

## Palm Oil Fuel Ash and Fly Ash for a Partial Replacement of Cement in High-Quality, Environmentally Friendly Mortar as a Solution to Industrial Waste

Shinta Marito Siregar<sup>1,2</sup>, Syahrul Humaidi<sup>1\*</sup>, Nurdin Bukit<sup>3</sup>, Erna Frida<sup>1</sup>

<sup>1</sup>Doctoral Program (Physics), FMIPA, Universitas Sumatera Utara, Jln Bioteknologi No 1, Medan, 20155, Indonesia

<sup>2</sup>Universitas Muslim Nusantara Al Washliyah, Jl. Garu II A No. 93, Medan, 20147, Indonesia

<sup>3</sup>Department of Physics, Universitas Negeri Medan, Jl. Willem Iskandar Pasar V, Medan, 20221, Indonesia

\*Corresponding author: syahrul1@usu.ac.id

### Abstract

This study explores the effects of incorporating palm oil fuel ash (POFA) and fly ash (FA) as partial cement substitutes on the mechanical properties and characteristics of high-quality mortar, specifically Engineered Cementitious Composites (ECC). ECC mortar was fabricated by milling POFA waste and FA through a top-down method utilizing a ball mill. The resulting material was subjected to tests for slump flow, water absorption, compressive strength, and characterized through XRF, FTIR, SEM/EDX, and XRD analyses. FTIR analysis verified the existence of Si–O and Al–O groups within the composite made of POFA-FA ECC. XRF analysis of FA and POFA showed cementitious properties, with  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  exceeding 50% and CaO surpassing 10%. SEM and XRD results indicated minimal cavity formation, suggesting a high compressive strength in the mortar. Particle size distribution analysis revealed prevalent particles in the  $1.5 \times 10^{-1}$  to  $2.0 \times 10^{-1}$   $\mu\text{m}$  range. The compressive strength test after 28 days, incorporating 15% FA and 10% POFA, yielded the highest strength at 59.30 MPa. The water absorption values ranged from 1.25% to 2.67%, indicating that POFA-FA assists in the cement hydration process and also serves as a filler. As a result, the material's density is very high, leading to fewer voids formed, thus reducing the trapped water, which significantly affects the mortar's strength.

### Keywords

POFA, Fly Ash, Cementitious Material, Industrial Waste

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## 1. INTRODUCTION

The coal sector and the palm oil industry play significant roles in supporting Indonesia's economy. The palm oil production serves multiple functions and has experienced a steady rise in worldwide demand. Additionally, coal-fired power stations are essential for meeting energy requirements across different sectors. The market for both industries has been growing steadily, driven by population growth. However, these industries also generate substantial amounts of byproducts that are often treated as waste (Zawawi et al., 2020; Ghazali et al., 2021; Muthusamy et al., 2020).

One of the essential agro-industries in Southeast Asia and Sub-Saharan Africa is Crude Palm Oil (CPO), which generates palm shells as waste during the processing phase. Indonesia, being one of the world's foremost palm oil suppliers, encounters a direct link between the cultivation of oil palms and the production of palm shells. As palm oil processing increases, so does the amount of palm kernel shells generated. Palm shells hold significant economic value for the palm oil processing industry

as they can be utilized as an energy source. When palm shells are used as fuel for power plants, they produce ash, commonly referred to as palm oil fuel ash (POFA). Consequently, the increasing global annual ash production from industries poses a risk of environmental pollution if not managed appropriately (Aldahdooh et al., 2014; Hamada et al., 2020; Khankhaje et al., 2018; Muthusamy et al., 2019; Thomas et al., 2017).

The increase in fuel oil prices has led many industries to switch to using coal as a fuel source, resulting in a continued increase in demand for coal in recent years. Consequently, the production of fly ash (FA) waste from coal-fired power plants has also elevated. Proper management of fly ash waste is essential as it incurs costs for the industry and, if mishandled, can result in environmental pollution and pose health risks to humans. Therefore, it is imperative to implement effective handling practices to mitigate the adverse effects of fly ash (Abdulkareem et al., 2014; Naganathan et al., 2015; Rafieizonooz et al., 2016).

In response to this challenge, several studies have explored the sustainable use of POFA and FA in the construction sec-

tor. As per [Sumra et al. \(2023\)](#), the addition of ground POFA (G-POFA) as a cement substitution in Ordinary Portland Cement (OPC) mortar increased its water absorption due to a more significant pozzolanic reaction. The mortar with 10% G-POFA substitution (G-POFA-10) indicated the highest tolerance to water absorption and sorptivity. Additionally, [Jose et al. \(2023\)](#) found that the water absorption of foamed concrete decreased with an increasing amount of POFA and red gypsum replacement. [Alex et al. \(2016\)](#) further demonstrated that mechanical strength increased with a decrease in fly ash size and a 20 wt% ash replacement in cement. Nonetheless, continuous endeavors are underway to enhance the results by considering a range of factors, including material characteristics, treatment techniques, mix proportions, construction methods, and methodologies. POFA-FA contains abundant cementitious compounds, making it suitable for application as a cementitious material in mortar-engineered cementitious composites (ECC). ECC mortar belongs to the High-Performance Fiber-Reinforced Cementitious Composites (HPFRCC) category, exhibiting high ductility and a moderate fiber fraction ([Adesina and Das, 2020](#); [Sherir et al., 2014](#); [Wang and Li, 2007](#)). ECC is a material that can be manufactured by considering microstructure, processing techniques, material properties, and performance. The micromechanical relationship between the mechanical performance of composites and their microstructure occurs ([Aldahdooh et al., 2014](#); [de Azevedo et al., 2022](#); [Hamada et al., 2018](#); [Muthusamy et al., 2020](#)). [Muthusamy et al. \(2021b\)](#) conducted a study using a composite of Oil Palm Shell (OPS) and FA to create concrete. The outcomes of compressive strength, flexural strength, and water absorption tests performed over a 28-day period demonstrated that the inclusion of 30% fly ash produced the most favorable results.

Moreover, the adoption of POFA-FA as a construction material is expected to reduce the requirement for and the manufacturing of conventional Portland cement, which has a substantial impact on carbon emissions and the environment. In the pursuit of developing environmentally friendly Portland cement, many studies have explored the potential of waste materials to serve as raw materials and provide sustainable solutions to various global environmental challenges ([Hamada et al., 2019](#); [Nath and Sarker, 2014](#)). Therefore, this study introduces the incorporation of POFA-FA into ECC mortar, as it is crucial as a substitute material for cement production. With this explanation in mind, this study aims to assess the mechanical characteristics and attributes of the newly created ECC mortar that utilizes POFA-FA as a key component.

## 2. EXPERIMENTAL SECTION

### 2.1 Materials and Instrumental

The POFA utilized was sourced from PT. Tinjowan Oil Palm Plantation (North Sumatra, Indonesia), while FA was obtained from PT. Sinarmas Oleochemical (Medan, North Sumatra, Indonesia). Other materials, such as fine sand and superplasticizer (SP) of the ViscoCrete-3115 N type, were obtained

from PT. SIKKA, Portland Cement Type I/Ordinary Portland Cement (OPC) from PT. Semen Padang.

The equipment used includes a drill mixer, slump-flow test equipment, cube mold measuring (15×15×15 cm), mix bucket, digital scales, meter, ball mill, compression test machine (ELE INTERNATIONAL, 2000 kN), oven, X-Ray Fluorescence (XRF, PANalytical Minipal 4), Fourier Transform Infra Red (FTIR, Shimadzu IRPrestige-21), X-Ray Diffraction (XRD, OHION), and Scanning Electron Microscopy-Energy Dispersive X-Ray (SEM-EDX Mapping, JEOL JSM-6510LA).

### 2.2 Methods

#### 2.2.1 ECC Mortar Manufacturing

As indicated in prior research, the ideal proportion for incorporating POFA is between 10-15% ([Danish et al., 2023](#)), and for FA in cementitious composites, it should not exceed 15% to avoid any notable impact on workability ([Lin et al., 2023](#)). For this reason, POFA-FA and other materials were weighed according to a predetermined mix proportion (Table 1). The mixing process began by combining the superplasticizer and water in a bucket using a drilling machine. Subsequently, the remaining materials, starting with POFA, FA, sand, and type 1 cement, were gradually added. Each addition was accompanied by stirring using a drill machine until the mixture was evenly mixed, after which the next material was added. The mixture in the bucket was continuously stirred until it reached a homogeneous state with good workability, as confirmed by the slump flow test. Following this, the mixed material was transferred to a mold and subjected to a compressive strength test after 28 days ([Huseien et al., 2023c](#)).

#### 2.2.2 Sample Preparation for Characterization

This preparation was conducted following the procedures outlined by [Satriawan and Awaluddin \(2021\)](#) and [Sumra et al. \(2023\)](#) with some modifications. The POFA-FA used for characterization was obtained by burning boilers at 800°C. The resulting POFA-FA ash was dried at 110°C for 24 hours, ground using a ball mill for 6 hours at 400 rpm and sieved through a 200-mesh sieve. Subsequently, it was further dried at 500°C for 4 hours and ground using a ball mill for an additional 6 hours at 400 rpm. The refined POFA-FA was characterized using XRF, FTIR, XRD, and SEM-EDX.

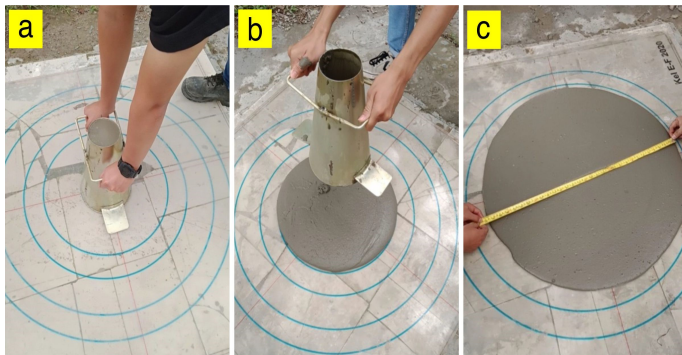
#### 2.2.3 Slump-Flow Test

The slump-flow test was performed to assess the ECC mixture's slump flow value and its filling capacity. The Abrams cone was positioned vertically on the base plate and firmly held in place. Within 30 seconds, the ECC mixture was poured into the Abrams cone funnel without any agitation. The Abrams cone was then gradually lifted in a single upward motion of approximately 30 cm, ensuring the mortar flow was undisturbed. Using a stopwatch, the time needed for the ECC mixture to achieve a 500 mm (T500) diameter was recorded immediately after lifting the Abrams cone. The maximum diameter was measured twice (D1 and D2) once the ECC mixture ceased

**Table 1.** Test Matrices with Various Cementitious Material Compositions

Cementitious Material	Fly Ash (FA) (%)				
	0	5	10	15	
Palm Oil Fuel Ash (POFA) (%)	0	0:0	0:5	0:10	0:15
	5	5:0	5:5	5:10	5:15
	10	10:0	10:5	10:10	10:15
	15	15:0	15:5	15:10	15:15

flowing (Akmal et al., 2017). The process of the slump-flow test is displayed in Figure 1.



**Figure 1.** Slump-Flow Test: (a) Placing Abrams Cone, (b) Lifting the Cone after Pouring the ECC Mixture, (c) Diameter Measurement

#### 2.2.4 Water Absorption Test

In this study, the water absorption test for the POFA-FA ECC mortar followed the method proposed by (Huseien et al., 2023b). The ECC mortar specimens were molded into dimensions of 15×15×15 cm, cured for 28 days, and then submerged in water for 24 hours. Subsequently, the POFA-FA ECC mortar samples were weighed after complete submersion in water ( $W_s$ ). After saturation, the composites were oven-dried for an additional 24 hours at approximately 105°C and then reweighed ( $W_d$ ). The water absorption values ( $WA\%$ ) for the mixtures were calculated using Equation (1).

$$WA(\%) = \frac{W_s - W_d}{W_d} \times 100 \quad (1)$$

#### 2.2.5 Compressive Strength Test

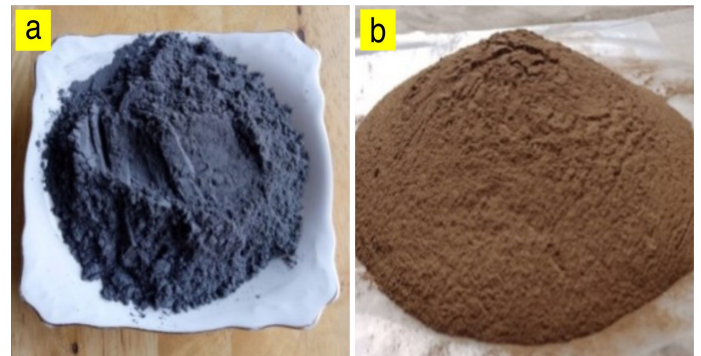
The compressive strength of the cured samples was assessed using a compression testing machine with a maximum capacity of 2000 kN. The POFA-FA ECC mortar specimens, aged for 28 days, were placed on the lower plate of the testing device. To accurately capture the failure pattern under uniaxial compression, a loading rate of 0.21 mm/min under displacement control was selected. Compressive strength was estimated by dividing the maximum applied force by the cross-sectional area

that resisted the load, following the methodology described by Chub-uppakarn et al. (2023) and Huseien et al. (2023a).

### 3. RESULTS AND DISCUSSION

#### 3.1 XRF Characterization of POFA and FA

XRF testing was conducted at the Central Laboratory of Minerals and Advanced Materials, State University of Malang, to identify the elements and compounds present in POFA and FA (Naganathan et al., 2015; Nugraha et al., 2007). After the XRF test, the POFA and FA were dried, pulverized in a ball mill, and then sieved through a 200 mesh. Figure 2 displays the appearance of the POFA and FA.



**Figure 2.** (a) POFA and (b) FA

The findings of the XRF test on POFA and FA are presented in Tables 2 and 3, respectively. Table 2 reveals that POFA has chemical compositions of 48.9%  $SiO_2$ , 3.4%  $Fe_2O_3$ , and 13.8%  $CaO$ , indicating its pozzolanic and cementitious properties (Alsubari et al., 2018). Similarly, Table 3 displays the chemical composition of FA, with  $SiO_2$ ,  $Al_2O_3$ ,  $Fe_2O_3$ , and  $CaO$  constituting 29.3%, 14.0%, 30.9%, and 18.3% respectively. These compositions also contribute to the pozzolanic and cementitious nature of FA.

#### 3.2 EDX and XRD Characterization

Figure 3 illustrates the outcomes of the EDX analysis performed on the POFA-FA ECC mortar. The analysis reveals the existence of different elements and oxides in the materials, with the following composition: C= 21.50%, MgO= 1.03%,  $Al_2O_3$ = 14.41%,  $SiO_2$ = 36%,  $SO_3$ =0.91%,  $K_2O$ = 3.10%,  $CaO$ = 12.10%, and  $FeO$ = 10.95%. According to the EDX data,  $SiO_2$  showed the highest value, primarily due to its elevated silica



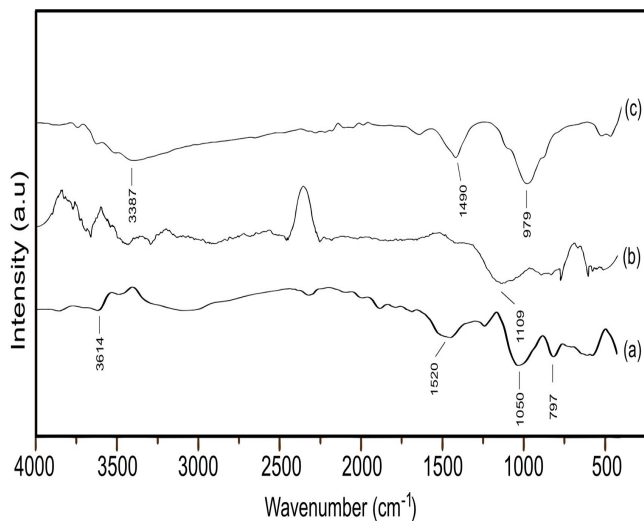
$2\theta$  angle, as depicted in Figure 4. The diffractogram of POFA indicates the presence of  $\text{SiO}_2$  peaks at  $2\theta$  angles of  $20.81^\circ$ ,  $21.72^\circ$ ,  $50.06^\circ$ ,  $59.85^\circ$ , and  $68.10^\circ$  (Figure 4). Additionally, peaks attributed to  $\text{CaCO}_3$  and  $\text{K}_2\text{SiO}_4$  were observed at  $2\theta$  angles of  $26.52^\circ$  and  $27.50^\circ$ , respectively.

Moreover, according to the XRD analysis of FA utilizing the data displayed in Table 4, it is evident that the crystal system exhibits hexagonal axes. The unit cell dimensions are identified as  $a = 4.8120 \text{ \AA}$ ,  $b = 4.8120 \text{ \AA}$ , and  $c = 5.3270 \text{ \AA}$ , with a corresponding density of  $2.80 \text{ g/cm}^3$ . According to Figure 4, the diffractogram of FA exhibits  $\text{SiO}_2$  peaks at  $2\theta$  angles of  $21.40^\circ$ ,  $50.74^\circ$ , and  $68.66^\circ$ , while  $\text{Al}_2\text{O}_3$  peaks are observed at  $2\theta$  angles of  $27.18^\circ$  and  $60.51^\circ$ .

The XRD analysis results of POFA-FA ECC mortar indicate an orthorhombic crystal system with unit cell parameters  $a = 8.314 \text{ \AA}$ ,  $b = 11.074 \text{ \AA}$ , and  $c = 18.296 \text{ \AA}$ , and a density of  $1.49 \text{ g/cm}^3$ . Figure 4 displays the  $\text{SiO}_2$  peaks at  $2\theta$  values of  $18.02^\circ$  and  $71.53^\circ$ . In addition, peaks corresponding to  $\text{CaCO}_3$  and  $\text{Fe}_2\text{O}_3$  were observed at  $2\theta$  values of  $27.14^\circ$  and  $56.21^\circ$ , respectively.

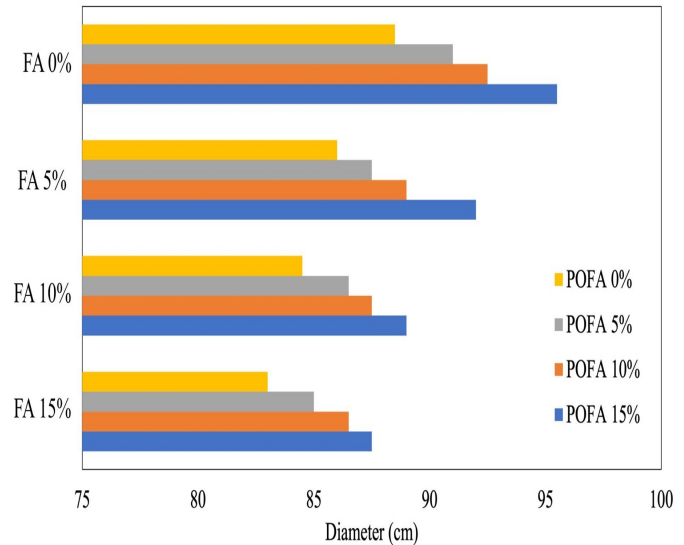
### 3.3 FTIR Characterization

The materials POFA, FA, and POFA-FA ECC mortar were subjected to FTIR characterization to identify their functional groups and assess any changes in functional groups following the creation of POFA-FA ECC mortar (Nandiyanto et al., 2019). Figure 5 presents the FTIR results.

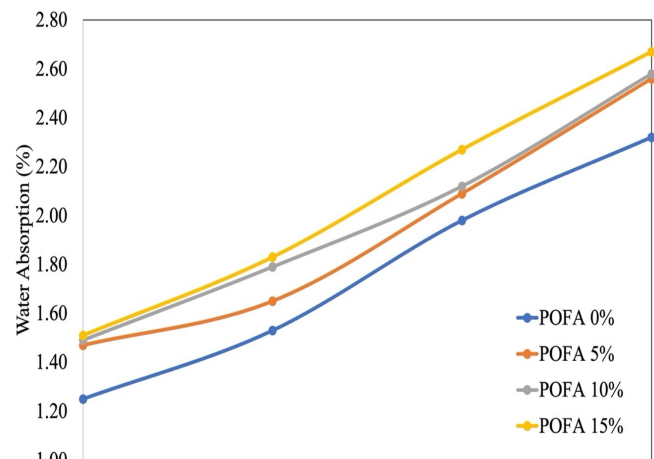


**Figure 5.** FTIR Spectra of (a) POFA, (b) FA, and (c) POFA-FA ECC Mortar

Based on Figure 5(a), the absorption band at around  $3614 \text{ cm}^{-1}$  suggests the moderate intensity of the O–H bond from the SiOH group. The peak at approximately  $1520 \text{ cm}^{-1}$  reveals the asymmetric stretching of the C–O bond (Satriawan and Awaluddin, 2021). Additionally, the band at  $1050 \text{ cm}^{-1}$  suggests the existence of Si–O bond asymmetric stretching vibration. Furthermore, the peak at  $797 \text{ cm}^{-1}$  corresponds to



**Figure 6.** Slump Flow Test Results



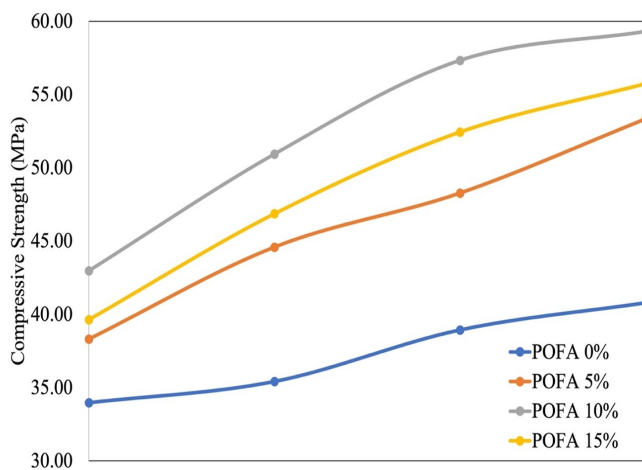
**Figure 7.** Water Absorption on POFA-FA ECC Mortar

the crystalline phase of quartz (Ranjbar et al., 2014; Wi et al., 2018).

Figure 5(b) demonstrates the stretching vibration of Si–O–Si, which is represented by a peak at  $1109 \text{ cm}^{-1}$  (Ryu et al., 2013). The FTIR spectra in Figure 5(c) reveal the presence of functional groups in POFA-FA ECC mortar. The wavenumber at  $3387 \text{ cm}^{-1}$  relates to the O–H stretching vibration from the SiOH group. Furthermore, the wavenumber of the asymmetric stretching C–O bond has shifted from  $1520$  to  $1490 \text{ cm}^{-1}$ . The change in the C–O groups is ascribed to the interconnection between the POFA-FA ECC mortar and carbon dioxide throughout the reaction process, resulting in the establishment of bonds between the composite and carbon dioxide (Satriawan and Awaluddin, 2021). Moreover, the peak of  $979 \text{ cm}^{-1}$  repre-

**Table 4.** XRD Data Analysis of POFA, FA, and POFA-FA ECC Mortar

Materials	Peak Position $2\theta$ (°)	FWHM $B_{size}$ (°)	D (nm)	hkl	d (Å)
POFA	20.81	0.27	31.26	100	4.1673
	21.72	0.27	31.30	041	2.4335
	26.52	0.26	32.80	10-1	11.0761
	27.50	0.25	34.18	-102	10.8581
	50.06	0.26	35.24	112	4.2109
	59.85	0.39	24.56	271	4.0262
	68.10	0.26	38.54	203	5.5381
FA	21.40	0.45	18.77	111	2.19272
	27.18	0.50	17.08	-122	1.17345
	50.74	0.81	11.34	228	1.09636
	60.51	0.75	12.81	1311	1.42871
	68.66	0.49	20.52	-206	1.64114
POFA-FA ECC Mortar	18.02	16.73	0.50	111	7.17398
	27.14	7.21	1.18	103	4.64035
	56.21	4.64	2.03	413	2.53024
	71.53	6.71	1.52	21-6	2.63930

**Figure 8.** Compressive Strength Test Results of POFA-FA ECC Mortar Aged 28 Days

sents Si–O–Si and Si–O–Al asymmetric stretching vibration (Liu et al., 2016; Ranjbar et al., 2014).

### 3.4 Slump-Flow Test on POFA-FA ECC Mortar

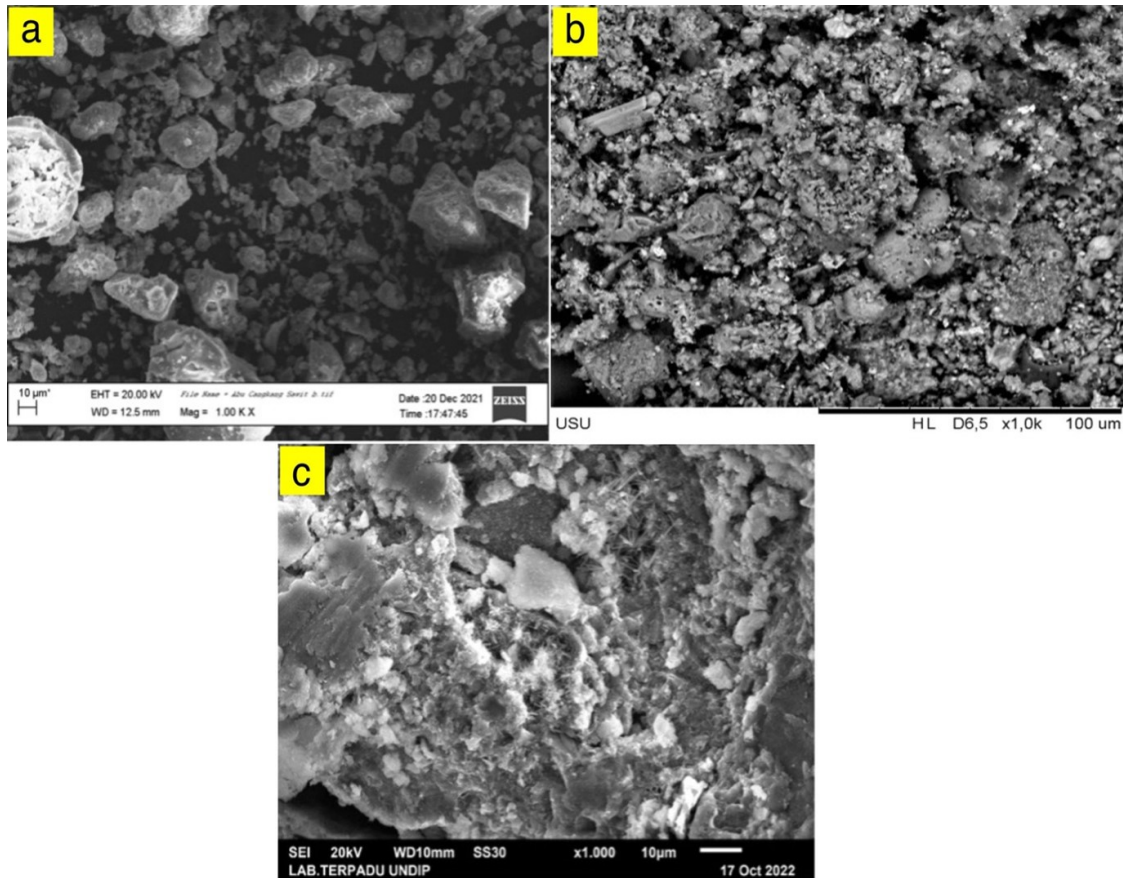
The ECC mortar's workability was assessed using a slump-flow test, and the results for the POFA-FA ECC mortar are presented in Figure 6. As the percentage of FA in the ECC mortar increased, there was a corresponding rise in the slump-flow value. This indicates that incorporating FA up to 15% in the ECC mixture enhances workability. Conversely, the addition of POFA resulted in a reduction in the slump-flow value (Nugraha et al., 2007). This can be explained by the higher water absorption capacity of POFA in comparison to cement or FA due to its larger granule size. Notably, the test results

demonstrated that a POFA:FA mixture with a ratio of 0%:15% exhibited the highest slump-flow value of 95.5 cm. In this research, the specimen with 15% POFA displayed superior slump flow values when contrasted with the other specimens, signifying an enhanced filling capacity of the POFA-FA composite. Additionally, all materials exhibited remarkable stability, with all mixtures achieving a flow time under 5 seconds to attain a 500 mm diameter. Consequently, it can be suggested that the addition of POFA improves the workability of POFA-FA ECC mortar, with greater workability observed at increased POFA content (Sabah et al., 2020; Zaimi et al., 2023).

### 3.5 Water Absorption of POFA-FA ECC Mortar

Figure 7 demonstrates that the water absorption value ranges from 1.25 to 2.67%. The addition of POFA and FA tends to increase the water absorption value of the mortar. However, it remains significantly below the standard water absorption value of around 5% for concrete (Muthusamy et al., 2020). The water absorption value is directly linked to the size of the pores in mortar or concrete. Excess water in the mortar evaporates, leading to the formation of cavities. When the mortar is soaked or exposed to water, these cavities become filled with water. The strength of mortar or concrete decreases with an increase in the number of pores (Muthusamy et al., 2021a).

According to Ayub et al. (2023), POFA has a pore volume of 0.011 cm<sup>3</sup>/g, which can increase up to 0.88 cm<sup>3</sup>/g when activated with KOH. Meanwhile, POFA-modified cementitious composites will exhibit a larger pore volume as the amount of POFA increases (> 0.9 cm<sup>3</sup>/g). This is because the pore volume of POFA is greater than that of cement. Therefore, optimizing the use of POFA in cement should be carefully considered to ensure that water absorption remains below 5% (Golewski, 2023; Tasnim et al., 2020; Zaimi et al., 2023).



**Figure 9.** Surface Morphological of (a) POFA, (b) FA, and (c) POFA-FA ECC Mortar at a Magnification of 1000×

### 3.6 Compressive Strength Measurements

The strength testing in this study was performed using only compressive strength. According to Golewski (2023), the strength of composites can be represented by compressive strength testing, although additional tests such as tensile strength, flexural strength, or others can be added. The trend in the findings of compressive strength testing generally reflects the same trend in other strength tests, such as tensile strength.

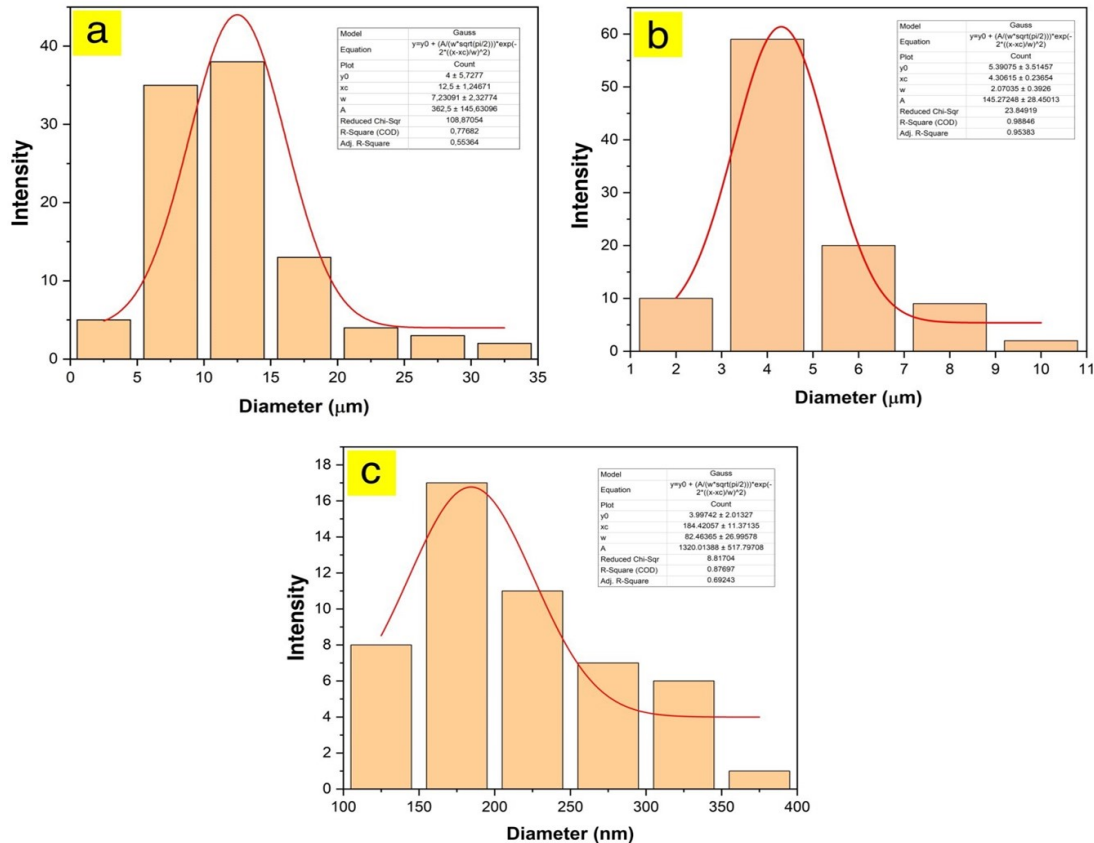
The compressive strength results of POFA-FA ECC mortar after 28 days are shown in Figure 8. The inclusion of FA and POFA results in an rise in compressive strength. Nonetheless, a reduction in strength is observed when POFA is added at a 15% ratio. This suggests that adding excessive POFA with a constant cement mass results in an excess of filler that is challenging to bind with cement as a matrix. Additionally, achieving the desired reaction from cementitious materials becomes difficult if the cement mass is not proportional to the cementitious material that will react with cement (Chousidis et al., 2015; Hossain et al., 2016; Ranjbar et al., 2014; Ting et al., 2020). The highest compressive strength (59.30 MPa) was obtained with a POFA:FA ratio of 10%:15%. Generally, the presence of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{CaO}$  in FA and POFA influences the crucial stages of the cement hydration process, resulting in increased compressive strength in FA-POFA ECC

mortar (Chousidis et al., 2015; Hossain et al., 2016).

### 3.7 SEM Morphology

The morphology of POFA, FA, and FA-POFA ECC mortar was examined through SEM analysis (Liu et al., 2016; Ting et al., 2020). Particle size distribution data were also obtained using SEM. Figure 9(a) shows that POFA exhibits an angular and irregular shape, characterized by granular clumps. The particle size of POFA predominantly falls within the range of 5-15  $\mu\text{m}$  (Figure 10(a)). This indicates a high proportion of extremely fine particles in POFA, potentially impacting the reactivity and workability of the ECC mortar. Conversely, FA (Figure 9(b)) appears as granules with varying shapes. The particle size distribution of FA, displayed in Figure 10(b), is mostly homogeneous at 4  $\mu\text{m}$ , with only a small fraction exhibiting heterogeneity due to agglomerates. Similar to POFA, this morphology influences the reactivity of the ECC mortar.

Meanwhile, Figure 9(c) of POFA-FA ECC mortar reveals the presence of gray color resulting from a lump of FA and black color due to the mixture process, resulting in cavity formation. This SEM image indicates the formation of a few cavities that could potentially impact the mechanical properties of the material. The presence of these cavities within the mortar results in a decrease in mechanical strength, thereby increasing the



**Figure 10.** Particle Size Distribution of (a) POFA, (b) FA, and (c) POFA-FA ECC Mortar

susceptibility to cracks or fractures (Siad et al., 2015). Furthermore, the particle size distribution of the manufactured material predominantly falls within the range of  $1.5 \times 10^{-1}$  to  $2.0 \times 10^{-1} \mu\text{m}$ , as shown in Figure 10(c).

#### 4. CONCLUSION

The XRF analysis of FA and POFA indicated that they possess cementitious properties, as evidenced by the presence of  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 > 50\%$  and  $\text{CaO} > 10\%$ . This suggests their potential contribution to the cement hydration binding process. The changes in functional groups observed in the cement materials after being incorporated into ECC mortar further validate this effect. The study demonstrates a direct relationship between the incorporation of FA and POFA and the compressive strength of ECC mortar, with the optimal POFA:FA composition of 10%:15% resulting in a strength of 59.30 MPa. SEM analysis was employed to evaluate the surface morphology and particle size distribution of the POFA-FA ECC mortar. The SEM results indicate minimal cavity formation, indicating a high compressive strength of the resulting mortar. Particle size distribution analysis revealed the dominance of particles ranging from  $1.5 \times 10^{-1}$  to  $2.0 \times 10^{-1} \mu\text{m}$ . XRD and EDX analysis confirmed the Si abundance, which contributes to the compressive strength of the POFA-FA ECC mortar. Consequently,

the utilization of POFA and FA waste as substitutes for cement presents an effective solution for managing industrial waste from POFA, FA, and Portland cement.

#### 5. ACKNOWLEDGMENT

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