

## Valorization of Sugarcane Bagasse and *Carica papaya* Seed Wastes into a Silica-Based Nanopesticide against *Callosobruchus maculatus*

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### Abstract

Agricultural waste valorization offers opportunities for developing sustainable materials for stored-product pest management. This study investigated the conversion of sugarcane bagasse and *Carica papaya* (*C. papaya*) seed wastes into a silica-based nanopesticide against *Callosobruchus maculatus*. Silica nanoparticles (SiNPs) were prepared from sugarcane bagasse ash by acid pretreatment, alkaline extraction and sol-gel processing, while *C. papaya* seed oil was obtained by Soxhlet extraction with n-hexane. The oil was loaded onto SiNPs to produce a silica-*C. papaya* seed oil nanocomposite (SiNC). GC-MS identified fatty acids including palmitic, palmitoleic, linoleic, oleic and stearic acids, together with squalene. FTIR, XRD, SEM-EDX and TGA analyses provided complementary evidence of oil incorporation and modification of the silica matrix while retaining its predominantly amorphous structure. Pesticidal activity increased with exposure time, with SiNC producing 55.17, 93.33 and 96.67% mortality after 24, 48 and 72 h, respectively, compared with 46.67–63.33% for *C. papaya* and 13.33–23.33% for SiNPs. The LC<sub>50</sub> of SiNC decreased from 27.20 to 8.83 mg/mL over 24–72 h. These findings demonstrate the potential of dual agricultural-waste valorization for developing effective silica-based botanical nanopesticides for sustainable stored-cowpea protection.

**Keywords:** *Callosobruchus maculatus*; silica nanoparticles; *Carica papaya* seed oil; nanopesticide; agricultural waste valorization

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### 1. Introduction

Postharvest insect infestation remains an important constraint to food security, particularly in tropical and subtropical regions where grain legumes constitute major sources of dietary protein. The cowpea weevil, *Callosobruchus maculatus* (F.) (*C. maculatus*), is one of the most destructive pests of stored cowpea and related pulses, causing deterioration in seed quality, weight, nutritional value and marketability during storage. Although synthetic insecticides and fumigants are widely used for its management, their repeated application raises concerns associated with pesticide residues, environmental contamination, toxicity to non-target organisms and the development of insecticide resistance. These limitations have intensified the search for effective pest-control materials derived from renewable resources and possessing reduced environmental footprints (Ibrahim et al., 2024; Zainab et al., 2024).

Nanotechnology provides an increasingly promising platform for developing more efficient pest-management systems. Nanopesticides can enhance the stability, dispersion, retention and controlled delivery of active pesticidal ingredients, potentially reducing the quantities required for effective control (Izuagie, et al., 2025). Among the nanomaterials investigated for this purpose, silica nanoparticles (SiNPs) are particularly attractive because of their high surface area, tunable porosity, physicochemical stability and capacity for surface modification. SiNPs can function either as active insecticidal materials or as carriers for other pesticidal compounds, thereby offering opportunities for combining different modes of pest control (Falk et al., 2019; Saw et al., 2023; Selvarajan et al., 2020). Recent comparative assessments further indicate that silica-based pesticide formulations can improve bioavailability, photostability and controlled-release characteristics relative to corresponding non-nano formulations, although environmental fate, field performance and regulatory issues still require careful evaluation (Zeng & Motola, 2025).

An additional sustainability advantage can be achieved when the silica precursor itself is obtained from agricultural residues. Sugarcane processing generates substantial quantities of bagasse, which may create disposal problems when it is inadequately managed. Following thermal treatment, sugarcane bagasse ash contains appreciable quantities of silica that can be recovered and converted into value-added silica materials. Recent studies have therefore highlighted sugarcane bagasse as a renewable and comparatively inexpensive feedstock for silica production, including nanosilica prepared through acid–base extraction, precipitation and sol–gel approaches. Valorization of this biomass residue simultaneously reduces waste accumulation and decreases dependence on conventional mineral silica sources, thereby strengthening the circular-economy credentials of nanomaterial production (September et al., 2023).

*C. papaya* seeds represent another underutilized agricultural by-product with considerable potential for value addition. *C. papaya* seeds contain substantial quantities of extractable oil together with fatty acids and other biologically active constituents. Recent chemical characterization has shown *C. papaya* seed oil to contain predominantly oleic acid together with palmitic, stearic and linoleic acids, as well as sterols, triterpenic alcohols and benzyl isothiocyanate (Goriainov et al., 2023). Importantly, the pesticidal potential of *C. papaya* seed-derived products is increasingly being demonstrated. Recent field investigations showed that *C. papaya* seed extracts reduced insect infestation and crop damage, supporting their potential as botanical components of more sustainable pest-management strategies (Siaw et al., 2025).

Combining a plant-derived pesticidal material with a silica nanostructure therefore offers an attractive strategy for integrating the intrinsic biological activity of botanical constituents with the high surface area, adsorption capacity and insecticidal/carrier properties of silica. Waste-derived silica itself has recently demonstrated pronounced toxicity against *C. maculatus*: Ibrahim et al. (2024), for example, reported concentration-dependent mortality of the pest following exposure to silica nanoparticles prepared from rice straw, confirming the feasibility of converting silica-rich agricultural residues into materials for stored-grain protection (Ibrahim et al., 2024). However, considerably less attention has been directed toward integrating sugarcane bagasse-derived silica nanoparticles with *C. papaya* seed oil as a composite pesticidal system.

Accordingly, the present study investigates the valorization of sugarcane bagasse and *C. papaya* seed wastes for the preparation of a silica-based nanopesticide against *C. maculatus*. Silica nanoparticles were prepared from sugarcane bagasse, while oil extracted from *C. papaya* seeds was incorporated into the silica material to obtain a silica nanocomposite. The study was designed to characterize the constituent materials and evaluate their pesticidal activity against the cowpea weevil, thereby exploring a dual-waste valorization strategy capable of contributing simultaneously to agricultural-waste management and sustainable stored-product pest control.

## 2. Materials and Methods

### 2.1 Materials and Reagents

Sugarcane bagasse was used as the precursor for the preparation of silica nanoparticles, whereas *C. papaya* seeds served as the source of the pesticidal oil. The chemicals reported for the study included n-hexane (95%, Fisher Scientific), acetone (99%, Fisher Scientific), sodium nitrate (99%, Fisher Scientific), hydrochloric acid (37%, Merck), sodium hydroxide, ethanol and distilled water. All reagents were of analytical grade and were used without further purification. Soxhlet extraction apparatus and other standard laboratory glassware were employed for oil extraction and sample preparation, while a Mettler Toledo analytical balance and Whatman filter papers were used for weighing and filtration, respectively.

### 2.2 Sample Collection, Pretreatment and Preparation

Sugarcane bagasse was obtained from a local vendor at Kasuwan Daji, Sokoto, Nigeria, and transported to the laboratory. The bagasse was thoroughly washed to remove adhering soil and other impurities, dried and converted to sugarcane bagasse ash by calcining at 650 °C for 2 h. The resulting ash was subsequently subjected to acid pretreatment followed by alkaline extraction and sol–gel processing to recover silica as described in section 2.4. This general approach is consistent with established methods for obtaining silica from sugarcane bagasse ash, in which acid pretreatment removes metallic impurities and alkaline extraction converts the silica fraction to soluble silicate prior to gel formation (September et al., 2023).

*C. papaya* seeds were also collected from Kasuwan Daji, Sokoto, Sokoto State, Nigeria. The seeds were washed, sun-dried for one week to reduce their moisture content and pulverized using a mortar and pestle. The resulting powder was stored in airtight containers to minimize moisture absorption and contamination prior to extraction. The use of dried, pulverized *C. papaya* seeds and n-hexane extraction is consistent with recent studies on the recovery and chemical characterization of *C. papaya* seed oil (Goriainov et al., 2023).

### 2.3 Extraction and GC–MS Analysis of *C. papaya* Seed Oil

Oil was extracted from the pulverized *C. papaya* seeds by continuous Soxhlet extraction using n-hexane as the extracting solvent. Following extraction, the oil fraction was recovered and filtered before being subjected to gas chromatography–mass spectrometry (GC–MS) for determination of its chemical constituents. Soxhlet extraction with n-hexane is an established approach for recovering *C. papaya* seed oil and has been employed in recent compositional investigations of *C. papaya* seed lipids (Goriainov et al., 2023).

### 2.4 Preparation of Silica Nanoparticles from Sugarcane Bagasse

Silica nanoparticles (SiNPs) were prepared from sugarcane bagasse ash using a sol–gel method. Briefly, 1 g of the ash was acid-pretreated by leaching with 10 mL of 1 M HCl under continuous stirring at 100 °C for 1 h to remove metallic impurities. The mixture was filtered, and the resulting residue was washed several times with distilled water. The washed residue was subsequently treated with 8 mL of NaOH solution and heated at 100 °C under continuous stirring for 1 h to extract the silica as soluble sodium silicate. The mixture was then filtered, and the sodium silicate-containing filtrate was titrated with 1 M HCl to induce gel formation. The resulting aquagel was allowed to age for 24 h and subsequently washed several times with distilled water to remove residual soluble species. It was then dried in an oven for 4 h to obtain a silica xerogel. The dried xerogel was weighed, ground to a fine powder using an agate mortar and sieved through a 200-mesh sieve to obtain relatively homogeneous silica nanoparticles. This preparation route follows the established sol–gel conversion of silica-rich biomass ash, in which alkaline extraction produces soluble sodium silicate followed by acid-induced gel formation and subsequent drying to obtain silica (Ibrahim et al., 2024; Izuagie, Salihu et al., 2025; September et al., 2023).

## 2.5 Preparation of Silica Nanoparticle–*C. papaya* Seed Oil Nanocomposite

The silica nanoparticle–*C. papaya* seed oil nanocomposite (SiNC) was prepared by loading the extracted *C. papaya* seed oil onto the synthesized silica nanoparticles (SiNPs) using a simple immersion/adsorption method. Briefly, 50 mg of SiNPs was suspended in 40 mL of acetone and thoroughly dispersed by sonication. Subsequently, 10 mg of the extracted *C. papaya* seed oil was added to the suspension, corresponding to an SiNP-to-oil mass ratio of 5:1. The mixture was stirred continuously at room temperature for 24 h to promote adsorption of the oil onto the nanoparticle surface. To enhance oil loading, the volume of the suspension was reduced to approximately 0.2 mL, after which the concentrated suspension was degassed by sonication for 10 min. The mixture was then stirred for an additional 6 h to facilitate uniform distribution and saturation of the oil within the silica matrix. Acetone was subsequently allowed to evaporate completely at room temperature and atmospheric pressure. The resulting oil-loaded material was washed four times with ethanol to remove unbound or loosely adsorbed oil from the surface of the nanoparticles. The recovered SiNC was dried in an oven at 30 °C, ground to a fine powder and stored in a sealed container until further characterization and pesticidal evaluation. Similar immersion and adsorption approaches have been employed for incorporating essential oils and other plant-derived pesticidal compounds into silica-based carriers (Attia et al., 2023; Ding et al., 2024).

## 2.6 Instrumental Characterization

GC–MS was employed to separate and identify the chemical constituents of the extracted oil by comparison of the recorded mass spectra with an appropriate reference mass-spectral database. The synthesized SiNPs and SiNC were characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM) and Thermogravimetric analysis (TGA). FTIR spectroscopy was used to identify characteristic functional groups and evaluate changes associated with incorporation of the *C. papaya* seed oil into the silica matrix. XRD analysis was employed to assess the structural and phase characteristics of the prepared SiNPs and SiNC, whereas SEM was used to examine their surface morphology and particle aggregation. GC-MS analysis was performed on an Agilent 6890N GC coupled to Agilent 5973N mass selective detector using DB 5MS (30 cm length, 0.250 mm ID) silica column and helium as a carrier gas at a flowrate of 1 mLmin<sup>-1</sup>. One microliter of the sample was injected into the column at an initial temperature set at 40 °C for 2 min before gradually increasing it to 200 °C at a rate of 5 °Cmin<sup>-1</sup>. It was then kept at this temperature for another 2 min before raising it to 300 °C at a rate of 5 °Cmin<sup>-1</sup> and held at this temperature for another 2 min. Data were acquired by GCMSD/Enhanced MassHunter software and Identification of compounds was by comparing results with those stored in Wiley Registry/NIST Mass Spectral Library 2023. Fourier transform infrared spectroscopy (FTIR) analyses were conducted on a Bruker Alpha spectrometer fitted with a Platinum ATR module (4000 – 500 cm<sup>-1</sup>). Spectra were recorded for solid samples after drying for at least 1 h. Scanning electron microscopy (SEM, Phonem Prox, Malaysia) coupled with Energy Dispersive X-ray (EDX), was operated at an accelerated voltage of 10 kV. XRD analysis was conducted using XRD, Philips X' Pert Pro with Cu K $\alpha$  radiation of wavelength of 0.15416 nm at the range of 5 – 80°. Thermogravimetric Analysis (TGA) was run on a Q600 SDT (TA Instruments). About 6 mg of sample was heated at a rate of 10 °Cmin<sup>-1</sup> from 50 to 800 °C in a 40  $\mu$ L platinum crucible under an atmosphere of high-purity continuous nitrogen gas (120 mL min<sup>-1</sup> flow rate).

## 2.7 Insecticidal Studies

### 2.7.1 *C. maculatus* Acquisition and Rearing

Adult *C. maculatus* were obtained from Sokoto, Nigeria, and transported to the Entomology Laboratory, Usmanu Danfodiyo University, Sokoto, for identification and laboratory culture. Adults were maintained on whole black-eyed beans in plastic containers (10 × 12 cm) covered with muslin cloth to provide ventilation and prevent escape.

Laboratory cultures were maintained at approximately 27.5 °C, 60 ± 5% relative humidity and a 12:12 h light–dark photoperiod. Comparable controlled-temperature rearing of *C. maculatus* on cowpea or black-eyed beans is widely used for laboratory insecticidal assays, including recent evaluations of agricultural waste-derived silica nanoparticles against this stored-product pest (Ibrahim et al., 2024).

### 2.7.2 Residual Toxicity

Residual/contact toxicity of the test materials against adult *C. maculatus* was evaluated using four treatment concentrations: 1.0, 1.5, 2.0, and 3.0% (m/v), with three replicates per treatment. Twenty grams of sterilized cowpea seeds were treated separately with the respective formulations and allowed to air-dry for 30 min before transfer into 12-cm-diameter Petri dishes. Five pairs of newly emerged adult *C. maculatus* were subsequently introduced into each experimental unit, while untreated cowpea seeds served as the negative control. The dishes were covered with muslin cloth, and mortality was assessed after 24, 48, and 72 h of exposure. Dead insects were removed and counted at each assessment period. Silica-based contact bioassays involving exposure of adult *C. maculatus* to treated cowpea seeds have similarly been used to establish concentration-dependent mortality and lethal-concentration values (Ibrahim et al., 2024).

Percentage mortality was calculated as:

$$\text{Mortality (\%)} = \frac{\text{Number of dead insects}}{\text{Total number of insects exposed}} \times 100$$

Where mortality occurred in the untreated control, corrected mortality was obtained using Abbott's correction:

$$\text{Corrected mortality (\%)} = \frac{M_t - M_c}{100 - M_c} \times 100$$

where  $M_t$  is the percentage mortality in the treatment and  $M_c$  is the percentage mortality in the untreated control.

## 2.8 Statistical Analysis

Experimental mortality data were evaluated for normality using the Shapiro–Wilk test and for homogeneity of variance using Levene's test. Differences among treatments were assessed by analysis of variance (ANOVA), and treatment means were separated using Tukey's honestly significant difference (HSD) test at a significance level of  $p < 0.05$ . Concentration–mortality relationships were analysed using log–probit regression to estimate the median lethal concentration ( $LC_{50}$ ) and 90% lethal concentration ( $LC_{90}$ ). Probit analysis remains a standard approach for analysing quantal concentration–mortality data and has recently been applied in studies of silica nanoparticle toxicity against *C. maculatus* (Ibrahim et al., 2024). Mortality in treated groups was corrected for mortality occurring in the untreated control using Abbott's method. Statistical analyses were performed using Microsoft Excel and SPSS version 20.

## 3. Results and Discussion

### 3.1 Product Yields

The percentage yields and physical appearance of the extracted *C. papaya* seed oil, silica nanoparticles (SiNPs) and silica nanoparticle–*C. papaya* seed oil nanocomposite (SiNC) are presented in **Table 1**. The extracted oil was brown and gave a yield of 29.3%, whereas the SiNPs were obtained as a white powder with a yield of 39.7%. Following incorporation of the seed oil into the silica material, a white SiNC product was recovered with a yield of 96.8%.

The relatively moderate yield of *C. papaya* seed oil demonstrates that the seeds constitute a potentially valuable source of extractable lipid. The value obtained in the present study is higher than the 18.3–27.0% range reported by Goriainov et al. (2023) for *C. papaya* seeds obtained from different geographical locations. Such differences may arise from variations in cultivar, seed maturity, geographical origin, moisture content and extraction conditions (Goriainov et al., 2023). Goriainov et al. similarly employed n-hexane/Soxhlet extraction and demonstrated that *C. papaya* seed oil is particularly rich in monounsaturated fatty acids. The 39.7% recovery of SiNPs also confirms the feasibility of converting sugarcane bagasse ash into a value-added silica material. Sugarcane bagasse has increasingly been recognized as an alternative silica precursor, with sol–gel extraction offering a relatively simple means of converting this agricultural residue into silica nanoparticles (Ni'mah et al., 2023). The high recovery of the SiNC following loading suggests minimal material loss during the immersion, solvent-removal and washing stages.

**Table 1.** Percentage yield and colour of prepared samples

| Sample               | % Yield | Colour |
|----------------------|---------|--------|
| Extracted Oil        | 29.3%   | Brown  |
| Silica NPs           | 39.7%   | White  |
| Silica Nanocomposite | 96.8%   | White  |

### 3.2 GC–MS Analysis of *C. papaya* Seed Oil

GC–MS analysis revealed a range of fatty acids and a triterpene hydrocarbon in the extracted *C. papaya* seed oil (Table 2). The compounds identified included tetradecanoic (myristic) acid, pentadecanoic acid, hexadecanoic (palmitic) acid, hexadecenoic (palmitoleic) acid, heptadecanoic (margaric) acid, octadecadienoic (linoleic) acid, 9-octadecenoic (oleic) acid, docosanoic (behenic) acid, octadecanoic (stearic) acid and squalene. The occurrence of oleic, palmitic, stearic and linoleic acids agrees strongly with the established fatty-acid profile of *C. papaya* seed oil. Goriainov et al. (2023), in a comparative analysis of *C. papaya* seed oils from different geographical origins, reported oleic acid as the predominant fatty acid at 68.7–74.9%, followed by palmitic acid (14.2–16.9%), stearic acid (4.8–5.9%) and linoleic acid (3.4–6.8%) (Goriainov et al., 2023). The presence of both saturated and unsaturated fatty acids in the current extract therefore supports its assignment as *C. papaya* seed oil. The detection of squalene additionally indicates the presence of an unsaponifiable hydrocarbon fraction, consistent with the chemically diverse minor constituents reported in *C. papaya* seed oils.

**Table 2.** GC-MS profile of *C. papaya* seed oil

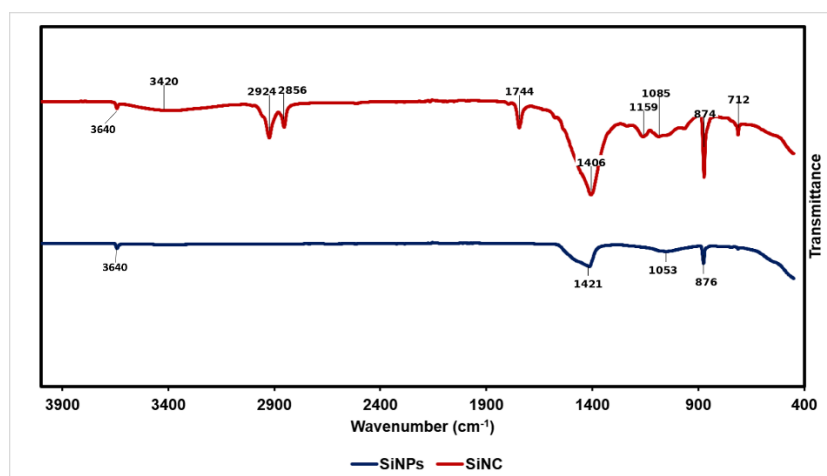
| Retention time (min) | Identified compound         | Common name / description | Molecular formula                              |
|----------------------|-----------------------------|---------------------------|------------------------------------------------|
| 13.962               | Tetradecanoic acid          | Myristic acid             | C <sub>14</sub> H <sub>28</sub> O <sub>2</sub> |
| 14.872               | Pentadecanoic acid          | Pentadecylic acid         | C <sub>15</sub> H <sub>30</sub> O <sub>2</sub> |
| 16.171               | <i>n</i> -Hexadecanoic acid | Palmitic acid             | C <sub>16</sub> H <sub>32</sub> O <sub>2</sub> |
| 16.571               | Hexadecenoic acid           | Palmitoleic acid          | C <sub>16</sub> H <sub>30</sub> O <sub>2</sub> |
| 17.058               | Heptadecanoic acid          | Margaric acid             | C <sub>17</sub> H <sub>34</sub> O <sub>2</sub> |
| 17.391               | Octadecadienoic acid        | Linoleic acid             | C <sub>18</sub> H <sub>32</sub> O <sub>2</sub> |
| 18.723               | Docosanoic acid             | Behenic acid              | C <sub>22</sub> H <sub>44</sub> O <sub>2</sub> |
| 18.980               | 9-Octadecenoic acid         | Oleic acid                | C <sub>18</sub> H <sub>34</sub> O <sub>2</sub> |
| 19.169               | Octadecanoic acid           | Stearic acid              | C <sub>18</sub> H <sub>36</sub> O <sub>2</sub> |
| 26.957               | Squalene                    | Triterpene hydrocarbon    | C <sub>30</sub> H <sub>50</sub>                |

### 3.3 FTIR Analysis

The FTIR spectra of the SiNPs and SiNC are presented in **Figure 1**. The SiNP spectrum displays the characteristic absorption region associated with silica, while the spectrum of the SiNC retains the silica-associated bands but exhibits additional and more prominent absorption features attributable to the incorporated organic component. In particular, the SiNC spectrum shows additional absorption in the aliphatic C–H stretching region around 3000–2800  $\text{cm}^{-1}$  and increased absorption in regions associated with the oxygen-containing functional groups of the seed oil.

Biomass-derived silica typically exhibits a broad O–H stretching band in the vicinity of 3400  $\text{cm}^{-1}$ , together with characteristic Si–O–Si vibrations in the approximately 1100, 800 and 450  $\text{cm}^{-1}$  regions. Ni'mah et al. (2023), for example, reported bands at 3421 and 1636  $\text{cm}^{-1}$  for surface hydroxyl/water species and at approximately 1050, 793 and 436  $\text{cm}^{-1}$  for Si–O–Si vibrations in silica prepared from sugarcane bagasse (Ni'mah et al., 2023).

The emergence of organic-associated bands in the SiNC spectrum provides evidence that *C. papaya* seed oil was retained by the silica after the washing and drying stages. Similar behaviour was observed by Attia et al. (2023), who found that silica nanoparticles loaded with cinnamon oil retained the principal silica bands while simultaneously displaying spectral features associated with the incorporated oil (Attia et al., 2023). Likewise, silica loaded with natural oils has been reported to display both Si–O–Si/Si–OH bands and C–H bands associated with the organic guest molecule (Lai et al., 2023). Thus, the FTIR results support formation of the SiNP–*C. papaya* oil composite rather than complete chemical replacement of the original silica framework.



**Figure 1.** FTIR spectra of silica nanoparticles (SiNPs) and silica–*C. papaya* seed oil nanocomposite (SiNC).

### 3.4 X-ray Diffraction Analysis

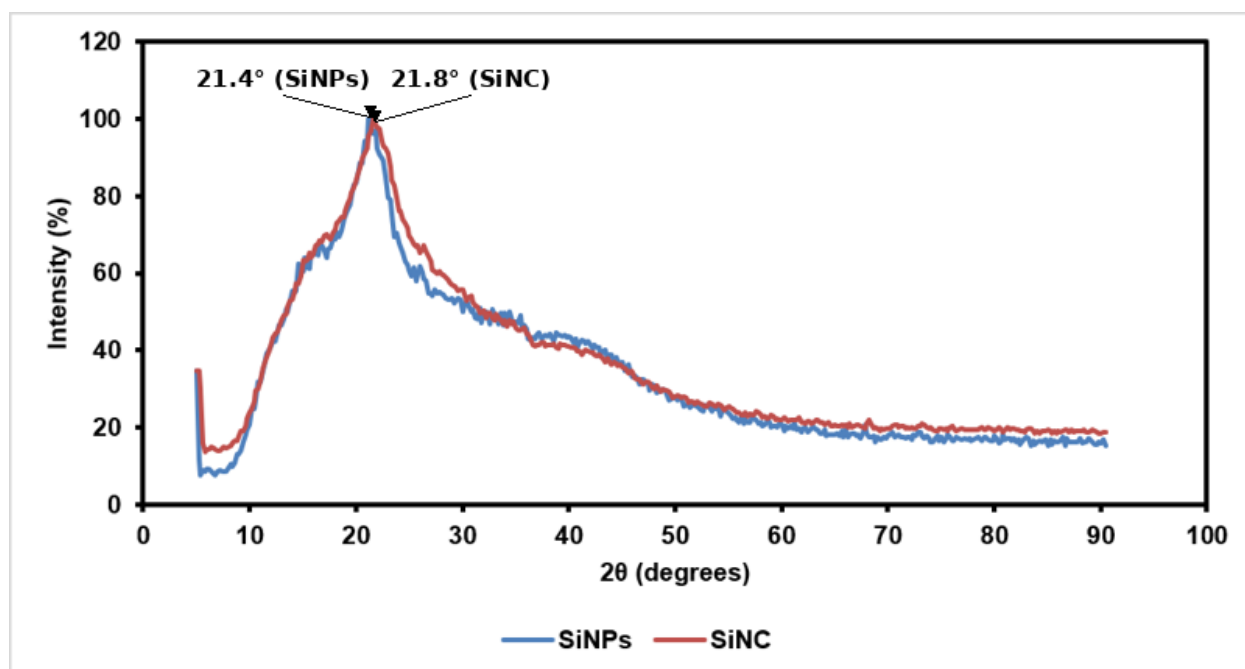
The XRD patterns of the silica nanoparticles (SiNPs) and silica nanoparticle–*C. papaya* seed oil nanocomposite (SiNC) are presented in **Figure 2**. Both samples exhibit a broad diffraction halo centred at approximately  $2\theta = 21\text{--}22^\circ$ , with no prominent sharp reflections over the investigated range. This feature is characteristic of predominantly amorphous silica and indicates the absence of substantial long-range crystalline ordering in the prepared materials. The broad nature of the diffraction maximum agrees with previous reports for silica obtained from sugarcane bagasse and other silica-rich agricultural residues by sol–gel processing. For example, Ni'mah et al. (2023) reported a broad diffraction feature at approximately  $2\theta = 22.6^\circ$  for silica nanoparticles derived from sugarcane bagasse.

Comparison of the two patterns shows that incorporation of *C. papaya* seed oil produced only modest changes in the diffraction profile. The principal broad maximum of the SiNPs occurs at approximately  $2\theta = 21.4^\circ$ , whereas

that of the SiNC is slightly shifted to about  $21.8^\circ$ . The SiNC pattern also appears marginally broader around the principal diffraction region and shows small differences in relative intensity across the higher-angle region. These changes are consistent with interaction of the organic components of the seed oil with the silica surface and/or pores, resulting in slight modification of the local structural environment of the silica without inducing a new crystalline phase.

Importantly, the absence of additional well-defined crystalline reflections in the SiNC pattern suggests that the incorporated seed oil remained predominantly in an amorphous or molecularly dispersed state within or on the silica matrix. The retention of the characteristic broad silica halo after oil loading further indicates that formation of the nanocomposite did not substantially alter the underlying amorphous  $\text{SiO}_2$  framework. Similar preservation of the silica diffraction profile following incorporation of botanical oils or other organic active compounds has been reported for silica-based delivery systems and is generally attributed to retention of the inorganic carrier structure during loading (Attia et al., 2023).

Generally, the XRD results support successful modification of the SiNPs with *C. papaya* seed oil while demonstrating that the nanocomposite remained predominantly amorphous.



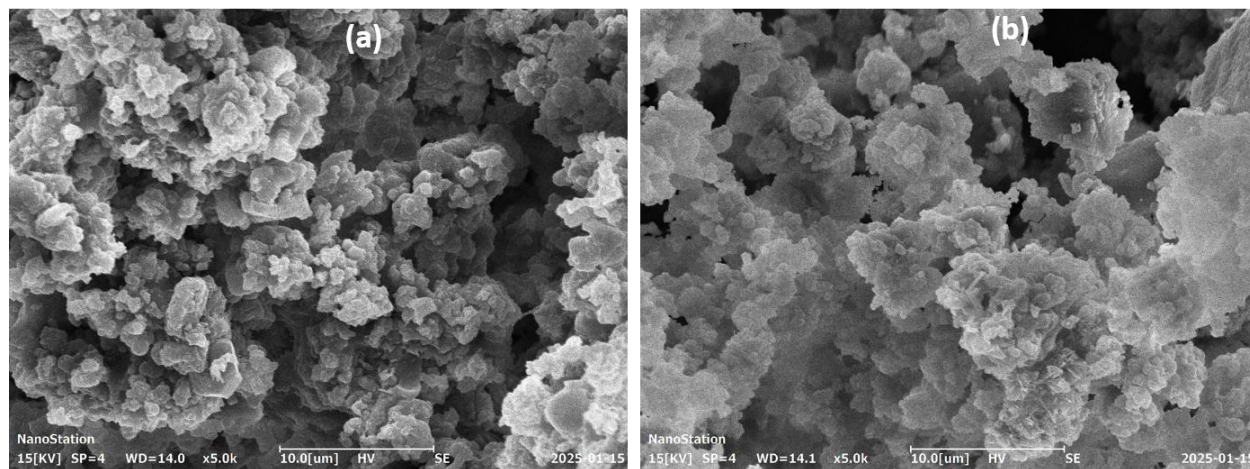
**Figure 2.** XRD patterns of silica nanoparticles (SiNPs) and silica-*C. papaya* seed oil nanocomposite (SiNC).

### 3.5 SEM Morphology

The SEM micrographs reveal strongly agglomerated particles in both the SiNPs and SiNC (**Figure 3**). The SiNPs appear as irregular clusters composed of smaller interconnected particles, producing a rough and heterogeneous surface. Following incorporation of the *C. papaya* seed oil, the SiNC retained the clustered morphology but appeared more compact, with the smaller silica domains embedded within or associated with a more continuous matrix.

Agglomeration is frequently observed in silica nanoparticles because surface silanol groups promote interparticle hydrogen bonding, particularly during drying. Recent characterization of sugarcane bagasse-derived silica has similarly revealed aggregated and porous particle networks (September et al., 2023). Changes in surface morphology after incorporation of an organic oil are also consistent with previous silica-supported botanical formulations. Attia et al. (2023) observed distinct surface changes when cinnamon oil was incorporated into

mesoporous silica, and complementary characterization confirmed occupation of the silica structure by the oil (Attia et al., 2023). The present SEM images therefore provide morphological evidence of modification of the silica following oil incorporation.



**Figure 3.** SEM micrographs of (a) silica nanoparticles (SiNPs) and (b) silica-*C. papaya* seed oil nanocomposite (SiNC).

### 3.6 EDX Analysis

The EDX results (**Figure 4** and **Table 3**) further demonstrate a marked compositional change following incorporation of *C. papaya* seed oil. The SiNP sample contained oxygen and silicon at atomic proportions of 82.10 and 17.90%, respectively, with no detectable carbon. In contrast, the SiNC contained 30.14% carbon, 67.94% oxygen and 1.92% silicon. The predominance of oxygen and silicon in the unloaded material is consistent with formation of an oxygen-rich silica phase. Similar EDX analyses of silica nanoparticles commonly identify Si and O as the dominant inorganic elements; Attia et al. (2023), for example, used the predominance of these elements as supporting evidence for SiO<sub>2</sub> formation (Attia et al., 2023). The appearance of a substantial carbon signal after loading is particularly significant because *C. papaya* seed oil is composed predominantly of carbon-rich fatty acids and other organic constituents. Thus, the transition from undetectable carbon in the SiNPs to 30.14 atomic% in the SiNC provides independent evidence for association of the oil with the silica phase.

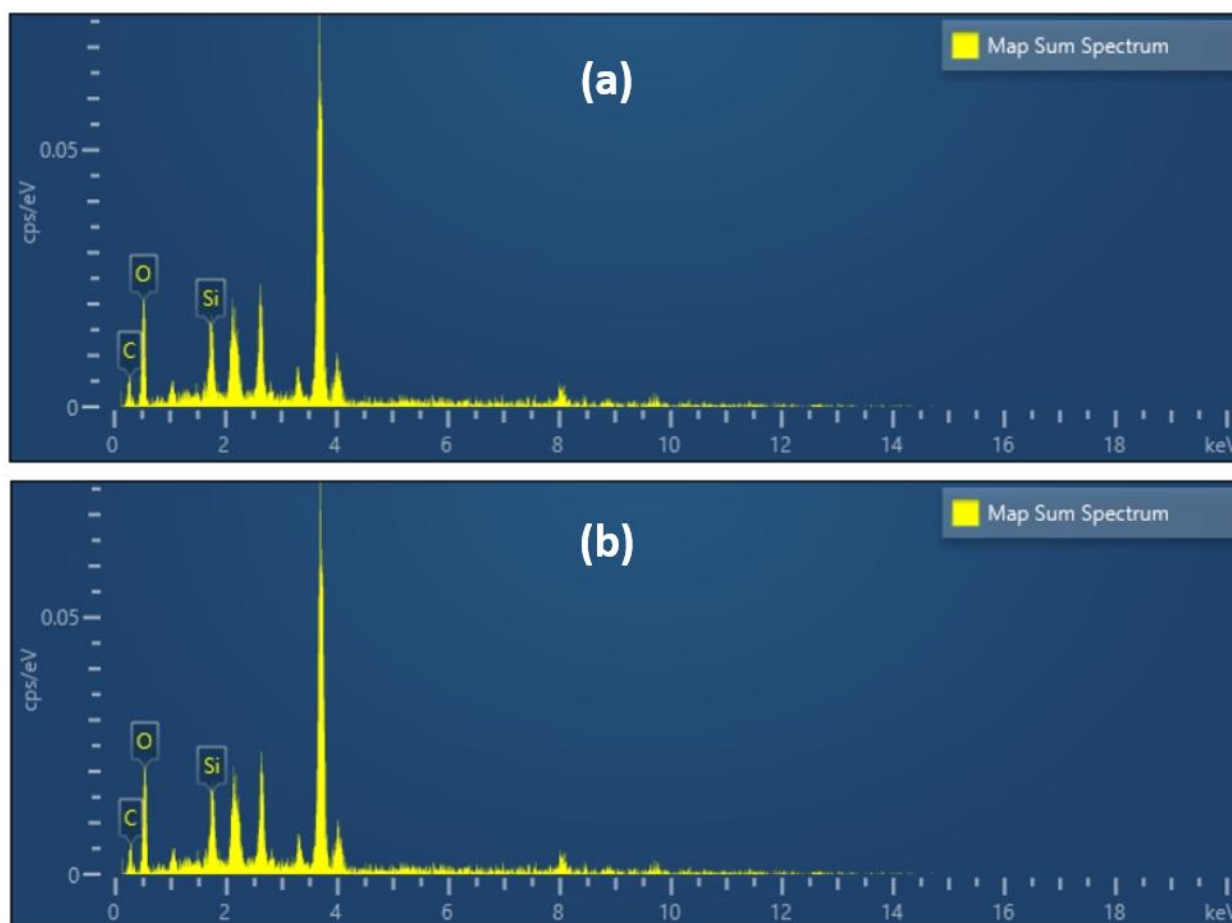
The corresponding reduction in the relative surface contribution of Si from 17.90 to 1.92 atomic% may also indicate coverage of silica domains by the organic component. Because EDX is a localized and semi-quantitative technique and the measured composition depends on the analysed surface region, these percentages should not be interpreted directly as bulk oil-loading percentages. Nevertheless, when considered together with FTIR, SEM and TGA, the EDX data strongly support successful formation of the SiNC.

**Table 3.** EDX-derived atomic percentages of C, O, and Si in silica nanoparticles (SiNPs) and the silica nanoparticle-*C. papaya* seed oil nanocomposite (SiNC).

| Element | Atomic % |        |
|---------|----------|--------|
|         | SiNPs    | SiNC   |
| C       | 0.00     | 30.14  |
| O       | 82.10    | 67.94  |
| Si      | 17.90    | 1.92   |
| Total   | 100.00   | 100.00 |

### 3.7 Thermogravimetric Analysis and Percentage Loading of Oil

The thermogravimetric profiles of the SiNPs and SiNC are shown in **Figure 5**. The SiNP material exhibited an overall mass loss of approximately 62.80%, whereas the SiNC showed a greater total mass loss of approximately 81.40%. The higher thermal mass loss of the SiNC is consistent with the presence of an additional organic fraction arising from the incorporated *C. papaya* seed oil.

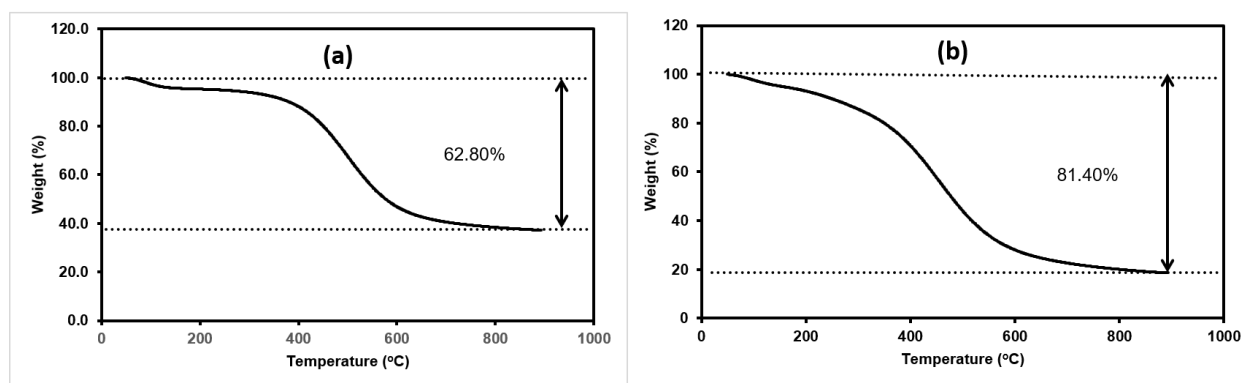


**Figure 4.** EDX spectra of (a) silica nanoparticles (SiNPs) and (b) silica-*C. papaya* seed oil nanocomposite (SiNC).

Thermogravimetric analysis is frequently used to confirm and estimate organic loading in silica-based delivery materials because the inorganic silica framework is comparatively thermally stable whereas the incorporated organic fraction volatilizes or decomposes during heating. Almaghrabi et al. (2023) demonstrated that TGA provides a reliable means of assessing guest loading in mesoporous silica materials (Almaghrabi et al., 2023). Similarly, Chakroun et al. (2023) reported substantially greater mass loss for essential-oil-loaded silica than for unloaded silica and attributed the difference principally to decomposition and volatilization of the entrapped oil. The increased mass loss recorded for the present SiNC therefore provides additional evidence of *C. papaya* seed oil incorporation and complements the carbon signal detected by EDX and the organic bands observed by FTIR.

### 3.8 Pesticidal Activity against *C. maculatus*

The pesticidal activities of SiNPs, *C. papaya* seed oil and the SiNC against adult *C. maculatus* showed clear differences among the three treatments and with increasing exposure time (**Figure 6** and **Table 4**). After 24 h, the maximum mortalities were 13.33% for SiNPs, 46.67% for *C. papaya* and 55.17% for SiNC. Mortality subsequently increased, reaching 17.24, 53.33 and 93.33%, respectively, after 48 h, and 23.33, 63.33 and 96.67%, respectively, after 72 h. Thus, SiNC consistently produced the greatest mortality, while the unloaded SiNPs displayed the lowest activity under the experimental conditions.



**Figure 5.** TGA profiles of (a) silica nanoparticles (SiNPs) and (b) silica-*C. papaya* seed oil nanocomposite (SiNC) illustrating the progressive thermal mass loss of the material with increasing temperature.

The progressive increase in mortality with exposure time is consistent with the time-dependent nature of stored-product insecticide activity. Ibrahim et al. (2024) demonstrated concentration-dependent toxicity of agricultural waste-derived silica nanoparticles against *C. maculatus* and reported increasing mortality with concentration and exposure. Their microscopy observations indicated strong adhesion of silica nanoparticles to the insect surface, together with cuticular abrasion and particle accumulation around body joints (Ibrahim et al., 2024). Silica particles may contribute to mortality through disruption of the protective cuticular surface, adsorption of cuticular lipids and increased water loss (Ibrahim et al., 2024).

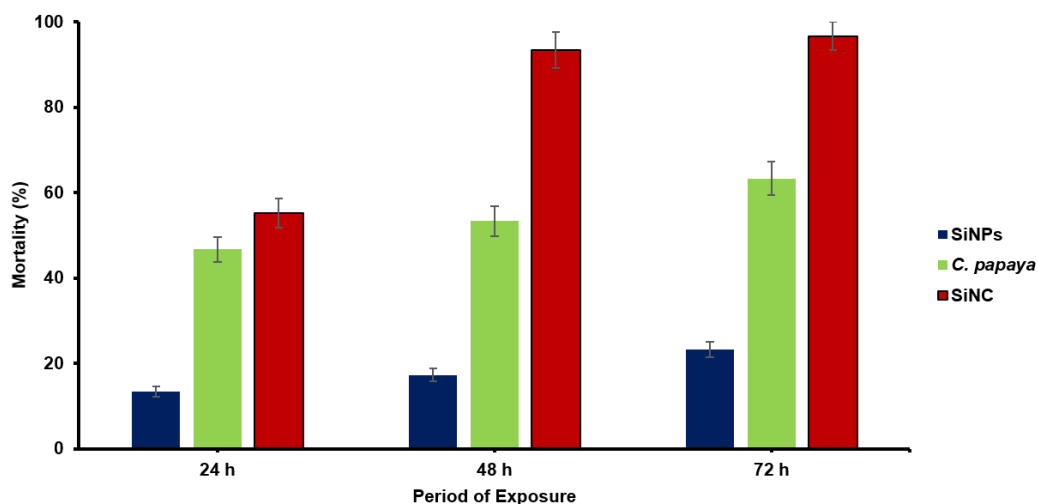
Nevertheless, the considerably greater activity of *C. papaya* compared with the SiNPs in the present study indicates that the botanical component made an important contribution to toxicity. *C. papaya* seeds contain several bioactive constituents in addition to their major fatty acids, and previous studies have demonstrated insecticidal effects of *C. papaya* seed-derived materials against *C. maculatus*. Idoko et al. reported increasing *C. maculatus* mortality with increasing amounts of *C. papaya* seed products, supporting the intrinsic pesticidal potential of the seed material (Idoko et al., 2018).

Most importantly, loading *C. papaya* onto the silica produced substantially higher mortality than either component alone, particularly at 48 and 72 h. This improvement is also reflected in the lethal-concentration estimates. The  $LC_{50}$  of SiNC decreased from 27.20 mg/mL at 24 h to 14.46 mg/mL after 48 h and 8.83 mg/mL after 72 h, while its corresponding  $LC_{90}$  values decreased from 88.84 to 27.75 and 18.92 mg/mL. *C. papaya* likewise displayed progressively decreasing  $LC_{50}$  values of 32.08, 26.05 and 20.48 mg/mL over 24–72 h, but these remained higher than those of SiNC at every corresponding exposure time. The lower  $LC_{50}$  and  $LC_{90}$  values of SiNC therefore demonstrate greater potency because a lower concentration was required to achieve equivalent mortality.

The enhanced activity of the SiNC may result from the complementary properties of the silica carrier and the bioactive seed oil. Silica-based nanocarriers provide a large surface area for association of active compounds and may improve their dispersion, retention and interaction with the target organism. Recent reviews indicate that silica carriers can enhance pesticide stability, bioavailability and controlled delivery and can reduce losses of active ingredients compared with conventional formulations (Zeng & Motola, 2025). Comparable enhancement has been reported for botanical-oil/silica formulations. Attia et al. (2023), for example, demonstrated that encapsulation of cinnamon oil in silica produced sustained insecticidal activity against a stored-product pest (Attia et al., 2023). The current results consequently support the use of SiNPs as a carrier for the pesticidal constituents of *C. papaya* seed oil.

The probit regression coefficients also provide information on the reliability of the concentration–mortality relationships. Most treatments produced relatively high  $R^2$  values, ranging from 0.9133 to 0.9928, indicating generally satisfactory agreement between the fitted models and mortality responses. The SiNC showed particularly strong fits after 24 and 48 h ( $R^2 = 0.9902$  and  $0.9845$ , respectively). However, its  $R^2$  decreased to  $0.8575$  at 72 h. This reduced value is plausibly associated with mortality approaching its upper response limit ( $96.67\%$ ), resulting in reduced discrimination among the higher concentrations.

Generally, the characterization and bioassay results are mutually supportive. FTIR indicated incorporation of organic functionalities into the silica-containing material, SEM revealed morphological modification after loading, EDX showed the appearance of a substantial carbon fraction, and TGA demonstrated increased thermal mass loss in the SiNC. These physicochemical changes coincided with a pronounced improvement in activity against *C. maculatus*, with the SiNC reaching  $93.33\%$  mortality after 48 h and  $96.67\%$  after 72 h compared with  $53.33$  and  $63.33\%$ , respectively, for the free *C. papaya* seed oil. The findings therefore demonstrate the potential of combining sugarcane bagasse-derived silica and *C. papaya* seed oil to generate a value-added nanopesticidal material from two agricultural waste streams. The result is consistent with current interest in silica-based nanopesticides as platforms for improving delivery and persistence of botanical active ingredients while simultaneously valorizing biomass residues (Saw et al., 2023; Zeng & Motola, 2025).



**Figure 6.** Mortality of *C. maculatus* exposed to silica nanoparticles (SiNPs), *C. papaya* seed oil, and silica nanoparticle–*C. papaya* seed oil nanocomposite (SiNC) after 24, 48, and 72 h of exposure. Error bars represent the standard deviation of replicate measurements.

**Table 4.** Mortality and lethal concentration parameters of SiNPs, *C. papaya* seed oil, and SiNC against *C. maculatus* at different exposure periods.

| Time (h) | Treatment        | Maximum mortality (%) | LC <sub>50</sub> (mg/mL) | LC <sub>90</sub> (mg/mL) | R <sup>2</sup> |
|----------|------------------|-----------------------|--------------------------|--------------------------|----------------|
| 24 h     | SiNPs            | 13.33                 | 149.10                   | 1023.86                  | 0.9759         |
|          | <i>C. papaya</i> | 46.67                 | 32.08                    | 113.41                   | 0.9133         |
|          | SiNC             | 55.17                 | 27.20                    | 88.84                    | 0.9902         |
| 48 h     | SiNPs            | 17.24                 | 191.55                   | 2520.95                  | 0.9882         |
|          | <i>C. papaya</i> | 53.33                 | 26.05                    | 89.43                    | 0.9145         |
|          | SiOC             | 93.33                 | 14.46                    | 27.75                    | 0.9845         |
| 72 h     | SiNPs            | 23.33                 | 131.81                   | 1679.73                  | 0.9928         |

|                  |       |       |       |        |
|------------------|-------|-------|-------|--------|
| <i>C. papaya</i> | 63.33 | 20.48 | 62.98 | 0.9402 |
| SiNC             | 96.67 | 8.83  | 18.92 | 0.8575 |

#### 4. Conclusion

This study demonstrated the successful valorization of sugarcane bagasse and *C. papaya* seed wastes into a silica-based nanocomposite with enhanced pesticidal activity against *C. maculatus*. Silica nanoparticles were successfully recovered from sugarcane bagasse ash, while GC–MS analysis confirmed the presence of several fatty acids and squalene in the extracted *C. papaya* seed oil. FTIR, XRD, SEM, EDX and TGA analyses collectively supported the incorporation of the seed oil into the silica matrix while retaining the predominantly amorphous character of the silica. Most importantly, the SiNC exhibited substantially greater pesticidal activity than either SiNPs or the free seed oil, achieving 93.33% mortality after 48 h and 96.67% after 72 h. Its  $LC_{50}$  decreased to 8.83 mg/mL at 72 h, further demonstrating enhanced potency with increasing exposure time. The findings generally indicate that combining waste-derived silica with *C. papaya* seed oil provides a promising approach for producing value-added botanical nanopesticides. Further studies should evaluate formulation stability, non-target toxicity, environmental fate and performance under practical grain-storage conditions before field-scale application.

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#### Conflict of Interest

The authors declare no conflict of interest.

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