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CHARACTERIZATION OF GAMMA-IRRADIATED CALCIUM SILICATES FROM GLASS WASTE

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ABSTRACT

With the rising concern over industrial glass waste, there is a growing need to develop sustainable strategies for material reuse. This study explores the potential of recycled calcium silicate ($CaSiO_3$) nano glass slides as eco-friendly and radiation-tolerant materials. The research focused on synthesizing ultrafine $CaSiO_3$ nanomaterials from used glass slides and evaluating their structural and morphological responses to gamma irradiation from a Cobalt-60 (Co-60) source. To assess these characteristics, three main analytical techniques were employed: X-ray Fluorescence (XRF) to determine elemental composition, X-ray Diffraction (XRD) to examine crystallographic structure, and Field Emission Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (FESEM-EDX) to study surface morphology and elemental distribution. XRF analysis confirmed calcium and silicon as the primary elements in the recycled samples, along with minor impurities likely introduced during processing. XRD results showed that as radiation doses increased (from 20 to 70 kGy), diffraction peaks became broader and less intense, indicating progressive structural disorder, though the fundamental crystal phase remained intact. FESEM-EDX imaging further revealed notable changes in the surface structure, including increased particle agglomeration and grain growth corresponding to higher radiation exposure. Overall, the study demonstrates that recycled $CaSiO_3$ nano glass retains its chemical and structural integrity even after significant gamma irradiation. These results highlight the material's promise for use in radiation-exposed environments, offering a meaningful step forward in transforming glass waste into valuable, sustainable nanomaterials suitable for environmental and industrial applications.

Keywords: Recycled Calcium Silicate ($CaSiO_3$); Gamma Irradiation; X-ray Diffraction (XRD); X-ray Fluorescence (XRF); Field Emission Scanning Electron Microscopy (FESEM-EDX)

INTRODUCTION

The growing volume of industrial and household glass waste worldwide necessitates a thorough examination of sustainable recycling methods to enhance the value of these materials. An effective approach involves converting waste glass into functional ceramic compounds, such as calcium silicate ($CaSiO_3$), with diverse applications in biomedical, environmental, and radiation-related fields. This sustainable and cost-effective method reduces landfill pressure and promotes material circularity by utilizing waste streams rich in silicates.

Numerous studies have explored the effects of ionizing radiation, particularly gamma rays, on the structure, heat, and light properties of silicate-based materials. For instance, Baltac and Mitran (2025) conducted a detailed analysis of gamma irradiation's impact on silica-based nanomaterials, highlighting changes in crystal structures and surface reactivity. Similarly, Büyükuslu et al. (2022) observed significant alterations in the morphology and luminescent properties of Nb-doped TiO_2 nanoparticles post-irradiation, indicating the formation of radiation-induced defects. The investigation of calcium silicate ceramics has attracted considerable academic interest due to their biocompatibility and responsiveness to radiation exposure.

Rare earth-doped polycrystalline $CaSiO_3$, synthesized by Gonzales-Lorenzo et al. (2018, 2020), shows promise for photon and neutron detection through thermoluminescence. The bioactivity and interfacial bonding of calcium silicates, as discussed by Hench et al. (1971) and Lin et al. (2007), suggest their suitability for biomedical scaffolds. Hao et al. (2018) demonstrated the significant sorption capacity of $CaSiO_3$ composites derived from industrial slag for removing hazardous metal ions in aquatic environments.

Despite extensive research on synthetic $CaSiO_3$ properties and its response to radiation, there is a lack of studies on utilizing recycled glass to produce $CaSiO_3$ nanomaterials and investigating the effects of Co-60 gamma irradiation on their structure and morphology. This gap presents a crucial area for advancing sustainable materials for nuclear and environmental applications.

This study aims to synthesize ultrafine calcium silicate ($CaSiO_3$) nanomaterials from used glass slides and examine how Co-60 gamma radiation influences their structure and morphology. Analytical characterizations using X-ray Fluorescence (XRF), X-ray Diffraction (XRD), and Field Emission Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (FESEM-EDX) were employed to determine composition, crystallinity, and surface morphology.

MATERIALS AND METHODS

Preparation of $CaSiO_3$ Glass Nanomaterials

Figure 1 illustrates the preparation of nano $CaSiO_3$ powder using the dry processing technique. It starts with the collection of discarded glass slides, which are then cleaned with distilled water to remove any surface residues or contaminants. This cleaning stage is crucial to eliminate any dirt that may affect the subsequent characterization process. After the cleaning process, the slides are manually ground using a mortar and pestle, reducing their size. This grinding is followed by ball milling technique to further crush the $CaSiO_3$ sample into finer particles. The mortar and pestle ensure efficient and controlled glass fragmentation, while the ball mill ensures that the sample size becomes finer, enhancing the uniformity of the powder at the ultrafine level. The sieved $CaSiO_3$ powder is then ready for characterization, a crucial step before exposure to Co-60 gamma radiation. The prepared nano material serves as a basis for analyzing structural and morphological changes after irradiation.

Figure 1: Preparation of $CaSiO_3$ Glass Nanomaterials before exposure

Characterization Techniques

X-ray Diffraction (XRD)

For XRD analysis, $CaSiO_3$ was ground and spread onto the sample holder to obtain a homogeneous surface area. The diffraction patterns were recorded using a Panalytical X'Pert PRO X-ray diffractometer (XRD) as shown in Figure 2 to analyse the structural properties of recycled nano $CaSiO_3$ glass slides before and after Co-60 gamma irradiation. This analysis was performed to confirm and evaluate changes in crystallinity, structural stability, and peak intensity in the samples after gamma irradiation. This analysis is crucial for evaluating its suitability in sustainable and radiation-resistant material applications.

Figure 2: Panalytical, X'Pert Pro

Field Emission Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (FESEM-EDX)

For FESEM-EDX analysis, the powder samples were mounted on a carbon tape and sputter-coated to reduce surface charging during image acquisition. The samples were observed using a Carl Zeiss/GeminiSEM 500 as shown in Figure 3 to assess physical features of $CaSiO_3$ samples before and after exposure to Co-60 gamma radiation, including particle size, morphology, surface texture, and agglomeration behavior of nanoscale particles, which are critical factors in assessing the quality and performance of materials. At the same time, EDX analysis was used to verify the elemental distribution on the sample surface.

Figure 3: Carl Zeiss/GeminiSEM 500

X-ray Fluorescence (XRF)

XRF is a non-destructive technique that was used to determine the elemental composition of a material. When the sample is irradiated with primary X-rays, electrons are ejected from the inner shell, which emit secondary X-rays. The emitted X-rays were used to identify the sample elements and measure their concentrations before and after irradiation.

It also detects any trace amounts of other substances, such as impurities or unwanted materials, that may have entered the glass during the recycling process. Understanding this information helps us assess whether the recycled glass retains good quality and is similar to the original, or if any changes could affect its functionality.

In this study, XRF was used to determine the elemental concentrations of recycled CaSiO3 nano-glass before and after irradiation. The powder samples were cleaned, placed in the sample holder, and then analyzed using a Shimadzu EDX-7000 spectrometer under ambient conditions. To ensure the reliability of elemental analysis measurements, the equipment was calibrated with appropriate reference standards.

Figure 4 shows a specialized machine called an X-ray Fluorescence (XRF) spectrometer used in this experiment. This machine helps to determine the types of small elements (such as atomic

components) in the recycled calcium silicate nanoglass slide. An interesting thing about XRF is that it does not harm the sample; it only emits strong X-rays onto it. These X-rays cause the atoms in the sample to emit their own small X-ray signals. By analyzing these signals, scientists can identify which elements are present in the sample and how much of each element is present.

Overall, XRF analysis provided valuable insights into the elemental composition and purity of the recycled $CaSiO_3$ nano glass slides, supporting the evaluation of the recycling process's effectiveness and consistency for reuse in scientific or industrial applications.

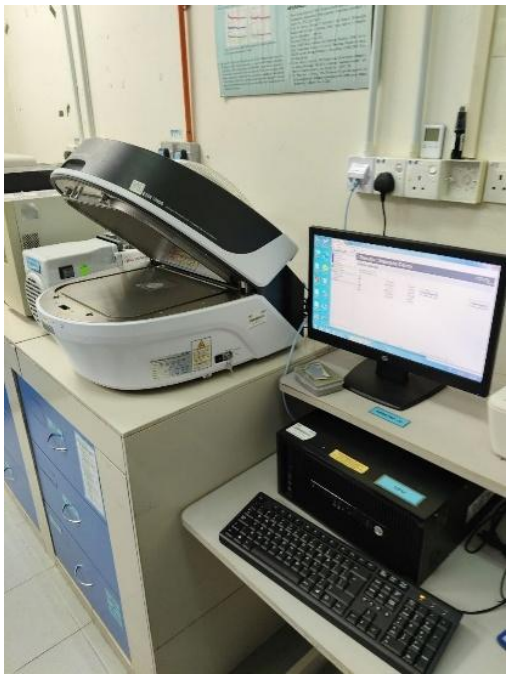


Figure 4: Shimadzu / EDX7000

Data collection

The data collection process will consist of two main steps: pre-radiation exposure and post-radiation exposure. Initially, the $CaSiO_3$ nanomaterials will undergo thorough examination through various tests before any radiation exposure. These tests will include radiation detectors to measure initial radiation levels, Energy Dispersive X-Ray Fluorescence (EDXRF) to determine elemental composition, FESEM-EDX to analyze surface morphology, and X-Ray Diffraction (XRD) to study crystal structure. These tests will provide a comprehensive understanding of the material's properties prior to radiation exposure.

Subsequently, the recycled $CaSiO_3$ nanomaterials will be subjected to gamma rays emitted from a Cobalt-60 (Co-60) source to observe the effects of high-energy radiation on the material. The same tests, FESEM-EDX and XRD, will be repeated post-radiation exposure to compare any structural or surface changes that may have occurred.

RESULTS AND DISCUSSION

XRF Analysis

Table 1 presents the XRF analysis results for $CaSiO_3$ samples pre-gamma irradiation. Silicon (Si) and calcium (Ca) were the predominant elements, comprising approximately 58.7% and 37.0% of the composition, respectively. These concentrations align with the expected stoichiometry of $CaSiO_3$, confirming successful compound formation. Minor elements like potassium (K), iron (Fe), sulfur (S), chlorine (Cl), silver (Ag), titanium (Ti), and others were detected in trace amounts (<2%), likely originating from feedstock recycling or processing contamination. The presence of high-energy characteristic peaks, such as Si Kα (1.74 keV) and Ca Kα (3.70 keV), ensured accurate elemental identification. Understanding the elemental distribution is crucial for assessing the suitability of recycled materials for high-performance applications, particularly in radiation environments.

Table 1: XRF results for $CaSiO_3$ samples before exposure

Analytical	Concentration (%)	Analytical	Energy (keV)
Si	58.732	Si Kα	1.74
Ca	37.048	Ca Kα	3.70
K	1.629	K Kα	3.32
Fe	1.029	Fe Kα	6.40
S	0.699	S Kα	2.30
Cl	0.274	Cl Kα	2.62
Ag	0.145	Ag Kα	22.02
Ti	0.143	Ti Kα	4.52
Mn	0.093	Mn Kα	5.90
Cr	0.067	Cr Kα	5.42
Sr	0.063	Sr Kα	14.16
Cu	0.040	Cu Kα	8.06
Rb	0.023	Rb Kα	13.36
Zn	0.016	Zn Kα	8.66

XRD Analysis

Figure 5(a) displays the XRD patterns of $CaSiO_3$ samples in grounded and ultra-grounded states. The ultra-grounded sample revealed sharper diffraction peaks, notably around 25° and 43°, indicating improved crystallinity. Both samples exhibited broad humps within 10°–35°, suggesting an amorphous phase presence, consistent with previous findings (Gimenez-Carbo et al., 2021). The detected phases were assigned to SiO_2, $CaCO_3$, and $CaSiO_3$, which should be indicated in the figure legend for clarity.

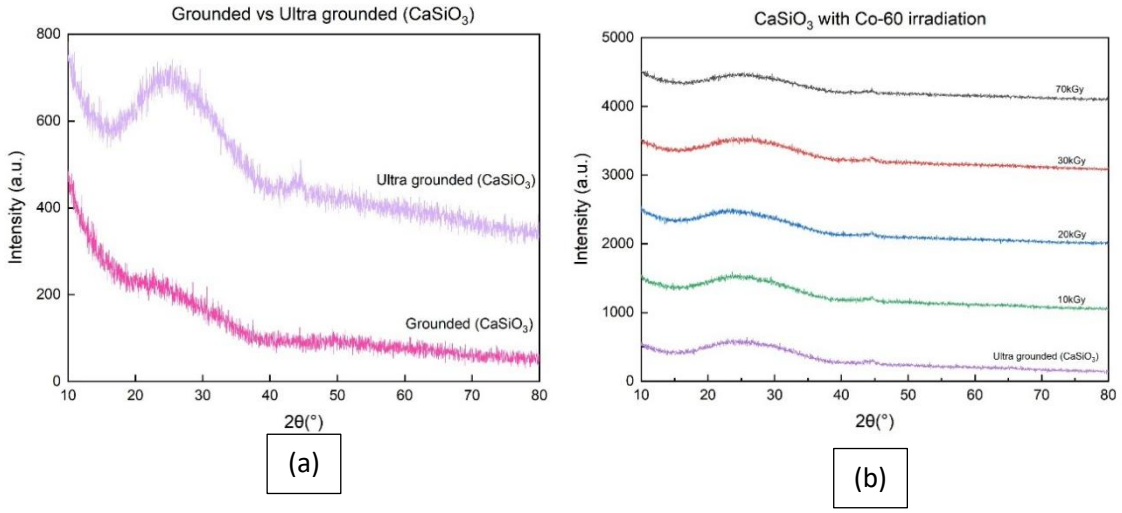


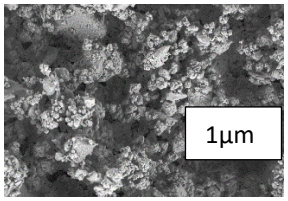
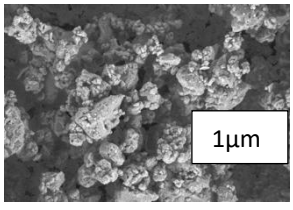
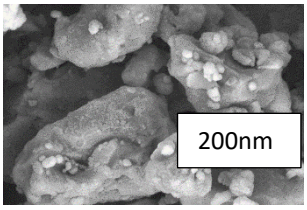
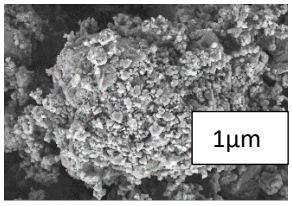
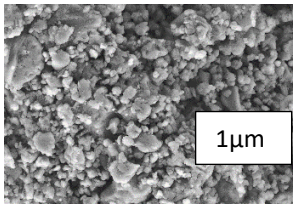
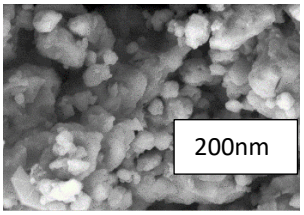
Figure 5 (a): XRD patterns of ground and ultra-ground $CaSiO_3$ samples
Figure 5 (b) shows the XRD patterns of $CaSiO_3$ after Co-60 gamma irradiation at doses ranging from 0 to 70 kGy.

As the irradiation dose increased, the diffraction peaks became broader and less intense, while the background signal gradually increased. These features suggest a reduction in crystallinity and the formation of radiation-induced defects, although the main crystalline phase remained detectable throughout the dose range studied, as previously observed in similar materials (Gonzales-Lorenzo et al., 2018).

FESEM Morphology and Particle Size Analysis

Table 2 and Table 3 present SEM images and average particle sizes of $CaSiO_3$ samples before and after gamma irradiation. Pre-irradiation samples showed rough, porous, and irregular particles. At 20 kGy, increased grain compaction and clustering were observed. By 30 and 40 kGy, partial sintering, smoothing, and densification became more evident. At 70 kGy, larger and more compact particles indicated significant radiation-induced diffusion and agglomeration.

Table 2: FESEM figures for $CaSiO_3$ samples before and after exposure

Magnification / **Radiation range**	**5kx**	**10kx**	**20kx**
Grounded			
20 kGy			

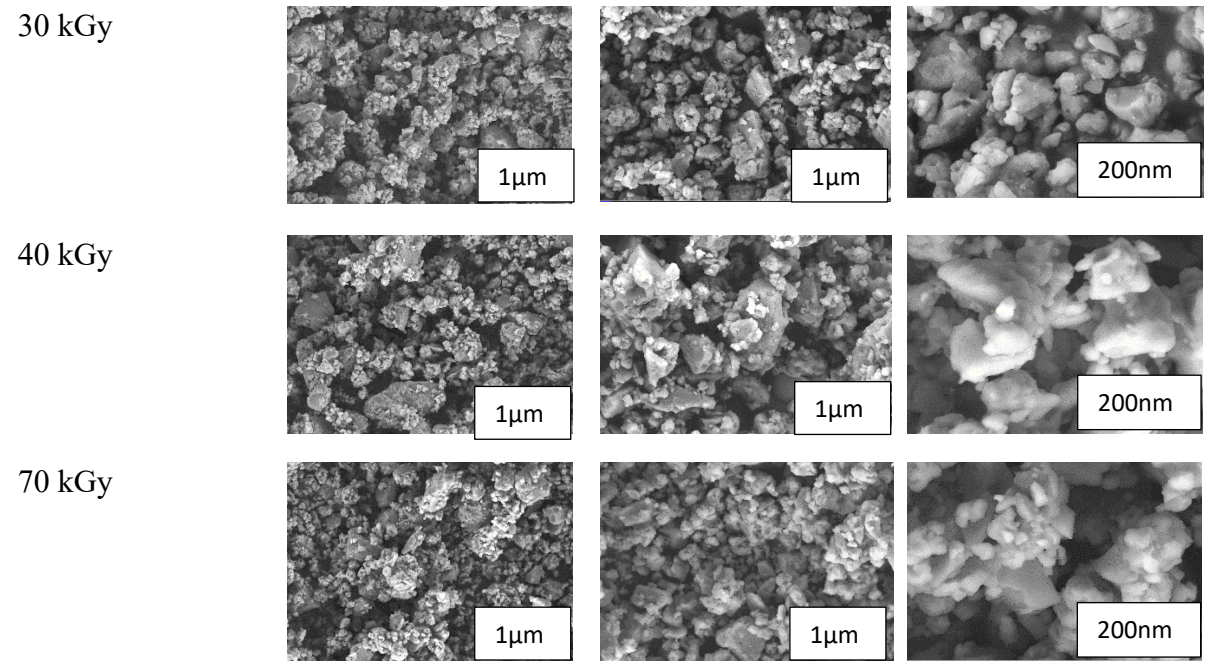


Table 3: Particle size distribution of $CaSiO_3$ samples measured at 20kx magnification

Magnification	**Control (µm)**	**20kGy (µm)**	**30kGy (µm)**	**40kGy (µm)**	**70kGy (µm)**
20kx	0.107	0.166	0.151	0.170	0.180

Table 3 summarises the particle sizes measured at 20kx magnification for the control and irradiated samples. The average particle size increased from 0.107 µm in the control sample to 0.166 µm after irradiation at 20 kGy, indicating initial agglomeration. The values at 30 and 40 kGy were 0.151 µm and 0.170 µm, respectively, while the 70 kGy sample showed the largest particle size of 0.180 µm. These results align with observed FESEM morphology and confirm gamma irradiation influences microstructure significantly.

CONCLUSION

The first objective was successfully achieved through this study: ultrafine calcium silicate ($CaSiO_3$) nanomaterials were synthesized from recycled glass slides via dry mechanical processing, including ball milling. The resulting gray powder showed sufficient fineness and homogeneity for further material testing.

Second, structural and morphological characterization confirmed the properties of the synthesized materials as expected. FESEM analysis revealed that the unirradiated sample consisted of irregular, porous particles with rough surfaces. Upon exposure to increasing doses of gamma radiation (20–70 kGy), the particles showed enhanced density, reduced porosity, and gradual grain growth with particle sizes at 20kx magnification increasing overall from 0.107 µm in the control sample to 0.180 µm at 70 kGy.

Third, the effect of Co-60 gamma irradiation was examined by XRD spectroscopy. The XRD pattern showed sharper peaks in the irradiated samples, indicating increased crystallinity.

In summary, this study demonstrated that recycled glass slides can be effectively modified into structurally stable $CaSiO_3$ nanomaterials. The materials exhibited durability under gamma irradiation, maintaining phase integrity and functional morphology. These findings support the potential use of recycled $CaSiO_3$ in radiation-resistant materials for environmental and industrial technologies.

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