DOI: https://doi.org/10.57233/ijsgs.v12i1.1139 ISSNp: 2488-9229; ISSNe: 3027-1118

IJSGS FUGUSAU VOL. 12(1) WEBSITE: https://fugus-ijsgs.com.ng

INTERNATIONAL JOURNAL OF SCIENCE FOR GLOBAL SUSTAINABILITY

(A PUBLICATION OF FACULTY OF SCIENCE, FEDERAL UNIVERSITY GUSAU, NIGERIA)

Sustainable Preparation of CaO–*Carica papaya* Seed Oil Nanocomposite from Agro-Wastes and Its Insecticidal Activity against *Callosobruchus maculatus*

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Received on: March, 2026 **Revised and Accepted on:** March, 2026 **Published on:** March, 2026

ABSTRACT

This study investigated the sustainable preparation of a calcium oxide–*Carica papaya* seed oil nanocomposite (CaONC) from chicken eggshell and pawpaw seed wastes and evaluated its insecticidal activity against *Callosobruchus maculatus*. Calcium oxide nanoparticles (CaONPs) were produced from eggshells by calcination, hydration and thermal dehydration, while *C. papaya* seed oil (CP) was extracted by Soxhlet extraction and incorporated onto CaONPs through solvent-assisted adsorption. GC–MS revealed fatty acids including myristic, palmitic, palmitoleic, linoleic, oleic and stearic acids, together with squalene. FTIR, XRD, SEM–EDX and TGA confirmed the crystalline CaO phase, aggregated morphology and successful incorporation of the organic oil fraction into CaONC. EDX showed 29.55 at.% carbon in CaONC compared with no detectable carbon in CaONPs, while TGA showed higher mass loss for CaONC (62.50%) than CaONPs (48.30%). Insecticidal assays demonstrated concentration- and time-dependent activity, with CaONC showing the highest efficacy. After 72 h, maximum mortality reached 87.00% for CaONC, compared with 63.33% for CP and 21.93% for CaONPs. CaONC also exhibited the lowest LC_{50} (7.77 mg/mL) and LC_{90} (17.41 mg/mL), indicating enhanced pesticidal performance and potential for sustainable stored-grain protection. These findings demonstrate a promising waste-valorization strategy that combines mineral and botanical components in a bio-based formulation for cowpea weevil management.

Keywords: Agro-waste valorization; calcium oxide nanoparticles; *Carica papaya* seed oil; *Callosobruchus maculatus*; nanopesticide.

1.0 INTRODUCTION

Cowpea (*Vigna unguiculata* L. Walp.) is an important grain legume and source of dietary protein, particularly in tropical and subtropical regions where it contributes significantly to household nutrition and food security. However, its postharvest preservation is seriously constrained by the cowpea weevil, *Callosobruchus maculatus* (F.), a highly destructive storage pest capable of causing substantial deterioration in seed quality, weight, viability and market value. Infestation begins either before harvest or during storage, while larval development within the grain makes control particularly difficult. Although synthetic insecticides and fumigants can provide effective protection, their repeated or inappropriate use is associated with pesticide residues, pest resistance, environmental contamination and potential risks to humans and non-target organisms. Consequently, increasing attention is being directed toward safer and renewable approaches for protecting stored grains(Kalpna et al., 2022; Mssillou et al., 2022).

Plant-derived insecticides, particularly botanical oils and extracts, have considerable potential for stored-product pest management because they contain chemically diverse bioactive constituents capable of causing contact toxicity, repellency, feeding deterrence and disruption of insect development. Several botanical oils have demonstrated mortality, reduced oviposition and suppression of progeny emergence in *C. maculatus* (Gupta et al., 2023; Mssillou et al., 2022). Similarly, agricultural residues containing bioactive phytochemicals have shown appreciable insecticidal activity against this pest, highlighting the possibility of simultaneously addressing crop protection and waste-management challenges(Ashamo et al., 2022). Nevertheless, botanical insecticides may be limited by poor stability, rapid degradation, volatility and relatively short persistence, creating a need for formulation strategies capable of improving their delivery and effectiveness.

Nanotechnology provides an opportunity to address some of these limitations through nanopesticides and nano-enabled formulations. The high surface-area-to-volume ratio and tunable physicochemical properties of nanoparticles can improve the dispersion, stability and interaction of pesticidal agents with target organisms (Izuagie et al., 2025). Nanoformulations may consequently enhance efficacy, protect active compounds from premature degradation and potentially reduce the quantity of pesticide required for effective pest control. Recent assessments nevertheless emphasize that these advantages must be considered alongside nanoparticle fate, non-target toxicity and appropriate risk assessment to ensure environmentally responsible application(Zainab et al., 2024). Green preparation of nanomaterials from biological and agricultural wastes is particularly attractive because it integrates nanotechnology with circular-economy principles.

Chicken eggshell waste represents an inexpensive and abundant calcium-rich feedstock for this purpose. Eggshell consists predominantly of calcium carbonate, which can be thermally converted to calcium oxide (CaO). Recent studies have demonstrated that controlled calcination of discarded eggshells can produce crystalline CaO nanoparticles without the extensive use of chemical precursors, providing a comparatively simple route for converting food waste into value-added nanomaterials (Adaikalam et al., 2024).

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Eggshell-derived CaO nanoparticles exhibit high surface reactivity and have demonstrated biological activities in addition to their established environmental and catalytic applications, making them interesting candidates for incorporation into functional nanocomposites.

Pawpaw (*Carica papaya* L.) seeds constitute another underutilized agro-food residue with potential for pest-management applications. The seeds contain an appreciable oil fraction and several biologically active constituents, including phenolics, terpenoid-related compounds and glucosinolate-derived benzyl isothiocyanate. Recent studies have confirmed the recovery of benzyl isothiocyanate and other bioactive constituents from papaya seeds and demonstrated important biological properties of the resulting extracts (Feltes et al., 2024). More recently, an insecticidal compound isolated from *C. papaya* seed oil exhibited strong activity against different developmental stages of *Anopheles gambiae*, further demonstrating that papaya seed oil is a promising reservoir of naturally occurring insecticidal constituents (Ogbonna et al., 2026). Combining such botanical constituents with inorganic nanoparticles could provide complementary mechanisms of action while improving their presentation and contact with target insects.

Despite advances in botanical insecticides and agro-waste-derived nanomaterials, information on the integration of eggshell-derived CaO nanoparticles with *C. papaya* seed oil for stored-grain protection remains limited. The present study therefore investigates the valorization of two common wastes—chicken eggshells and pawpaw seeds—through the preparation of a CaO–pawpaw seed oil nanocomposite and evaluation of its insecticidal activity against *C. maculatus*. The pawpaw seed oil was chemically profiled by gas chromatography–mass spectrometry, while the prepared CaO nanoparticles and nanocomposite were physicochemically characterized. Their insecticidal performances were subsequently evaluated to determine whether combining the inorganic and botanical components could provide an effective waste-derived formulation for sustainable management of the cowpea weevil.

2.0 MATERIALS AND METHODS

2.1 Materials and Reagents

Waste chicken eggshells were used as the precursor for the preparation of calcium oxide nanoparticles (CaONPs), while seeds of *Carica papaya* L. were used as the source of pesticidal oil. Both materials were selected because of their availability as food-related wastes and were collected from locations within Sokoto metropolis, Sokoto State, Nigeria. The chemicals used included *n*-hexane (95%, Fisher Scientific), acetone (99%, Fisher Scientific), ethanol, and distilled water. All reagents were of analytical grade and were used without further purification. A Soxhlet extraction apparatus and 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 and Pretreatment

2.2.1 Collection and Pretreatment of Eggshells

Waste chicken eggshells were collected from local sources within Sokoto metropolis, Nigeria. The eggshells were thoroughly washed with warm tap water to remove adhering organic matter and other surface impurities and subsequently rinsed with distilled water. The cleaned shells were dried in a hot-air oven at 120 °C for 24 h. The dried eggshells were crushed using a blender and passed through a 60-mesh sieve to obtain a relatively uniform powder prior to calcination. Cleaning, drying, size reduction, and high-temperature calcination are commonly employed during the conversion of waste eggshells, which consist predominantly of calcium carbonate, into CaO-based materials (Adaikalam et al., 2024; Fasanya et al., 2022).

2.2.2 Collection and Preparation of C. papaya Seeds

C. papaya seeds were collected from Kasuwan Daji in Sokoto, Sokoto State, Nigeria. The seeds were initially washed with tap water, rinsed with distilled water, and sun-dried for one week to reduce their moisture content. The dried seeds were pulverized into a fine powder using a mortar and pestle and stored in airtight containers to minimize moisture uptake and contamination prior to oil extraction. Drying and pulverization of papaya seeds before solvent extraction are consistent with procedures recently employed for the recovery and characterization of *C. papaya* seed oil (Goriainov et al., 2023).

2.3 Extraction and GC–MS Analysis of *Carica papaya* Seed Oil

2.3.1 Extraction of Carica papaya Seed Oil

Oil was extracted from the pulverized *C. papaya* seeds by continuous Soxhlet extraction using *n*-hexane as the extracting solvent. The powdered seeds were placed in the Soxhlet apparatus and subjected to continuous solvent extraction. Following extraction, the recovered oil was filtered and preserved for subsequent chemical analysis and nanocomposite preparation. Soxhlet extraction with *n*-hexane has been successfully employed for the recovery of oil from *C. papaya* seeds in recent compositional and process studies (Goriainov et al., 2023).

2.3.2 Gas Chromatography–Mass Spectrometry Analysis

The chemical composition of the extracted *C. papaya* seed oil was determined by gas chromatography–mass spectrometry (GC–MS). Analysis was performed using an Agilent 6890N gas chromatograph coupled to an Agilent 5973N mass-selective detector and equipped with a DB-5MS silica capillary column. Helium was used as the carrier gas at a flow rate of 1.0 mL min^{-1}, and 1 µL of the oil sample was injected. The oven temperature was initially maintained at 40 °C for 2 min, increased to 200 °C at 5 °C min^{-1}, and held for 2 min. The temperature was subsequently increased to 300 °C at 5 °C min^{-1} and maintained for a further 2 min. Data were acquired using GC/MSD Enhanced MassHunter software. Individual constituents were identified by comparing their recorded mass spectra with reference spectra available in the Wiley Registry/NIST Mass Spectral Library 2023. GC-based analysis has similarly been employed to establish the characteristic lipid and chemical profiles of *C. papaya* seed oils(Goriainov et al., 2023).

2.4 Preparation of Calcium Oxide Nanoparticles from Eggshells

Calcium oxide nanoparticles were prepared from the powdered eggshells through calcination followed by hydration and subsequent thermal dehydration. Briefly, the powdered eggshells were calcined in a muffle furnace at 900 °C for 3 h. At elevated temperature, the calcium carbonate ($CaCO_3$) constituting the major mineral component of eggshell was thermally decomposed to calcium oxide (CaO), with the liberation of carbon dioxide. High-temperature calcination at approximately 850–900 °C has been reported as an effective approach for converting waste chicken eggshell $CaCO_3$ to CaO (Adaikalam et al., 2024; Fasanya et al., 2022). The resulting CaO was refluxed in water at 60 °C for 6 h, thereby promoting its hydration to calcium hydroxide [$Ca(OH)_2$]. The resulting material was subsequently dried in a hot-air oven at 105 °C overnight. The dried solid was ground using a blender and subjected to a second calcination at 870 °C for 3 h to thermally dehydrate $Ca(OH)_2$ and regenerate CaO. The resulting CaO material was cooled and stored under vacuum in a desiccator until further use. The CaO/$Ca(OH)_2$ transformation accompanying hydration and subsequent thermal treatment of calcined eggshell-derived materials has also been documented in recent studies (Adaikalam et al., 2024; Chen et al., 2022). The final product was designated as eggshell-derived calcium oxide nanoparticles (CaONPs) and subjected to instrumental characterization prior to nanocomposite formulation.

2.5 Preparation of CaO Nanoparticle–Carica papaya Seed Oil Nanocomposite

The CaO nanoparticle–*C. papaya* seed oil nanocomposite (CaONC) was prepared by loading the extracted seed oil onto the CaONPs using a solvent-assisted immersion/adsorption approach. Briefly, 50 mg of CaONPs was suspended in 40 mL of acetone and thoroughly dispersed by sonication. Subsequently, 10 mg of *C. papaya* seed oil was added to the suspension, corresponding to a CaONP-to-oil mass ratio of 5:1. The suspension was continuously stirred at room temperature for 24 h to promote adsorption and interaction of the oil constituents with the nanoparticle surface. The suspension was subsequently concentrated to approximately 0.2 mL and sonicated for 10 min for degassing. It was then stirred for a further 6 h to facilitate uniform distribution of the oil within the nanoparticle matrix. Acetone was 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. The recovered CaONC was dried in an oven at 30 °C, ground into a fine powder, and stored in a sealed container until characterization and insecticidal evaluation. Solvent-assisted immersion and adsorption are established approaches for incorporating bioactive molecules and oils into nanoparticulate carrier systems (Fakhariha et al., 2025).

2.6 Instrumental Characterization

The prepared CaONPs and CaONC were characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM–EDX), and thermogravimetric analysis (TGA). These complementary techniques were employed to establish the functional groups, crystalline phases, morphology, elemental composition, and thermal characteristics of the materials. FTIR analysis was performed using a Bruker Alpha spectrometer equipped with a Platinum ATR module over the range of 4000–500 cm^{-1}. Samples were dried for at least 1 h prior to analysis. The spectra were used to identify characteristic functional groups in the CaO-NPs and evaluate spectral changes associated with incorporation of *C. papaya* seed oil into the CaO matrix. XRD analysis was conducted using a Philips X'Pert Pro diffractometer with Cu Kα radiation (λ = 0.15416 nm) over a 2θ range of 5–80°. The diffraction patterns were used to evaluate the crystalline phases of the prepared materials and confirm the formation of CaO. XRD has been extensively employed to verify the conversion of the predominantly calcitic $CaCO_3$ phase of eggshell into crystalline CaO following high-temperature calcination (Adaikalam et al., 2024). Surface morphology and elemental composition were examined using a Phenom ProX scanning electron microscope coupled with an energy-dispersive X-ray detector and operated at an accelerating voltage of 10 kV. SEM was employed to evaluate surface morphology and particle aggregation, while EDX was used to determine the elemental composition of the samples. Thermogravimetric analysis was conducted using a Q600 SDT instrument (TA Instruments). Approximately 6 mg of each sample was placed in a 40 µL platinum crucible and heated from 50 to 800 °C at a rate of 10 °C min^{-1} under a continuous flow of high-purity nitrogen at 120 mL min^{-1}. TGA was used to compare the thermal behaviour of CaONPs and CaONC and to provide evidence of the incorporation of organic constituents from *C. papaya* seed oil into the nanocomposite.

2.7 Insecticidal Studies

2.7.1 Acquisition and Rearing of C. maculatus

Adult *C. maculatus* were obtained in Sokoto, Nigeria, and transported to the Entomology Laboratory, Usmanu Danfodiyo University, Sokoto, for identification and laboratory culture. The insects were maintained on whole black-eyed cowpea seeds in plastic containers (10 × 12 cm) covered with muslin cloth to allow ventilation and prevent escape. The cultures were maintained at approximately 27.5 °C, 60 ± 5% relative humidity, and a 12:12 h light/dark photoperiod. Laboratory rearing of *C. maculatus* on cowpea seeds under controlled environmental conditions is commonly used for evaluating the insecticidal activity of nanoparticulate materials against this stored-product pest (Ibrahim et al., 2024).

2.7.2 Residual Toxicity Assay

Residual/contact toxicity of the test materials against adult *C. maculatus* was evaluated at concentrations of 1.0, 1.5, 2.0, and 3.0% (m/v), with three replicates per treatment.

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IJSGS FUGUSAU VOL. 12(1) WEBSITE: https://fugus-ijsgs.com.ng

Twenty grams of sterilized cowpea seeds were separately treated with the respective formulations and allowed to air-dry for 30 min before being transferred into 12-cm-diameter Petri dishes. Five pairs of newly emerged adult *C. maculatus* were subsequently introduced into each experimental unit. Untreated cowpea seeds served as the negative control. The Petri dishes were covered with muslin cloth, and insect mortality was assessed after 24, 48, and 72 h of exposure. Dead insects were counted and removed at each assessment period. Concentration- and exposure-time-dependent contact toxicity assays are commonly employed for evaluating nanoparticle-based materials against adult *C. maculatus* (Ibrahim et al., 2024).

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

Where mortality occurred in the untreated control, the observed mortality was corrected using Abbott's equation:

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

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

2.8 Statistical Analysis

Statistical analyses were performed using Microsoft Excel and IBM SPSS Statistics version 20. Mortality data were assessed for normality using the Shapiro–Wilk test and for homogeneity of variance using Levene's test. Differences among treatment groups were evaluated using analysis of variance (ANOVA), followed by Tukey's honestly significant difference (HSD) test for separation of treatment means 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}) at the respective exposure periods. Probit modelling is routinely employed for estimating lethal concentrations from quantal mortality responses and has recently been applied to nanoparticle toxicity studies involving *C. maculatus* (Ibrahim et al., 2024).

3.0 RESULTS AND DISCUSSION

3.1 Product Yields

The percentage yields and physical appearance of the prepared samples are presented in Table 1. The extracted *C. papaya* seed oil was brown and gave a yield of 29.3%, whereas the CaONPs were obtained as a white powder with a yield of 31.2%. The relatively moderate oil recovery confirms that papaya seeds represent a potentially useful lipid-rich agricultural by-product. Papaya seed-oil yield can vary considerably with cultivar, geographical origin, moisture content and extraction conditions. For example, Goriainov et al. (2023) reported oil yields of 18.3–27.0% for seeds originating from different geographical regions, demonstrating the strong influence of raw-material and processing variables (Goriainov et al., 2023). The white appearance of the CaONPs is consistent with calcium-rich oxide/hydroxide materials produced from thermally treated biogenic calcium carbonate.

Table 1: Percentage yield and physical appearance of extracted *C. papaya* seed oil, CaONPs and CaONC.

Sample	% Yield	Colour
Extracted *C. papaya* seed Oil	42.3%	Brown
CaONPs	31.2%	White
CaONC	98.3%	White

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

The GC–MS profile presented in Table 2 revealed several saturated and unsaturated fatty acids. The compounds identified included myristic acid, pentadecanoic acid, palmitic acid, palmitoleic acid, margaric acid, linoleic acid, behenic acid, oleic acid and stearic acid. The occurrence of oleic, palmitic, stearic, linoleic, palmitoleic and myristic acids agrees well with published compositional studies of papaya seed oil. Goriainov et al. (2023), for example, identified oleic, palmitic, stearic and linoleic acids as important constituents of papaya seed oils collected from different geographical origins (Goriainov et al., 2023). Similar fatty-acid profiles have also been reported in recent studies of papaya seed oil, with oleic acid generally constituting the major fatty-acid fraction (Santana et al., 2022). The presence of both unsaturated fatty acids and the triterpene hydrocarbon squalene indicate that the oil contains chemically diverse lipophilic constituents that could contribute to its biological activity.

Table 2: GC–MS-identified constituents of *C. papaya* seed oil showing retention times, compound identities and molecular formulae.

Retention time (min)	Identified compound	Common name / description	Molecular formula
13.962	Tetradecanoic acid	Myristic acid	$C_{14}H_{28}O_2$
14.872	Pentadecanoic acid	Pentadecylic acid	$C_{15}H_{30}O_2$
16.171	*n*-Hexadecanoic acid	Palmitic acid	$C_{16}H_{32}O_2$
16.571	Hexadecenoic acid	Palmitoleic acid	$C_{16}H_{30}O_2$
17.058	Heptadecanoic acid	Margaric acid	$C_{17}H_{34}O_2$
17.391	Octadecadienoic acid	Linoleic acid	$C_{18}H_{32}O_2$
18.723	Docosanoic acid	Behenic acid	$C_{22}H_{44}O_2$
18.980	9-Octadecenoic acid	Oleic acid	$C_{18}H_{34}O_2$
19.169	Octadecanoic acid	Stearic acid	$C_{18}H_{36}O_2$
26.957	Squalene	Triterpene hydrocarbon	$C_{30}H_{50}$

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3.3 FTIR Analysis

The FTIR spectra of CaONPs and CaONC are compared in Figure 1. Both spectra contain a sharp absorption in the region around 3600–3650 cm⁻¹, attributable primarily to O–H stretching associated with surface hydroxyl groups and/or adsorbed moisture. Bands in the region around 1400–1500 cm⁻¹ and near 870 cm⁻¹ are consistent with carbonate-related vibrations, whereas absorption below approximately 600 cm⁻¹ can be assigned to Ca–O vibrations. Comparable bands at approximately 3647, 1400, 875 and 512 cm⁻¹ have recently been reported for eggshell-derived CaO materials (Adaikalam et al., 2024).

In comparison with CaONPs, the CaONC spectrum shows additional or more pronounced features in the C–H stretching region around 2920–2850 cm⁻¹ and the carbonyl-containing region around 1700–1750 cm⁻¹ (Figure 1). These changes are compatible with incorporation of the organic oil components identified by GC–MS into the calcium-based material. Thus, the FTIR result provides chemical evidence for the coexistence of the inorganic calcium phase and organic constituents in CaONC. The carbonate and hydroxyl bands additionally indicate that the surface of the calcium material is susceptible to hydration and atmospheric carbonation, phenomena frequently reported for CaO nanoparticles (Adaikalam et al., 2024).

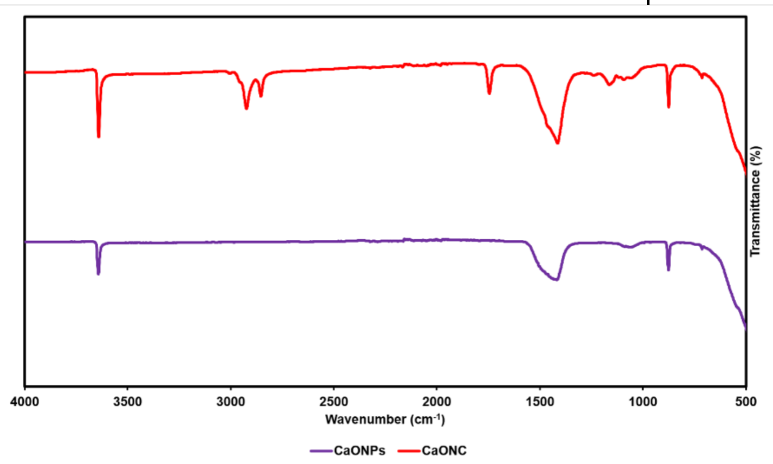

Figure 1: FTIR spectra of CaONPs and CaONC showing characteristic calcium–oxygen/hydroxyl vibrations and changes associated with incorporation of the organic component.

3.4 X-ray Diffraction Analysis

The XRD patterns of CaONPs and CaONC are presented in Figure 2. Both samples exhibit distinct diffraction peaks at approximately 32.2°, 37.3°, 53.9°, 64.2°, and 67.4° (2θ), confirming a highly crystalline calcium oxide phase. These reflections agree closely with the characteristic peaks reported for cubic CaO, including those at about 32.25°, 37.42°, 54.04°, 64.47°, and 67.74° (Bin Mobarak et al., 2022; Sahadat Hossain et al., 2023). The absence of the prominent $Ca(OH)_2$ reflection near 18° suggests that extensive hydration of CaO was not evident in the samples. Importantly, CaONC retained essentially the same diffraction positions as CaONPs, although with slightly reduced intensities. This indicates that oil modification did not substantially alter the crystalline structure of CaO, while the decreased intensities may reflect surface coating by the organic component.

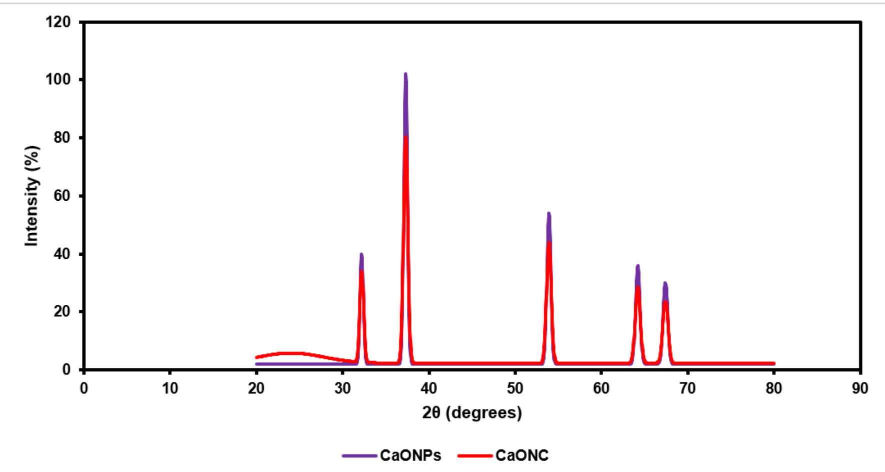

Figure 2: X-ray diffraction patterns of CaONPs and CaONC showing the crystalline calcium-containing phases.

3.5 SEM Morphology

The SEM micrographs in Figure 3(a) and (b) reveal irregular particles assembled into relatively large agglomerated clusters. The particles are non-uniform in shape and show rough, textured surfaces with numerous interparticle boundaries and voids. Agglomeration is commonly observed for biogenic calcium oxide nanoparticles because of their high surface energy and strong interparticle interactions. Recent SEM studies of eggshell-derived CaO have similarly described aggregated or clustered nanoparticle morphologies (Bhuvaneshwari et al., 2024).

The CaONC micrograph (Figure 3b) retains the aggregated morphology but appears comparatively compact, suggesting that association with the organic fraction modifies particle packing without destroying the underlying calcium-based particles.

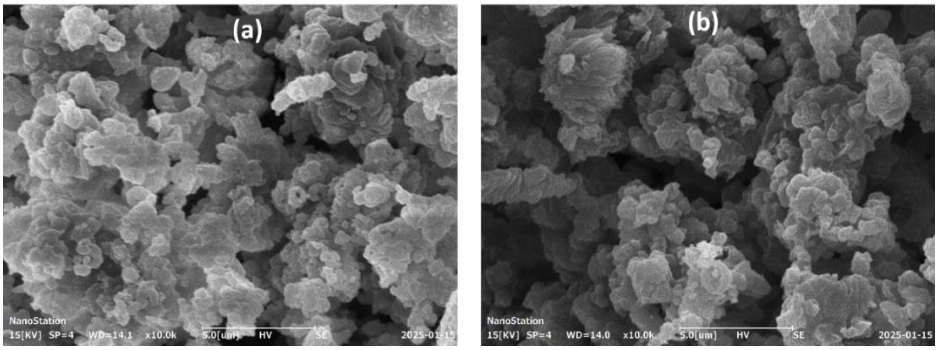

Figure 3: SEM micrographs showing the surface morphology and particle agglomeration of (a) CaONPs and (b) CaONC at ×10,000 magnification.

3.6 EDX Analysis

The elemental spectra and quantitative composition are shown in Figure 4 and Table 3, respectively. CaONPs contained only Ca and O within the reported analysis, at 47.45 and 52.55 atomic %, respectively. In contrast, CaONC contained 29.55 atomic % C, 46.56 atomic % O and 23.89 atomic % Ca. The CaONP spectrum in Figure 4a displays the characteristic oxygen signal around 0.52 keV and prominent Ca K lines around 3.69 and 4.01 keV, confirming a calcium–oxygen-based material.

The near-equivalent atomic proportions of Ca and O in CaONPs are broadly consistent with a calcium oxide-derived material, although the slightly higher oxygen content is also compatible with the surface hydroxylation indicated by FTIR and XRD. For CaONC, the marked appearance of carbon together with the reduction in relative calcium content (Figure 4b; Table 3) provides strong evidence for incorporation or surface coverage by the organic component. This observation complements the additional organic functional-group bands observed in Figure 1, supporting successful formation of the composite.

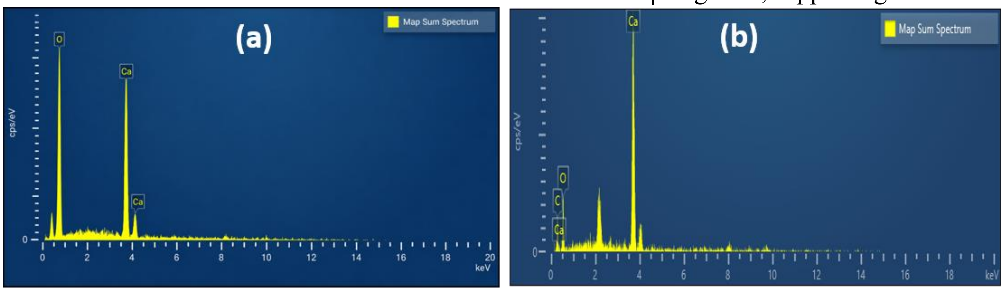

Figure 4: EDX spectra of (a) CaONPs and (b) CaONC showing the characteristic elemental signals of Ca, O and, in the composite, C.

Table 3: EDX-derived atomic composition of CaONPs and CaONC.

Element	Atomic %	
	CaONPs	CaONC
C	0.00	29.55
O	52.55	46.56
Ca	47.45	23.89
Total	100.00	100.00

3.7 Thermogravimetric Analysis and Oil Loading

The thermal behaviour of CaONPs and CaONC is presented in Figure 5(a) and (b). CaONPs showed an overall mass loss of approximately 48.30%, whereas CaONC exhibited a greater mass loss of approximately 62.50%. The curves indicate relatively small changes at lower temperatures followed by the principal mass-loss region at intermediate temperatures before approaching a more stable residual mass at high temperature.

The larger mass loss of CaONC—approximately 14 percentage points greater than that of CaONPs—is consistent with decomposition of the incorporated organic fraction in addition to thermal transformations of the calcium-containing phase. Meanwhile, the substantial mass loss of the nominal CaONPs is consistent with the FTIR and

XRD evidence that the sample contains some surface hydroxylated species. Eggshell-derived CaO readily undergoes hydration and carbonation during handling, and these phases subsequently undergo dehydroxylation and/or decarbonation upon heating (Lanzón et al., 2023). Thus, the TGA results in Figure 5 are internally consistent with the structural information obtained from FTIR and XRD.

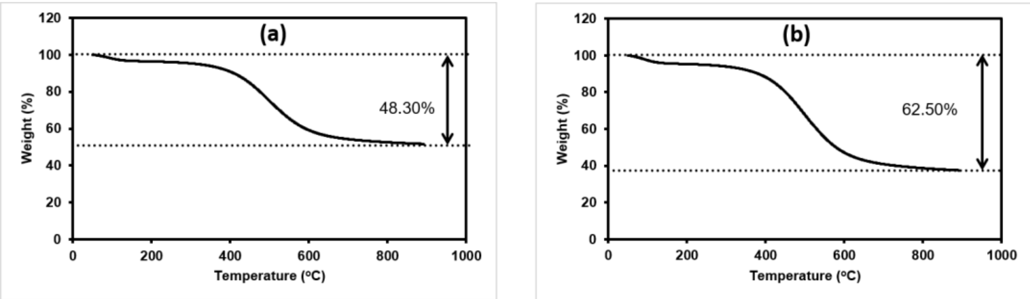

Figure 5: Thermogravimetric profiles of (a) CaONPs and (b) CaONC showing total mass losses of approximately 48.30 and 62.50%, respectively.

3.8 Pesticidal Activity against *C. maculatus*

The mortality results in Figure 6 show a clear exposure-time dependence, with CaONC producing the highest mortality, followed by CP, while CaONPs alone were considerably less active. This trend is confirmed by the probit parameters presented in Table 4. At 24 h, maximum mortality was 12.26, 46.67 and 48.55% for CaONPs, CP and CaONC, respectively. By 72 h, mortality increased to 21.93% for CaONPs, 63.33% for CP and 87.00% for CaONC.

The superior activity of CaONC is more clearly reflected by its decreasing lethal concentrations. Its LC_{50} decreased from 24.75 mg/mL at 24 h to 13.01 mg/mL at 48 h and 7.77 mg/mL at 72 h, while LC_{90} decreased from 79.07 to 17.41 mg/mL. At 72 h, the LC_{50} of CaONC was therefore about 2.6-fold lower than that of CP (20.48 mg/mL) and almost 15-fold lower than that of CaONPs (114.67 mg/mL). The results suggest that combining the calcium-based particles with the papaya-derived organic fraction substantially enhances pesticidal performance. This may arise from complementary contact effects of calcium-based particles and the bioactivity of plant-derived organic constituents, although demonstration of true chemical synergism would require a formal synergy analysis.

The trend is supported by recent evidence showing that biogenic CaO nanoparticles can control *C. maculatus*. Madesh et al. (2025) reported dose-dependent mortality of *C. maculatus* following treatment with shell-derived CaO nanoparticles and attributed part of the activity to particle adherence, dehydration and desiccation of insects (Madesh et al., 2025). Botanical oils and plant-derived compounds are also well established as alternative control agents against *C. maculatus (Mssillou et al., 2022)*.

Most R^2 values in Table 4 were approximately 0.91–0.94, indicating generally acceptable agreement of the regression models with the mortality data. Overall, Figure 6 and Table 4 identify CaONC as the most effective formulation, particularly after 48–72 h.

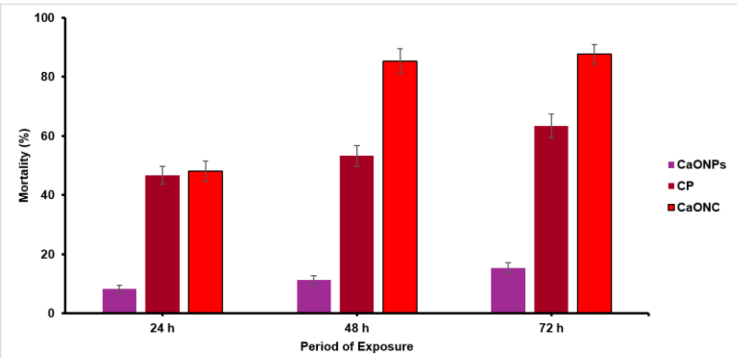

Figure 6: Mortality of *C. maculatus* following exposure to CaONPs, CP and CaONC for 24, 48 and 72 h. Error bars represent the standard deviation of replicate measurements.

Table 4. Mortality and probit-derived lethal concentration parameters (LC_{50} and LC_{90}) for CaONPs, CP and CaONC against *C. maculatus* at different exposure periods.

Time (h)	Treatment	Maximum mortality (%)	LC_{50} (mg/mL)	LC_{90} (mg/mL)	R^2
24 h	CaONPs	12.26	131.21	952.19	0.9271
	CP	46.67	32.08	113.41	0.9133

	CaONC	48.55	24.75	79.07	0.9308
48 h	CaONPs	16.03	164.73	2268.86	0.9388
	CP	53.33	26.05	89.43	0.9145
	CaONC	83.06	13.01	25.81	0.9057
72 h	CaONPs	21.93	114.67	1528.55	0.9432
	CP	63.33	20.48	62.98	0.9402
	CaONC	87.00	7.77	17.41	0.7803

4.0 Conclusion

This study demonstrated the successful valorization of chicken eggshells and *C. papaya* seeds into a CaO–seed oil nanocomposite with enhanced insecticidal activity against *C. maculatus*. Characterization by FTIR, XRD, SEM–EDX and TGA showed that the CaO crystalline structure was largely retained following oil incorporation, while the increased carbon content and greater thermal mass loss of CaONC provided evidence of successful loading of the organic fraction. GC–MS further revealed several fatty acids and squalene in the papaya seed oil. Among the tested materials, CaONC consistently exhibited the greatest pesticidal performance, reaching 87.00% mortality after 72 h and producing the lowest LC_{50} and LC_{90} values of 7.77 and 17.41 mg/mL, respectively. These findings indicate that combining eggshell-derived CaONPs with papaya seed oil substantially improves activity relative to the individual components. The developed nanocomposite therefore represents a promising waste-derived formulation for sustainable protection of stored cowpea, although further studies on storage stability, non-target effects, residue behaviour and practical application are recommended before wider use.

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