Effect of Particle Size Reduction on the Reactivity of Ground Industrial Ash for Sustainable Cement Substitute

Mehul Rangani[1], Sanjay Joshi[2] Ajaysinh Vaghela[3], Dr. M. Mohan Babu[4], Dhaval Kumar P Advani[5]*

[1]Research Scholar, Department of Civil Engineering, Faculty of Technology, RK University, Rajkot-360020, Gujarat, India

[2]Professor, Department of Civil Engineering, Faculty of Technology, RK University, Rajkot-360020,

[3]Assistant Professor, Department of Civil Engineering, Faculty of Technology, RK University, Rajkot-360020,

[4]Professor, Department of Civil Engineering, Sri Venkateswara College of Engineering & Technology (Autonomous), RVS Nagar, Chittoor, Andhra Pradesh, India

[5]Assistant Professor, Applied Mechanics Department, Shantilal Shah Engineering College, Bhavnagar-Gujarat, India.

Abstract

This research examines the impact of particle size decrease on industrial ash properties and its applicability as a sustainable supplementary cementitious material (SCM). Raw industrial ash was first analyzed using X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF), and surface area to determine its mineralogical and physical makeup. The ash was ground mechanically with a Los Angeles abrasion machine for 1 to 20 Hours (first 1 to 5 hours in 1-hour intervals and after that keep 5-hour intervals up to a total grinding time of 20 hours), to progressively decrease particle size. Particle Size Analysis (PSA) of the ground samples was used to evaluate degree of size reduction. The environmental significance of this work is that utilizing industrial ash, a significant industrial waste product, in cementitious use not only alleviates landfill disposal but also lowers the carbon impact of traditional Portland cement. The research identifies links between particle size reduction and reactivity and aids in the development of greener, more sustainable construction materials.

Key Words: *Industrial Ash, Particle size reduction, Mechanical Grinding, Sustainability, Supplementary cementitious materials (SCM), Los Angeles Machine.*

1. Introduction

Industrial ash, a waste product of many combustion and metallurgical processes, raises serious environmental concerns because it is large in volume and has few disposal outlets. Its use as a supplementary cementitious material (SCM) in cement and concrete is becoming more popular over the last few decades not only because of its technical advantages but also because of its benefits to the environment. Nevertheless, its use as a supplementary cementitious material or a filler within construction materials has aroused considerable interest. One of the significant parameters affecting the reactivity and applicability of industrial ash is its particle size. Yet, raw industrial ash tends to be poorly reactive owing to its coarse particle size and incomplete amorphous content. Mechanical grinding is thus required to increase its fineness and upgrade its capability as a cement replacement. Mechanical grinding is extensively used to improve the fineness and surface area of powders. Grinding and milling operations are important in altering the physical and chemical properties of industrial residues like fly ash (FA), palm oil fuel ash (POFA), and bottom ash (BA). The creation of nano- or micro-sized pozzolanic products increases their reactivity, ultimately leading to the enhancement of the mechanical and durability characteristics of cement-based composites. Different researchers have employed different grinding methods and periods to study the influence of fineness on material performance in concrete and mortar purposes. Yu Xuan Liew et al. (2023) [1] researched the applicability of employing a Los Angeles abrasion (LAA) machine at low speeds of grinding in order to produce POFA of nano sized. Their research compared how different sizes of POFA particles (with sizes ranging from 982 nm to 150 nm) impacted mortar flowability and compressive strength. The research established that finer particle of POFA contributed greatly to compressive strength because of greater surface area and pozzolanic activity. In the same context, Khan R.A. et al. (2016) [2] discussed the effects of grinding BA by the LAA machine. The original bottom ash (OBA) and ground bottom ash (GBA) which was ready after 30 minutes of grinding were compared on the basis of use in concrete mixtures. The research demonstrated how grinding greatly enhanced the performance of BA in cementitious materials. Tambichik et al. (2020) [3] also used the LAA machine to mill POFA and rice husk ash (RHA). The materials were milled using 10 steel balls to a fineness of less than 150 microns for 2 hours. Refining was found to enhance pozzolanic reactivity as well as compatibility in blended cement. Yong-Sik Chu et al. (2019) [4] used both vibration and attrition mills to mill fly ash. A surface area of 5900 cm^2/g was achieved when milling was done for 1 hour. By using an attrition mill (HAS-1), the surface area of the fly ash was 5200 cm^2/g, reflecting the success of high-energy, short-duration milling in increasing particle surface area. Ghasan Fahim Huseien et al. (2018) [5] sieved POFA and ground it for 6 hours in an LAA milling machine holding 15 stainless steel balls (50 mm in diameter) with a speed range of 32–35 rpm. It was proven in the study that extended grinding duly decreased particle size and enhanced the ash fineness, thus impacting the pozzolanic activity and microstructure of blended concrete. A research work by Mohd Warid Hussin et al. (2009) [6] examined the two levels of fineness of POFA, which are 45 μm and 10 μm. The first was obtained after grinding and wet sieving for 3.5 hours, whereas the second took 10 hours of grinding to obtain a median particle size of below 10 μm. The findings highlighted the need for grinding time in the acquisition of ultrafine POFA with enhanced cement replacement efficiency. Rosas-Casarez et al. (2018) [7] also conducted mechanical grinding of FA employing a ball mill in consecutive 1-hour intervals for the purpose of material passage through #200 and #325 sieves. This resulted in average particle sizes of 74 μm and 45 μm for FA-2 and FA-3, respectively, illustrating satisfactory control of particle size through prolonged grinding. Belal Alsubari et al. (2018) [8] ground sieved POFA in an LAA machine for 16 hours at 33 rpm. The high LOI in the produced G-POFA was eliminated by calcination at 600 °C for 2 hours, then subjected to an extra 2-hour grinding process. The integrated thermal and mechanical treatment greatly improved the material's reactivity and fineness. In Sajjad Ali Mangi et al.'s (2019) [9] research, coal bottom ash was oven dried for 24 hours at 110 ± 5 °C and ground for 2 hours using LAA grinding before it was ground in a ball mill for 20, 30, and 40 hours to investigate the effect of grinding time on particle fineness and effectiveness in concrete mixes with different levels of cement replacement. Lastly, Hui Li et al. (2016) [10] investigated high-energy milling (HEM) of fly ash at 700 rpm with a 10:1

weight ratio of ball to powder. The milling time was changed to assess its effect on particle size and reactivity. The process was effective in grinding fly ash into nano-sized particles and making its performance as supplementary cementitious material better. Through various high-energy milling methods have been documented in literature, the use of the Los Angeles abrasion machine — usually employed for aggregate toughness test — for long-term grinding of ash is not very extensively studied. This research attempts to investigate the time-dependent influence of grinding on particle size distribution in industrial ash. The main scope is to determine the influence of grinding time on particle size reduction and hence the usability of the ash in sustainable concrete usage. The findings are presented not merely in physical terms but also in their implication towards saving the environment through reduction in CO_2 emissions and waste utilization.

2. Materials and Methods:

2.1 Materials: Raw Industrial Ash (Fly Ash): The raw industrial ash used as a supplementary material was procured from Nayara Energy Pvt. Ltd., Jamnagar, through Stallion Energy Pvt. Ltd. It was obtained in powder form as an industrial by-product.

2.2 Initial Characterization:

2.2.1: Chemical Composition of Industrial Ash: The Chemical characterization of the raw industrial ash was conducted using X-ray fluorescence (XRF), and the detailed oxide composition is provided in Table 1.

Table 1 Chemical Composition of Industrial Ash

Components	Results	Unit	Components	Results	Unit
SiO_2	59.8	Mass%	Cl	0.0265	Mass%
Al_2O_3	16.6	Mass%	ZrO_2	0.0207	Mass%
Cao	10.9	Mass%	NiO	0.0133	Mass%
Fe_2O_3	6.55	Mass%	ZnO	0.0075	Mass%
MgO	3.66	Mass%	CuO	0.0070	Mass%
K_2O	1.04	Mass%	Rb_2O	0.0060	Mass%
TiO_2	0.854	Mass%	Y_2O_3	0.0029	Mass%
SO_3	0.477	Mass%	PbO	0.0023	Mass%
SrO	0.0370	Mass%	As_2O_3	0.0011	Mass%

2.2.2: Particle Size Analysis: Particle size was determined on the Malvern Mastersizer 3000 instrument using dry dispersion. Values were between 1.63 µm (Dv10) and 97.5 µm (Dv100), with a median (Dv50) of 11.4 µm. Mean diameters were D [3,2] = 2.81 µm and D [4,3] = 17.2 µm. Span was 3.584, showing a moderately broad distribution. Specific surface area was 2133 m²/kg. Particle size histogram is presented in Figure 1.

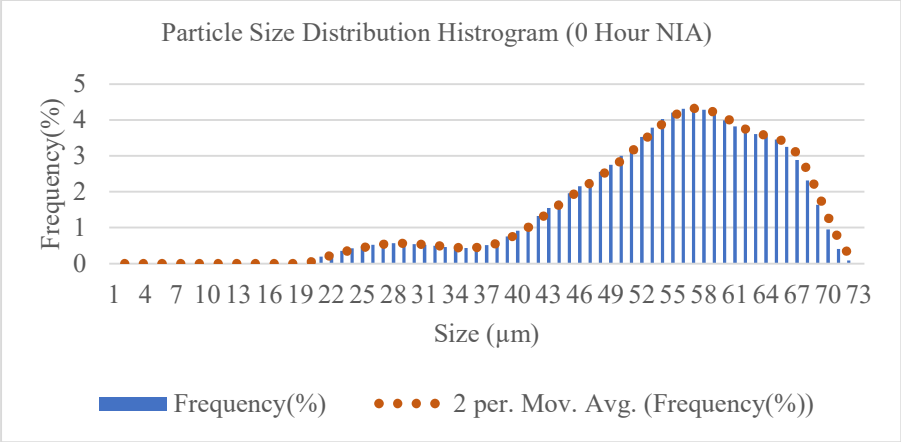

Figure 1 PSD Histogram of Raw Industrial ash

Table 2 Result of Particle Size Distribution of Raw Industrial ash

Concentration	0.0010%	Dv (10)	1.63 µm
Uniformity	1.070	Dv (50)	11.4 µm
Specific Surface Area	2133 m²/kg	Dv (90)	42.6 µm
D [3,2]	2.81 µm	Dv (95)	52.9 µm
D [4,3]	17.2 µm	Dv (99)	70.9 µm
Span	3.584	Dv (100)	97.5 µm
Result Unit	Volume		

2.2.3. Surface Area Analysis with Pore Size Distribution for Raw Industrial Ash:

2.2.3.1. Surface Area Analysis: Surface area was analyzed by multiple BET (Brunauer, Emmet, and Teller) on instrument Quanta chrome Novae2200. The Graph of Relative Pressure vs 1 / [W((Po/P) - 1)] is shown in figure -2 and Testing result of BET analysis is indicated in table-3.

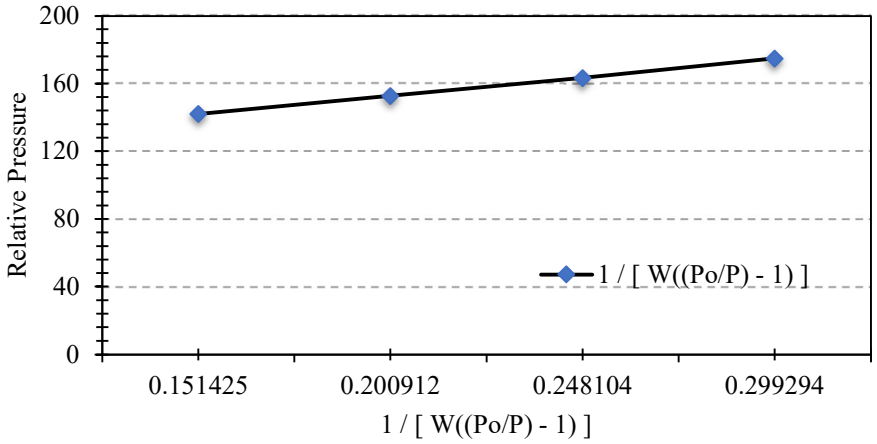

Figure 2. Graph of Relative Pressure vs 1 / [W((Po/P) - 1)]

Table 3: Result of Multipoint BET Analysis

Result Multi Point BET			Multipoint BET Summary/Results	
Relative Pressure	Volume Adsorbed (cc/g)	1 / [W((Po/P) - 1)]	Isotherm Branch	Adsorption
0.151425	1.00446	142.143	Slope	222.097
0.200912	1.31753	152.6866	Intercept	108.33
0.248104	1.61554	163.4211	Correlation coeff., r	0.999905
0.299294	1.95399	174.9002	C constant	3.05018
			Surface area (m^2/g)	10.539

2.2.3.2.: Pore Size Distribution of Raw Industrial Ash by BJH Method:

The Graph of Pore diameter vs Cumulative Pore Volume (cc/g) vs dV(d)[cc/nm/g] is shown in figure-3 and result of BJH method is mentioned in table-4.

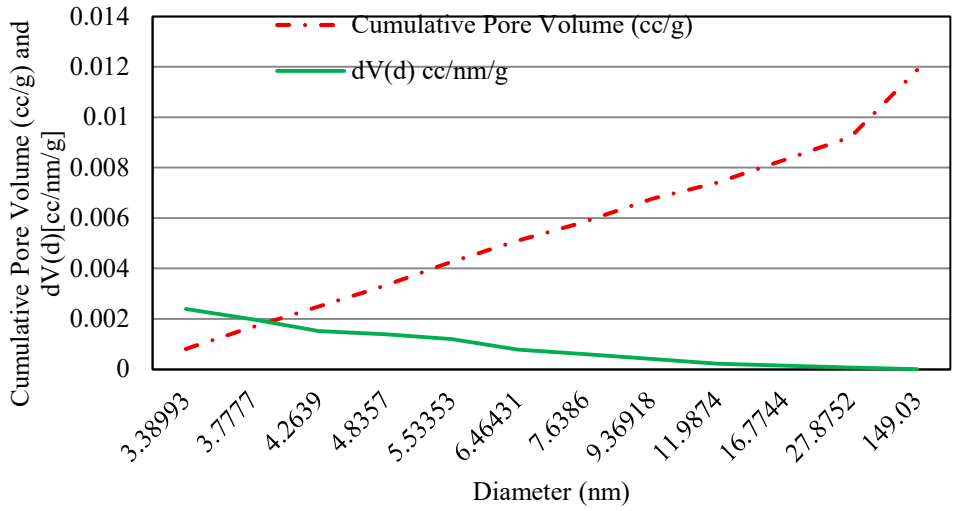

Figure 3. Graph of Pore diameter vs Cumulative Pore Volume (cc/g) vs dV(d)[cc/nm/g]

Table 4. Result of BJH adsorption

BJH Pore Size Distribution-Adsorption			BJH adsorption Summary/Results	
Diameter (nm)	Pore Volume (cc/g)	dV(d) cc/nm/g	Surface Area (m^2/g)	5.93915
3.38993	0.000807769	0.002395872	Pore Volume (cc/g)	0.0118793
3.7777	0.001676021	0.001980569	Pore Diameter Dv(d)	3.38993
4.2639	0.002486639	0.001517983		
4.8357	0.00332838	0.001380808		
5.53353	0.004265535	0.001192246		
6.46431	0.00510026	0.000776114		
7.6386	0.005860389	0.00059708		

9.36918	0.006750107	0.000406621		
11.9874	0.007419955	0.000219733		
16.7744	0.0083065	0.00013586		
27.8752	0.009226165	5.86668E-05		
149.03	0.01187934	1.17069E-05		

2.2.4: Mineralogical Study of Industrial Ash: XRD Analysis

X-Ray diffraction method is successfully used to understand the relation of morphology phenomenon. It is globally accepted non-destructive technique to identify the structural information of the material. It is sole technique to comprehend the crystal structure and chemical. Figure-5 illustrates the XRD of industrial ash.

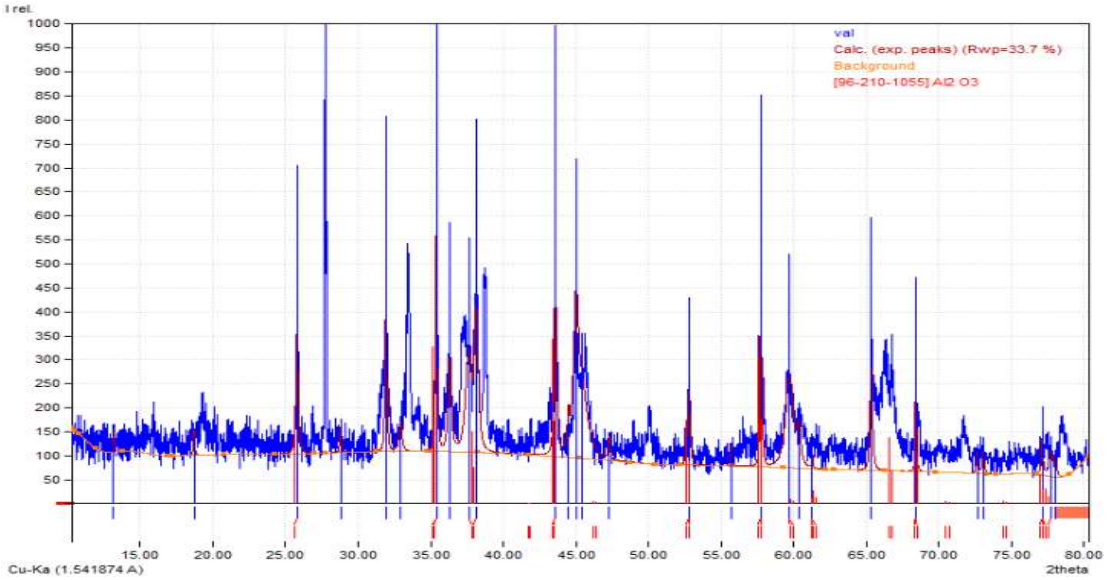

Figure 5 XRD of Industrial ash

2.3 Grinding Procedure:

2.3.1: Material Preparation by Using Los Angeles Machine and Grided Material Identification by Sophisticated Testing:

Experimental study was conducted through a Los Angeles machine, an abrasive grinding machine. A steel drum containing a chain of steel balls and a grinding charge is the composition of the machine. The fly ash was grided through utilizing the varying diameter ball size for varying time periods from 1 to 5 hours, followed by intervals of 5 hours from 5 to 20 Hours.

2.3.2: Grided Material Identification by Sophisticated Testing:

2.3.2.1: Particle Size Analysis of Grided Industrial Ash by Ball Milling:

In this study work the effect of grinding time on particle size and surface area of fly ash. The sample has been taken with the specified interval time and it is analyzed by the sophisticated testing and the results are concluded. The results of samples are mentioned in table-5 and few particle size distribution histograms of the samples are given in figure-6 to 8. For measuring the particle size, the samples of different griding times were analyzed and tested with a Master-Sizer 3000 particle size analyzer. The particle size was tested with the dry method.

Table 5. Result of Particle Size Analysis of 1,3,5,10, and 15 Hours Grided Industrial ash

Time Period (Hours)	1 Hours	3 Hours	5 Hours	10 Hours	15 Hours
Concentration	0.0010%	0.0009%	0.0008%	0.0007 %	0.0008 %
Uniformity	1.121	1.048	1.075	0.879	0.926
Specific Surface Area	2247 m²/kg	2296 m²/kg	2468 m²/kg	3106 m²/kg	3647 m²/kg
D [3,2]	2.67 µm	2.61 µm	2.43 µm	1.93 µm	1.65 µm
D [4,3]	16.1 µm	14.6 µm	13.6 µm	8.88 µm	7.38 µm
Span	3.719	3.465	3.532	2.816	2.966
Result Unit	Volume	Volume	Volume	Volume	Volume
Dv (10)	1.47 µm	1.44 µm	1.25 µm	0.776 µm	0.583 µm
Dv (50)	10.4 µm	9.85 µm	8.99 µm	6.82 µm	5.49 µm
Dv (90)	40.1 µm	35.6 µm	33.0 µm	20.0 µm	16.9 µm
Dv (95)	50.9 µm	45.1 µm	43.7 µm	23.4 µm	19.8 µm
Dv (99)	70.1 µm	62.3 µm	63.6 µm	29.3 µm	24.6 µm
Dv (100)	97.6 µm	86 µm	86.3 µm	35.3 µm	31.0 µm

Particle Size Distribution Histogram of Grided Industrial Ash

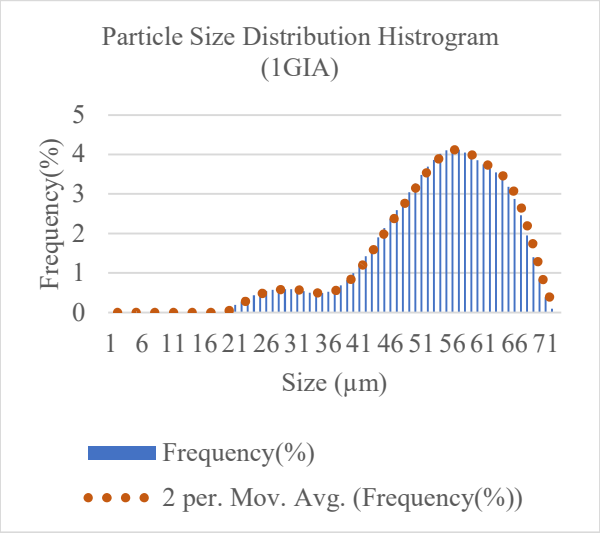

Figure 6 Particle Size Distribution of 1 GIA

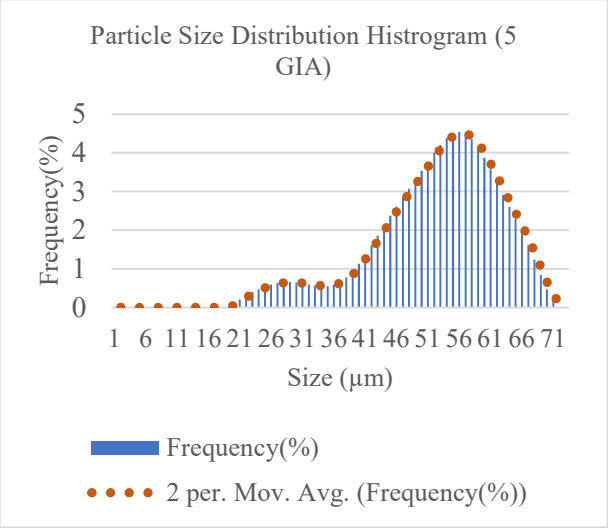

Figure 7 Particle Size Distribution of 5 GIA

Table 6. Physical Properties of various Grinding Times of Industrial ash.

Griding Time (Hours)	Specific surface area (m^2/kg)
1	2247
2	2281
3	2296
4	2392
5	2468
10	3106
15	3647
20	4201

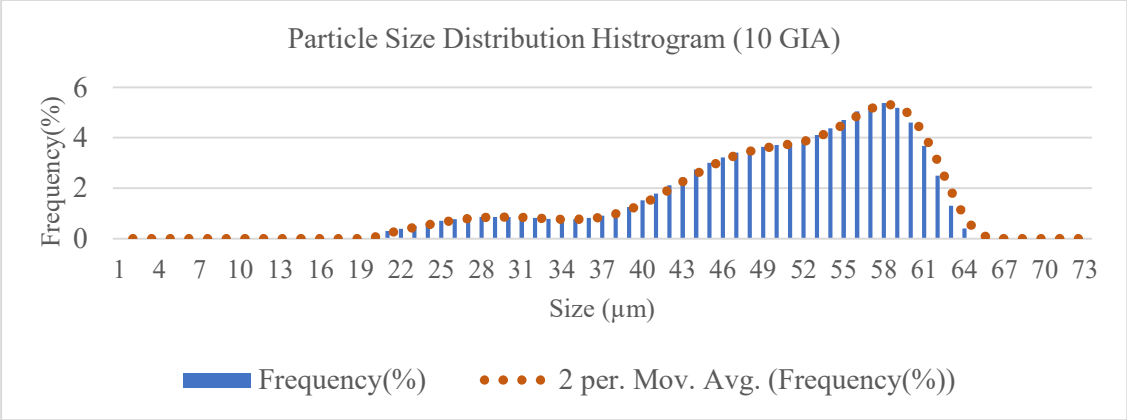

Figure 8 Particle Size Distribution of 10 GIA

3. Results and Discussion:

3.1 Effect of Grinding Duration on Particle Size and Surface Area with Time: Present a Table-6 display the result of surface area at different grinding times (0, 1, 2, 3, 4, 5, 10, 15, 20 h).

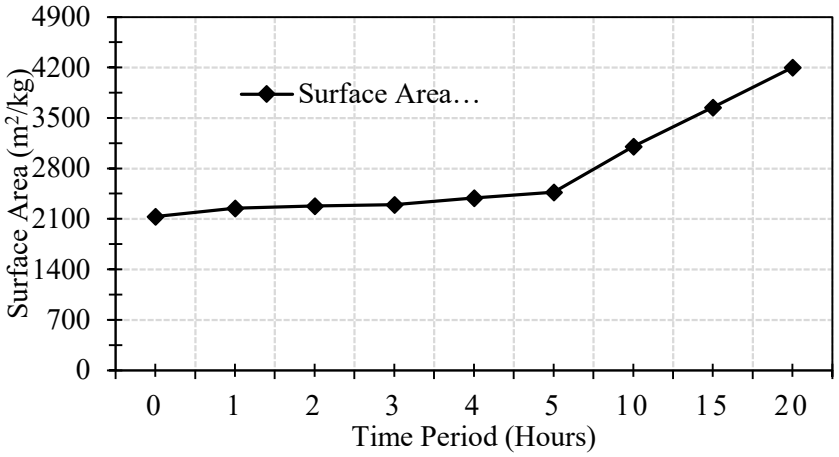

Figure 9. Graph for Griding Time vs Surface Area (m^2/kg)

The Graph in figure 9 illustrates the way that surface area of industrial ash rises consistently with grinding time to more than 4200 m²/kg at 20 hours.

Table 7. Particle Size Analysis vs Griding Time Period

Hours	Dv(10) µm	Dv(50) µm	Dv(90) µm	Dv(95) µm	Dv(99) µm	Dv (100) µm
0	1.63	11.4	42.6	52.9	70.9	97.5
1	1.47	10.4	40.1	50.9	70.1	97.6
2	1.45	10.1	37.77	48.11	66.25	92.2
3	1.44	9.85	35.6	45.1	62.3	86
4	1.36	9.48	34.4	44.47	62.9	86.2
5	1.25	8.99	33	43.7	63.6	86.3

10	0.776	6.82	20	23.4	29.3	35.3
15	0.583	5.49	16.9	19.8	24.6	31
20	0.385	4.22	14.1	16.38	20.12	26.2

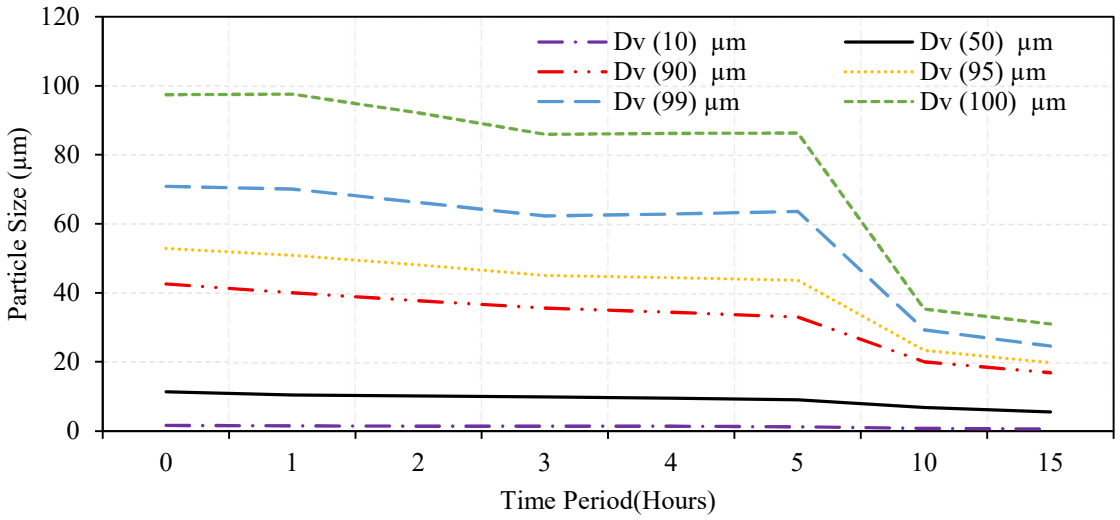

Figure 10. Graph of Particle Size (Dv (µm) vs Time Period

Discussion on Particle Size Reduction and Surface Area Development:

From the figure-10 the particle size measurement demonstrates the profound effect of grinding time in the Los Angeles Abrasion (LAA) machine on the fineness of industrial ash particles. The raw ash initially had a median size (Dv50) of 11.4 µm, a maximum size (Dv100) of 97.5 µm, and a specific surface area of 2133 m²/kg. Through progressive grinding, uniform decrease in particle size was noted in all characteristic values (Dv10, Dv50, Dv90, Dv95, and Dv100), with uniform growth in surface area. During initial grinding (0–5 hours), size reduction was moderate, which largely accounted for the disintegration of big agglomerates. For instance, after 5 hours, the Dv50 came down to 8.99 µm, while the surface area grew up to 2468 m²/kg. This period can be taken as the primary fragmentation stage, during which the machine impact and abrasion forces mainly focus on coarse particles. After 5 hours, grinding became more effective in generating ultrafine fractions. At 10 hours, the Dv50 dropped dramatically to 6.82 µm, along with a tremendous rise in surface area to 3106 m²/kg. With prolonged grinding for 15 and 20 hours, the purification was more significant, with Dv50 being 5.49 µm and 4.22 µm, respectively. The smallest fraction, Dv10, was 0.385 µm at 20 h, and Dv100 went down to 26.2 µm. The specific surface area almost doubled, being 4201 m²/kg. Generally, the results indicate two phases of grinding: gradual disintegration (0–5 h) and intensive grinding (10–20 h). This optimization enhances surface reactivity, packing density, and possible hydration performance, asserting the efficacy of LAA grinding in the production of sustainable ultrafine ash for cementitious purposes.

4. Conclusion:

This study underlines the capability of mechanical grinding with a Los Angeles abrasion machine to effectively decrease the particle size of industrial ash in 20 hours. The median particle size (D50) fell from 11.40 µm to 4.2 µm, and (D100) fell from 97.5 µm to 26.2 µm with a very large increase in particles smaller than 45 µm and the ash surface area increased in all cases with gridding, from 2133 m²/kg (raw ash) to 4201 m²/kg (20 hours milling). These finer particles are better suited for use as supplementary cementitious materials in concrete. In addition to the technical advancements, this reduction in particle size holds significant environmental significance. Through facilitating the utilization of industrial ash as a

partial substitute for Portland cement, this process can contribute to lower CO_2 emissions for the cement and concrete industry.

References:

[1] Yu Xuan Liew et.al, Suitability of Using LA Abrasion Machine for the Nano Manufacturing of Palm Oil Fuel Ash and Incorporating in Mortar Mixture, The Open Civil Engineering Journal (2023).

[2] Khan R A et.al, The effect of coal bottom ash (CBA) on mechanical and durability characteristics of concrete, Build. Mater. Struct.3:31-42(2016).

[3] M A Tambichik et.al, Combining pozzolanic material as ternary binder in Recycled Aggregate Concrete (RAC) to develop a new sustainable concrete, IOP Conf. Series: Materials Science and Engineering 713, 012013 (2020).

[4] Yong-Sik Chu et.al, Reactivity of fly ashes milled in different milling devices, Rev. Adv. Mater. Sci.; 58:179–188 (2019).

[5] Ghasan Fahim Huseien et.al, Synergism between palm oil fuel ash and slag: Production of environmental-friendly alkali activated mortars with enhanced properties, Construction and Building Materials 170, 235–244 (2018).

[6] Mohd Warid Hussin et.al, Durability of high strength concrete containing palm oil fuel ash of different fineness, Malaysian Journal of Civil Engineering 21(2): 180-194(2009).

[7] Carlos Antonio Rosas-Casarez et.al, Influence of Size Reduction of Fly Ash Particles by Grinding on the Chemical Properties of Geopolymers, Appl. Sci., 8, 365; doi:10.3390/app8030365 (2018).

[8] Belal Alsubari et.al, Heat-treated palm oil fuel ash as an effective supplementary cementitious material originating from agriculture waste, Construction and Building Materials 167, 44–54 (2018).

[9] SAJJAD ALI MANGI et.al, Effects of ground coal bottom ash on the properties of concrete, Journal of Engineering Science and Technology Vol. 14, No. 1 338 – 350 (2019).

[10] Hui Li et.al, Comparative study on the characteristics of ball-milled coal fly ash, J Therm Anal Calorim ,124:839–846(2016).