

A Study into the effects of the milling time on the Physiochemical and Morphological Characteristics of Cow Bone Powder (CBP)

Omolayo M. Ikumapayi*^{1,2}

^{1,2} Department of Mechanical Engineering Science, University of Johannesburg, Auckland Park
Kingsway Campus, Johannesburg, 2006, South Africa

^{1,2} Modern and Advanced Manufacturing Systems (MAMS) Research Group, University of Johannesburg
oikumapayi@uj.ac.za ; Ikumapayi.omolayo@gmail.com

Esther T. Akinlabi^{1,2}

^{1,2} Department of Mechanical Engineering Science, University of Johannesburg, Auckland Park
Kingsway Campus, Johannesburg, 2006, South Africa

^{1,2} Modern and Advanced Manufacturing Systems (MAMS) Research Group, University of Johannesburg
etakinlabi@uj.ac.za

Abstract

Cow bone has been regarded as bio-waste and environmental pollutant, nevertheless, many researchers have developed ways to utilize this biomaterial and make it environmental friendly especially in the areas of reinforcement, activated carbon, filler, additive as well as water purification. It has also been established in the recent time that performance, efficiency and effective utilization of CBP depend solely on whether is in the form of nano-, micro-, and macro- particles (Oladele, I, 2016). The bone of the cow head (Skull) was collected from the abattoir, washed and cleaned from meat, sun-dried for 6 weeks and washed again with de-ionized water to remove impurities and contaminants, it was then transferred to the oven set at 50 °C to dry for 5 hours before crushing, it was then crushed and pulverised. The pulverized powder then dried in an electric oven set at 50° C for a week to ensure total dryness and sieved according to ASTM standard using 150 µm size. In this study, Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDX), X-Ray Fluorescence (XRF) have been employed to characterize the powder at different milling time varying from 0, 20, 40 and 60 Mins. Digital Vibratory Disc Milling Machine (VDMM) rated 380V/50Hz at 940 rpm was used for the milling. The SEM Images was set to 1.00kx at 50 µm, 2.00x at 20 µm and 5.00kx at 10 µm. The particles size got reduced from 150 µm to average diameter of 300 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: Cow Bone Powder, Milling time, SEM-EDX, XRD, XRF

1.0 Introduction

Cow bones are readily available at the abattoirs in South Africa as a result of a large number of cows being slaughtered daily in order to meet the demand of the people. Research showed that these bones are apparently become waste and pollute the environments making it unfit to live in. The study investigates how what regarded as environmental pollutants can be converted to useful application in engineering and other fields. Cow bone being natural animal fibre is expected to have good surface compatibility in addition to the structural compatibility requirements as biomaterials (Ahmad et al., 2014; Aramide, Ibitoye, Oladele, & Borode, 2010; Oladele & Adewole, 2013).

The performance of cow bone powder in any application has been traced to whether CBP is in form of nano-, micro-, and macro- particles. CBP has been established to have improved strength and wear properties when used as reinforcement and fillers (Adewole, 2015; Agunsoye, Talabi, Awe, & Kelechi, 2013). Some researchers have confirmed that CBP has absorption capacities to remove heavy metals such as Cadmium from the body of palm oil mill, also for energizers in case hardening of mild steel and other metals (Ahmad et al., 2012; Ihom, 2013). CBP has been applied in many applications such as removal of fluoride from the body of water, removal of lead in aqueous solution, and as the same time for the treatment of high strength industrial waste water (Abdulrahman, Latiff, Daud, Ridzuan, & Jagaba, 2016; Deydier, Guilet, Sarda, & Sharrock, 2005).

Researchers have done great works to investigate into natural fiber composite (i.e. biocomposites) as it applied in reinforcement either in natural or synthetics. Investigations have spanned into the distributions of CBP particles size in order to study its influence on mechanical and metallurgical properties (Asuke et al., 2012; Oladele & Adewole, 2013). It was revealed that fine cow bone particles improve strength while coarse particles will give improved toughness. It was revealed from the study that CBP either fine or coarse is structurally compatible and it is expected that this will also aid compatibility with the surface conditions as biomaterials since this is derived from animal fibers based particles. Cow bone particles have been proven to in a composite material of polymer to have increased tensile strength and hardness values (Daramola & Adewole, 2014) while rigidity and impact strength decreased. The study showed that the addition of CBP has a significant impact on wear behavior of the composite but there is no effect between the interaction of load and time (Oladele, I, 2016).

It was established by (Abdulrahman et al., 2016; Mohammed, Aboje, Auta, & Jibril, 2012) that activated carbon derived from cow bones are more effective and efficient in lowering the acidity content of palm oil when compare with dog bones, chicken bones, and goat bones.

Value of the data

- The data revealed the microstructures of the cow bone (Skull) powder at different milling time, this will be useful to the users as to know the likely trends and patterns of the CBP micrographs which will enable them to predict the absorption rate when using CBP in any application.
 - The data revealed the chemical compositions of CBP through the use of XRF, this will also be of great value to the user
 - The elemental compositions of the CBP were also revealed via the use of EDX, this will guide the users on the likely elements that are present in CBP
 - The data obtained can be used in investigating surface modification, surface texture, and processing which will be applicable in the reinforcement of metals, polymer, and ceramics composites, it will be used as filler, aggregate, activated carbon etc.
 - The data obtained can be used as the basis for determining other animal bones chemical compositions under the same experimental conditions
-

2.0 Material:

The bone of the cow heads (Skulls), de-ionized water, acetone was used in this study. The bones were collected from the abattoir as shown in [Figure 1a](#), washed and cleaned from meat, sun-dried for 6 weeks as shown in [Figure 1b](#) and washed again then dried, crushed and pulverized.

2.1 Experimental Methodology

After the Cow Bone (Skulls) Samples collected from the abattoir, washed and cleaned from meat, sun-dried for 6 weeks and washed again with de-ionized water to remove impurities and contaminants, it was then transferred to the oven set at 50 °C to dry for 5 hours and also cleaned with acetone before crushing, it was then crushed and pulverised. After which it was dried in the oven at 50 °C for seven days in order to remove the water content, after drying it was then sieved using ASTM meshes standard range by employed KingTest Sieve (see [Figure 1c](#)) of 150 µm size on KingTest Sieve Shaker (VB 200/300) having operating voltage 220V/50 Hz and 5 A. The CBP proportion that passed through the Mesh size of 150 µm was then taken for milling. The flowchart procedure of the powder preparation and characterizations is showed in [Figure 2](#).



Figure 1: (a) Fresh Cow Skull Bone Obtained (b) Dried Cow Bone (c) Cow Bone Powder (CBP) in a 150 µm ASTM standard Sieve

2.2 CHEMICAL COMPOSITIONS OF COW BONE POWDER

In this research, the chemical composition of Cow Bone Powder (CBP) was analyzed at 150 µm with the aid of X-Ray Fluorescence (XRF) spectroscopy with model PHILIP PW1404 XRF Wavelength Disperse Spectrometer and the outcome of the analysis is as depicted in [Table 1](#):

Table1: The Chemical Composition Analysis of CBP using XRF

CHEMICAL FORMULA	WFA (%)
CaO	45.06
SiO ₂	0.08
MgO	0.65
TiO ₂	0.06
Fe ₂ O ₃	0.25
Al ₂ O ₃	0.16
MnO	0.01
Na ₂ O	0.17
P ₂ O ₅	34.60
K ₂ O	0.12
ZnO	0.02
LOI	16.28
	Σ= 97.46

2.3 Vibratory Disc Milling Machine (VDMM)

A Digital Vibratory Disc Grinding Mill Lab Pulveriser (740 x 740 x 950 mm) as depicted in [figure 3](#) 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 pulverized cow bone powder (CBP). The machine was thoroughly washed, dried and cleaned with acetone before and after use to remove any contaminants that may be present. 100 g of CBP 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 and more depending on the rise in the temperature of the containing disc.

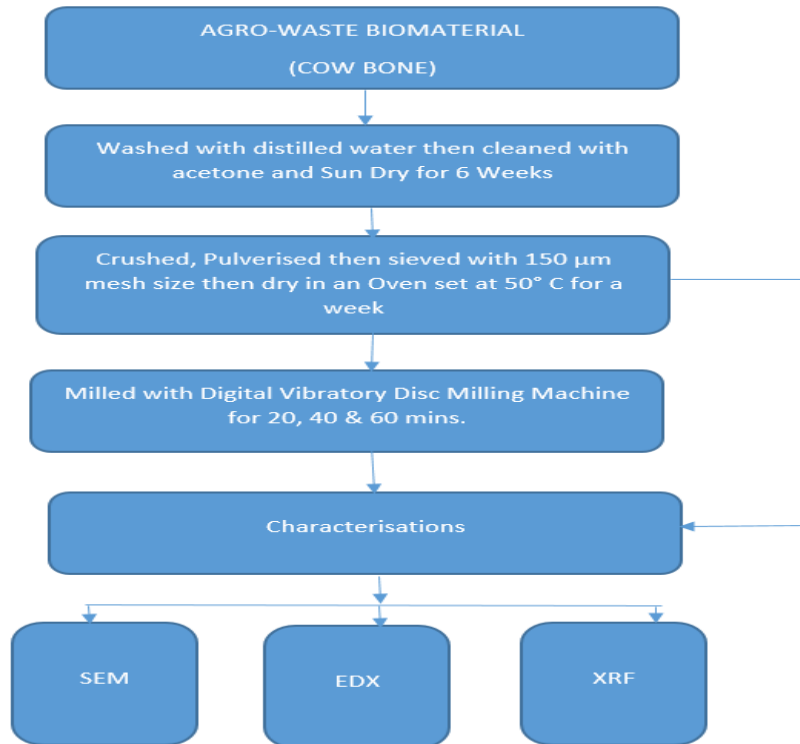


Figure 2: Flowchart for the preparation and characterizations of CBP



Figure 3: Diagram of Vibratory Disc Milling Machine (VDMM)

2.4 Microstructural Analysis

Cow Bone Powder (CBP) obtained was then characterized by the use of scanning electron microscopy (SEM) and Energy dispersive X-ray spectroscopy (EDXS). These tools were employed to determine the morphology and elemental composition at different milling time of 20, 40, 60 Mins and unmilled samples.

2.5 Data Acquisition and Characterizations

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

2.6 Scanning Electron Microscope (SEM)

TESCAN model, type VEGA 3 LMH and model no VG9731276ZA (Figure 4) 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 20KV, all micrographs were taken at SEM magnification of 1.00kx, 2.00kx and 5.00kx for different milling times of 20, 40 and 60 mins. The elemental compositions of CBP were also analyzed at different milling times by the use of EDS. The SEM micrographs of different milling time were presented in Figure 5 and the EDXS data were presented in Figure 6 and the summary has been presented in Table 2. Sizes of CBPs were determined using SEM/software.



Figure 4: Photograph of the SEM-EDX Analysis Machine used

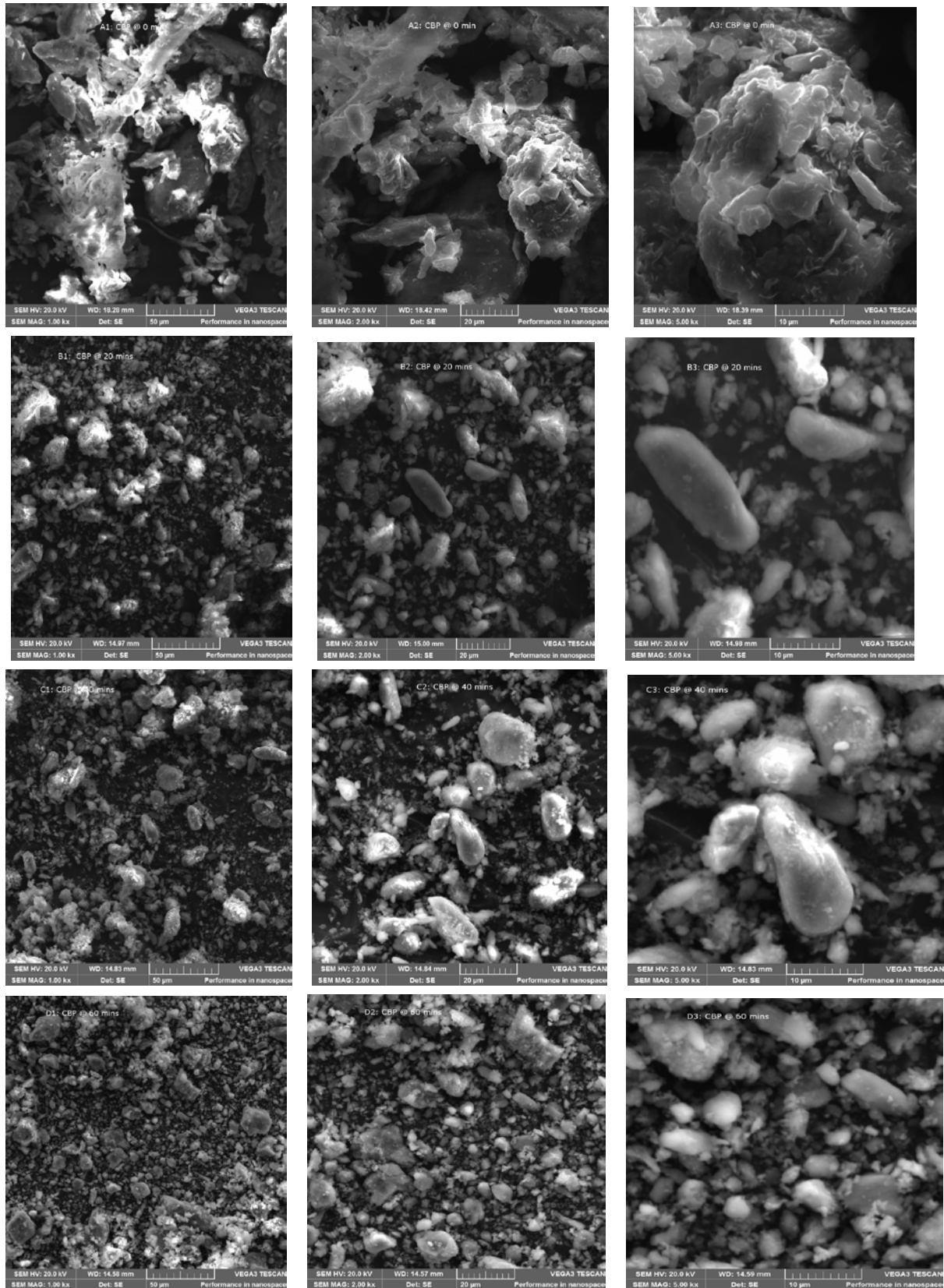


Fig 5: SEM Micrographs of Cow Bone Powder (CBP) at different SEM magnifications of 1.0kx, 2.0kx and 5.0kx and at different milling time of 0, 20, 40 and 60 Mins.

Figure 5 above, label A1, A2, A3 on the micrographs represent the Unmilled (0 min) Cow Bone Powder at different magnifications of 1.0kx, 2.0kx and 5.0kx resulting to 50 μm , 20 μm , and 10 μm respectively, this show coarse particle size since it was 150 μm sieved (Unmilled). B1, B2, and B3 show the micrographs of 20 Minutes milling at SEM magnifications of 1.0kx, 2.0kx and 5.0kx resulting to 50 μm , 20 μm , and 10 μm respectively. C1, C2, and C3 presented the micrographs of 40 minutes of milling at 1.0kx, 2.0kx and 5.0kx resulting to 50 μm , 20 μm and 10 μm respectively while D1, D2, and D3 represent the micrographs of 60 minutes of milling at 1.0kx, 2.0kx and 5.0kx resulting to 50 μm , 20 μm , and 10 μm respectively. 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 crystalline is arranged. D1 revealed smallest grain size structures when compared with C1, B1, and A1 of the same 1.0kx SEM MAG. Likewise, D2 and D3 were smaller in particle size structures when compared with other members of the same SEM MAG.

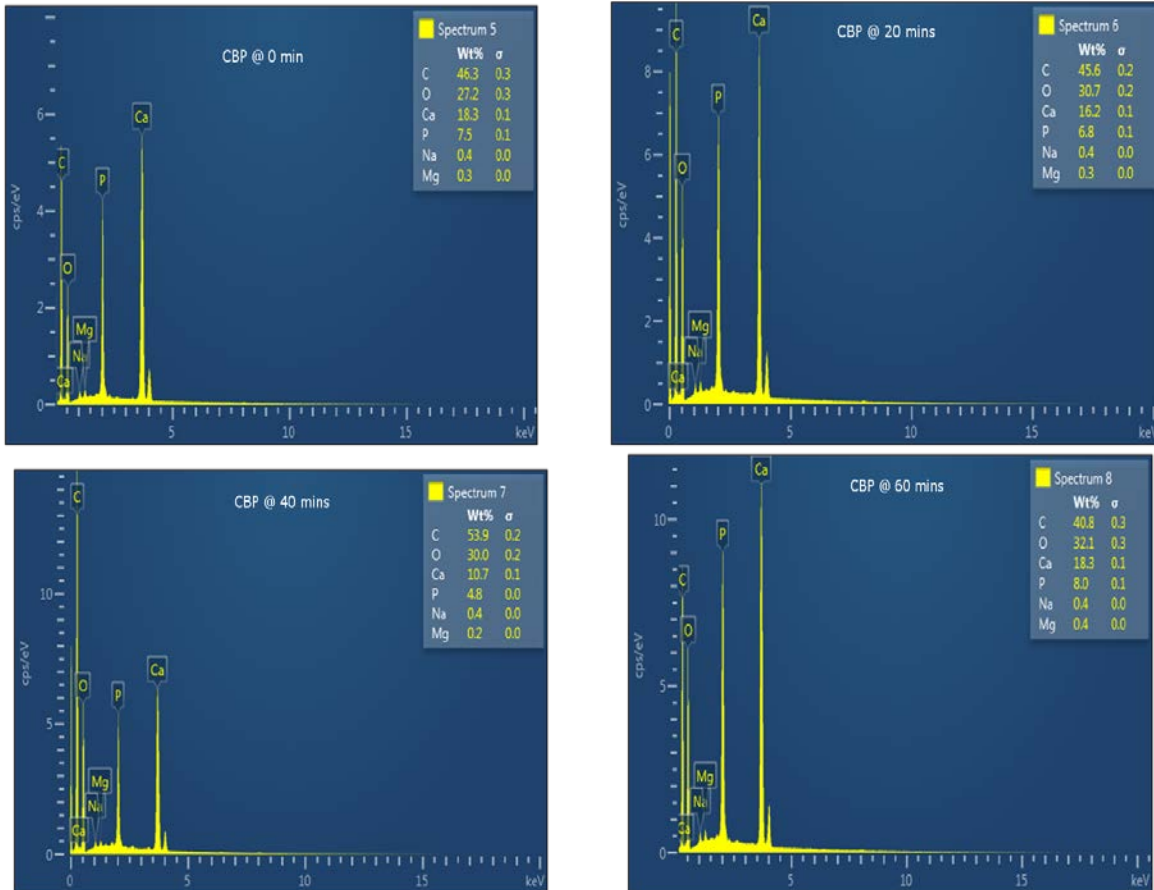


Figure 6: Elemental Compositions of CBP at different Milling time as analyzed by EDXS

Table 2: Variable in Elemental Compositions of CBP as analyzed by EDXS

POWDER	ELEMENT	ELEMENTAL COMPOSITION AT DIFFERENT MILLING TIMES			
		0 Min (Unmilled)	20 Mins	40 Mins	60 Mins
CBP	C	46.3	45.6	53.9	40.8
	O	27.2	30.7	30.0	32.1
	Ca	18.3	16.2	10.7	18.3
	P	7.5	6.8	4.8	8.0
	Na	0.4	0.4	0.4	0.4
	Mg	0.3	0.5	0.2	0.4

Figure 6 and Table 2 showed the variations in elemental compositions of Cow Bone Powder. It was observed from table 2 that Carbon (c), Oxygen (O) and Calcium (Ca) have a relatively high percentage of compositions. The percentage composition of Phosphorous (P) is higher than of Sodium (Na) and Magnesium (Mg). Mg and Na have the least composition in Cow Bone as analyzed by EDX. The elemental composition varied along with the milling time from 0 – 60 mins.

3.0 Conclusion

From the results obtained and their discussions, the following conclusions were drawn from the research carried out: The particle size reduced from 150 µm to an average size of 300 nm after milling for 60 mins using digital vibratory disc milling machine shown in (Figure 2). EDX revealed different elemental compositions that were contained in CBP which are Calcium, Phosphorous, Sodium, Magnesium, Carbon and Oxygen in which any user of CBP in near future can easily use as the basis of their research work. XRF also established different chemical compositions contained in milled CBP. It can be concluded that CBP can be used in different science and engineering applications varying from reinforcement in metal, polymer and ceramics composites, fillers, activated carbon, water purifier, heavy metals absorber, etc.

References

- Abdulrahman, A., Latiff, A. A. A., Daud, Z., Ridzuan, M. B., & Jagaba, A. H. (2016). Preparation and Characterization of Activated Cow Bone Powder for the Adsorption of Cadmium from Palm Oil Mill Effluent. *IOP Conference Series: Materials Science and Engineering*, 136(1), 1–7. <https://doi.org/10.1088/1757-899X/136/1/012045>.
- Adewole, T. (2015). Effect of Cow Bone Ash Particle Size Distribution on the Mechanical Properties of Cow Bone Ash-Reinforced Polyester Composites. *Chemistry and Materials Research*, 7(3), 40–47.
- Agunsoye, J. O., Talabi, S. I., Awe, O., & Kelechi, H. (2013). Mechanical Properties and Tribological Behaviour of Recycled Polyethylene/Cow Bone Particulate Composite. *Journal of Materials Science Research*, 2(2), 1–39. <https://doi.org/10.5539/jmsr.v2n2p41>.
- Ahmad, M., Lee, S. S., Lim, J. E., Lee, S. E., Cho, J. S., Moon, D. H., ... Ok, Y. S. (2014). Speciation and phytoavailability of lead and antimony in small arms range soil amended with mussel shell, cow bone, and biochar: EXAFS spectroscopy and chemical extractions. *Chemosphere*, 95(1), 433–441. <https://doi.org/10.1016/j.chemosphere.2013.09.077>.
- Ahmad, M., Soo Lee, S., Yang, J. E., Ro, H. M., Han Lee, Y., & Sik Ok, Y. (2012). Effects of soil dilution and amendments (mussel shell, cow bone, and biochar) on Pb availability and phytotoxicity in military shooting range soil. *Ecotoxicology and Environmental Safety*, 79, 225–231. <https://doi.org/10.1016/j.ecoenv.2012.01.003>.
- Aramide, F. O., Ibitoye, S. A., Oladele, I. O., & Borode, J. O. (2010). Pack carburization of mild steel, using a pulverized bone as carburized: Optimizing process parameters. *Leonardo Electronic Journal of Practices and Technologies*, 9(16), 1–12.
- Asuke, F., Aigbodion, V. S., Abdulwahab, M., Fayomi, O. S. I., Popoola, A. P. I., Nwoyi, C. I., & Garba, B. (2012). Effects of a bone particle on the properties and microstructure of polypropylene/bone ash particulate composites. *Results in Physics*, 2, 135–141. <https://doi.org/10.1016/j.rinp.2012.09.001>.
- Daramola, O. O., & Adewole, A. T. (2014). Comparative Study of the Reinforcement Efficiency of Cow Matrix Composites for Biomedical. *ACTA TEHNICA CORVINIENSIS – Bulletin of Engineering*, 4, 1–29.
- Deydier, E., Guilet, R., Sarda, S., & Sharrock, P. (2005). Physical and chemical characterization of crude meat and bone meal combustion residue: “Waste or raw material?” *Journal of Hazardous Materials*, 121(1–3), 141–148. <https://doi.org/10.1016/j.jhazmat.2005.02.003>.
- Ihom, P. A. (2013). Case hardening of mild steel using cow bone as Energiser. *African Journal of Engineering Research*, 1(October), 97–101.
- Mohammed, A., Aboje, A. A., Auta, M., & Jibril, M. (2012). A Comparative Analysis and Characterization of Animal Bones as Adsorbent. *Pelagia Research Library Advances in Applied Science Research*, 3(5), 3089–3096. Retrieved from www.pelagiaresearchlibrary.com.
- Oladele, I. O. (2016). Development of Bone Particulate Reinforced Epoxy Composite For Biomedical Application. *Journal of Applied Biotechnology & Bioengineering*, 1(1), 1–6. <https://doi.org/10.15406/jabb.2016.01.00006>.
- Oladele, I. O., & Adewole, T. A. (2013). Influence of Cow Bone Particle Size Distribution on the Mechanical

Properties of Cow Bone-Reinforced Polyester Composites. *Biotechnology Research International*, 5, 1–6.
<https://doi.org/10.1155/2013/725396>.

Biographies

Omolayo M. Ikumapayi is a Ph.D. candidate at the Department of Mechanical Engineering Science, University of Johannesburg South Africa. He obtained MSc in Mechanical Engineering (option in Design and Production) from the University of Lagos, Nigeria and his BEng in Mechanical Engineering from the Federal University of Agriculture Makurdi, Nigeria. He is currently a contract staff (Lecturer) at the University of Johannesburg, South Africa. He was a one-time Lecturer at Afe Babalola University, Ado Ekiti and Covenant University Ota, Nigeria. He was also a one-time teaching assistant at the University of Lagos, Nigeria. He is a registered Engineer with COREN, Member of Nigerian Society of Engineer (MNSE), Member of Nigerian Institution of Mechanical Engineer (MNIMEchE), Member of Chartered Institute of purchasing and supply management of Nigeria (MCIPSM), Member of Academy for Entrepreneurial Studies (M.AES) and Associate Member of the Certified Institute of Shipping, Nigeria (ACIS) among others. His research interests include manufacturing, simulation, processing using agro-wastes powders, surface modifications, characterizations, tribocorrosion, Friction stir processing/welding, electron beam processing/welding and nanotechnology.

Esther T. Akinlabi is currently a Full Professor in the Department of Mechanical Engineering Science and Vice Dean, Teaching and Learning, Faculty of Engineering and Built Environment (FEBE), University of Johannesburg South Africa. She has authored several peer-reviewed scholarly Journals, Books, and Book Chapters. Her areas of interests are in Energy, Friction Stir Welding/Processing, additive manufacturing, laser manufacturing, AutoCAD, Research Design etc.