



## Effect of Particle Size on Material Recovery and Elemental Distribution in the Recycling Of End-Of-Life Polycrystalline Silicon Photovoltaic Modules

\*<sup>1</sup>Albashir Yasir, <sup>2</sup>S.M Gumel, <sup>1</sup>Waheed Islamiyat O, <sup>1</sup>Tahir Ibrahim D, <sup>1</sup>Ndagi Mohammad, <sup>3</sup>Sule Erten Ela and <sup>4</sup>Hauwa Yahaya Umar

<sup>1</sup>Department of Pure and Industrial Chemistry, Bayero University, Kano State, Nigeria.

<sup>2</sup>Department of Industrial Chemistry, Federal University, Dutsinma, Dutsinma, Katsina State, Nigeria.

<sup>3</sup>Department of Chemistry, Federal University, Dutsinma, Dutsinma, Katsina State, Nigeria.

<sup>4</sup>Ege University, Solar Energy Institute, Bornova, 35100 Izmir, Turkey.

<sup>5</sup>Department of Chemistry, College of Education (T), Gombe State, Nigeria.

\*Corresponding authors' email: [ayasir@fudutsinma.edu.ng](mailto:ayasir@fudutsinma.edu.ng)

### ABSTRACT

The rapid deployment of photovoltaic (PV) systems has raised concerns regarding the management of end-of-life (EoL) solar modules. This study investigates the recovery efficiency of polycrystalline silicon PV panels subjected to sequential mechanical and thermal treatments, with emphasis on the influence of particle size distribution on downstream chemical and structural characterization. Disassembled PV modules ( $\approx 2$  kg per test) were crushed using mortar-and-pestle and hammer-impact methods, followed by sieving into coarse ( $>5$  mm), medium (1–5 mm), and fine (0.08–0.40 mm) fractions. Thermal treatment at 650 °C facilitated the removal of encapsulant materials, after which the recovered fractions were characterized using X-ray diffraction (XRD) and X-ray fluorescence (XRF) analyses. Results indicated improved recovery efficiency in finer fractions, with glass yields increasing from 71.3% in the coarse fraction to 83.4% in the fine fraction. XRD analysis revealed dominant phases of quartz ( $\text{SiO}_2$ ), albite ( $\text{NaAlSi}_3\text{O}_8$ ), and lime ( $\text{CaO}$ ), while XRF analysis confirmed enrichment of silicon (Si) and antimony (Sb) in finer fractions. Statistical analysis using one-way ANOVA ( $p < 0.05$ ) showed significant differences among particle size fractions in terms of recovery yield and elemental composition, indicating the practical importance of particle size optimization in PV recycling processes. The findings demonstrate that particle size engineering plays a critical role in improving material recovery efficiency and supports the development of sustainable circular photovoltaic recycling strategies.

**Keywords:** Photovoltaic Waste; Particle Size Distribution; Recycling; XRD; XRF; Circular Photovoltaics; Material Recovery

### INTRODUCTION

The rapid expansion of photovoltaic (PV) technology has resulted in unprecedented growth in solar module deployment worldwide. Global PV capacity surpassed 1 TW in 2022 and is projected to exceed 4.5 TW by 2030, leading to a substantial increase in end-of-life (EoL) PV waste generation. Estimates suggest that cumulative PV waste could exceed 78 million tonnes globally by 2050 (International Renewable Energy Agency [IRENA], 2016). Effective management of EoL PV modules is therefore essential for minimizing environmental impacts and supporting circular economy objectives.

Improper disposal of PV modules can result in the loss of valuable materials and the release of hazardous substances such as lead, cadmium, and antimony into the environment. Consequently, recycling has emerged as a critical strategy for sustainable PV waste management, enabling the recovery of valuable materials including glass, silicon, aluminum, silver, and other metals.

Crystalline silicon (c-Si) PV modules, which dominate the global solar market, are primarily composed of glass, aluminum frames, silicon wafers, polymeric encapsulants, and smaller quantities of metallic components (Dias et al., 2016). Various recycling approaches involving mechanical, thermal, and chemical treatments have been developed to recover these materials efficiently (Granata et al., 2014; Pagnanelli et al., 2017). Mechanical pretreatment methods such as hammer and rotor crushing, followed by thermal delamination, have demonstrated effectiveness in improving

material liberation and enhancing recovery efficiency (Fiandra et al., 2019).

Despite recent advances in PV recycling technologies, the influence of particle size distribution on material recovery efficiency remains insufficiently investigated. Particle size reduction can improve liberation of embedded materials and enhance downstream recovery processes; however, finer particles may also concentrate hazardous elements and increase handling risks during recycling operations (Kang et al., 2012; Xu et al., 2018). Understanding the relationship between particle size distribution and material recovery is therefore essential for developing efficient and environmentally safe recycling systems.

This study investigates the recovery efficiency of mechanically crushed c-Si PV modules by comparing coarse ( $>5$  mm), medium (1–5 mm), and fine (0.08–0.4 mm) particle fractions obtained after sequential mechanical and thermal treatments. Structural and elemental characterization using X-ray diffraction (XRD) and X-ray fluorescence (XRF) analyses was performed to evaluate the effect of particle size distribution on material composition and recycling efficiency.

### MATERIALS AND METHODS

#### Materials

End-of-life polycrystalline silicon photovoltaic (c-Si PV) modules were used as the input material for this study. Approximately 2 kg of PV module material was processed per experimental run. Prior to treatment, modules were manually

dismantled to remove aluminum frames, junction boxes, and external electrical components.

The following equipment was used during the mechanical pretreatment process:

- i. Two-blade rotor crusher
- ii. Hammer crusher equipped with a 5 mm sieve
- iii. Mortar and pestle
- iv. Standard laboratory sieves (8 mm, 5 mm, 1 mm, 0.4 mm, and 0.08 mm)
- v. Sample collection bottles

### Chemicals

Analytical-grade reagents were used throughout the study:

- i. Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, 96%, Sigma-Aldrich)
- ii. Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>, 35% v/v, Sigma-Aldrich)
- iii. Deionized water for sample preparation and dilution

### Instruments and Analytical Equipment

The following instruments were employed for thermal treatment and material characterization:

- i. Resistance furnace (maximum operating temperature: 650 °C)
- ii. X-ray diffractometer (XRD, PANalytical X'Pert Pro, Co-K $\alpha$  radiation,  $\lambda = 1.789 \text{ \AA}$ )
- iii. X-ray fluorescence spectrometer (XRF, SPECTRO XEPOS)
- iv. Microwave digestion system (Milestone Ethos 900)
- v. Atomic Absorption Spectrophotometer (AAS)
- vi. Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES, Varian Vista-MPX CCD simultaneous)

### Recycling Procedure

The recycling process consisted of sequential mechanical pretreatment, thermal delamination, chemical digestion, and physicochemical characterization.

### Mechanical Pretreatment

The dismantled PV modules were initially fragmented using a two-blade rotor crusher to reduce the module size and facilitate subsequent liberation of encapsulated materials. Secondary crushing was performed using a hammer crusher fitted with a 5 mm sieve.

To ensure reproducibility, each crushing stage was performed under identical operating conditions with three repeated crushing cycles per batch. The crushed material was subsequently sieved into three principal particle size fractions:

- i. Coarse fraction: >5 mm
- ii. Medium fraction: 1–5 mm
- iii. Fine fraction: 0.08–0.4 mm

The fractions were weighed to determine mass distribution and material recovery yield.

### Thermal Treatment

Thermal treatment was carried out in a resistance furnace at 650 °C for 1 h under atmospheric air conditions to degrade and remove the ethylene-vinyl acetate (EVA) encapsulant layer binding the PV components.

The furnace heating rate was maintained at 10 °C min<sup>-1</sup> from ambient temperature to the target temperature. After cooling

to room temperature, the thermally treated material was sieved into five size fractions using standard laboratory sieves (8 mm, 5 mm, 1 mm, 0.4 mm, and 0.08 mm). Fractions with particle sizes  $\leq 1$  mm were selected for subsequent chemical and structural analyses due to their higher liberation efficiency.

### Chemical Digestion

Glass-rich fractions (1–0.08 mm) were subjected to microwave-assisted acid digestion. Approximately 0.2 g of each sample was treated with a mixture of 9 mL concentrated H<sub>2</sub>SO<sub>4</sub> (96%) and 1 mL H<sub>2</sub>O<sub>2</sub> (35%) in Teflon digestion vessels.

Digestion was performed at 220 °C using a Milestone Ethos 900 microwave digestion system. After digestion, the solutions were cooled, filtered, and diluted with deionized water prior to elemental analysis.

To ensure analytical reliability, all digestion experiments were conducted in triplicate, and blank digestions were included for quality control.

### Material Characterization

#### Mass Balance Analysis

Recovery efficiency was evaluated gravimetrically by measuring the mass distribution of sieved fractions after crushing and thermal treatment.

#### X-ray Diffraction (XRD)

Crystalline phases present in the recovered materials were identified using a PANalytical X'Pert Pro diffractometer with Co-K $\alpha$  radiation ( $\lambda = 1.789 \text{ \AA}$ ). Data were collected over a  $2\theta$  range of 10–80° with a step size of 0.02° and a scanning rate of 2° min<sup>-1</sup>.

#### X-ray Fluorescence (XRF)

Elemental composition of the recovered fractions was determined using a SPECTRO XEPOS XRF spectrometer. Measurements were performed on homogenized powdered samples prepared according to the manufacturer's recommendations.

#### ICP-OES and AAS Analysis

Digested solutions were analyzed for trace metals including Pb, Sb, Ag, and Cd using ICP-OES and AAS techniques. ICP-OES was employed for multi-element quantitative analysis, while AAS was used to validate selected heavy metal concentrations.

Calibration standards and procedural blanks were analyzed alongside samples to ensure measurement accuracy.

#### Statistical Analysis

All experiments were conducted in triplicate, and results are presented as mean  $\pm$  standard deviation.

One-way analysis of variance (ANOVA) was used to evaluate the effect of particle size on material recovery efficiency and elemental concentration. Tukey's honestly significant difference (HSD) post-hoc test was applied to compare differences between mean values at a significance level of  $\alpha = 0.05$ .

Statistical analyses were performed using OriginPro 2023 software.

**Table 1: Comparative Summary of Recycling Procedures Adopted for Different Particle Size Fractions**

Processing Step	D5 (Coarse Fraction, >5 mm)	D1 (Medium Fraction, 1–5 mm)	D0.08 (Fine Fraction, 0.08–0.4 mm)
Input Material	Polycrystalline silicon PV module ( $\approx 2$ kg)	Polycrystalline silicon PV module ( $\approx 2$ kg)	Polycrystalline silicon PV module ( $\approx 2$ kg)
Manual Disassembly	Removal of aluminum frame, junction box, and external electrical components	Removal of aluminum frame, junction box, and external electrical components	Removal of aluminum frame, junction box, and external electrical components
Primary Crushing	Two-blade rotor crusher	Two-blade rotor crusher	Two-blade rotor crusher
Secondary Crushing	Hammer crusher fitted with 5 mm sieve	Hammer crusher fitted with 5 mm sieve	Hammer crusher fitted with 5 mm sieve
Thermal Treatment	650 °C for 1 h in resistance furnace under atmospheric air	650 °C for 1 h in resistance furnace under atmospheric air	650 °C for 1 h in resistance furnace under atmospheric air
Sieving Procedure	Sieved into 8 mm, 5 mm, 1 mm, 0.4 mm, and 0.08 mm fractions	Sieved into 8 mm, 5 mm, 1 mm, 0.4 mm, and 0.08 mm fractions	Sieved into 8 mm, 5 mm, 1 mm, 0.4 mm, and 0.08 mm fractions
Selected Fraction	Particles >5 mm retained for analysis	Particles between 1–5 mm retained for analysis	Particles between 0.08–0.4 mm retained for analysis
Chemical Digestion	0.2 g sample digested with 9 mL H <sub>2</sub> SO <sub>4</sub> (96%) and 1 mL H <sub>2</sub> O <sub>2</sub> (35%) at 220 °C using microwave digestion	Same digestion conditions applied	Same digestion conditions applied
Characterization Techniques	XRD, XRF, ICP-OES, and AAS	XRD, XRF, ICP-OES, and AAS	XRD, XRF, ICP-OES, and AAS
Measured Parameters	Mass recovery yield, crystalline phases, and elemental composition	Mass recovery yield, crystalline phases, and elemental composition	Mass recovery yield, crystalline phases, and elemental composition

**Note:** D5, D1, and D0.08 represent coarse (>5 mm), medium (1–5 mm), and fine (0.08–0.4 mm) particle size fractions obtained after sequential crushing and sieving of polycrystalline silicon photovoltaic modules.

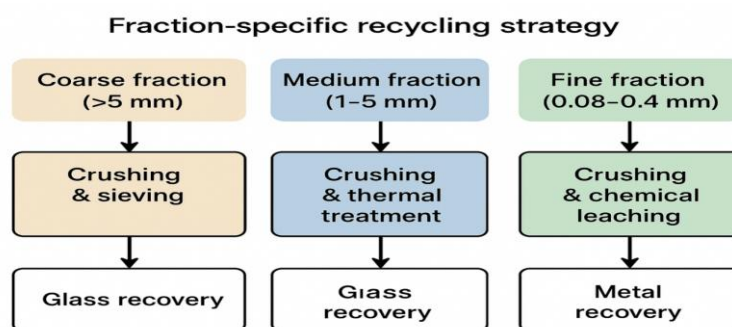


Figure 1: Fraction Specific Recycling Strategy

## RESULTS AND DISCUSSION

### Effect of Particle Size on Glass Recovery

The effect of particle size on glass-rich fraction recovery after mechanical and thermal treatment is presented in Table 2. Glass recovery increased significantly with decreasing particle size, with the fine fraction (D0.08, 0.08–0.4 mm) exhibiting the highest recovery yield of  $83.4 \pm 2.4\%$ , followed by the medium fraction (D1, 1–5 mm) at  $77.8 \pm 1.7\%$ , and the coarse fraction (D5, >5 mm) at  $71.3 \pm 2.1\%$ .

The observed increase in recovery efficiency with decreasing particle size can be attributed to enhanced liberation of glass particles from encapsulant and metallic components during

crushing and sieving operations. Finer fractions exhibited reduced contamination by polymeric residues and improved separation efficiency after thermal delamination.

One-way ANOVA revealed statistically significant differences among the particle size fractions ( $F(2,6) = 102.5$ ,  $p < 0.0001$ ). Tukey's HSD post-hoc analysis confirmed that all pairwise comparisons were statistically significant ( $p < 0.001$ ), indicating that particle size had a strong influence on recovery efficiency.

These findings are consistent with previous studies reporting improved liberation and material recovery at reduced particle sizes during PV recycling processes.

**Table 2: Mass Balance and Glass-Rich Fraction Recovery at Different Particle Sizes**

Particle Size Fraction	Feed Mass (g)	Mass Recovered (g)	Mass Loss After Furnace (%)	Glass-Rich Fraction Yield (%)
D5 (>5 mm)	2000	$1625 \pm 30$	$12.5 \pm 0.6$	$71.3 \pm 2.1$
D1 (1–5 mm)	2000	$1538 \pm 25$	$14.2 \pm 0.5$	$77.8 \pm 1.7$
D0.08 (0.08–0.4 mm)	2000	$1486 \pm 28$	$16.9 \pm 0.7$	$83.4 \pm 2.4$

**Table 3: Tukey's HSD Post-Hoc Comparison of Recovery Yields**

Comparison	Mean Difference	p-value	95% CI Lower	95% CI Upper	Significant ( $\alpha = 0.05$ )
D5 vs D0.08	+12.07	<0.001	11.53	12.60	Yes
D5 vs D1	+6.50	<0.001	5.97	7.03	Yes
D1 vs D0.08	-5.57	<0.001	-6.10	-5.03	Yes

### XRD Analysis of Recovered Fractions

The XRD patterns of the recovered fractions are presented in Figures 3–5. Quartz (SiO<sub>2</sub>) and albite (NaAlSi<sub>3</sub>O<sub>8</sub>) were identified as the dominant crystalline phases across all particle size fractions, while minor lime (CaO) peaks were also detected.

The fine fraction (D0.08) exhibited sharper and more intense diffraction peaks compared with the coarse fraction, indicating increased crystallinity and improved liberation of glass-rich phases after crushing and thermal treatment. Minor

peaks potentially attributable to Sb-containing phases were also observed in the fine fraction. However, due to the low intensity of these peaks, definitive identification of Sb<sub>2</sub>O<sub>3</sub> requires further confirmation using complementary techniques such as SEM-EDS or Rietveld refinement analysis.

The relative phase abundance presented in Table 4 should therefore be regarded as semi-quantitative estimations derived from peak intensity comparison rather than absolute quantitative phase analysis.

**Table 4: XRD-Identified Crystalline Phases in Recovered Fractions**

Particle Size Fraction	Main Crystalline Phases Detected	Relative Abundance (Semi-Quantitative %)
D5 (>5 mm)	Albite (NaAlSi <sub>3</sub> O <sub>8</sub> ), Quartz (SiO <sub>2</sub> ), Lime (CaO)	Albite 66%, Quartz 23%, Lime 11%
D1 (1–5 mm)	Quartz (SiO <sub>2</sub> ), Albite (NaAlSi <sub>3</sub> O <sub>8</sub> ), Lime (CaO)	Quartz 69%, Albite 20%, Lime 11%
D0.08 (0.08–0.4 mm)	Quartz (SiO <sub>2</sub> ), Lime (CaO), minor Sb-containing phases	Quartz 74%, Lime 15%, Minor traces

### XRF Elemental Composition

The elemental compositions of the recovered fractions determined by XRF analysis are summarized in Table 5. Silicon, calcium, and antimony were identified as the dominant constituents in all particle size fractions, while Pb, Fe, and Ag occurred in lower concentrations.

The concentration of Si increased progressively with decreasing particle size, from 26.8 ± 0.5 wt% in the coarse fraction to 29.9 ± 0.7 wt. % in the fine fraction. This trend indicates enrichment of silica-rich glass phases in finer

particles due to enhanced liberation efficiency during crushing.

Similarly, Sb concentration increased slightly in finer fractions, suggesting preferential partitioning of Sb-containing glass additives into smaller particle sizes during mechanical treatment. Trace concentrations of Pb and Ag were also detected, indicating partial retention of metallic impurities within the recovered glass-rich material.

These results agree with previous reports indicating enrichment of heavy metals and glass-forming constituents in fine PV recycling fractions.

**Table 5: XRF Major Element Composition of Recovered Fractions**

Element (wt. %)	D5 (>5 mm)	D1 (1–5 mm)	D0.08 (0.08–0.4 mm)
Si	26.8 ± 0.5	28.4 ± 0.6	29.9 ± 0.7
Al	1.10 ± 0.04	1.28 ± 0.05	1.36 ± 0.04
Ca	5.35 ± 0.09	5.66 ± 0.08	5.88 ± 0.10
Sb	20.9 ± 0.4	21.5 ± 0.5	22.2 ± 0.5
Fe	0.041 ± 0.002	0.046 ± 0.002	0.050 ± 0.003
Pb	0.022 ± 0.001	0.024 ± 0.001	0.027 ± 0.001
Ag	0.162 ± 0.004	0.171 ± 0.005	0.183 ± 0.006

### ICP-OES Analysis of Trace Metals

ICP-OES analysis of digested fractions revealed the presence of trace metals including Pb, Ag, Sb, Cu, and Zn (Table 6). Antimony exhibited the highest concentration among the analyzed metals and increased from 1,850 ± 40 ppm in the coarse fraction to 2,115 ± 55 ppm in the fine fraction.

Lead and silver concentrations also increased with decreasing particle size, indicating progressive enrichment of metallic

constituents within finer fractions during crushing and sieving. Cadmium concentrations remained below detectable limits in most fractions, with only trace levels detected in D0.08.

The enrichment of heavy metals in finer fractions highlights the need for controlled handling and safe recycling practices to minimize occupational and environmental exposure risks.

**Table 6: ICP-OES Analysis of Trace Metals in Recovered Fractions**

Metal (ppm)	D5 (>5 mm)	D1 (1–5 mm)	D0.08 (0.08–0.4 mm)
Cd	<0.05	<0.05	0.08
Pb	92 ± 3	101 ± 4	117 ± 5
Ag	68 ± 2	74 ± 2	81 ± 3
Sb	1850 ± 40	1960 ± 50	2115 ± 55
Cu	11 ± 1	13 ± 1	16 ± 1
Zn	5.1 ± 0.3	5.6 ± 0.3	6.4 ± 0.4

### Statistical Evaluation

Statistical analysis confirmed that particle size significantly influenced both recovery efficiency and elemental composition of the recycled fractions ( $p < 0.05$ ). Fine

fractions differed significantly from coarse and medium fractions in terms of glass recovery yield and concentrations of Si and Sb.

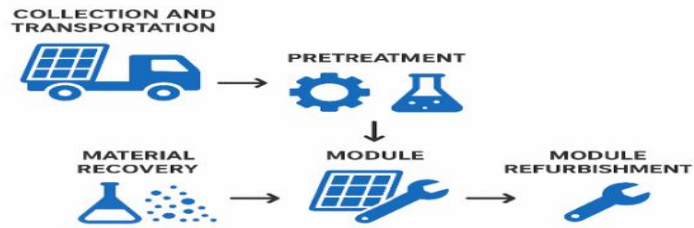


Figure 2: Fraction Obtained After Crushing and Thermal Treatment of Polycrystalline Module

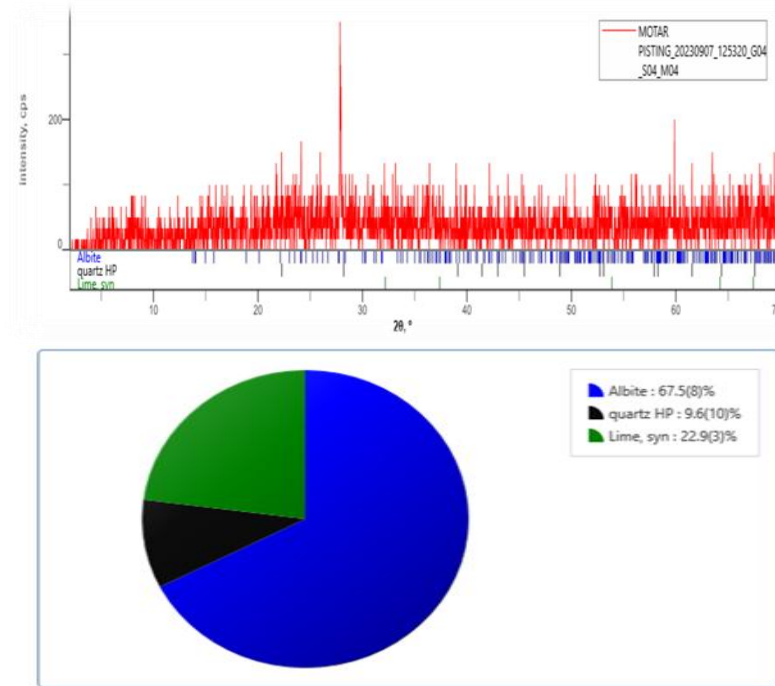


Figure 3: Size Distribution after Treatment of Polycrystalline Module by Mortar Pistol Crushing

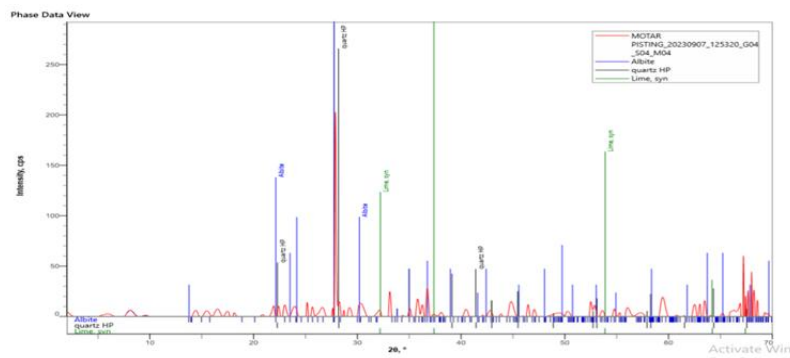


Figure 4: XRD Pattern of Obtained Products after Crushing Plus Hammer Crushin

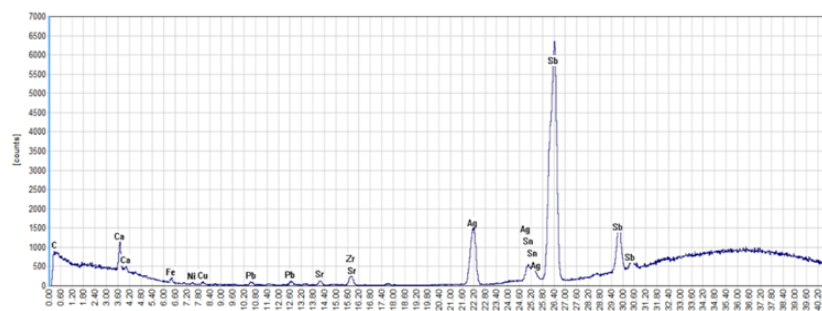


Figure 5: XRD Pattern of Obtained Products after Crushing plus Thermal Treatment

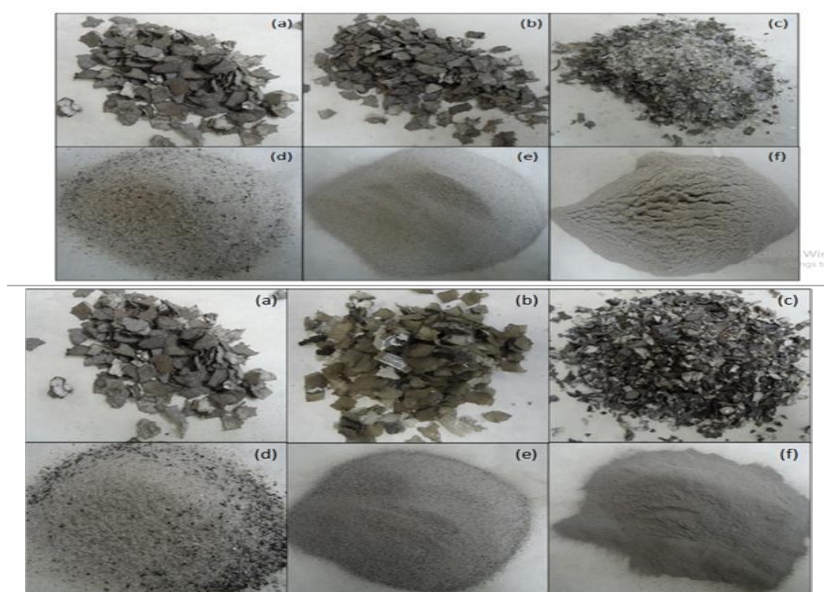


Figure 6: Glass-rich Fractions Obtained after Crushing and Thermal Treatment of Polycrystalline Silicon Photovoltaic (c-Si PV) Modules

Table 7: Semi-Quantitative Phase Distribution Obtained from XRD Analysis

Dataset/Weight Fraction (wt. %)	Albite (%)	Quartz (%)	Lime (%)
Mortar–pestle crushed fraction	67.5 ± 0.8	9.6 ± 1.0	22.9 ± 0.3

Table 8: Representative Peak List from XRD Analysis

No.	2θ (°)	d-spacing (Å)	Peak Height (cps)	FWHM	Intensity (cps)	Crystallite Size (Å)
1	27.842 ± 0.008	3.2017 ± 0.0009	302 ± 49	0.047	24 ± 3	1805 ± 410

Table 9: Phase Identification from XRD Analysis

No.	2θ (°)	Identified Phase	Chemical Formula	PDF Card Number	Profile Type
1	27.842 ± 0.008	Quartz	SiO <sub>2</sub>	01-083-0542	Split pseudo-Voigt

Table 10: Qualitative Phase Analysis of Recovered Fractions

Phase Name	Chemical Formula	Figure of Merit	Space Group	Database Card Number
Albite	NaAlSi <sub>3</sub> O <sub>8</sub>	2.770	C-1	00-001-0739
Quartz	SiO <sub>2</sub>	0.987	P3121	01-083-0542
Lime	CaO	2.770	Fm-3m	00-004-0777

## Discussion

### Effect of Particle Size on Mass Recovery and Glass Yield

The results demonstrate that particle size reduction significantly influenced glass recovery efficiency during recycling of polycrystalline silicon photovoltaic (c-Si PV) modules. Fine fractions (0.08–0.4 mm) achieved the highest glass-rich fraction yield (83.4 ± 2.4%), followed by medium fractions (77.8 ± 1.7%) and coarse fractions (71.3 ± 2.1%). The improved recovery observed in finer fractions is primarily attributed to enhanced liberation of glass particles from EVA encapsulant residues and metallic interlayers during crushing and thermal treatment. Reduction in particle size increases the exposed surface area and weakens bonding between glass and polymeric phases, thereby improving separation efficiency during sieving. Similar liberation-enhanced recovery trends have been reported in previous PV recycling investigations and other comminution-based waste recovery systems.

The higher mass loss observed after furnace treatment in fine fractions may be associated with increased decomposition of residual EVA and volatilization of minor organic components due to the larger reactive surface area of smaller particles. Although fine grinding improved recovery efficiency,

additional milling and sieving stages may increase operational energy demand and dust generation in industrial-scale recycling systems. Consequently, the economic benefit of higher recovery must be balanced against processing costs and occupational safety considerations.

Statistical analysis confirmed that particle size had a significant effect on glass recovery efficiency ( $F(2,6) = 102.5$ ,  $p < 0.0001$ ). However, beyond statistical significance, the practical implication is that finer particle production may enhance downstream material purification and critical metal recovery, particularly for glass-rich fractions intended for secondary reuse.

### Elemental Distribution and Enrichment Behavior

XRF analysis revealed progressive enrichment of Si and Sb with decreasing particle size. Silicon concentration increased from 26.8 wt% in coarse fractions to 29.9 wt% in fine fractions, while Sb increased from 20.9 wt% to 22.2 wt%.

The increase in Si concentration in fine fractions indicates preferential liberation of silica-rich glass phases during crushing. This trend suggests that mechanical size reduction promotes selective separation of glass components from metallic and polymeric materials. Similar enrichment

behavior has been reported in mechanically treated PV waste, where brittle glass phases preferentially fracture into smaller particles.

The enrichment of Sb and other trace metals such as Pb and Ag in fine fractions is particularly important from both environmental and resource recovery perspectives. Antimony-containing compounds are commonly associated with PV glass additives and flame-retardant materials. During crushing, these phases may preferentially accumulate in finer particles because of differential fracture behavior and increased surface exposure.

Although the observed increase in heavy metal concentration was relatively moderate, fine fractions may represent a potentially hazardous dust stream if improperly handled during large-scale recycling. At the same time, the concentration of valuable metals within these fractions could improve the feasibility of downstream hydrometallurgical recovery processes.

#### **Phase Evolution and XRD Interpretation**

XRD analysis identified quartz ( $\text{SiO}_2$ ), albite ( $\text{NaAlSi}_3\text{O}_8$ ), and lime (CaO) as the dominant crystalline phases across all particle size fractions. Quartz peaks became more prominent in finer fractions, reflecting increased exposure of silica-rich glass material after mechanical liberation.

However, caution should be exercised when interpreting increased peak sharpness as evidence of higher crystallinity. The observed sharpening of diffraction peaks in fine fractions may partially result from particle-size-related diffraction effects and improved sample homogeneity rather than a true increase in crystalline order. Therefore, the XRD results should be interpreted primarily as evidence of enhanced phase exposure and liberation rather than structural crystallization. Minor Sb-containing phases were detected in fine fractions after thermal treatment. These phases may be associated with oxidation or concentration of antimony-containing additives during milling and furnace treatment. Nevertheless, definitive identification of  $\text{Sb}_2\text{O}_3$  requires confirmation through complementary analytical techniques such as SEM-EDS, Raman spectroscopy, or Rietveld-refined XRD analysis.

The progressive dominance of quartz in finer fractions indicates that comminution effectively separates brittle glass-rich phases from tougher composite layers within the PV module structure. This observation has practical implications for selective liberation strategies in industrial PV recycling operations.

#### **Heavy Metal Concentration and Environmental Implications**

ICP-OES analysis demonstrated progressive enrichment of Pb, Ag, Sb, Cu, and Zn in the finer particle fractions. Lead concentration increased from 92 ppm in coarse fractions to 117 ppm in fine fractions, while Sb exceeded 2,100 ppm in the finest fraction.

This enrichment behavior indicates that fine particles may act as sinks for hazardous and valuable metals during mechanical processing. From a recycling perspective, this concentration effect may be advantageous because it enables targeted recovery of economically important metals from smaller high-grade fractions. Concentrating metals into fine fractions could reduce reagent consumption during subsequent leaching or hydrometallurgical extraction stages.

Conversely, the accumulation of toxic elements in fine powders raises concerns regarding occupational exposure, airborne particulate emissions, and environmental contamination during industrial handling. Fine particulate matter containing Pb and Sb may pose inhalation risks if

adequate dust collection and containment systems are not implemented.

Although the present study identified metal enrichment trends, environmental mobility and leachability of these elements were not evaluated. Future studies should therefore include leaching tests such as TCLP or SPLP analysis to assess the environmental behavior of recovered fine fractions under disposal or reuse conditions.

#### **Implications for PV Recycling Process Design**

The combined mass balance, XRF, XRD, and ICP-OES results demonstrate that particle size is a critical parameter governing both recovery efficiency and contaminant distribution during PV recycling.

Fine fractions provided the highest glass recovery and greatest enrichment of valuable metals, indicating strong potential for downstream resource recovery. However, the increased concentration of hazardous metals and the likelihood of higher dust generation may complicate industrial handling and environmental management.

Medium-sized fractions (1–5 mm) may therefore represent a practical compromise between liberation efficiency, processing cost, and occupational safety. These fractions achieved relatively high recovery efficiency while potentially minimizing excessive energy consumption associated with ultrafine grinding.

The findings suggest that an optimized PV recycling strategy should adopt fraction-specific processing routes, including:

- i. selective grinding to avoid excessive fine dust formation,
- ii. targeted hydrometallurgical treatment of fine metal-rich fractions,
- iii. controlled thermal delamination for improved liberation efficiency,
- iv. And secondary reprocessing of coarse fractions to maximize overall material recovery.

Such an integrated approach could improve the economic and environmental sustainability of crystalline silicon PV recycling systems.

#### **Statistical Interpretation of Particle Size Effects**

The one-way ANOVA and Tukey HSD analyses confirmed that particle size reduction significantly influenced glass recovery efficiency and elemental distribution among the recovered fractions.

While statistical significance demonstrates measurable differences between fractions, the practical significance lies in the process engineering implications. The approximately 12% increase in recovery observed between coarse and fine fractions represents a potentially meaningful improvement for industrial recycling systems, particularly when processing large PV waste volumes.

However, the benefits of increased liberation must be balanced against additional grinding energy requirements, dust management costs, and heavy metal concentration in fine fractions. Therefore, optimization of particle size should consider both recovery efficiency and operational sustainability rather than maximizing liberation alone.

#### **CONCLUSION**

This study investigated the influence of particle size on the recycling efficiency of end-of-life polycrystalline silicon photovoltaic (c-Si PV) modules using sequential mechanical, thermal, and chemical treatment processes. The results demonstrated that particle size plays a critical role in determining glass recovery efficiency, elemental distribution, and phase liberation during PV recycling.

Fine fractions (0.08–0.4 mm) achieved the highest glass-rich fraction recovery (approximately 83%), confirming that particle size reduction improves liberation of glass and embedded metallic constituents. In contrast, coarse fractions (>5 mm) exhibited lower recovery efficiency due to incomplete separation of encapsulant and composite layers. Elemental analysis further revealed progressive enrichment of Si, Sb, Pb, and Ag in finer fractions, indicating selective concentration of both valuable and hazardous components during crushing and sieving.

XRD analysis identified quartz (SiO<sub>2</sub>), albite (NaAlSi<sub>3</sub>O<sub>8</sub>), and lime (CaO) as the dominant crystalline phases in the recovered materials. Minor Sb-containing phases detected in fine fractions suggest that thermal and mechanical treatment can promote concentration of antimony-bearing compounds within smaller particle sizes. ICP-OES analysis confirmed that toxic metals such as Pb and Sb preferentially accumulated in fine powders, highlighting the need for effective dust control and environmentally safe handling during recycling operations.

The findings demonstrate that although fine particle fractions improve recovery efficiency and enhance the potential for critical metal extraction, they also present increased environmental and occupational risks because of heavy metal enrichment. Consequently, an optimized recycling strategy for crystalline silicon PV waste should adopt a fraction-specific approach in which medium-sized fractions (1–5 mm) are prioritized for efficient and safer glass recovery, while fine fractions are subjected to controlled hydrometallurgical or chemical recovery processes for valuable metal extraction.

Overall, this study contributes to the development of sustainable and circular photovoltaic recycling systems by demonstrating how particle size distribution influences both resource recovery and contaminant behavior in end-of-life PV modules. The results provide useful insight for the design of safer and more efficient PV recycling processes aimed at supporting circular economy objectives in the rapidly growing solar energy sector.

Future studies should investigate the leachability and environmental mobility of heavy metals in recovered fractions, as well as the economic feasibility and energy requirements of large-scale particle-size-based PV recycling systems.

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