica, which originates from rocks, progressively breaks down to produce monosilicic acid ( $\text{H}_4\text{SiO}_4$  or  $\text{Si}(\text{OH})_4$ ), also known as orthosilicic acid, and weathering leads to this process [4,12]. Both silicon sources that exist in organisms are biologically deposited silica and monosilicic acid [18]. Indeed, the Poaceae family is easily obtained; however, many species are still not being extracted or synthesized via the sol-gel method.

The progressive interest in silica nanoparticle synthesis was due to its widespread consumption as a fundamental raw material in the development of new classes of revolutionary materials [19]. Silica nanoparticles have been generated using Chemical Vapour Condensation (CVC) [20], Reverse Micro Emulsion (RME) [21], precipitation method [22], etc. However, the sol-gel method was applied due to its ability to provide pure, suitable products in a variety of conditions. It was used to synthesize silica, porcelain materials, and glass [23]. To fulfill the growing need for multifunctional nanosystems, a range of nanoparticles with exact shapes and sizes were produced using a comprehensive grasp of the sol-gel method [24]. This method has dual advantages: it can produce relevant silica particles at a lower cost, reduce disposal, and address pollution challenges [25]. Consequently, aqua gel and xerogel were involved in this method before the silica nanoparticles were produced.

In essence, this review article has evaluated and compiled several works that have been conducted regarding the extraction of silica nanoparticles from several examples of the Poaceae family, including the green technique of sol-gel. The compilation works associated with the Poaceae family species are Bambusoideae (bamboo) [26], *Hordeum vulgare* (barley) [27], *Zea mays* (corn) [28], *Saccharum munja* (pin red grass) [29], *Oryza sativa* (rice) [30], *Saccharum officinarum* (sugarcane) [31], *Eragrostis tef* (teff) [32], and also *Cymbopogon citratus* (lemongrass) [33].

Considering the various extraction methods and conditions employed by researchers to obtain silica nanoparticles from Poaceae family plants, there is significant scope for further investigation into systematically optimizing these parameters to achieve better yields and control over particle size distribution, surface properties, and other physicochemical characteristics. Therefore, the parameters elucidated in this

review article offer valuable insights for optimizing outcomes in this domain.

## II. EXTRACTION OF SILICA NANOPARTICLES IN PLANTS

Plant silicon concentration can be measured using a variety of methods. The following are descriptions of the six most utilized approaches in Table II, with ground plant material as the starting point. The simplest extraction method was DeMaster's technique, labelled as (A) in Table II, which used a 1%  $\text{Na}_2\text{CO}_3$  solution. A total of 30 mg of plant matter was extracted for 5 hours at 85°C in a 1%  $\text{Na}_2\text{CO}_3$  solution, then filtered with the pore size of 0.45  $\mu\text{m}$  and evaluated for this approach [12,34].

Table II. Summary of Primary Plant Silicon Extraction and Measurement Techniques

	Extractant	Procedure	Reference
(A)	Solution of 1% $\text{Na}_2\text{CO}_3$	Plant matter (~30 mg) extracted in 1% $\text{Na}_2\text{CO}_3$ solution at 85 °C	[12,34]
(B)	Solution of 0.5M NaOH	Plant material (~100 mg) extracted in 0.5 M NaOH solution at 85°C	[35]
(C)	2-step HF	Step 1: Plant matter (~100 mg) digested in a mixture of nitric acid, distilled water, and hydrofluoric acid (40%) at 190°C Step 2: hydrofluoric acid is neutralized by 10 mL and 4% boric acid solution at 150°C	[36]
(D)	Lithium metaborate fusion	Plant matter was ashed at 500°C and mixed with lithium meta-tetraborate at 1000 °C. The gained bead is exchanged into nitric acid.	[37]
(E)	Tiron ( $\text{C}_6\text{H}_4\text{Na}_2\text{O}_8\text{S}_2$ )	Plant matter was combined with the solution of tiron, which was buffered at pH 10.5 at 85°C.	[38]
(F)	No extractant, yet XRF	Plant matter (~100 mg) homogenized to a powder and required calibration.	[39,40]

The next technique, labelled as (B), was also simple and utilized the solution of 0.5 M NaOH. A total of 100mg of plant matter was extracted for 5 hours at 85°C in a 0.5 M NaOH solution, then filtered with the pore size of 0.45  $\mu\text{m}$  and evaluated for this technique [35].

Hydrofluoric acid (HF) was a more complex technique that involved real digestion, besides the extraction approach, which was labeled as (C). According to Puppe et al. [36], 2-step extraction is performed via a microwave system with a closed vessel. Firstly, a total of 100mg of plant matter was combined with the mixture of 5 mL of 65% nitric acid solution, 4 mL distilled water, and 1 mL of 40% solution of hydrofluoric acid at 190°C. Then, 10 mL boric acid (4%) was utilized in the neutralization process at 150°C before we analyzed it.

Another technique provided in extracting Si from plant matter was the lithium metaborate method, labelled as (D). The muffle furnace was used to ashing the plant matter in a porcelain crucible by progressively raising the temperature to 500°C and maintaining it for a minimum of 1 hour. Next, the Si was extracted by mixing 5-100 mg of ash with lithium meta-tetraborate (0.4 g  $\text{Li}_2\text{B}_4\text{O}_7$  and 1.6 g  $\text{LiBO}_2$ ) in the muffle furnace for 5 min at 1000°C. Then, the yielded bead was placed in a conical flask and agitated until it dissolved at 90°C before the solution was diluted to 100 mL with nitric acid (10%) [37].

The next method used in extracting plant Si was the tiron technique (labeled as (E)). Since the sample was extracted at 85°C, it was analogous to  $\text{Na}_2\text{CO}_3$  and  $\text{NaOH}$  techniques. A total of 50 mg of plant matter was mixed with 30 mL tiron solution (0.1 M), which was buffered at pH 10.5 and set up at 85°C within 2 hours. Only 10 mL of this 30 mL was taken and combined with 10 mL  $\text{H}_2\text{O}_2$  (30%) before being heated to 85°C for 1 hour to eliminate the tiron, then filtered with the pore size of 0.2  $\mu\text{m}$  before we evaluated it [38].

Another simple technique in analyzing Si was via X-ray fluorescence spectrometry, in which no extraction was needed, labeled as (F). The content of Si in a total of 100 mg of plant matter was measured directly with a requirement of calibrations [39-40]. According to Nakamura et al. [37], the borate fusion technique resulted in a high Si concentration compared to the  $\text{Na}_2\text{CO}_3$  technique. Nevertheless, a simple equation could be utilized to adjust the difference in the data.

### III. SOL-GEL METHOD IN EXTRACTING SILICA NANOPARTICLES

A sol-gel method is included in the technique of extracting silica from plants and contributes many benefits to the synthesis process. According to Pierre [41], this technique permits material synthesis not only with any oxide composition but also with new hybrid organic-inorganic

materials that are not naturally present. Generally, the first steps in this technique are carried out at low temperatures, and thus, kinetics in a variety of chemical reactions can simply be controlled, which is assisted by green technology. Moreover, sol-gel associates the solid colloidal state with a liquid medium, whereas any pollution from the dust's eventual dispersion can be avoided. Hence, the synthesis of sol-gel is applied in the biggest industries to control critical pollution [41].

One of the broadly utilized methods to synthesize particles of silica is the method of sol-gel, and this method produces silica nanoparticles in pure form with the potential to manage the size of particles, size of distribution, and morphology via the reaction parameters in systematic monitoring [42-43]. The sol-gel method, which is also known as a soft chemistry process, affects the ambient temperature in either basic or acidic circumstances [3]. This procedure necessitates metal alkoxides, or inorganic salts, to be hydrolyzed and condensed in the existence of a mineral base or acid as a catalyst. Both silane reactions are shown in Figure 1.

The entire silica structure is created when the silanol groups condense between them or between ethoxy groups, which leads to siloxane bridges (Si-O-Si) [42,44].

Determining whether the condensation or the hydrolysis step is quicker depends on whether a base or an acid is the catalyst in this process. Hydrolysis is quicker than condensation in the case of an acid-catalyzed reaction, and then it will contribute to the formation of a large number of tiny silica particles. These particles clump together to create a gel-like form. As opposed to acid-catalyzed reaction, the condensation step in the base-catalyzed reaction is faster than the hydrolysis step, resulting in a bigger size of silica nanoparticles [3]. The silica particles are supposed to be nonporous and prone to produce between solid spheres (base-catalyzed) or gel networks (acid-catalyzed) if only these three compounds are used, as shown in Figure 2.

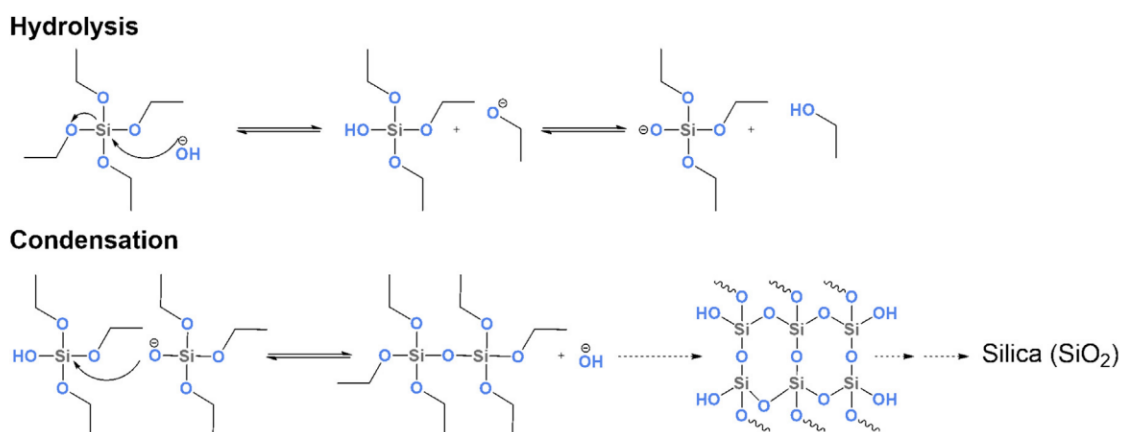


Figure 1. Hydrolysis and Condensation Reactions of Silanes [3]

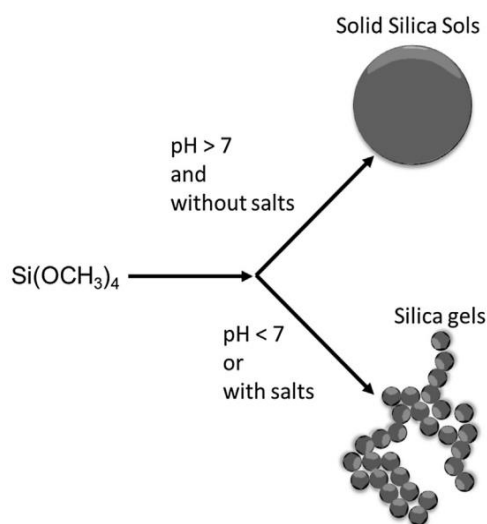


Figure 2. Silica Synthesis Pathways under Acidic (gels) and Basic Conditions (sols) [3].

Apart from that, there are a few precursors that can be utilized in the sol-gel method to produce mesoporous silica nanoparticles. One of them was organically modified precursors that did not require an oxygen bridge and resisted hydrolysis since an organic group was directly connected to the Si atom. Organo-silica nanoparticles are thought to have superior qualities, such as a larger area of the surface, a less condensed siloxane form, and a lower density [45]. Organic templates are only used in a few practical applications due to their limited accessibility and high cost. Tetrakis (2-hydroxyethyl) orthosilicate, tetraethyl orthosilicate (TEOS) or tetramethoxysilane (TMOS) and are some of the most commonly used silica precursors [46-47].

Other silica precursors to sol-gel-derived silica were glycerol-derived polyol-based silane precursors and sodium metasilicate. For glycerol-derived polyol-based silane precursors, they were very sensitive toward the ionic strength of sol even though not pH-dependent [48]. This silica precursor utilized orthosilicic acid in the past. However, owing to the longer time engagement and demand for freshly synthesized acid, it was no longer extensively utilized [49]. Subsequently, the pure alkoxy silanes and alkoxides were examples of sodium metasilicate precursors in the sol-gel-derived silica [5].

#### IV. Methodology For Extracting Silica Nanoparticles From Poaceae Species By Sol-Gel

##### A. *Bambusoideae* (Bamboo)

*Bambusoideae*, also known as bamboo, is one of the species in the Poaceae family. According to Nazri & Sapawe [51], fresh bamboo leaves were obtained from the forest in Alor Gajah, Melaka, Malaysia. The leaves were rinsed with water before using the ionized water to remove the undesired particles. The rinsed leaves were then dried at  $105^\circ\text{C}$  and continuously calcined at  $600^\circ$  to obtain the ashes.

The sol-gel method was started by isolating the sodium silicate from bamboo leaves ash (BLA) overnight in the acidic solution, HCl, and then stirring it with NaOH solution for 4 hours. Next, the filtration process was used to remove undesired particles, such as carbon residue, via filter paper Whatman-41 before the distilled water rinsed the mixture to produce the gel residues. After the sodium silicate was produced, the solution pH was acidified by 3M HCl until the neutral pH was reached. The mixture was aged for 18 hours before being dried at  $105^\circ\text{C}$  for 4 hours to obtain the silica powder [51]. Figure 3 shows the methodology for extracting silica nanoparticles from bamboo leaves.

In an additional method, silica nanoparticles were then extracted from leaves ashes and combined with water to produce the mesoporous silica nanoparticles. Cetyltrimethylammonium bromide (CTAB) and ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) were stirred constantly for 30 minutes to perform the silica water-modification based [51].

##### B. *Cymbopogon citratus* (Lemongrass)

*Cymbopogon citratus* (lemongrass) is a perennial grass; hence, it is categorized as part of the Poaceae family. As reported by Firdaus et al. [33], the lemongrass was collected from Stesen Agroteks UniMAP, Perlis, Malaysia, and cultivated on heavy clay soil that contains a high silica content. Before undergoing the process of treatment, the raw material was ground, sifted, and desiccated.

The leaching treatment was proceeded by hydrochloric acid (HCl) with 37% concentrated lemongrass immersed in 5M HCl at  $110^\circ\text{C}$  for 3 hours. Subsequently, the sample of lemongrass was washed with distilled water and then heated at  $105^\circ\text{C}$  in the furnace overnight. After that, the dehydrated lemongrass was calcined at  $600^\circ\text{C}$  until white ash yielded [33].



Figure 3. Extraction of Silica Nanoparticles from Bamboo Leaves [26]

### C. *Eragrostis tef* (Teff)

*Eragrostis tef*, also known as teff, is an annual grass and family of poaceae. Brown teff straw was utilized and collected from South Gondar, Ethiopia, in the study of Wassie & Srivastava [32]. Teff straw was rinsed with distilled water and heated at 80°C in the electric oven before it was ground to pass over 0.1mm sieve mesh and rinsed and heated back. Then, it was stored at room temperature in a sealed plastic bag. To avoid the crystallization temperature, the combustion of silica nanoparticles had been made at 600°C. The sample of teff straw was heated for 4 hours to 600°C in a muffle furnace at a rate of 10 °C/min [32].

Various approaches were used to characterize the combusted sample. 100 g/L of acid solution (10% acid solution of HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub> mixture at 4:1 v:v ratio) was treated with the teff straw ash for 30 minutes. Next, the teff straw was rinsed a few times using distilled water before it was centrifuged at 4000 rpm. It was heated again at the same temperature, holding time, and heating rate in a muffle furnace [32].

### D. *Hordeum vulgare* (Barley)

*Hordeum vulgare*, also called barley, is a cereal plant in the grass family Poaceae. According to Akhayere et al. [52], the barley obtained from Haspolat, Lefkosa farms was heated and stored at 100°C. The barley sample was rinsed with distilled water to eliminate impurities, contaminants, and dust before drying it at 100°C for 24 hours. After the miller turned the powder into powder, the powdered barley was heated to 10% w/v HNO<sub>3</sub> and washed a few times with distilled water. Next, the solid was heated for 24 hours at 60°C. The prepared sample was refluxed with 2M HCl for 6 hours and divided into four for the heating process at 700°C in a furnace [52].

### E. *Oryza sativa* (Rice)

*Oryza sativa* is rice and is part of the Poaceae family. Based on the study of Mahmud et al. [53], the rice husks collected from Perak, Malaysia, were rinsed through clear water to remove dirt and continuously heated at 110°C in an oven for 24 hours. The prepared husks were then subjected to reflux heating at 100°C by continuous stirring for 2 hours in anhydrous citric acid solutions. Then, the residue husks were filtered and washed frequently until the neutral pH was reached. Husks were then heated at 110°C for 24 hours in an air oven before they were weighted and pulverized subsequently in the furnace for 2 hours at 700°C until the whitish amorphous silica nanoparticles formed [53]. The extraction of silica nanoparticles from rice husk is shown in Figure 4.

### F. *Saccharum munja* (Pin red grass)

*Saccharum munja*, also called pin red grass, is part of the Poaceae family. According to Khan et al. [29], the dried *Saccharum munja* (obtained from the Bannu region, Khyber Pakhtunkhwa, Pakistan) was acidified with a solution of 30% sulfuric acid and 10% HCl. After that, the prepared sample was heated for 4 hours at 600°C in the furnace to change all the incorporated hydrocarbons before continuing to the acid washing step. The calcined *Saccharum munja* ash was acid-leached for 2 hours at 100°C with 10% HCl solution under reflux conditions in this step. All the materials were filtered and washed a few times with distilled water until a neutral pH was gained.

Next, the treatment of alkaline was done for *Saccharum munja* ash via NaOH with the condenser in the heating mantle, and then the solution was refluxed for 5 hours to prepare a solution of sodium silicate. The solution of silicate was filtered and rinsed with boiling water before being cooled to room temperature [29].

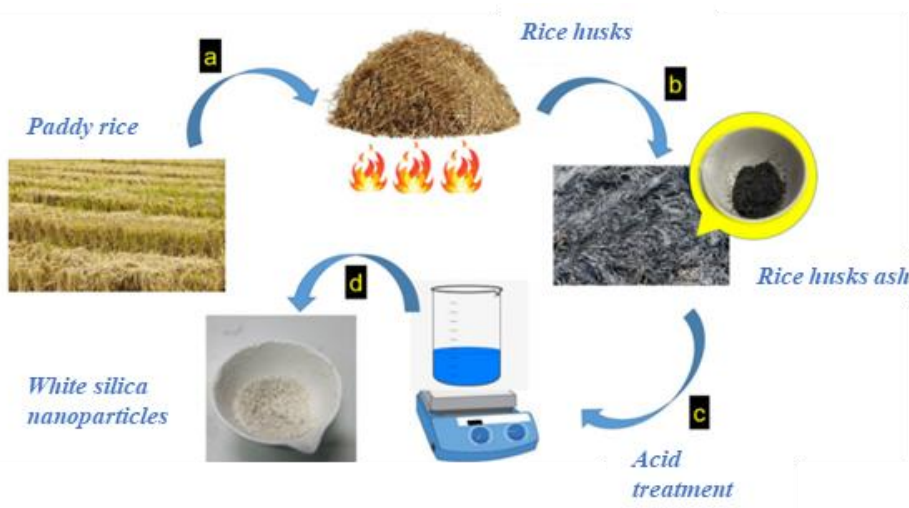


Figure 4. Extraction of Silica Nanoparticles from Rice Husks [53]

Subsequently, the sodium silicate produced was gently added to the solution (SDS, butanol, and water) and stirred at 60°C for 30 minutes before H<sub>2</sub>SO<sub>4</sub> was included. The mixture was then aged at 60°C for 8 hours and filtered before rinsing with distilled water several times and calcined at 550°C for 4 hours in atmospheric conditions [29].

### G. *Saccharum officinarum* (Sugarcane)

*Saccharum officinarum*, or sugarcane, is one of the plants from the Poaceae family. As reported by Falk et al. [31], sugarcane bagasse ash (SBA) was the precursor of silica from Ribeirao Preto, Brazil. SBA was sieved and then leached in HCl with a continuous stirring for 1 hour at 100°C. The prepared ash was sieved and rinsed with distilled water a few times before NaOH was combined under constant stirring at 100°C for 1 hour.

Then, the yielded sodium silicate was chilled to room temperature. Next, the agitation process was done by titration of HCl, and after 24 hours of ageing, the gel was washed with deionized water a few times and dehydrated until silica xerogels were produced [31]. Figure 5 shows the extraction of silica from sugarcane bagasse ash via sol-gel.



Figure 5. Extraction of Silica Nanoparticles from Sugarcane Bagasse Ash [31]

## V. QUANTIFICATION OF SILICA NANOPARTICLES EXTRACTED BY POACEAE SPECIES

To quantify the silica nanoparticles, the precipitated silica from each Poaceae species was weighed on the analytical balance. The percentage yield of silica was obtained by the precipitation as shown in Equation 1, whereas  $\eta$  (%) will be the silica percentage yield,  $m_{ext}$  (g) will represent the mass of the silica nanoparticles (SiO<sub>2</sub>) produced, and  $m_{SiO_2}$  (g) is the weight of the extracted compound chose [54].

$$\eta (\%) = \frac{m_{ext}}{m_{SiO_2}} \times 100 \quad (1)$$

The values (%) of silica nanoparticle composition in each Poaceae family species are represented in Table III. From the tables, the production of silica nanoparticles from *Oryza sativa*, also known as rice, was the highest content of other Poaceae species extracted.

Table III. Summary of the Values (%) of Silica Nanoparticles Composition in Poaceae Family Species

Poaceae Family Species	Value (%)	Reference
Bambusoideae (Bamboo)	85.57	[51]
<i>Cymbopogon citratus</i> (Lemongrass)	98.59	[33]
<i>Eragrostis tef</i> (Teff)	97.01	[32]
<i>Hordeum vulgare</i> (Barley)	94.1	[52]
<i>Oryza sativa</i> (Rice)	99.77	[53]
<i>Saccharum munja</i> (Pin red grass)	3.3	[29]
<i>Saccharum officinarum</i> (Sugarcane)	96.8	[31]

As previously mentioned in the methodology of Bambusoideae, bamboo leaves were utilized in this research compared to other parts since they contained the richest silica component, which is over 70% of the composition. According to Nazri & Sapawe [51], 85.57% of the silica component was composed successfully of 203.54g of bamboo leaves usage by calcination at a temperature of 600°C.

Next, the *Cymbopogon citratus* gained a value of 98.59% in silica content from the treated lemongrass ash. The parameter used to obtain the highest content was similar to Bambusoideae since the calcination temperature was 600°C, yet the leaching temperature was 110°C [33].

For *Eragrostis tef*, the teff straw was combusted and treated with acid at 600°C for four hours to obtain a silica content of 97.01%. The treatment used is also known as thermo-chemical treatment [32].

Subsequently, *Hordeum vulgare* obtained 94.1% silica content in them by conducting the barley grass waste at 700°C for combustion and undergoing the acid treatment method. Barley grass sustained some treatment processes to remove the metallic impurities, which further affected quality and integrity [52].

*Oryza sativa* gained the highest content among the researched Poaceae family species, with a silica content value of 99.77%. Based on Mahmud et al. [53], the acid treatment proceeded with anhydrous citric acid and was done by pulverization in the furnace for 2 hours at 700°C.

Then, for *Saccharum munja*, the obtained silica content value is only 3.3%, which is the lowest amount of silica in the Poaceae family species that was researched. The utilized parameter was the acid treatment method via 10% HCl and 30% H<sub>2</sub>SO<sub>4</sub>, and the calcination process at 550°C in atmospheric conditions [29].

Finally, the *Saccharum officinarum* was synthesized via the sol-gel polymeric pathway via sugarcane bagasse ash. The ashes were leached with 1M HCl 37% in producing silica xerogels with 96.8% of silica purity [31].

## VI. CHARACTERIZATION OF SILICA NANOPARTICLES IN POACEAE SPECIES

Some methods can be utilized to identify the nano-silica in the plant matter, like colourimetric, gravimetric, x-ray, and spectrophotometric. These methods can be categorized as gravimetric, spectrophotometric after solubilization (colourimetric, atomic absorption (AA) and inductively coupled plasma (ICP)) and non-destructive spectrophotometric (x-ray fluorescence, near-infrared) [4]. The identification of  $\text{Si}(\text{OH})_4$  utilizing Si-molybdate complexes via UV-Vis (colourimetry) or the identification of silica using inductively coupled plasma-optimal emission spectroscopy (ICP-OES) is two of the most popular analytical methods for silica quantification [55]. TGA and elemental studies reveal how the amino groups modified the surface of silica nanoparticles and how this process varied depending on the surface area and the size of the produced nanoparticles [56]. X-ray Fluorescence (XRF) is utilized to gain the chemical components of silica without chemical treatment [57].

Furthermore, the solubilized samples can also be identified by other techniques such as Inductively Coupled Plasma-Atomic Emission Spectrophotometry (ICP-AES) and Absorption Spectrophotometry (AAS). Experiments on chloride acid and fluoride acid extraction are completed, proving that there is no difference between the two methods [58-59]. The identification of components in silica for bamboo lignocellulosic waste liquid (black liquor) is one of the studies in the determination of silica by ICP-AES [60].

Generally, there are several instruments utilized to characterize silica and silica nanoparticles from the Poaceae species, such as x-ray fluorescence analysis (XRF), thermogravimetric analysis (TGA), morphological analysis by Fourier transforms infra-red (FTIR), scanning electron microscopy (SEM) and x-ray diffraction (XRD). However, in this research, the selected instruments to analyze the silica nanoparticles were focused on the FTIR analysis only.

### A. Fourier Transform Infra-Red (FTIR) Analysis

All major chemical groups that exist in the silica nanoparticles were analyzed in the range of  $4000\text{--}350\text{ cm}^{-1}$ . Without further preparation, FTIR was utilized for liquid or solid state probing. The Attenuated Total Reflection (ATR) sampling methodology was utilized to examine the functional groupings of samples in each of the Poaceae species. Initially, the FTIR background spectrum was recorded. The samples were then transferred to the sample plate. The metal tip's handle was turned clockwise until it was neared to the sample. After commencing the scanning procedure, the spectrum emerged. The handle was turned anticlockwise, and the metal tip was raised after the spectrum scanning process was completed [61]. Figure 6 illustrates an FTIR spectrometer fitted with diamond ATR sample cell features, a close-up of the sample platform, a sketch of ATR's sample slot, and a schematic diagram depicting how the source's radiation interacts with the sample.

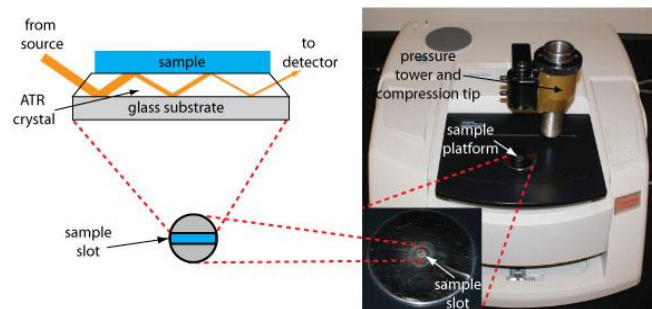


Figure 6. FTIR Spectrometer Equipped with a Diamond ATR Sample Cell

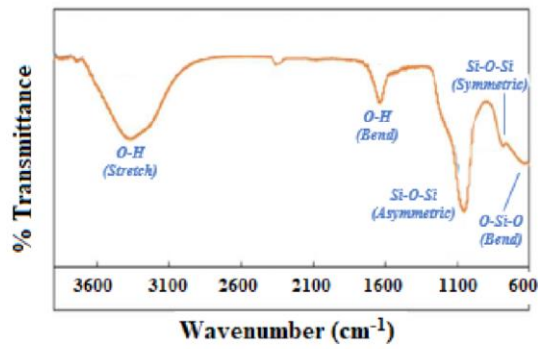
Figure 7 shows how similar the pattern of principal chemical groups exists in the silica extracts in each Poaceae family species, which were identified using FTIR spectra analysis. Generally, the bending vibration of the O-H bond from silanol groups (Si-OH) was responsible for the broadband at  $1633\text{ cm}^{-1}$  to  $1645\text{ cm}^{-1}$ . Meanwhile, the adsorbed water molecules on the surface of silica [62-64] were caused by the stretching vibration of the O-H bond from Si-OH silanol groups starting from  $3000\text{ cm}^{-1}$  to  $3700\text{ cm}^{-1}$ . The bending vibration network of O-Si-O was linked to band  $534\text{ cm}^{-1}$  to  $800\text{ cm}^{-1}$ , while the symmetric stretching vibration of Si-O-Si was associated with band  $601\text{ cm}^{-1}$  to  $807\text{ cm}^{-1}$ . In addition, the presence of band  $1044\text{ cm}^{-1}$  to  $1092\text{ cm}^{-1}$  was caused by Si-O-Si stretching vibration in asymmetric types. The general results of major chemical groups in the selected Poaceae family species [65-67] were also revealed in Table IV.

Table IV. IR Frequency Range and Band Assignment for Poaceae Family Species

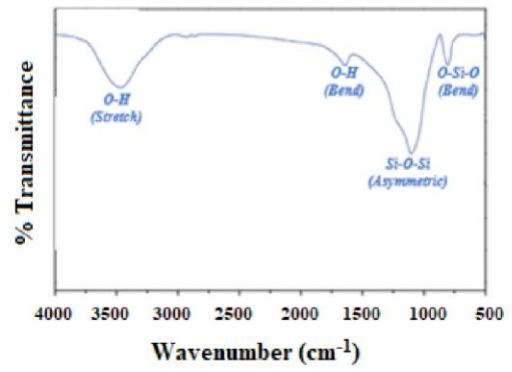
Band Assignment		Wavenumber ( $\text{cm}^{-1}$ )
O-H bond in Si-OH (Silanol groups)	Bending vibration	1633 - 1645
	Stretching vibration	3000-3700
O-Si-O	Bending vibration	534-800
Si-O-Si	Symmetric stretching vibration	601 - 807
	Asymmetric stretching vibration	1044 - 1092

According to Figure 7, all Poaceae family species obtained five significant peaks as bending and stretching vibration of the O-H bond in the silanol group, bending vibration in the O-Si-O bond, symmetric stretching vibration, and asymmetric stretching vibration in the Si-O-Si bond, respectively. However, the *Hordeum vulgare* (barley) and *Oryza sativa* (rice) were lacking in the symmetric stretching vibration of the Si-O-Si bond. The parameter influenced one of the reasons why it occurred in the methodology process, such as temperature, etc.

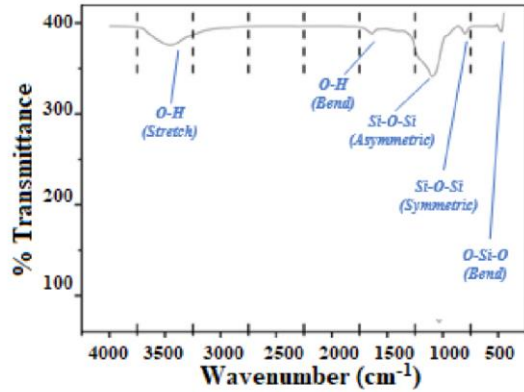




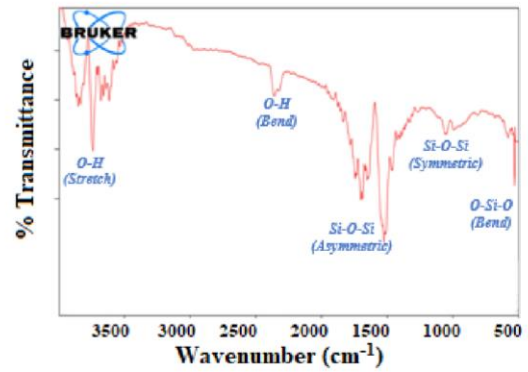
a) *Bambusoideae* [51]



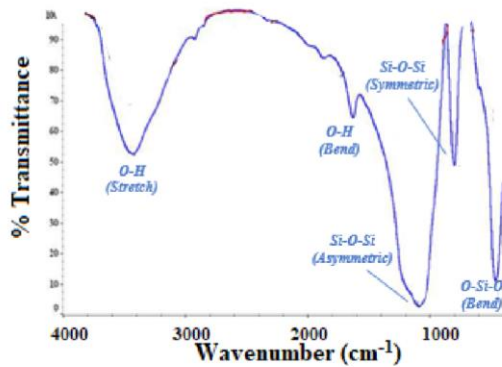
e) *Oryza sativa* [53]



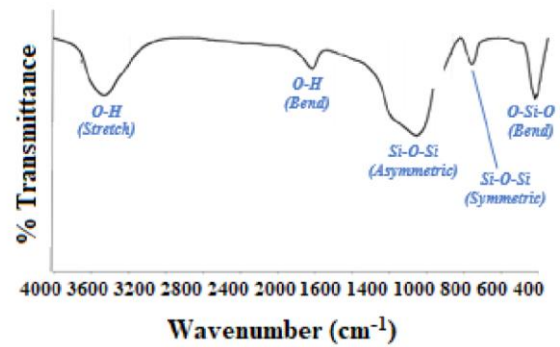
b) *Cymbopogon citratus* [33]



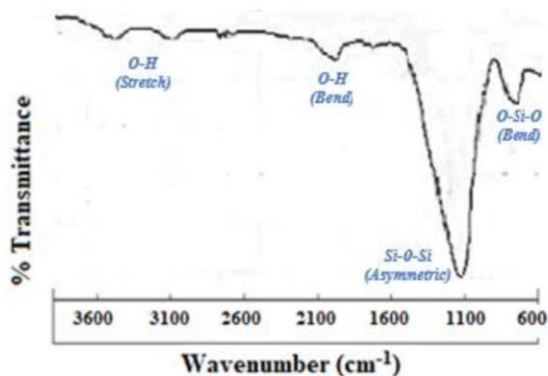
f) *Saccharum munja* [29]



c) *Eragrostis tef* [32]



g) *Saccharum officinarum* [31]



d) *Hordeum vulgare* [52]

Figure 7. FTIR Spectra of Produced Silica Nanoparticles in a) *Bambusoideae*, b) *Cymbopogon citratus*, c) *Eragrostis tef* d) *Hordeum vulgare*, e) *Oryza sativa*, f) *Saccharum munja* g) *Saccharum officinarum*

## VIII. CONCLUSIONS

Silica nanoparticles are well known as a major section in the nanoparticle research industry, specifically in the medicine section, since they have the properties of large pore sizes and volume, high surface area, high biocompatibility, modifiable surfaces, and short diffusion-reaction pathways. They also emphasize biomedical fields regarding their stability and low toxicity in the human body. Since most of the Poaceae family species contain a high value of silica content and are easily acquired, the research focused on the pathway to extracting the silica nanoparticles via the green technology method, which is sol-gel. This method has dual advantages, which are that it can produce relevant silica particles at a lower cost and reduce disposal as well as the pollution challenges.

Based on the quantification results, the *Oryza sativa* obtained the highest value of silica nanoparticles among the other Poaceae family species. To produce the highest silica content, which is 99.77%, the accurate parameter in methodology was crucial. Since the rice husk ash was treated with anhydrous citric acid instead of hydrochloric acid, rice yielded a high content of silica. In addition, when other Poaceae family species calcined at temperate below 600°C, the rice was pulverized for 2 hours at 700°C in the furnace.

Ultimately, in characterizing the major chemical groups that exist in the silica nanoparticles, FTIR analysis in the range of 4000–350 cm<sup>-1</sup> was utilized. All researched Poaceae family species showed the presence of bending and stretching vibration of the O-H bond in the silanol group, bending vibration in the O-Si-O bond, symmetric stretching vibration, and asymmetric stretching vibration in the Si-O-Si bond, respectively, except for the *Hordeum vulgare* (barley) and *Oryza sativa* (rice) which were lack of the peak of symmetric stretching vibration of Si-O-Si bond.

Given the diverse extraction methods and conditions utilized by researchers to derive silica nanoparticles from Poaceae family plants, there remains ample opportunity for future exploration aimed at systematically refining these parameters. Enhancements are needed to attain improved yields, better control over particle size distribution, enhanced surface properties, and other pertinent physicochemical characteristics.

## CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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