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Structural and Crystallographic Evolution of Nano-POFA using XRD Based Crystallite Analysis

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Abstract:
Palm oil fuel ash (POFA) is one of the main by-products of the palm oil industry, predominantly composed of silica (SiO_2) and various other metal oxides. Nano-POFA (P_1) was synthesized using a top-down approach via high-energy ball milling process at a rotational speed of 1300 rpm for 1 hour using 4 mm-diameter milling balls. The X-ray diffraction patterns revealed that the dominant crystalline phases were Quartz (SiO_2) and Mullite ($Al_6Si_2O_{13}$), accompanied by minor phases such as Cristobalite (SiO_2), Hematite (Fe_2O_3), Calcite ($CaCO_3$), CaO, and Al_2O_3. The XRD analysis results of samples P_0 (without ball milling) and P_1 (1 hour of ball milling) revealed a significant decrease in peak intensity and peak broadening after 1 hour of the milling process. This phenomenon indicates partial amorphization and crystal fragmentation. The average crystallite size decreased from 92.14 nm in sample P_0 to 47.52 nm in sample P_1. These results indicate that the high-energy ball milling process induces lattice distortion, structural disorder, and amorphization within the Si-O framework of the POFA. The Williamson-Hall analysis indicated an increase in microstrain from $\varepsilon = 3.38 \times 10^{-3}$ to $\varepsilon = 1.227 \times 10^{-2}$. The structural transformations increase surface defect density and reactivity, signifying nano-POFA's potential as material heterogenous catalyst for related chemical processes

Keywords: Ball milling; crystallite size; microstrain; nano-POFA; x-ray diffraction

1. Introduction

Palm Oil Fuel Ash (POFA) is as solid waste generated from the combustion of palm biomass, abundantly available in tropical palm oil-producing countries, particularly in Indonesia. Chemically, POFA is primarily composed of silica (SiO_2) along with other metal oxides such as aluminum oxide (Al_2O_3), calcium oxide (CaO), magnesium oxide (MgO) and iron(III) oxide (Fe_2O_3) (Hanifa et al., 2020; Yusof et al., 2020; Sufian et al., 2025; Abdulhaleem et al., 2025). This chemical composition makes POFA a potential precursor for functional materials, such as adsorbents, matrix fillers, or supporting materials for heterogenous catalysts. The utilization of POFA not only helps reduce waste but also provides an opportunity to produce value-added, environmentally friendly materials.

The direct use of untreated POFA limits its specific surface area and the number of active surface sites available for catalytic reactions Transforming POFA into its nanoscale form (nano-POFA) offers an attractive approach to enhance its surface properties, catalytic potential, pozzolanic reactivity, and interfacial bonding properties. According to Liew et al. (2024) the reduction of POFA particle size to the nanoscale increases the specific surface area and dissolution rate of amorphous silica, thereby improving material reactivity. One of the widely techniques for this purpose is high-energy ball milling. This is mechanochemical activation method has proven effective in reducing particle size, creating lattice defects, and increasing the amorphous fraction of materials – including silica-based and industrial waste material (Vasconcelos et al., 2025) The ball milling process is governed by impact and frictional forces between the milling balls and material particles, which can alter the crystalline structure, resulting in reduced crystallite size, increased lattice defects, and possible phase transformations. According to Mashri et al., (2022), the mechanical milling process significantly reduces the

particle size and modifies the physical characteristics of POFA such as morphology, surface area, and particle size distribution. Extended milling duration leads to finer and more homogenous particles, which in turn enhance the dissolution rate of amorphous silica and reactivity of POFA. According to Snellings et al.,2021), mechanical activation via milling promotes particles fragmentation, amorphization, and the formation of lattice defects with particle fly ash particles.

The crystallographic characterization of POFA is therefore crucial for understanding its catalytic potential. X-ray diffraction (XRD) remains one of the most fundamental techniques for identifying crystalline phases, crystal structure, and crystallite size of the examined material (Ali et al., 2022) X-ray diffraction remains a fundamental tool for assessing this microstructural transformation. By analyzing diffraction line broadening, the crystallite size and lattice strain can be estimated through the Scherrer equation and the Williamson-Hall (W-H) approach. Unlike Rietveld refinement, these methods provide a rapid and reliable evaluation of crystallite dynamics for heterogeneous materials such as POFA.

Several previous studies have investigated the synthesis and characterization of POFA based materials. Ranjbar et al. (2014) demonstrated that POFA can be effectively used as a supplementary material due to its pozzolanic properties. Tangchirapat et al.,(2009) reported that calcinated POFA exhibited improved reactivity, attributed to the removal of unburned carbon and the transformation of crystalline phase. Altwair et al., (2012) further confirmed that ultrafine POFA particles produced through extended milling showed enhanced surface area and improved cementitious activity. Additionally, XRD analysis has been applied to other biomass ashes such as rice husk (Wang et al., 2024). Prior research has shown that the XRD peak intensities of POFA decrease significantly after intensive milling, indicating an enhancement in amorphous characteristics and a reduction in crystalline size. However, quantitative studies focusing on the effects of short-duration high-energy ball milling on the phase evolution and crystallite size of POFA remain relatively limited. At relatively short milling durations, some crystalline phases such Quartz (SiO_2) and mullite ($Al_6Si_2O_{13}$) can still be detected, which may contribute to thermal stability and catalytic activity Therefore, understanding the relationship between milling conditions (such as duration, speed, and ball size) and the crystallographic characteristic of nano-POFA is crucial for designing optimized catalytic materials.

The milling speed of 1300 rpm was selected based on preliminary experimental trials and the operational specifications of the Retsch Emax high-energy ball mill, which indicate that this speed provides an optimal balance between impact energy and thermal stability. At this rotational speed, sufficient mechanical energy is delivered to induce effective mechanochemical activation while minimizing excessive heat generation that could potentially lead to phase decomposition. Furthermore, a milling duration of 1 hour was applied as a short-time processing condition to examine the early-stage accumulation of lattice strain. This timeframe enables the observation of initial structural modifications before reaching the amorphization saturation commonly associated with prolonged milling processes (Mashri et al., 2022).

This study aims to provide a preliminary investigation into the utilization of Nano-POFA as a heterogenous catalyst and to examine the phase transformation and crystallite size evolution of Nano-POFA synthesized via high-energy ball milling at a speed of 1300 rpm for milling durations of 0 and 1 hours, using 4 mm diameter ball. The results of this research are expected to provide a comprehensive understanding of the mechanochemical effects on the crystalline structure of POFA and serve as an initial study for the development of value-added and environmentally friendly heterogenous catalytic materials.

2. Method

Materials and initial preparation

The Palm Oil Fuel Ash (POFA) waste was obtained from the Blankahan Palm Oil Mill, Kuala Village, Langkat, North Sumatera. The POFA waste was dried at 110^{o}C for 24 hours to remove moisture. The raw POFA was then sieved using an 80 µm mesh to eliminate large particles and other contaminants such as sand. The fine powder obtained was subsequently calcinated at 600^{o}C using furnace for 120 minutes to remove unburned carbon residues.

Synthesis of nano-POFA

The synthesis of nano-POFA was carried out using a top-down approach through high-energy ball milling (Retsch Emax) with grinding jars made of stainless steel, tungsten carbide, and zirconium oxide. The

ball-to-powder ratio was maintained at 15 mL: 15 mL, a total of 15 mL of POFA powder was used, with a grinding jar capacity of approximately 50 mL and using grinding balls with a diameter 4 mm. The milling was performed at a rotational speed of 1300 rpm. The experiments were conducted under two milling durations, 0 hours (P_0) as the control and 1 hour (P_1) to evaluate the effect of short-duration milling on phase transformation and crystallite size. After milling, the powder was dried in an oven at 80^{o}C for 2 hours and stores in an airtight container prior to characterization.

X-ray diffraction analysis

X-ray diffraction (XRD) analysis was performed using a Bruker D8 Advance diffractometer with a Cu Kα radiation source (λ = 1.5406 Å) with 2θ range of 10° -90°, with a step size of 0.02° and scanning rate of 2°/min. The diffraction patterns were obtained after the scanning process was completed. Initial phase identification was conducted by comparing the diffraction peaks with standard data from the Crystallography Open Database (COD) using CIF files. The CIF files used for this analysis correspond to the following crystalline phase such as Quartz (COD #5000035), Mullite (COD #9010159), Cristobalite (COD #1010954), Lime (COD #1011095), Hematite (COD #9015964), Corundum (COD #9007634), and Calcite (COD #1010962). The crystallite size was estimated using the Scherrer equation (Eq. 1).

$$D = \frac{K\lambda}{\beta cos\theta}, \quad (1)$$

Where D is the crystallite size (nm), K is the crystallite shape factor (0.9), λ is the X-ray wavelength, β is the full width at half maximum (FWHM) of the diffraction peak (in radians), and θ is the diffraction angle.

The microstrain (ε) was determined using the Williamson-Hall method (Eq.2).

$$\beta cos\theta = \frac{K\lambda}{D} + 4\varepsilon\, sin\theta \quad (2)$$

3. Results and Discussion

XRD Patterns of Unmilling POFA and Nano-POFA

X-Ray diffraction (XRD) analysis provides essential information regarding the crystalline phases and structural evolution of POFA before and after mechanochemical activation. The XRD patterns of POFA samples before and after the ball milling process are presented in Figure 1. The diffraction pattern of untreated POFA without milling (P_0) is shown in Figure 1(a). In the unmilled POFA sample (P_0), the diffraction pattern is dominated by sharp and intense peaks corresponding to quartz (SiO_2), observed at 2θ ≈ 20.83°, 26.68°, 39.39°, 54.89° and 60.09°. This confirms that silica is present primarily in biomass combustion ashes, as high-temperature burning promotes the formation of stable silicate minerals (Abdulhaleem et al., 2025)

In addition to quartz, aluminosilicate phases such as mullite ($Al_6Si_2O_{13}$) were detected at 2θ ≈ 26.61°, 39.39° and 59.93°. Several minor diffraction peaks were identified, associated with calcite ($CaCO_3$), hematite (Fe_2O_3), Corundum (Al_2O_3), Cristobalite (SiO_2), and Lime (CaO). The presence of these phases indicates that palm oil fuel ash (POFA) still contains various metal oxides that can be utilized as functional materials. The sharp diffraction peaks, predominantly attributed to silicate mineral such as quartz, reflect the high degree of crystallinity characteristic of biomass combustion-derived materials. Previous studies have noted that ashes derived from biomass combustion exhibit sharp and intense diffraction peaks corresponding to crystalline silicate phases, suggesting that significant crystallinity is retained despite their origin as combustion residues. (Elanthikkal et al., 2023).

After 1 hour of high-energy ball milling, the peak intensities decreased significantly, particularly for the strongest quartz peak located at 2θ = 26.67°. This reduction indicates a decrease in crystallinity due to the fragmentation of dominant crystalline structures into smaller domains. Furthermore, the milling process resulted in noticeable peak broadening, as shown in Figure 2. This broadening is attributed to the decrease in crystallite size and the increase in lattice strain caused by high-energy impacts during milling.

Similar phenomena have also been reported for mechanically activated fly ash and rice husk ash, where milling promotes structural refinement and partial amorphization (Snellings et al., 2021). In addition, the slight increase in intensity within the 20°-35° region suggests for the formation of a semi-amorphous silica phase,

likely resulting from plastic deformation and lattice disorder induced by the impact forces during ball milling. The repeated collisions in the high-energy milling process leads to particle fragmentation and disruption of Si-O-Si atomic coordination, producing lattice defects and increasing the specific surface area of material (Kweon et al., 2025,Gauthier et al., 2024).

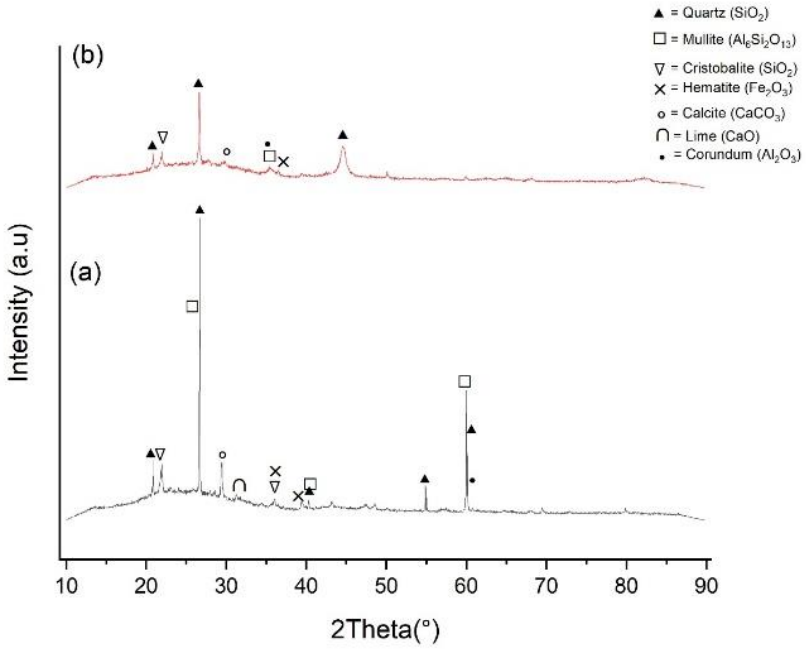

Figure 1. X-ray Diffraction (XRD) patterns of (a) POFA before ball milling (P_0) and (b) POFA after 1 hour of ball milling (P_1)

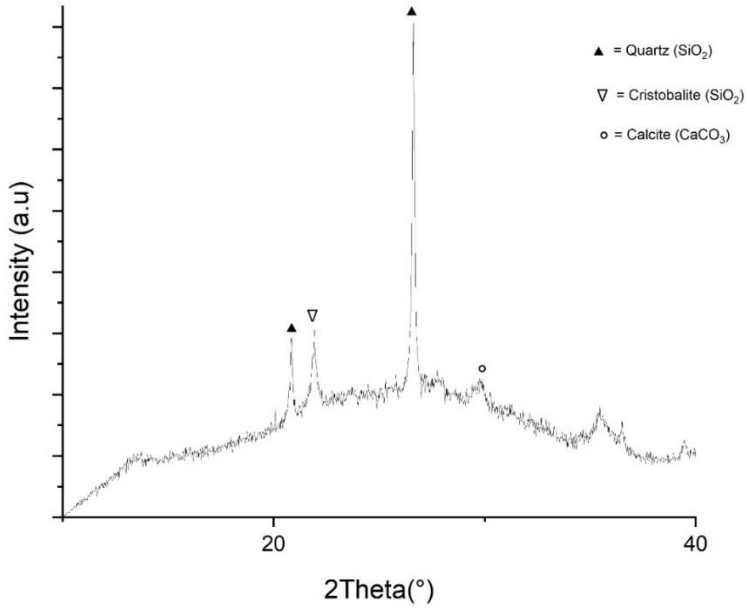

Figure 2. X-ray diffraction (XRD) pattern of ball-milled POFA (P_1) in the 2θ range of 10°-40°

Crystallite size and microstrain analysis

The crystallite size (D) was calculated using the Scherrer equation based on the main quartz (SiO_2) diffraction peak. The average crystallite size of POFA prior to ball milling (P_0) was approximately 92.07 nm. After 1 hour of high-energy ball milling, the crystallite size decreased significantly to 47.48 nm, indicating effective crystallite refinement induced by mechanochemical activation To further analyze the origin of diffraction peak broadening, the Williamson-Hall (W-H) method was applied. This approach allows the separation of size-induced broadening from strain-induced broadening by considering both crystallite size and lattice microstrain contribution, as expressed in equation (2). The Williamson-Hall plot for the unmilled POFA sample is presented in Figure 3a. Linear fitting of $\beta \cos \theta$ versus $4 \sin \theta$ produced a negative slope of -0.00338, indicating the presence of compressive strain ($\varepsilon = 3.38 \times 10^{-3}$) within the crystal lattice. The intercept value of 0.00793 was used to estimated the crystallite size based on the W-H method, yielding approximately 15.48 nm, calculated using the Scherrer constant ($k = 0.9$) and the Cu Kα wavelength ($\lambda = 0.15406$). The negative microstrain value indicates that the lattice crystal experiences internal stress as a result of calcination at 600°C. High-temperature treatment induces thermal expansion and partial recrystallization, leading to an imbalance of stress among neighboring crystallites. Upon cooling to room temperature, differences in thermal contraction rates between crystalline and amorphous regions can generate lattice shrinkage, leading to compressive strain within the material.

After high-energy ball milling, the microstrain increased markedly to $\varepsilon = 1.227 \times 10^{-2}$. This higher microstrain value indicates the presence of crystal defects and lattice distortions that serve as additional active sites. The positive slope observed in the W-H plot for the milled sample (Figure 3b) suggests the development of tensile microstrain, implying that the crystal lattice undergoes tensile deformation as a result of repeated high-energy impacts during the milling process. Simultaneously, the reduction in crystallite size produces a nanocrystalline structure with substantially larger surface-to-volume ratio. These structural modifications are highly relevant to the application of POFA as a heterogenous catalyst. Therefore, the combination of smaller crystallite size and higher microstrain enhances the surface area and facilitates the diffusion of reactant molecules, thereby indicating that ball-milled POFA exhibits higher potential as a heterogenous catalyst.

The crystallite size obtained from Scherrer equation is larger compared to that derived from the Williamson-Hall analysis, shown in Table 1. This discrepancy indicates that broadening of the diffraction peaks in not solely attributed to the small crystallite size but also to the presence of internal lattice strains, as identified by negative slope in the W-H plot at Figure 3a. Therefore, the W-H method provides a more realistic estimation of crystallite size, as it considers the contribution of strain to peak broadening. calculated crystallite size for POFA ball milled (≈ 24.2 nm) was obtained considering the absolute value of the intercept. The negative intercept in the W-H plot suggests that strain effects dominate peak broadening, hence this size should be interpreted as an approximate value.

Table 1. Comparison of crystallite size and microstrain values for POFA samples determined by Scherrer and William-Hall methods

Sample	D Scherrer (nm)	D W-H methods (nm)	Microstrain (ε)	Slope (W-H)
P_0 (0 h milling)	92.07	15.48	3.38×10^{-3}	-0.00338 (compressive)
P_1 (1 h milling)	47.48	24.2	1.227×10^{-2}	+0.01227 (tensile)

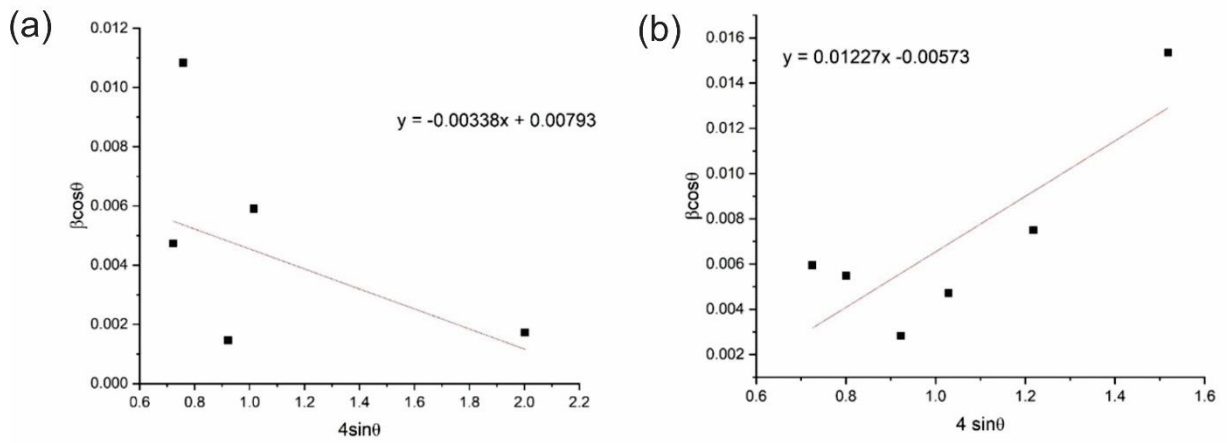

Figure 3. Structural Implications for Heterogenous Catalysts Williamson-Hall plots for (a) POFA without ball milling process and (b) POFA with ball milling process

The reduction in crystallite size and the increase in microstrain are expected to significantly enhance the surface reactivity and increase the density of active sites in nano-POFA, thereby improving its effectiveness as a catalytic material. High-energy ball milling induces partial transformation of the crystalline quartz (SiO2) phase into a more amorphous structure due to intense impact forces and repeated lattice deformation. This mechanochemical process generates a material with a higher degree of structural disorder, which is closely associated with improved catalytic performance.

The formation of a disordered or semi-amorphous silica framework contributes to the creation of a greater number of surface defects, such as broken siloxane bonds and unsaturated coordination sites. These defects can serve as reactive centers that facilitate adsorption and activation of reactant molecules. In particularly, ball-milled POFA contains an increased fraction of amorphous SiO_2 which promotes the formation of silanol (Si-OH) groups on the surface (Yusof et al., 2020). These silanol groups play an essential role in heterogeneous catalysis, as they provide active adsorption sites and can act as anchoring points for catalytic species during surface-mediated reactions. In addition, the presence of basic oxide phases such as CaO, Fe_2O_3, and Al_2O_3 further enhances the surface acid-base characteristics of nano-POFA. Calcium oxide contributes strong basic sites, which are particularly favorable for base-catalyzed reactions such as transesterification in biodiesel production. Meanwhile, iron oxide phase may introduce redox-active centers that are beneficial for catalytic oxidation processes, including the degradation of organic pollutants in wastewater treatment. Alumina, on the other hand, can provide additional Lewis acid sites, improving the bifunctional catalytic behavior of the material.

The synergistic combination of nanoscale crystallite refinement, defect generation, and multifunctional oxide composition suggests that nano-POFA possesses enhanced catalytic versatility compared to untreated POFA. The increased surface area resulting from crystallite size reduction improves mass transfer and accessibility of reactants, while microstrain induced lattice distortions increase surface energy and reactivity. These properties are crucial for heterogeneous catalytic systems, where catalytic efficiency depends strongly on surface-active site availability and diffusion processes. Furthermore, mechanochemical activation offers a sustainable and cost-effective approach for transforming palm oil industry waste into value-added catalytic materials. Ball milling is a solvent-free and scalable method that enables the production of defect-rich nanostructured silica frameworks without the need for complex treatments. Therefore, nano-POFA represents a promising environmentally friendly catalyst precursor for renewable energy applications, such as biodiesel synthesis, biomass upgrading into liquid fuels (Durimin-Iya et al., 2024), and advances oxidation processes for wastewater remediation (Yusof et al., 2020).

4. Conclusion

This study successfully revealed the structural and phase transformations of Palm Oil Fuel Ash (POFA) induced by high-energy mechanical activation, as evidenced by X-ray diffraction (XRD) analysis combined with Rietveld refinement. The unmilled POFA (P_0) exhibited a dominant crystalline Quartz (SiO_2) phase, followed by mullite ($Al_6Si_2O_{13}$), and minor phase such as calcite, hematite, dan Al_2O_3, with a relatively low fraction of amorphous content. After high-energy ball milling for 60 minutes at a rotational speed 1300 rpm, the diffraction peak intensities decreased significantly and the main peaks became broader, indicating a reduction in crystallite size, accompanied by an increase in lattice distortion and atomic disorder within silica framework. These results demonstrate that mechanical activation effectively induces structural disorder and partial amorphization, which may enhance the surface reactivity and catalytic potential of resulting nano-POFA material.

The crystallite size, calculated using the Scherrer equation, decreased from 92.07 nm to 47.48 nm. Furthermore, Williamson-Hall analysis indicated an increase in microstrain from $\varepsilon = 3.38 \times 10^{-3}$ to $\varepsilon = 1.227 \times 10^{-2}$, suggesting the formation of lattice defects and an expansion of the active surface area. This condition leads to the formation of a greater number of surface-active sites, which are crucial for enhancing catalytic performance.

Chemically, the combination of amorphous SiO_2 phases, metal oxide (Fe_2O_3, CaO, Al_2O_3) and nanoscale morphology contributes to enhances adsorption capacity, the formation of base sites, and improved thermal stability. The findings of this study elucidate the correlation between structural deformation induced by milling, reduced crystallinity, and increased amorphization as key factors responsible for enhancement of active sites in silica-based waste materials. This structural transformation establishes nano-POFA as a promising candidate for environmentally friendly and sustainable waste-derived catalytic materials.

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