Characterizations of the evolving properties during the milling time of Wood Fly Ash Nanoparticles (WFA-NPs)

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Abstract

Wood Fly Ash (WFA) as a waste product from the combustion of wood has been regarded as environmental pollution since the generation of wood fly ash is much across the continent predominantly in the villages and most cities of the world. In recent time, effective and reliable utilization of WFA has been developed in areas like filler, additive, aggregate, reinforcement, water purification, activated carbon and most especially construction industries. Its efficiency and performance have been traced to whether is in the form of nano-, micro-, and macro- particles. Effects of milling time on WFA has been studied, its characterizations using Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDX) analysis, X-Ray Fluorescence (XRF), and X-Ray Diffraction (XRD) has been studied. The SEM magnification was set to 1000x at 50 µm and the milling time was carried out at 0, 20, 40 and 60 mins. Digital Vibratory Disc Milling Machine (VDMM) rated 380V/50 Hz at 940 rpm was used for the milling. WFA was received at micro-sized particles, it was washed with distilled water to remove impurities and dry in an oven set at 80°C for 48 hours then sieved according to ASTM standard using 75 µm size. The particles size got reduced from 75 µm to average diameter of 200 nm. It was established in the study that the duration of the milling affects volume, surface area, particle size, pore size distributions, microstructure and some other mechanical properties as well as the morphology of the powder.

Keywords: SEM-EDX, Milling Time, Nano Particle, Vibratory Disc Milling, Wood Fly Ash

1.0 Introduction

Fly ash has been described as the fine particulate by-product that was released into the atmosphere together with gases as a result of combustion processes (Gianoncelli et al., 2013; Rose, 2018; Rudolf Hela, 2013). Fly Ashes either from municipal solid waste or thermal plant solid waste has been regarded as hazardous waste due to toxic contaminations like chloride salts, heavy metals, dioxins as well as some organic compounds present within (Mubeen, Buekens, Chen, Lu, & Yan, 2017). Fly ash can be described as fine and spherical glassy particles which contain fundamentally of Al_2O_3, SiO_2 as well as CaO as the main constituent (Feng & Clark, 2011). It was established by several researchers that Wood Fly Ash belongs to type F fly ash in the aggregate of Al_2O_3 + $Fe2O_3$ + SiO_2 mass exceeds about 70% sum total of fly ash mass (Liu et al., 2018). High levels of macronutrients are produced from wood ash made from hardwood than conifers, and it has been reported that silica content is usually low (Pitman, 2018; Xu & Shi, 2018). Soil type and application rate are primary effects of wood ash.
It has been reported by (Hamidi, Man, Azizli, Ismail, & Nuruddin, 2014) that Fly ash has great potentials to be used in many applications as a result of its abundance and low cost. Notwithstanding, its larger particle size has resulted

in low properties of the resultant product and low reactivity (Ramu, 2016), these reasons propel this current research. High energy milling (HEM) has been used by many researchers to carry out milling of powders (Z. Chen et al., 2016; Dvorak & Hájková, 2016; Li, Chen, Cao, Liu, & Li, 2016; Paul, Manna, & Nando, 2007) and one of the HEM types is high-speed grinding (HSG). The like of (Kumar, Kumar, & Mishra, 2018; Rajak, Raj, Guria, & Pathak, 2017) carried out mechanical activation process by the use of high energy planetary ball mill and validate the experimental result with a milling simulation called back-calculation method for accuracy and acceptability.

Fly Ash has been used by various researchers such as (R. Chen, Li, Xiang, & Li, 2016; Cross, Stephens, & Vollmer, 2005; Dinaharan, Nelson, Vijay, & Akinlabi, 2016; Hemalatha & Ramaswamy, 2017; Kayali, 2005; Ma et al., 2018; Roberto, José, & Andrade, 2017) for activation carbon, water purification, additive/replacement used in Portland cement, as reinforcement for bricks, composite matrix in metal, polymers and ceramics as well as filler in many applications.

Nanotechnology is a control, understanding and restricting of matter in the form of nanometers (i.e. <100 nm) to create materials with basically new properties and functions. Nanotechnology can have applications in engineering especially structural composites, construction, nano-sensors, material coatings, concrete etc(Mehrinejad, Mohseni, Ali, & Sarker, 2015). The effect of milling into nano form was to make the utilization of WFA in any application easier and faster since the particle size greatly affects the mechanical properties and its utilization in another form.

1.1 Application of Fly Ash

The performance of Fly Ash (FA) either wood or coal in any application depends wholly on whether is in form of nano-, micro-, and macro- particles. It has been established by various authors that tend to investigate the effect of milling on Fly Ash that reductions in particle size improve mechanical properties, morphology, and also absorption rate is increased when in finest form. Despite the hazardous and environmental unfriendliness of fly ash, so many benefits can be derived in it. Some of the uses and emerging uses of Fly Ash as established by (Chatterjee, 2010) among others are:

1. Fly Ash has potential applications in cement, brickmaking, and concrete, this is the most famous application of Fly Ash which has been thriving for decades. It was established that cement constitute up to 40- 45 % Fly ash by weight (Rycroft, 2017; Tiwari, 2016)
2. It has been established that FA has potential applications in geopolymers concrete. Though, the market for this application is developing slowly.
3. FA finds application in the area of Zeolites. These are crystalline solid structures that comprise Oxygen, Aluminium, and Silicon. These Zeolites can be used for water purification, petrochemical cracking and water softening.
4. Fly Ash can be used to remove Acid Mine drainage also known as AMD which is the contaminant in mine water as well as mine backfilling.
5. FA also very useful in Agriculture to reduce the poor quality of agricultural land as well as to rehabilitate mine land. FA efficacy can be utilized to modify soil health and the performance of crop yielding. Organic fertilizer can be made through it and it can as well serve as bio-leaching.
6. FA can also be used as road surface and filler applications. FA finds new interest in asphalt filler and also as embankments layer for tar roads.
7. FA can be used as stiffeners and fillers in plastics and rubber products. It was established that fly ash increases the strength as well as elasticity of the product which eventually helps in curing and molding. In facts, FA used in composite insulators of silicone rubber improves tensile strength, dielectric strength, and resistivity while maintaining its hydrophobic characteristics.
8. It can also be used in the mining of coal ash for rare earth and metals.
9. It can be used as ceramic glazes. It acts as flux thereby reducing the melting point of the glaze.
10. It can serve as compost for landfilling, forestry and agricultural applications.
11. It can be used for making soap. Potassium hydroxide (Caustic Potash) is made through it which is one of the ingredients for making soap.

Value of the data

- The data is very valuable in a manner that the users will be able to know the variation in elemental compositions at different milling time as well as unmilled wood fly ash nanoparticle (WFA-NPs) as revealed by EDX.
- The data is of value to the users because the users will be able to know likely crystalline structures and minerals that are likely present in WFA as revealed by XRD
- The data obtained can be used in investigating surface processing and modifications as well as aggregate, filler and reinforcement in ceramics, polymer and metal matrix composite at different milling time.
- The data is of value because the users will be able to know the type of chemical composition present in wood fly ash as revealed by XRF.
- The data revealed the micrographs of the milled and unmilled wood fly ash at different milling time, the users will be able to observe the trend and patterns of the milled wood fly ash which can also predict the absorption rate of WFA-NPs at any applications.

2.0 Material:

The Wood Fly Ash (WFA) used in this study was collected after a bunch of firewood was burned into charcoal and further undergo combustion process which turned it into ash. The ash was then collected, sieved, washed with distilled water to remove any foreign and unwanted particles, which was then discharged and drained. An electric oven set at 80° C was used to dry it to dryness for 48 hours. The Chemical Composition was analyzed by X-Ray Fluorescence (XRF) as shown in Table 1.

2.1 Experimental Methodology

After the Fly Ashes Samples collected were washed with distilled water to remove impurities and they were dry in the oven at 80° C for 48 hours in order to remove the water content, after drying it was then sieved using ASTM meshes standard range by employed KingTest Sieve of 75 µm size on KingTest Sieve Shaker (VB 200/300) having operating voltage 220V/50 Hz and 5 A. The Fly Ashes proportion that passed through the Mesh size of 75 µm was then taken for milling. The flowchart procedure of the powder preparation and characterizations is showed in Figure 1.

2.2 Chemical Compositions of Raw Fly Ashes

In this research, the chemical composition of WFA in the as-received state (raw state) was analyzed with the aid of X-Ray Fluorescence (XRF) and the outcome of the analysis is as depicted in Table 1:

Table1: The Chemical Composition Analysis of WFA using XRF

CHEMICAL FORMULA	**WFA (%)**
Al_2O_3	10.7
SiO_2	46.31
MgO	0.36
TiO_2	0.933
Fe_2O_3	17.28
CaO	3.002
MnO_2	0.0471
Na_2O	2.48
P_2O_5	0.113
K_2O	2.53
SO_3	3.423
LOI	8
	Σ= 87.17

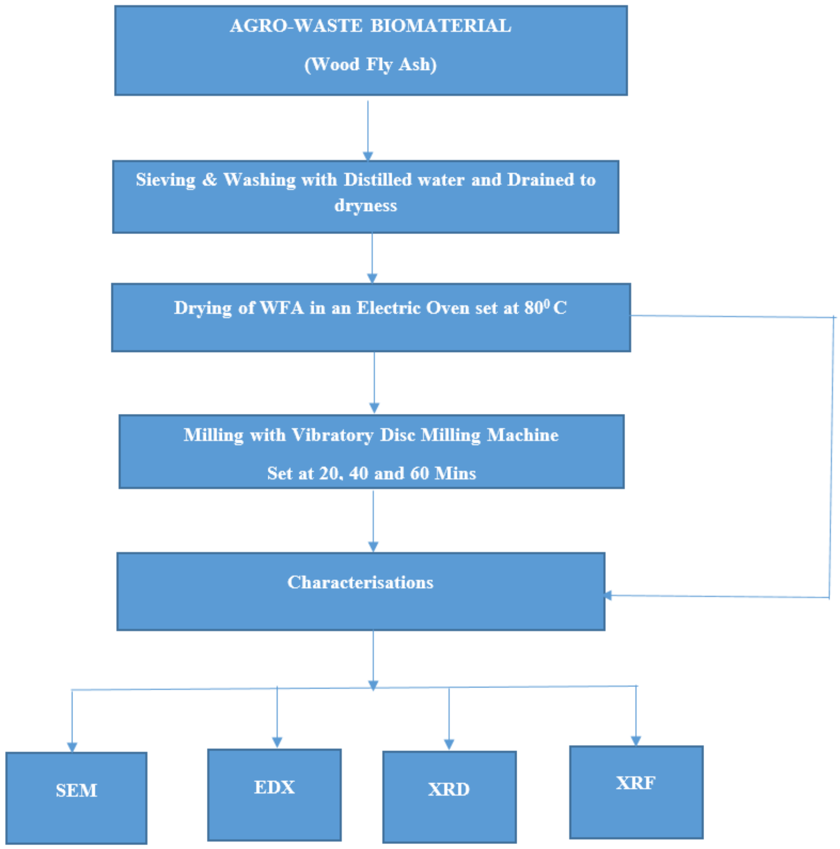

Figure 1: Flowchart for the preparation and characterizations of WFA

2.3 Vibratory Disc Milling Machine (VDMM)

A Digital Vibratory Disc Grinding Mill Lab Pulveriser (740 x 740 x 950 mm) with Machine Model 2MZ-200 supplied by FTLAB Technology has 2 pieces of bowl with capacity of 200 g per bowl, feed size of less than 15mm, pulverize time of 3 -5 minutes or more depends on the nature of the materials, while the operating voltage of 380V/50 Hz, motor capacity of 1.5 kW with rotational speed of 940rpm was employed to carry out dry mechanical milling (DMM) at varying processing time of 20, 40 and 60 mins which enables the reduction in particle size from micron to nano levels for wood fly ash powder. The machine was thoroughly washed, dried and cleaned with acetone before and after use to remove any contaminants that may be present. 40 g of WFA samples was charged into each bowl and then set for running. The machine was interrupted every 5 mins of operation in order to avoid a rise in temperature and at the same time to limit adherence of the powder within the container walls, the cooling interval before the next running was 20 mins.

2.4 Data Acquisition and Characterizations

Mechanical dry milling (MDM) was carried out with different process milling time of 20, 40 and 60 mins. Wood Fly Ash Nanoparticles (WFA-NPs) obtained was then characterized by the use of Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDX) analysis, X-Ray Fluorescence (XRF), and X-Ray Diffraction (XRD). These tools were employed to determine the morphology and elemental and chemical compositions at different milling time of 0, 20, 40, 60 mins.

2.5 Scanning Electron Microscope

TESCAN model, type VEGA 3 LMH and model no VG9731276ZA (Figure 2) with the following details 50/60 Hz, 230 V and 1300 VA was the type of SEM machine that was employed for the studies. In order to have the sample more conductive and to have better resolution, the samples were sputter coated with a thin layer of carbon just before the scanning electron microscope analyses coupled with Energy Dispersive X-ray spectrometer (EDXS) analyses. The beam intensity used in the analysis was 12 and the accelerating voltage used was 20 kV, all micrographs were taken at SEM magnification of 1.00 kx which is 50 µm for different milling times of 20, 40 and 60 mins. The elemental compositions of WFA-NPs were also analyzed at different milling times by the use of EDS. The SEM micrographs of different milling time were presented in Figure 3 and the EDXS data were presented in Figure 4 and the summary has been presented in Table 2. Sizes of WFA-NPs were determined using SEM/software.

Figure 2: Photograph of the SEM-EDX Analysis Machine used

B1, B2, and B3 presented in figure 5 represent the micrographs of unmilled at 0 min, and that of milled at 20, 40 and 60 mins respectively at 50 µm for an SEM Magnification of 1000x. The micrographs revealed that unmilled has the largest particle sizes when compared to other milled powder. The duration of milling greatly affects the distribution of the particles and how the crystallites are arranged.

Table 2: Variable in Elemental Compositions of WFA-NPs as analyzed by EDXS

POWDER	ELEMENT	ELEMENTAL COMPOSITION AT DIFFERENT MILLING TIMES			
		0 Min (Unmilled)	20 mins	40 mins	60 mins
WFA-NPs	Ca	76.6	85.0	85.4	84.9
	K	10.3	3.6	3.6	3.8
	Mg	5.4	3.7	3.4	3.0
	Si	4.7	4.6	4.5	5.0
	Al	1.5	1.5	1.3	1.5
	Fe	1.5	1.6	1.9	1.8

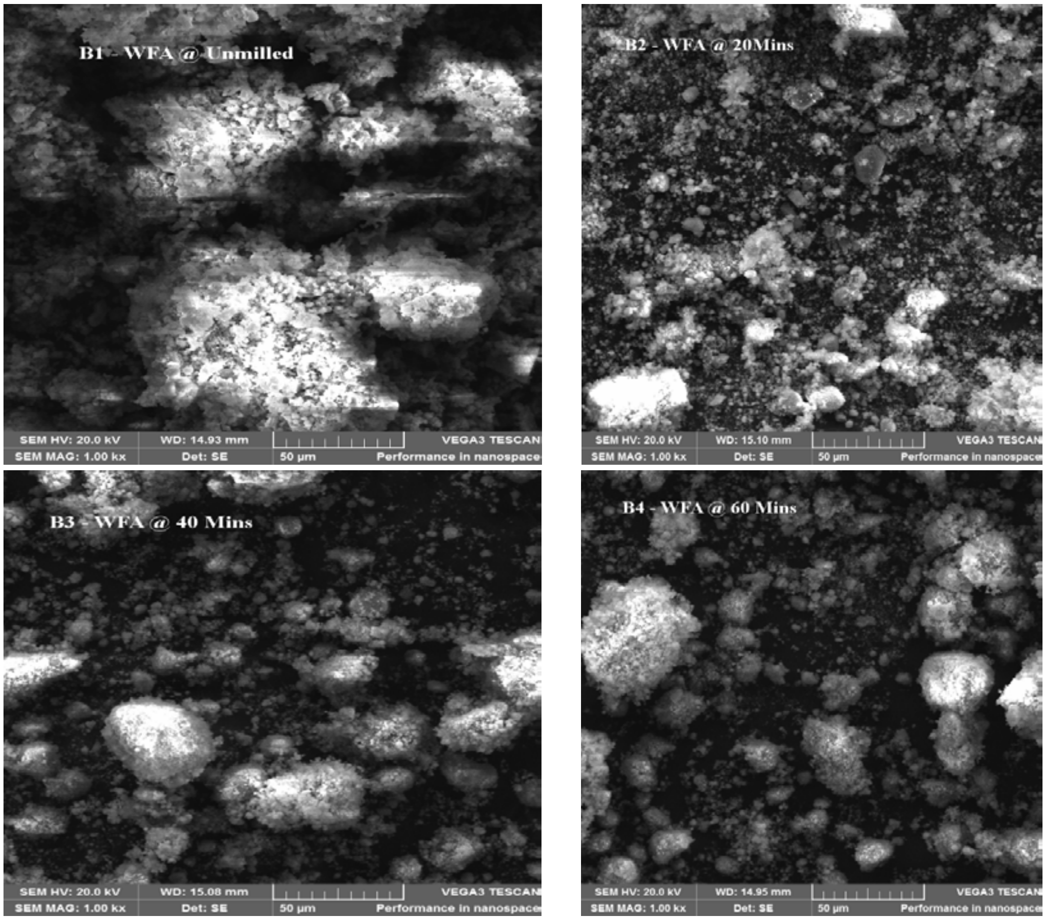

Fig 3: SEM Micrographs of Wood Fly Ash nanoparticles (WFA-NPs) at 1000x SEM MAG at different milling time of 0, 20, 40 and 60 mins.

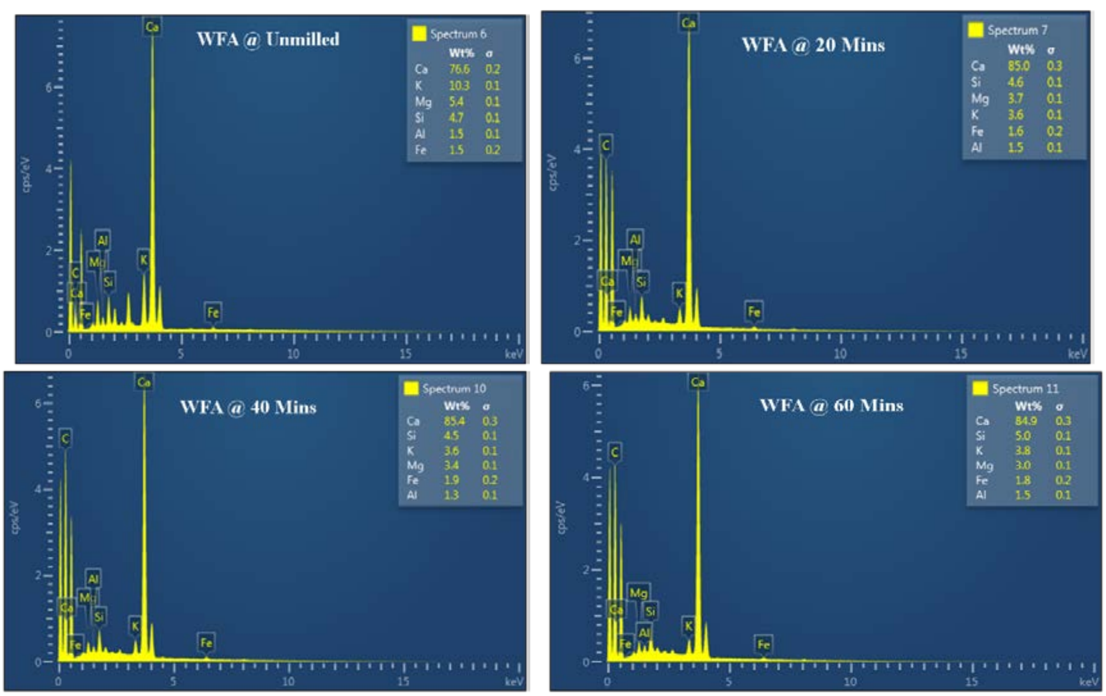

Figure 4: WFA Elemental Compositions at different Milling time as analyzed by EDXS

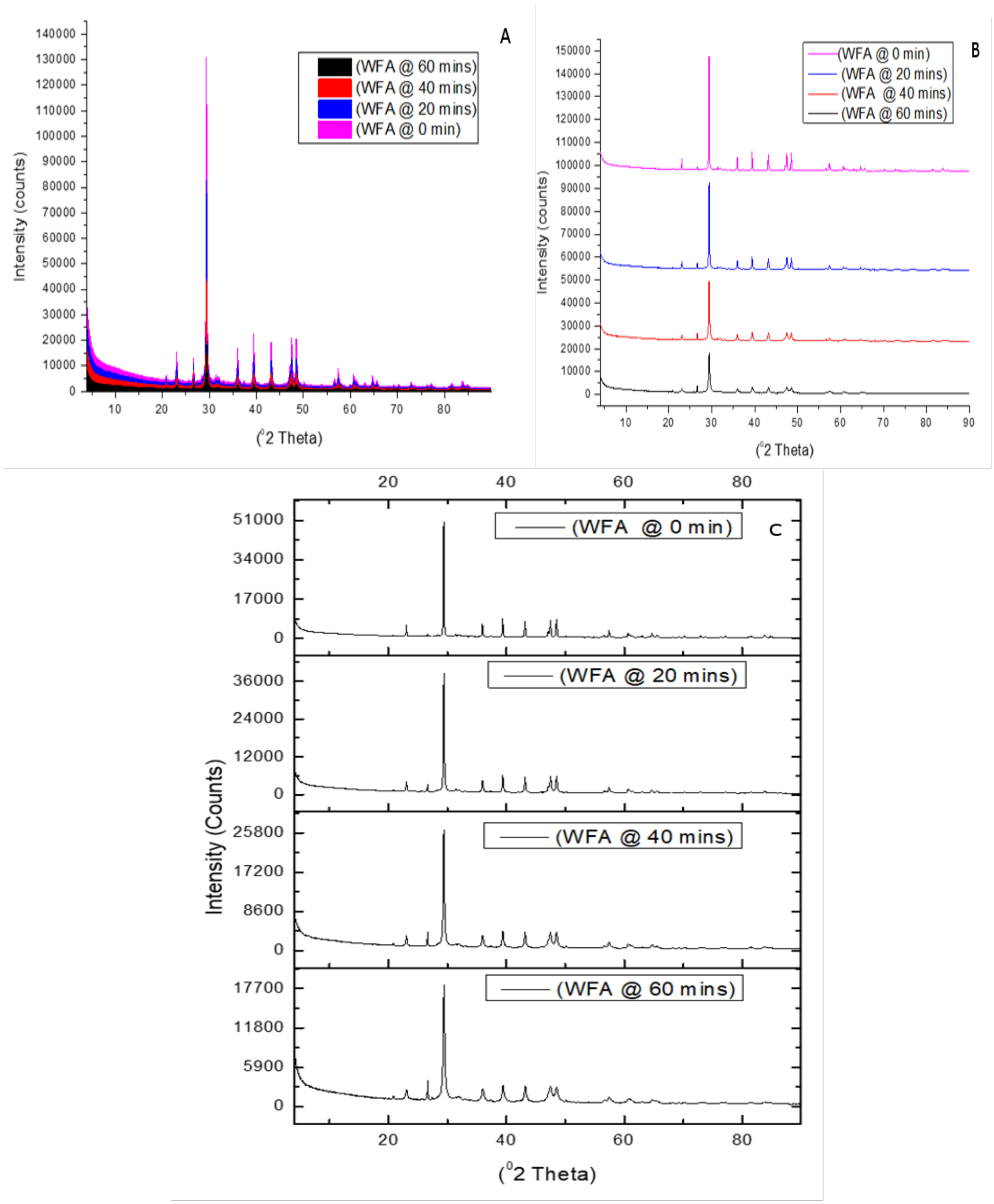

Figure 5: (a & b) showed Cumulative Intensity of XRD diffractograms for Wood Fly Ash Nano-Particles (WFA-NPs) at different Milling time (c) X-ray diffraction pattern (XRD) for WFA at different milling time

2.6 X-Ray Diffraction (XRD):

PHILIPS X'Pert which was made in Holland with the model number (12NC: 943003040601) and operating parameters, PW: 3040/60, 240 V, 8.5 kVA, and 50 Hz was employed to study and obtain the ultimate quality of diffraction data of the samples and to confirm the crystal structure of the samples used. Milled WFA-NPs at 20, 40 and 60 mins and unmilled samples were analyzed with the aid of X-Ray diffraction (XRD) for determination of the existence of crystalline phases before and after disc milling and mineralogical composition was obtained. The crystal phases detected by the XRD are rhombohedral, hexagonal, cubic and tetragonal. After the completion of the milling process, the samples of WFA were analyzed by X-ray diffractometer with the following processing parameters: Current 40 mA, excitation voltage of 40 kV, scanning rate 0.05 (2ϴ/seg), angle 2ϴ between 10° and 80°, respectively. The diffraction pattern at different milling times is as depicted in figure 5. The XRD was acquired using copper Kα radiation **(λ = 1.5406 Å)** and kβ radiation **(λ = 1.39225 Å)** and an automatic divergence slit; i.e., an irradiated sample length that is independent of the Bragg angles ((2θ) in degree). XRD analysis showed that the most dominant Minerals in wood fly ash are calcite ($CaC0_3$), quartz (SiO_2), Sylvite (KCl), Lime (CaO), maghemite-Q (Ɣ-Fe_2O_3), nitratetine ($Na(NO_3)$) and magnetite (Fe_3O_4) phases.

An XRD has widely been used in the characterizations of the nanoparticle to study the crystal structure, peak, and crystallite size. Both milled and unmilled WFA powders are characterized by x-ray diffractometer, X-pert software is employed to analyze the phase transformation and structural changes micro to nano particles. XRD sample preparation is done by following the standard particle. Figure 5 (a, b & c) showed that XRD of the WFA powder before and after VDMM was used. Figure 5c shows the x-ray diffraction pattern of wood fly ash at different milling time, it was revealed that at 0 min (unmilled) WFA has the highest peak at 30° (2ϴ), this is where the highest intensity was attained, followed by 20 mins milled and the least peak was observed at 60 mins milled. Figure 5 (a and b) also show a similar pattern of x-ray diffraction but in a cumulative pattern. It is clearly shown in Figure 5b that unmilled WFA has the highest peak while 60 mins milled has the least peak. Figure 5a shows the area pattern of different milling time with respect to WFA. It was observed that unmilled has the highest peak and 60 mins milled has the least peak. It was observed in Figure 5c that at 0 min (unmilled), the intensity is at 51,000 counts, 36,000 counts at 20 mins, 25,800 counts at 40 mins and 17,700 counts at 60 mins. Origin 8.5 software is used to plot XRD graphs from the data acquired from the XRD analysis.

3.0 Conclusion

In conclusion, from the experiments carried out and the analysis, we, therefore, establish that the particle size of WFA has been reduced from micrometer to nanometer through the help of vibratory disc milling machine (VDMM). The particle size reduced from 75 µm to an average size of 200 nm after 60 mins milling with VDMM. It was observed that there is a reduction in crystallite size and the powder becomes amorphous. The crystalline structure, surface texture of the WFA has changed over the period of milling time as revealed by XRD and SEM. The nanoparticles of WFA can be useful in reinforcement, filler, activated carbon, water purification, it can also be utilized in construction industries as alternative or replacement to cement as well as aggregate in metal, polymer and ceramics matrices. SEM analysis revealed the morphology of WFA particles which was observed to be spherical in shape and the particle sizes reduce as the milling time increases, and the volume and the of the particles increases with increase in milling time. EDX revealed most elemental compositions that are dominant in WFA as presented in Table 2 and the chemical composition has revealed by XRF contained in Table 1. XRD analysis showed that the most dominant Minerals in wood fly ash are calcite ($CaC0_3$), quartz (SiO_2), Sylvite (KCl), Lime (CaO), maghemite-Q (Ɣ-Fe_2O_3), nitratetine ($Na(NO_3)$ and magnetite (Fe_3O_4) phases. The crystal phases detected by the XRD are rhombohedral, hexagonal, cubic and tetragonal.

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