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Original Research Article

Hydrogen Peroxide-Assisted Extraction and FTIR Characterization of Silica from Bambusa vulgaris Leaves Using 650°C Calcination

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Abstract

Valorization of silica-bearing plant residues offers a route for converting low-value biomass into functional inorganic materials. This study investigated silica recovery from Bambusa vulgaris leaves using a multistage process comprising hydrochloric-acid leaching, hydrogen-peroxide pretreatment, calcination, alkaline extraction, acid precipitation, washing, and drying. Freshly collected leaves were washed and oven-dried, after which a 50.044 g powder batch was leached with 1 M HCl at 80°C for 1 h. The recovered dry mass after leaching was 31.8 g. A 3.0 g acid-leached sub-batch was then treated with 1 mL of 30% H2O2 in 20 mL distilled water at 60°C for 1 h and dried to 1.942 g before calcination at 650°C for 3 h. A representative gravimetric mass balance showed that approximately 100 g of dry leaf powder produced 20 g of calcined ash and approximately 10 g of dried silica-based powder, corresponding to a 20% ash yield, a 10% recovered-product yield on a dry-leaf basis, and a 50% ash-to-product recovery. The calcined material was subjected to alkaline extraction with 2 M NaOH at 90°C for 1 h, and the resulting sodium-silicate-containing filtrate was acidified with HCl to approximately pH 3. The formed gel was washed and dried at 100°C for 12 h to obtain a white silica-based powder. FTIR analysis over 4000-400 cm-1 showed a broad O-H band centered at 3398 cm-1, a prominent Si-O-Si asymmetric stretching band at 1062 cm-1, a Si-O-related band near 800 cm-1, and a broad Si-O-Si bending region at approximately 470-425 cm-1. These features are consistent with hydrated, silanol-containing silica and with the broad spectral profile commonly reported for biogenic amorphous silica. However, FTIR alone cannot establish crystallographic phase or quantitative purity. The study therefore demonstrates a feasible H2O2-assisted extraction route with competitive gravimetric recovery, while identifying XRD, XRF/EDX, replicated yield measurements, and a matched no-H2O2 control as essential next-stage analyses.

Keywords: Bambusa Vulgaris, Bamboo Leaf Waste, Biogenic Silica, Hydrogen Peroxide, Acid Leaching, Alkaline Extraction, FTIR, Biomass Valorization.

1. INTRODUCTION

Silica is an important inorganic material used across adsorbents, catalysts, composites, ceramics, coatings, and other advanced material systems. Conventional silica production relies heavily on mineral feedstocks, whereas plant-derived silica provides an alternative route in which biogenic silicon accumulated during plant growth is recovered from residual biomass [10, 11]. The attraction of agricultural and forestry residues is not simply that they are renewable; their ash can contain silica in a chemically accessible form that can be converted to sodium silicate under alkaline conditions and subsequently re-precipitated as silica [8, 9]. For a viable bio-silica process, however, ash composition, organic removal, metallic impurities, thermal history, extraction pH, and washing efficiency all influence the quality and reactivity of the final material.

Bamboo is especially relevant because members of the Bambusoideae accumulate silicon in leaf tissues as phytoliths. Recent microscopy and elemental

work on tropical bamboo has shown that silica deposition is concentrated in epidermal tissues and increases with leaf age, supporting the use of senescent or waste leaves as a silica-bearing feedstock [1]. Earlier work on bamboo leaf ash likewise reported a strongly siliceous composition. Frías *et al*., characterized bamboo leaf ash produced at 600°C using XRF, XRD, FTIR, and thermal methods [2], while Moraes *et al*., reported an ash containing 74.23% silica, with most of that silica assessed as amorphous by complementary characterization [3]. These findings establish bamboo leaves as a plausible biogenic silica source, although composition varies with species, leaf maturity, soil, climate, and combustion conditions.

Several processing strategies have been used to recover silica from bamboo leaves. Rangaraj and Venkatachalam used high-temperature treatment followed by acid washing, NaOH extraction, acid precipitation, drying, and additional heat treatment to produce amorphous silica nanoparticles from Dendrocalamus strictus [4]. Fatimah *et al*., extracted silica from bamboo leaf ash by refluxing with NaOH and precipitating a silica gel by acidification [5]. More recently, Vinai *et al*., demonstrated that bamboo leaf ash generated over a 550-800°C range can serve as a source for bio-derived sodium silicate [6], and Bindar *et al.,* optimized a bamboo-leaf bio-silica route involving acid washing, combustion at 700°C, NaOH extraction, gelation, and drying [7]. Together, these studies show that alkali dissolution and acid precipitation are robust steps, but thermal and pretreatment conditions remain process-specific.

Acid leaching is widely used in biomass-silica production because alkali and alkaline-earth metals can promote unwanted mineral phases during heating and remain as contaminants in the recovered product. Classical rice-husk work by Kalapathy *et al.,* demonstrated that alkaline extraction followed by acid precipitation can produce silica gels and that acid pretreatment and final washing reduce mineral contamination [8, 9]. Contemporary reviews similarly identify pretreatment, controlled thermochemical conversion, and thorough washing as major determinants of bio-silica quality [10, 11]. These principles are transferable to bamboo leaves, but the lignocellulosic matrix introduces an additional challenge: cellulose, hemicellulose, lignin, and extractives must be sufficiently disrupted or combusted to expose the inorganic fraction.

Hydrogen peroxide is an oxidative reagent with an established role in bamboo delignification, particularly under alkaline conditions. Studies on bamboo biomass have shown that peroxide-based pretreatments can cleave or oxidatively modify lignin structures and improve removal of the lignin-rich matrix [12, 13]. Atmospheric-pressure alkaline hydrogen-peroxide treatment has also produced substantial delignification in bamboo [15]. The present work incorporated an H2O2 treatment after acid leaching and before calcination. Importantly, the current experiment did not include a matched no-H2O2 control, so the contribution of this step to ash quality, silica yield, or required calcination temperature cannot be isolated experimentally. It is therefore treated here as an exploratory process feature rather than as a proven energy-saving modification.

The downstream motivation of the project is textile materials processing. Silanes and silicones are widely used in textile finishing, and silica/siloxane chemistry can provide routes toward surface modification and functional coatings [14]. Nevertheless, the present manuscript is intentionally limited to the extraction and characterization stage. It does not claim that the recovered powder is equivalent to a commercial silicone softener, nor does it use the fabric-mechanical data from the broader thesis project. This separation allows the silica-recovery chemistry to be evaluated on its own evidence.

Accordingly, the objectives of this study were to (i) recover a silica-based powder from Bambusa vulgaris leaves using acid leaching, H2O2 pretreatment, 650°C calcination, alkaline extraction, and acid precipitation; (ii) document stage-specific mass changes and quantify representative gravimetric ash and silica-based-product yields; and (iii) evaluate the recovered material by FTIR spectroscopy. The study also benchmarks the observed recovery against published bamboo-leaf silica routes that did not employ H2O2 pretreatment, while recognizing that a matched peroxide-free control is required to establish a causal yield advantage. Additional analytical evidence is still required for a stronger materials claim, particularly XRD for crystallographic phase and XRF or EDX for elemental composition and quantitative purity.

2. MATERIALS AND METHODS

2.1 Raw Material and Reagents

Bambusa vulgaris leaves were hand-collected from mature bamboo stands on the NITER campus and nearby areas in Dhaka, Bangladesh. The initially harvested mass was approximately 1.034 kg. Leaves were visually sorted and washed with distilled water to remove adhering soil, dust, and loosely attached debris. The cleaned wet mass was 555 g, and the recorded dry mass after oven drying was 395 g. The main experimental summary records the initial drying step at 80°C, while one narrative laboratory entry reports 100°C. Because the original records do not fully resolve this discrepancy, the process should be regarded as oven drying within the 80-100°C range; all subsequent masses are reported exactly as recorded.

Analytical or laboratory-grade hydrochloric acid (HCl), sodium hydroxide (NaOH, 98%), hydrogen peroxide (H2O2, 30%), and distilled water were used.

HCl was used for both the initial leaching step and later acid precipitation. NaOH was used to convert silica in the calcined material into soluble sodium silicate. The H2O2 treatment was performed as an oxidative pretreatment before calcination.

2.2 Leaf Preparation and Acid Leaching

The dried leaves were cut and ground with a blade grinder to obtain a coarse powder with increased surface area. A recorded batch of 50.044 g was treated with 500 mL of 1 M HCl prepared from 41.7 mL concentrated HCl and distilled water. Leaching was carried out at 80°C for 1 h with the pH maintained at approximately 1.5-2. The solid fraction was then filtered and washed repeatedly with distilled water until the wash filtrate approached pH 6.5. The formal method records oven drying at 80°C for 4 h, yielding 31.8 g of acid-leached dry material. A separate notebook extract lists 1 h for this drying step; the 4 h condition is retained in the manuscript because it is the value repeated in the consolidated methodology and experimental-condition table, while the documentation inconsistency is acknowledged as a limitation.

2.3 Hydrogen-Peroxide Oxidative Pretreatment

A 3.0 g portion of the acid-leached dry powder was treated with 1 mL of 30% H2O2 dispersed in 20 mL distilled water. The suspension was stirred continuously at 60°C for 1 h, filtered, washed with distilled water, and oven-dried at 80°C for 20 min. The recovered dry mass was 1.942 g. The source thesis labels this step as delignification. Because neither lignin content nor a no-peroxide control was measured, the term oxidative pretreatment is used in this manuscript, while the established delignifying action of peroxide in bamboo under optimized alkaline conditions is used only as mechanistic context [12-15].

2.4 Calcination

The H2O2-treated material was placed in a ceramic crucible and calcined in a muffle furnace at 650°C for 3 h under air. The purpose of this step was to combust the remaining lignocellulosic material and produce a silica-enriched inorganic ash. The ash was described visually as grey to white. For the selected 1.942 g analytical sub-batch, a separate post-calcination mass was not preserved. In the broader process mass balance, however, approximately 100 g of dry processed leaf powder produced approximately 20 g of calcined ash, corresponding to a gravimetric ash yield of 20%.

2.5 Alkaline Extraction and Sodium Silicate Formation

The calcined material from the selected batch was subjected to alkaline digestion using 1.58 g NaOH (98%) dissolved in 19.4 mL distilled water, corresponding to an approximately 2 M NaOH solution in the recorded protocol. The mixture was stirred at 90°C for 1 h. Under strongly alkaline conditions, silica dissolves to form sodium silicate according to Eq. (1):

SiO2 + 2NaOH -> Na2SiO3 + H2O (1)

The slurry reached a strongly alkaline pH (recorded at approximately 13) and was filtered to separate insoluble residues from the sodium-silicate-containing filtrate. Alkali extraction followed by acid precipitation is a well-established approach for recovering silica from silica-rich biomass ashes [8, 9].

2.6 Acid Precipitation, Washing, and Drying

The sodium silicate filtrate was acidified by dropwise addition of an HCl solution prepared using 8.33 mL concentrated HCl in a total solution volume of 50 mL. Acid was first added toward neutrality and then continued until approximately pH 3. The nominal precipitation reaction is shown in Eq. (2):

Na2SiO3 + 2HCl -> SiO2 + 2NaCl + H2O (2)

The acidified system was covered and allowed to rest for 12 h to promote gelation. A dark green gel was observed at this stage. The original project attributed this color to co-precipitated plant pigments; because no chemical assay of the colored species was conducted, it is more cautiously described here as residual plant-derived chromophore contamination. The gel was filtered using ash-free filter paper and washed repeatedly with hot distilled water until the filtrate approached pH 6.5-7. The washed material was dried at 100°C for 12 h, producing a white silica-based powder. On the representative 100 g dry-leaf basis, approximately 10 g of dried silica-based powder was recovered from approximately 20 g of ash.

2.7 FTIR Characterization

Fourier transform infrared spectroscopy was performed on the recovered powder (Sample 035) at CRIR-NITER on 2 April 2026. The spectrum was collected in transmission mode over 4000-400 cm-1 at 1 cm-1 resolution and reported as transmittance (%T). The analytical objective was to identify vibrational bands associated with silanol and siloxane groups and to compare the spectral profile with published biogenic silica spectra. FTIR was not used as a stand-alone quantitative purity method and was not treated as a definitive crystallographic technique.

2.8 Data Treatment

Mass retention and loss were calculated only for stages where both an input and an output mass were available for the same batch or for the representative process mass balance. The raw-to-washed mass change was not interpreted as moisture content because visual sorting and removal of external debris occurred during washing. Moisture-related loss was instead estimated from the washed wet mass to the recorded dry mass. Because different sub-batches were used after acid leaching, the early stage-specific percentages were not multiplied to estimate overall recovery. Instead, the representative 100 g dry-leaf mass balance was used directly to calculate ash yield and recovered-product

yield. No inferential statistics were applied because the extraction stages and FTIR analysis were not performed as replicated independent batches.

Gravimetric yields were calculated as follows: ash yield (%) = (mass of calcined ash / mass of dry leaf powder) x 100; recovered silica-based-product yield on a dry-leaf basis (%) = (mass of dried recovered powder / mass of dry leaf powder) x 100; and ash-to-product recovery (%) = (mass of dried recovered powder / mass of calcined ash) x 100. For the representative mass balance (100 g dry leaf -> 20 g ash -> 10 g dried product), these values were 20%, 10%, and 50%, respectively. Because elemental purity was not measured by XRF/EDX, the 10% value is reported as gravimetric recovered-product yield rather than quantitative pure-SiO2 yield.

Table 1: Consolidated experimental conditions used for silica recovery from Bambusa vulgaris leaves

Stage	Input / reagent	Condition	pH / endpoint	Recorded outcome
Leaf preparation	1.034 kg raw leaves	Wash; oven dry; grind	-	555 g washed; 395 g dry
Acid leaching	50.044 g powder; 1 M HCl, 500 mL	80°C, 1 h	1.5-2; wash to ~6.5	31.8 g dry solid
H2O2 pretreatment	3.0 g acid-leached material; 1 mL 30% H2O2 + 20 mL water	60°C, 1 h; dry 80°C, 20 min	Not recorded	1.942 g dry solid
Calcination	H2O2-treated solid	650°C, 3 h, air	-	Grey-white ash; ~20 g per 100 g dry-leaf basis (20% ash yield)
Alkaline extraction	1.58 g NaOH in 19.4 mL water	90°C, 1 h	~13	Sodium-silicate-containing filtrate
Acid precipitation	HCl prepared from 8.33 mL concentrated acid in 50 mL total	Dropwise acidification; rest 12 h	~7 then ~3	Silica gel
Washing / drying	Hot distilled water	Wash; 100°C, 12 h	Final wash ~6.5-7	White silica-based powder; ~10 g per 100 g dry-leaf basis (10% dry-leaf yield; 50% ash-to-product recovery)
FTIR	Sample 035	4000-400 cm-1; 1 cm-1 resolution	-	Silica-related vibrational bands

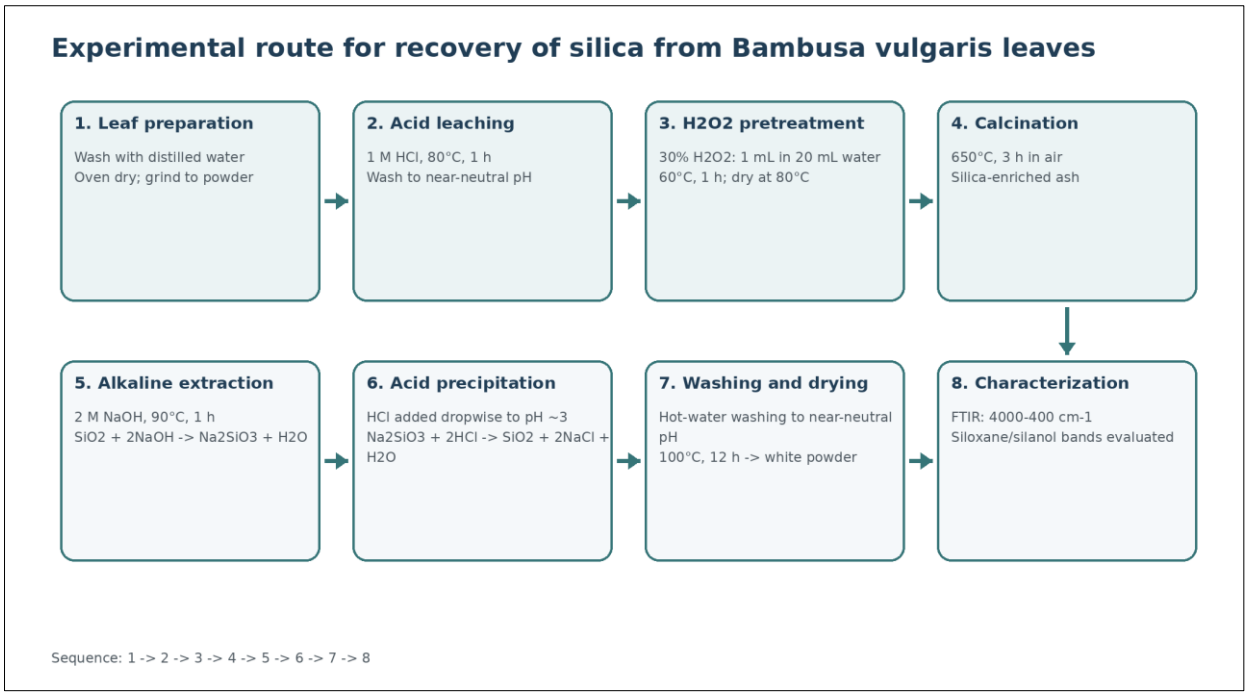

Figure 1: Process flow used in the present study. The diagram summarizes the recorded sequence and conditions; it does not imply that the independent effect of H2O2 was experimentally isolated

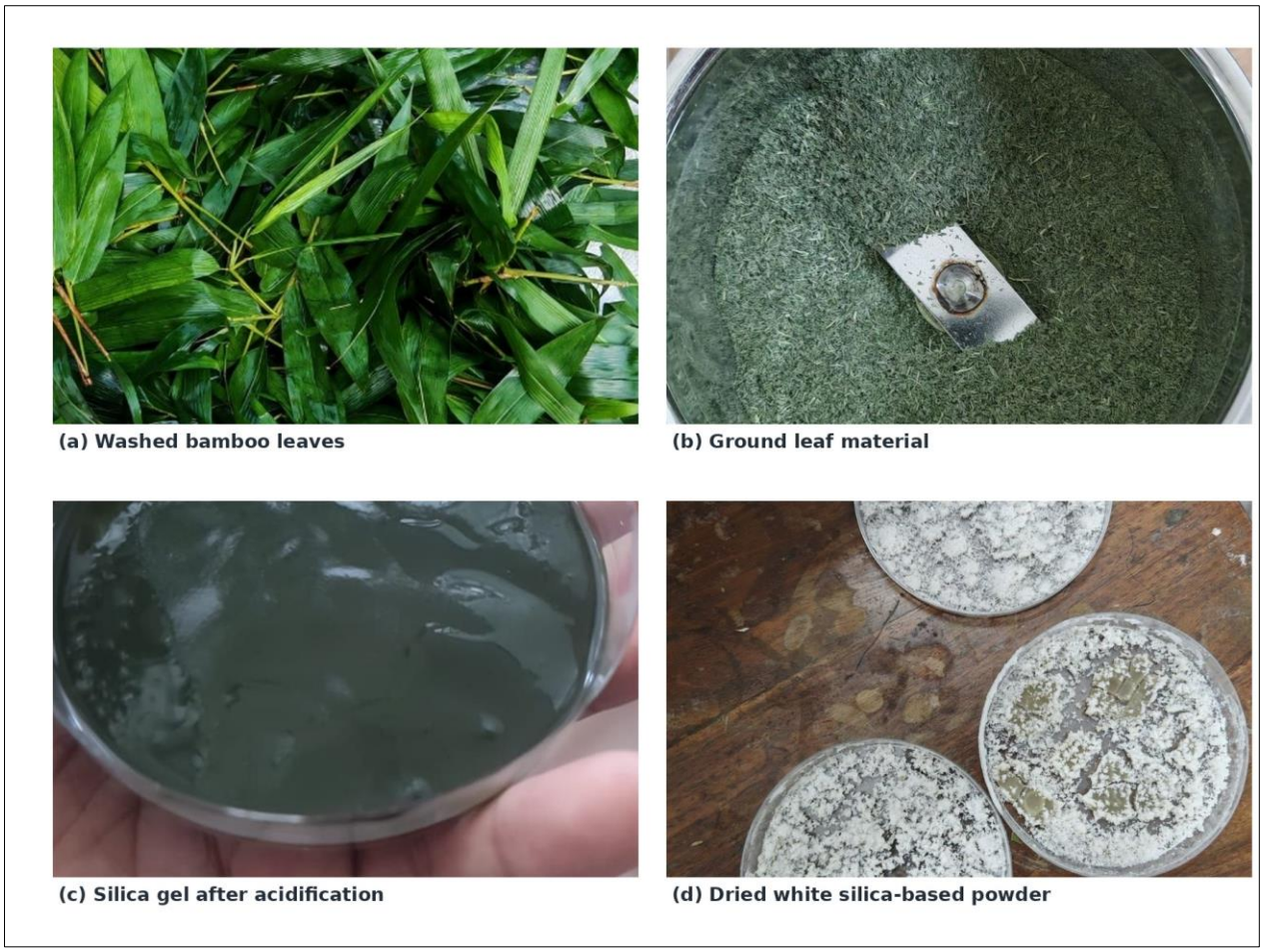

Figure 2: Representative laboratory observations from the extraction sequence: (a) washed Bambusa vulgaris leaves, (b) ground leaf material, (c) gel observed after acidification of the sodium silicate filtrate, and (d) dried white silica-based powder. Images originate from the authors' experimental records.

3. RESULTS AND DISCUSSION

3.1 Recorded Mass Changes and Gravimetric Silica-Product Yield

The initial 1.034 kg leaf collection decreased to 555 g after washing and manual removal of unwanted material, corresponding to 53.7% mass retention relative to the as-collected mass. This decrease should not be treated as simple moisture loss because soil, damaged material, and other external matter were removed during cleaning. Subsequent oven drying of the 555 g washed leaves produced 395 g dry material, equivalent to 71.2% retention and a 28.8% mass decrease relative to the washed wet mass. This latter change provides the most defensible estimate of water and volatile loss during the initial preparation stage.

For the acid-leaching batch, 31.8 g remained from 50.044 g of dry powder, giving 63.5% mass retention and 36.5% loss. The decrease is consistent with removal of acid-soluble inorganic constituents together with soluble or hydrolyzed organic components, although no elemental analysis of the leachate was performed. Acid pretreatment is widely reported to reduce metallic impurities in biomass-derived silica processes and can improve the chemical quality of the subsequently recovered silica [8-11]. In the present study, however, the identities and quantities of removed Ca, K, Mg, Fe, or Al were not directly measured and should therefore not be stated as experimentally confirmed.

The H2O2-treated sub-batch decreased from 3.000 to 1.942 g, corresponding to 64.7% retention and 35.3% mass loss. This observation shows substantial removal or conversion of material during the oxidative treatment and subsequent washing/drying, but the experiment cannot assign that loss specifically to lignin. Bamboo delignification studies demonstrate that peroxide systems can remove or oxidatively fragment lignin [12-15], yet those studies generally employ controlled alkaline peroxide conditions. The present treatment used a lower peroxide dose without a recorded alkaline pH adjustment, so its chemistry and efficiency should not be assumed to match alkaline hydrogen-peroxide pretreatment.

The representative process mass balance provides the overall gravimetric recovery requested for evaluation of extraction efficiency. Approximately 100 g of dry processed leaf powder yielded 20 g of calcined ash

(20% ash yield), from which approximately 10 g of dried silica-based powder was recovered. The corresponding recovered-product yield was therefore 10% on a dry-leaf basis, while the ash-to-product recovery was 50%. These values describe mass recovery of the FTIR-confirmed silica-based product; they should not be interpreted as 10 wt.% analytically pure SiO2 in the original leaf because quantitative elemental purity was not determined.

Table 2: Stage-specific mass changes and representative gravimetric recovery during silica extraction

Stage	Input mass (g)	Output mass (g)	Retention (%)	Mass decrease (%)
Collection -> washed	1034	555.0	53.7	46.3
Washed -> oven dry	555	395.0	71.2	28.8
Acid leaching batch	50.044	31.8	63.5	36.5
H2O2 sub-batch	3.000	1.942	64.7	35.3
Dry leaf -> calcined ash (representative basis)	100.0	20.0	20.0	80.0
Calcined ash -> dried silica-based product (representative basis)	20.0	10.0	50.0	50.0

Note: The first four rows are stage-specific measurements from the recorded batches. The final two rows present the representative 100 g dry-leaf mass-balance basis used to calculate ash yield and recovered silica-based-product yield. The 50% ash-to-product value is a gravimetric recovery and does not represent elemental SiO2 purity.

3.2 Role of Acid Leaching, Peroxide Pretreatment, and Calcination

The process integrates three different functions before alkaline extraction. First, HCl leaching is intended to remove acid-soluble mineral species and reduce the probability that non-silica minerals remain in or react with the ash. Second, the H2O2 step acts oxidatively on the organic matrix before thermal treatment. Third, calcination oxidizes the remaining carbonaceous fraction and concentrates the inorganic residue. The representative mass balance of the complete H2O2-assisted route gave a 20% ash yield, a 10% dry-leaf-basis recovered-product yield, and a 50% ash-to-product gravimetric recovery. These values provide a quantitative basis for comparing the present route with published bamboo-leaf silica processes.

The selected calcination temperature of 650°C lies within the broader range used in bamboo-leaf research. Frías et al. characterized bamboo leaf ash obtained at 600°C [2], Vinai *et al*., investigated ashes prepared between 550 and 800°C [6], and Bindar *et al*., used 700°C in an optimized bio-silica workflow [7]. By contrast, other bamboo silica studies have used substantially higher temperatures, including approximately 750-900°C [4, 5]. Because bamboo ash mineralogy depends strongly on temperature, time, and impurity chemistry, a lower numerical furnace setting cannot by itself be equated with lower process energy or superior amorphous-silica preservation. Establishing that the H2O2 step enabled 650°C calcination would require a controlled comparison against otherwise identical untreated material.

Published bamboo-leaf silica routes that did not include an H2O2 pretreatment provide useful external benchmarks. Rangaraj and Venkatachalam [4], reported a 50.2% silica-nanoparticle yield from bamboo leaf ash after thermal treatment, acid washing, alkaline extraction, and acid precipitation, while Bindar *et al*., [7], reported an optimized bio-silica yield of 42.33% following HCl washing, combustion at 700°C, NaOH extraction, gelation, and drying. The present H2O2-assisted route gave a 50.0% ash-to-product gravimetric recovery at 650°C. Numerically, this is 7.67 percentage points higher than the optimized yield reported by Bindar *et al*., and essentially comparable with the 50.2% ash-based yield reported by Rangaraj and Venkatachalam. This comparison indicates that the present lower-temperature H2O2-assisted sequence can achieve competitive silica-product recovery. However, differences in bamboo species, feed preparation, yield definitions, thermal conditions, reagent concentrations, and washing protocols prevent the cross-study comparison from proving that H2O2 itself caused the higher recovery; a matched no-H2O2 control remains necessary for that causal conclusion.

The grey-to-white ash observed after heating is consistent with extensive combustion of organic matter. However, color is only a qualitative indicator. Residual carbon, alkali metals, alkaline-earth metals, and minor silicate phases can persist even when the ash appears pale. The broader mass balance recorded approximately 20 g ash from 100 g dry processed leaf powder, but no loss-on-ignition measurement was performed and the selected 1.942 g FTIR sub-batch did not have a separately preserved post-calcination mass. TGA/LOI in future replicated batches would provide a direct measure of residual volatile or carbonaceous content.

3.3 Alkaline Dissolution and Acid Precipitation

The alkaline extraction stage follows the pH-dependent solubility behavior exploited in many biomass-silica processes: silica is converted to soluble silicate under strongly alkaline conditions, whereas lowering the pH promotes polymerization and precipitation of hydrated silica [8, 9]. The recorded rise to approximately pH 13 is consistent with the NaOH-rich extraction environment, and the subsequent appearance of a gel after HCl addition is consistent with silica condensation as the sodium silicate solution is acidified.

The initially dark green gel became white after repeated washing and drying. This visual change is important as a process observation because it indicates that colored soluble or dispersible species were removed during purification. Nevertheless, visual whiteness cannot be used as evidence of high chemical purity. In published bamboo silica studies, claims of high silica content or purity are supported by XRF, EDX, ICP, or other elemental methods, often together with XRD and microscopy [3-7]. The current product is therefore described as a white silica-based powder rather than as high-purity nanosilica.

3.4 FTIR Identification of Silica-Related Functional Groups

The FTIR spectrum of Sample 035 showed the expected cluster of silica-related vibrational bands (Figure 3). The broad envelope centered at 3398 cm-1 is assigned to hydrogen-bonded O-H stretching from surface silanol groups and adsorbed water. The most prominent mid-infrared feature occurred at 1062 cm-1 and is assigned to asymmetric Si-O-Si stretching. A further band near 800 cm-1 is associated with symmetric Si-O/Si-O-Si vibrations, while the broad absorption in the approximately 470-425 cm-1 region is consistent with Si-O-Si bending modes. Comparable bands have been reported for bamboo-derived silica and other biogenic silica materials [2-4].

The 1062 cm-1 band was broad rather than sharply resolved, and the low-wavenumber features likewise formed a broad envelope. Such broadening is commonly associated with disordered silicate networks and is consistent with an amorphous or poorly ordered silica-rich material. This interpretation agrees qualitatively with bamboo-leaf silica studies in which FTIR was combined with XRD to demonstrate an amorphous phase [3-7]. In the present work, however, XRD was not completed. The correct conclusion is therefore that the FTIR spectrum is consistent with a silanol-containing, amorphous-like silica network, not that the crystallographic phase has been definitively proven.

A narrow feature in the approximately 2350-2160 cm-1 region was treated as an atmospheric CO2/background-subtraction artifact rather than as a sample band. This is also consistent with the original instrumental interpretation. No strong conclusion about carbon-containing impurities was made from the remaining weak features because FTIR band intensity alone is not a quantitative impurity assay.

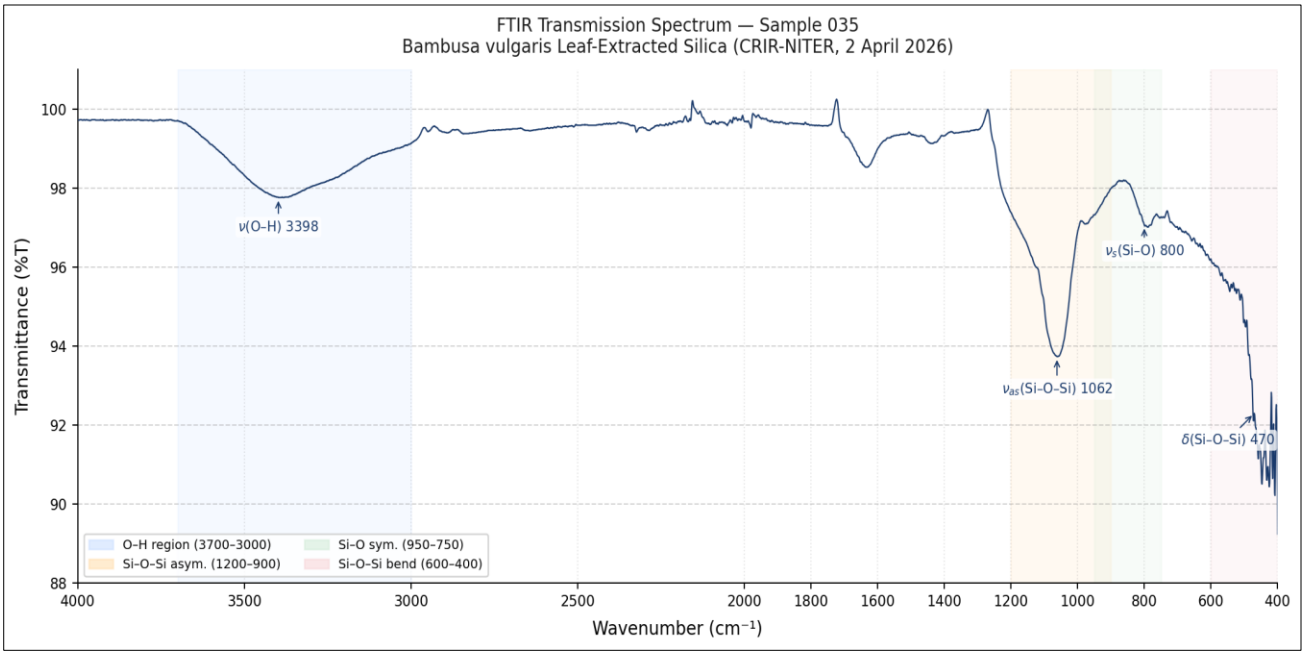

Figure 3: FTIR transmission spectrum of Sample 035 (Bambusa vulgaris leaf-derived silica-based powder). The spectrum was recorded at CRIR-NITER on 2 April 2026 over 4000-400 cm-1 at 1 cm-1 resolution. Annotated bands are those interpreted in the original project data

Table 3: Principal FTIR bands observed for Sample 035 and conservative assignments

Band position (cm-1)	Observed character	Assignment	Interpretation
3398	Broad O-H envelope	O-H stretching	Surface silanols and/or adsorbed water
1062	Strong, broad band	Si-O-Si asymmetric stretching	Primary silica-network fingerprint
~800	Moderate band	Si-O / Si-O-Si symmetric stretching	Consistent with silica network

Band position (cm-1)	Observed character	Assignment	Interpretation
~470-425	Broad low-wavenumber envelope	Si-O-Si bending	Consistent with silica network
~2350-2160	Narrow/spike feature	Atmospheric CO2 / background artifact	Not assigned to sample chemistry

3.5 Comparison with Published Bamboo-Leaf Silica Routes

The present route fits within the general chemical logic of reported bamboo-leaf silica recovery while differing in its pretreatment sequence and analytical completeness. Published studies commonly combine thermal conversion with alkaline dissolution and acid-induced gel formation, but they vary widely in calcination temperature, alkali concentration, precipitation pH, and final heat treatment [4-7]. Unlike the comparison routes in Table 4, the present process includes an H2O2 oxidative pretreatment before calcination. Its 50.0% ash-to-product gravimetric recovery is higher than the 42.33% optimized yield reported by Bindar *et al*., [7], and essentially matches the 50.2% bamboo-leaf-ash yield reported by Rangaraj and Venkatachalam [4]. This favorable numerical benchmarking strengthens the case for the H2O2-assisted route as a competitive process at 650°C, while the absence of an internally matched peroxide-free control means the magnitude of the H2O2-specific benefit cannot yet be isolated experimentally.

Table 4: Yield-oriented comparison of published bamboo-leaf silica routes with the present H2O2-assisted process

Study	Bamboo / feedstock	H2O2 pretreatment	Key thermal / chemical route	Reported yield / comparison
[2] Frías *et al*., (2012)	Bamboo leaf waste / ash	No	Calcination at 600°C; ash characterization	Extraction yield not reported in the present comparison; demonstrates silica-rich ash near 650°C.
[4] Rangaraj & Venkatachalam (2017)	Dendrocalamus strictus leaves	No	Thermal combustion; HCl washing; NaOH extraction; acid precipitation	50.2% silica-nanoparticle yield from bamboo leaf ash; essentially comparable with present 50.0% ash-to-product recovery (-0.2 percentage point).
[5] Fatimah *et al*., (2019)	Gigantochloa apus leaf ash	No	900°C ash; 4 M NaOH reflux; acid gelation	Yield basis not used for direct comparison; confirms non-peroxide sodium-silicate/acid-gel route.
[3] Moraes *et al*., (2019)	Bamboo leaf ash	No	Auto-combustion	Reported 74.23% silica content in ash (composition, not extraction yield).
[6] Vinai *et al.,* (2022)	Bamboo leaves / ash	No	Calcination 550-800°C; NaOH conversion to sodium silicate	No directly comparable isolated-silica yield used here; demonstrates bamboo ash as silicate source.
[7] Bindar *et al*., (2024)	Fallen bamboo leaves	No	HCl washing; 700°C combustion; NaOH extraction; gelation/drying	Optimized reported bio-silica yield 42.33%; present 50.0% recovery is numerically +7.67 percentage points.
Present study	Bambusa vulgaris leaves	Yes: 30% H2O2, 1 mL/3 g acid-leached material	1 M HCl; H2O2 pretreatment; 650°C; 2 M NaOH; HCl to ~pH 3	20% ash yield; 10% recovered-product yield on dry-leaf basis; 50% ash-to-product gravimetric recovery.

3.6 Sustainability and Potential Textile Relevance

Bamboo leaf valorization is attractive because the feedstock can be an underused residual biomass and its silicon is biologically accumulated rather than mined as a dedicated mineral raw material. Converting such residues to silica is consistent with circular-material strategies described for agricultural-waste-derived silica [10, 11]. The present process also operates at atmospheric pressure and uses common reagents. These features support the concept of decentralized or locally adapted silica production, especially where bamboo biomass and textile processing coexist.

At the same time, the process should not yet be labeled environmentally superior to commercial silica production. HCl and NaOH consumption, repeated

washing, wastewater neutralization, oven drying, and furnace heating all carry environmental burdens. No life-cycle assessment, energy balance, reagent-recovery study, or wastewater characterization was performed. A future sustainability comparison should therefore quantify energy per kilogram of recovered silica, acid/base consumption, water demand, salt generation, and opportunities for reagent or heat recovery.

For textile applications, the recovered material is best viewed as a potential silica precursor rather than as a finished silicone softener. Textile silicone systems usually rely on organofunctional silanes or polysiloxanes whose molecular flexibility and reactive functionality govern handle, durability, lubrication, and surface effects [14]. The FTIR-confirmed silanol/siloxane chemistry of the present powder is relevant because surface silanols can participate in subsequent silane coupling or sol-gel chemistry. Demonstrating textile-grade suitability, however, would require quantified purity, controlled particle size, reproducible surface area, formulation stability, and performance testing against commercial finishing agents.

3.7 Study Limitations and Required Validation

- No XRD measurement was completed; therefore, the amorphous character is inferred from FTIR band broadness and literature consistency rather than crystallographically confirmed.
- No XRF, EDX, ICP-OES, or ICP-MS analysis was completed; therefore, silica purity and residual elemental impurities are unknown.
- A representative process mass balance recorded approximately 100 g dry processed leaf powder -> 20 g ash -> 10 g dried silica-based product, allowing gravimetric yield calculation. However, this mass balance was not replicated independently, and the selected 1.942 g FTIR sub-batch did not retain a separate post-calcination mass; therefore, yield uncertainty and batch-to-batch reproducibility cannot yet be quantified.
- The extraction was not run as replicated independent batches, so confidence intervals, statistical significance, and reproducibility of the 20% ash yield, 10% dry-leaf product yield, and 50% ash-to-product recovery cannot be quantified.
- No matched control without H2O2 was performed. The literature benchmarking therefore shows that the 50.0% ash-to-product recovery is competitive with non-peroxide bamboo-leaf routes [4,7], but it does not establish that H2O2 itself is responsible for the numerical difference in yield.
- The source records contain minor inconsistencies in the initial drying temperature and post-acid-leach drying time; the consolidated conditions used here are reported transparently, but future work should use time-stamped standardized batch sheets.
- No SEM/TEM, particle-size analysis, BET surface-area measurement, TGA/LOI, or zeta-potential measurement was performed, limiting conclusions about morphology, porosity, residual organics, and dispersion behavior.

4. CONCLUSIONS

This study recovered a white silica-based powder from Bambusa vulgaris leaves through a sequence of acid leaching, H2O2 oxidative pretreatment, calcination at 650°C, NaOH extraction, HCl-induced gel formation, washing, and drying. The process produced measurable mass reductions during pretreatment, including 36.5% loss during acid leaching of the 50.044 g batch and 35.3% loss during H2O2 treatment of the selected 3.0 g sub-batch. A representative overall mass balance yielded approximately 20 g ash and 10 g dried silica-based powder from 100 g dry processed leaf powder, corresponding to 20% ash yield, 10% recovered-product yield on a dry-leaf basis, and 50% ash-to-product gravimetric recovery. This 50.0% recovery is numerically higher than the 42.33% optimized yield reported for the non-peroxide bamboo-leaf route of Bindar *et al*., [7], and is essentially comparable with the 50.2% ash-based yield reported by Rangaraj and Venkatachalam [4]. These literature benchmarks indicate competitive recovery for the present H2O2-assisted route at 650°C, although a matched no-H2O2 control is required before attributing the yield difference specifically to peroxide pretreatment. FTIR analysis showed a broad O-H band at 3398 cm-1, a strong Si-O-Si asymmetric stretch at 1062 cm-1, a band near 800 cm-1, and a low-wavenumber Si-O-Si bending region near 470-425 cm-1. These features provide strong spectroscopic evidence for a silanol-containing silica network and are consistent with the broad profiles reported for biogenic amorphous silica. The evidence does not, however, justify a quantitative high-purity claim or definitive crystallographic phase assignment. The most important next experiments are XRD, XRF/EDX, replicated mass-balance measurements, TGA/LOI, SEM/BET characterization, and a matched no-H2O2 control.

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DECLARATIONS

Ethics Approval: Not applicable. The study involved no human participants or animals.

Consent to Participate: Not applicable.

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Conflict of Interest: None declared.

REFERENCES

1. Rotmuenwai N, Aryuyo K, Kruethaworn N, Wattananit W, Yookongkaew N. Exploring silica accumulation in bamboo leaves: a study on phytolith morphology and epidermal patterning in the tropical giant bamboo Dendrocalamus copelandii. Annals of Botany. 2025;135(4):757-768. https://doi.org/10.1093/aob/mcae209
2. Frías M, Savastano H, Villar E, Sánchez de Rojas MI, Santos SF. Characterization and properties of blended cement matrices containing activated bamboo leaf wastes. Cement and Concrete Composites. 2012;34(9):1019-1023. https://doi.org/10.1016/j.cemconcomp.2012.05.005
3. Moraes MJB, Moraes JCB, Tashima MM, Akasaki JL, Soriano L, Borrachero MV, Payá J. Production of bamboo leaf ash by auto-combustion for pozzolanic and sustainable use in cementitious matrices. Construction and Building Materials. 2019;208:369-380. https://doi.org/10.1016/j.conbuildmat.2019.03.007
4. Rangaraj S, Venkatachalam R. A lucrative chemical processing of bamboo leaf biomass to synthesize biocompatible amorphous silica nanoparticles of biomedical importance. Applied Nanoscience. 2017;7:145-153. https://doi.org/10.1007/s13204-017-0557-z
5. Fatimah I, Prakoso NI, Sahroni I, Musawwa MM, Sim YL, Kooli F, Muraza O. Physicochemical characteristics and photocatalytic performance of TiO2/SiO2 catalyst synthesized using biogenic silica from bamboo leaves. Heliyon. 2019;5(11):e02766. https://doi.org/10.1016/j.heliyon.2019.e02766
6. Vinai R, Ntimugura F, Cutbill W, Evans R, Zhao Y. Bio-derived sodium silicate for the manufacture of alkali-activated binders: use of bamboo leaf ash as silicate source. International Journal of Applied Ceramic Technology. 2022;19(3):1235-1248. https://doi.org/10.1111/ijac.14004
7. Bindar Y, Ramli Y, Steven S, Restiawaty E. Optimization of purity and yield of amorphous bio-silica nanoparticles synthesized from bamboo leaves. The Canadian Journal of Chemical Engineering. 2024;102(4):1419-1430. https://doi.org/10.1002/cjce.25148
8. Kalapathy U, Proctor A, Shultz J. A simple method for production of pure silica from rice hull ash. Bioresource Technology. 2000;73(3):257-262. https://doi.org/10.1016/S0960-8524(99)00127-3
9. Kalapathy U, Proctor A, Shultz J. An improved method for production of silica from rice hull ash. Bioresource Technology. 2002;85(3):285-289. https://doi.org/10.1016/S0960-8524(02)00116-5
10. September LA, Kheswa N, Seroka NS, Khotseng L. Green synthesis of silica and silicon from agricultural residue sugarcane bagasse ash - a mini review. RSC Advances. 2023;13:1370-1380. https://doi.org/10.1039/D2RA07490G
11. Hamidu I, Afotey B, Kwakye-Awuah B, Anang DA. Synthesis of silica and silicon from rice husk feedstock: a review. Heliyon. 2025;11(4):e42491. https://doi.org/10.1016/j.heliyon.2025.e42491
12. Huang C, Zhan Y, Cheng J, Wang J, Meng X, Fang G, Ragauskas AJ. The bamboo delignification saturation point in alkaline hydrogen peroxide pretreatment and its association with enzymatic hydrolysis. Bioresource Technology. 2022;359:127462. https://doi.org/10.1016/j.biortech.2022.127462
13. Zhan Y, Cheng J, Liu X, Huang C, Wang J, Han S, Fang G, Meng X, Ragauskas AJ. Assessing the availability of two bamboo species for fermentable sugars by alkaline hydrogen peroxide pretreatment. Bioresource Technology. 2022;349:126854. https://doi.org/10.1016/j.biortech.2022.126854
14. Chruściel JJ. Modifications of textile materials with functional silanes, liquid silicone softeners, and silicone rubbers - a review. Polymers. 2022;14(20):4382. https://doi.org/10.3390/polym14204382
15. Mun JS, Mun SP. Alkaline hydrogen peroxide delignification of three lignocellulosic biomass under atmospheric pressure. BioResources. 2024;19(1):998-1009. https://doi.org/10.15376/biores.19.1.998-1009.