Microstructural Characterization of Cement Kiln Dust as a Mineral Filler for Asphalt Production

Kabiru Usman Rogo[1], Hassan Aliyu[1], Yakubu Mamman Wakawa[2] & Samaila Saleh[3]

[1]Department of Civil Engineering Technology, School of Engineering Technology, Nuhu Bamalli Polytechnic, Zaria
[2]Department of Civil Engineering, Federal Polytechnic, Mubi, Adamawa State
[3]Department of Civil Engineering Technology, Hassan Usman Katsina Polytechnic, Katsina State
Corresponding Author's email: krusman07@nubapoly.edu.ng

Abstract: The construction industry grows in an unprecedented manner, as such demand for construction materials must be matched with production. Cement, being a key construction material and a filler for asphalt production is consumed in billions of tons globally, particularly sub-Saharan Africa. Cement production produces huge by-product which is passed through Air Pollution Control Devices (APCDs) also known as electrostatic precipitators in the form of flue dust popularly referred to as Cement Kiln Dust (CKD). Despite recycling coarser CKD back into production line, yet, large quantities of finer fractions end of as waste dumped in landfills. Rising production cost, environmental safety concerns, and sustainable consumption/production sought alternatives for cement for construction particularly, asphalt production. Little amount of cement used in asphalt production, nonetheless, multiplier effect of the huge quantity of asphalt required for road construction makes this amount significant. This study adopts some physical, mechanical, chemical, structural/morphological laboratory tests to ascertain the properties of CKD. Essentially, this study assessed the microstructural properties of CKD in comparison to ordinary Portland cement (OPC) using toxicity characteristic leaching procedure (TCLP), specific gravity, sieve analysis, Xray diffraction (XRD), and Xray fluorescence (XRF) tests. Results compared to established standards and pavement production/construction manual suggests that the texture, crystallinity, chemical composition of CKD satisfies the ASTM C150 for hydraulic cement for use as a filler in asphalt.

Keywords: Asphalt Production, Cement Kiln Dust, Microstructure, Mineral Filler, landfill.

INTRODUCTION

Industrial-bye products have wide ranging chemical composition dependent on the type/nature of parent raw material or the industrial process that generated the waste. Often times, the type, extent, concentration and toxicity levels of the compounds dictates the strategy of utilisation either re-use, recycling, or disposal (Sorvari & Wahlström, 2014). Sustainable construction drives for the incorporation of industrial by-products and waste material in construction requires them be assessed by characterisation. Several research efforts were geared towards characterizing and evaluating the environmental impacts of industrial by-products for use as replacement of construction materials like aggregate, fillers, reinforcement and cement (Ali Jattak *et al.*, 2019; Peploe & Zealand, 2006). Cement is the most expensive construction material aside reinforcing bars in Nigeria, thus, finding a suitable material to replace it becomes pertinent. Additionally, cement manufacture produces significant by-product termed as cement bypass flue dust (CBFD) (Sultan *et al.*, 2018) or most popularly, cement kiln dust (CKD). About 15 - 20% by weight of cement raw clinker is produced as by-product (CKD) hence need to reuse (Kunal *et al.*, 2012). Cement is used as a filler for asphalt production which enhances antistripping and improves stiffness of the mix (Hainin *et al.*, 2013). It was established that fines or fillers are significant component in asphalt as it enhances mixture performance (Kalaitzaki *et al.*, 2017). A number of studies explored replacing cement with various industrial by-products or waste for construction purposes ranging from concrete, asphalt, mortar, bricks

production to block production. Materials used to replace cement in concrete, mortar or brick/block production include fly ash, bio-pozzolanas like rice husk ash, maze cob ash, white cowpea pod ash, waste glass and fibres of various types and grades (Adebayo Mujedu *et al.*, 2020; Usman *et al.*, 2022).

However, among the numerous by-products used for cement replacement in construction, CKD is among the cost-effective, versatile and effective (because it has inherent properties as that of the raw cement) material which its utilisation makes the environment more safer than the other waste material because its cancerous if inhaled due to open dumping (M. Kassim, 2014), it is worth noting that CKD in itself safe under normal condition, just like cement, but easily contaminated if dumped as waste in landfills (Kunal *et al.*, 2012). CKD is use for myriad of applications (Amadi & Lubem, 2014; Arulrajah *et al.*, 2017; Mackie *et al.*, 2010; Najim *et al.*, 2014) but limited studies explored the microstructural properties of CKD, particularly for asphalt production to satisfy the requirement highlighted in the literature (Gautam *et al.*, 2018).

A number of studies have shown that CKD can effectively replace lime, cement or fly ash in construction applications (Ismail & Belal, 2016; Usman *et al.*, 2023). As road construction consume greater resources than building due to the lengthy stretch it covers, thus, replacing cement with CKD for asphalt production will ameliorate CKD's disposal challenges. Therefore, this study explored the micro-characteristics of CKD with the aim of establishing its suitability and potential of use as cement replacement for asphalt production.

MATERIALS AND TEST METHODS

Material

Cement

Ordinary Portland normal setting cement (OPC) produced by the Dangote Obajana factory was employed for this study. The Nigerian construction industry utilises this cement more than similar brands for regular construction works. The OPC was emptied from the sack and stored in air-tight containers to assure its pristineness



Plate 1. Ordinary setting Portland cement from Dangote Obajana factory

Cement Kiln Dust (CKD)

The fine powdery light grey uniformly graded CKD was acquired from Dangote Cement industry located at Obajana in Kogi state. The factory utilises state-of-the-art dust precipitator facility which ensures all dust particles are captured and minimal to zero amount is released into the air. Because CKD's grain texture depends on the manufacturing technology in place, technique of dust collection, and chemical composition of the parent raw materials. Fig. 2 depicts the particular CKD sample tested for this study.



Plate 2. CKD collected from air control pollution devices (ACPD)

Table 1 indicated that CKD has lower LOI than OPC perhaps due to the fact that CKD has undergone the production process and has lower 'volatile' constituents that may lead to loss of weight. Moreover, the fineness of CKD may be more desirable than OPC in asphalt for the specific high surface which result in greater reactivity. The CKD used in this study is unusually whitish to light-grey as compared to the OPC. This could not be far-fetched from the fact that the clinker has been subjected to a higher temperature, especially when the coarse CKD is reinjected back into the production line.

Table 1. Physical Properties of CKD and Cement

Physical Properties	CKD	OPC	Remark
Colour (appearance)	Tan grey	Grey	The CKD looks like gypsum
Specific gravity	2.88	3.10	The specific gravity is similar
Blaine Fineness (B.F) (cm^3/g)	7260	4300	CKD has higher B.F
Soundness (mm)	1.4	1.0	OPC is better than CKD
Percent passing sieve 10 µm (%)	36	23	CKD is finer than OPC
Loss on ignition (LOI) at 1000 ^oC (%)	6.3	10.2	

Microstructural Test Evaluation

The study was conducted almost entirely in the laboratory and the field. The research is phased into three (3) stages viz; material collection and characterisation, then physical, mechanical, and chemical testing are conducted under controlled environment.

Toxicity characteristics leaching procedure (TCLP)

This test was introduced by the United States environmental protection agency (US EPA) 1311 (1999). Trace toxic elements often contaminates waste and industrial by-products and if used for construction may leach into the soil thereby polluting groundwater or cause serious illnesses like cancer if airborne. Possible heavy metals targeted to be detected include Barium (Ba), Lead (Pb), Arsenic (As), Selenium (Se), Silver (Ag), Cadmium (Cd), Copper (Cu), Chromium (Cr) and Zinc (Zn). Distilled water was used to prepare the test specimens in the ratio 1:20 then centrifuged at 30 – 40 rpm for 24 hours. The filtrate was subjected to inductively coupled plasma mass spectrometry (ICP–MS) to detect the heavy metals.

Gradation test (Sieve analysis)

A dry sieve analysis under ASTM C136/C136M-14 standard procedures was employed and subsequently employed the 90 and 75 µm sieve sizes due to the mixture's fineness (ASTM C136, 2010). The method invariably measures the fineness of cement

and CKD as well. The cement and CKD samples

were dried to ensure moisture traces is expelled and then sieved through 600 µm to 75 µm set of sieves stacked in reducing aperture sizes from top to bottom. The sieves were shook using a mechanical shaker and manually to ensure completeness of the sieving.

Specific gravity test

The specific gravity is conducted according to ASTM C 188 (ASTM, 2011) which measures the density of a standard hydraulic cement. The methods which use the Le Chatelier flask, indirectly measure the specific gravity of the cement by determining and calculating from laboratory test, the density of the cement and CKD alike. The specific gravity of the cement is mathematically computed by dividing the density of the cement to that of distilled water at 4 ^{o}C.

X-ray fluorescence (XRF)

The extent of chemical composition in a crystalline sample is best determined by the XRF test. XRF works on the principle that unpaired electrons in the outer atomic orbitals detaches and replaced by another from sub-orbital lower energy levels creating fluorescence effect with a consequent energy differential. The energy is captured, recorded and matched with a library of stored similar fluorescence in the XRF machine which affirms the type of chemical compound so detected with reasonable accuracy.

X-ray Diffraction (XRD)

This test was employed in this study to determine the crystallinity of CKD and cement. spent garnet at atomic and elemental levels was evaluated using X-ray diffraction. Copper (Cu) Kα radiation source was used in an X-ray diffractometer set at 0.02^{o} scanning at 0.5^{o}/min within the range of 2θ from 0° to 100°.

RESULTS

Physical, morphological and mechanical characteristics of CKD and Cement

The CKD is largely light whitish-grey while the cement is dark-grey when pristine but turns darker when exposed to moisture. The CKD has higher specific surface area with smooth-textured grains similar to the cement. The particle size distribution of cement and CKD are presented in Fig. 1. The physical properties of the constituent material in this study are highlighted in Table 1 while the chemical composition is presented in Table 2. The chemical composition of the two materials indicated similarity in the percentages of the active cementing ingredients.

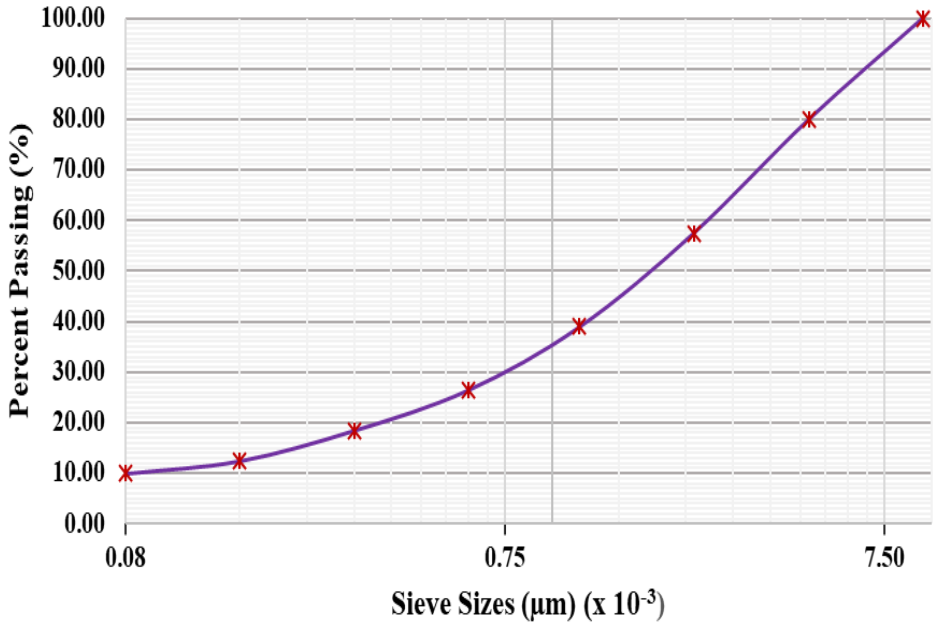

Fig. 3. Gradation for CKD and Cement

It can be seen that from Fig. 3 that both CKD and cement 100% passed through the 4.25 mm but 35% passed the 750 µm sieve. While the CKD has a percentage retained on the pan of 7.29 g, the cement has 12.89 g on the pan. The result signifies that cement is finer than the CKD. Nonetheless, it is interesting to note that from the laboratory data both CKD and cement records highest retained weights on the 150 µm sieve.

Importantly, each material satisfies the fineness modulus requirement for hydraulic cements of minimum percentage retained of 10 % on the 90 µm sieve.

Table 2 presents the extent of the chemical composition of the CKD in comparison to that of cement.

Table 2. Xray Fluorescence (XRF) result on CKD and cement samples

Compound (%)	CKD	OPC	ASTM C150 Requirement for Hydraulic Cements (max. %)
Al_2O_3	3.89	5.11	6
Fe_2O_3	2.72	3.28	6
CaO	63.81	63.43	N.A
SiO_2	14.88	20.66	N.A
MgO	1.34	1.98	6
K_2O	1.02	0.49	-
Na_2O	0.20	0.37	-
Ti_2O	0.36	0.29	-
SO_3	3.49	3.64	-
LOI	29.48	3.10	-
Others	0.54	1.66	-

The morphology or rather crystallinity of the CKD and Cement samples present similar peaks as seen in Fig. 4. The crystalline latices of the active ingredients in cementitious materials notably tricalcium silicate, dicalcium silicate, and tetra calcium Alumno-ferrite within 30 – 40 degrees were detected. Moreover, CKD has more peaks within the said range signifying better bond.

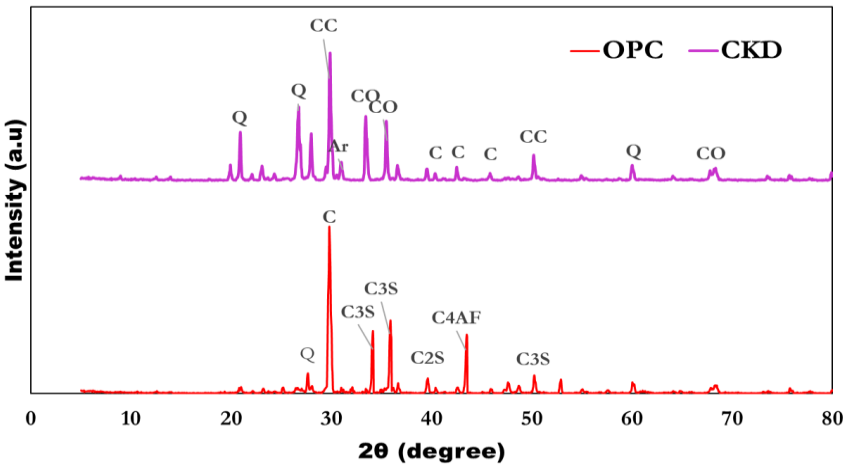

Fig. 4. XRD pattern of CKD in comparison to that of Cement

Chemical properties of CKD and Cement

X-Ray fluorescence (XRF) and TCLP constitutes the chemical composition and extent of contamination respectively. The various percentages of the constituent compounds present in CKD and Cement is presented in Table 2.

Moreover, the test which determines the suitability/safety or otherwise of a material to be used for construction is the TCLP test. The test result is presented in Table 3.

Table 3. Result of Heavy metal concentration test

Heavy Metal Concentration (mg/L)	CKD	TCLP Standard Levels	REMARK
Ni	0.001	2.00	
Cr	0.000842	5.00	
Pb	0.000014	0.005	
Cu	11.00	25.00	Cu is high though well below limit
Ar	0.02	5.00	
Ba	15.00	100.00	
Zn	17.80	250.00	
Sr	-	-	
Th	7	-	
Cd	0.00021	1.00	
Co	-	8.00	Cobalt is not detected in the CKD sample

The CKD sample exhibited that it is free from contamination by heavy toxic metals as indicated in Table 3. The result in Table 3 shows that Zinc (Zn), Barium (Ba) and Copper (Cu) represents the elements with the highest concentration. Despite having the highest concentration, yet they are well below the maximum set limits. Thus, CKD is safe to be employed for construction purposes including asphalt production and pavement construction.

DISCUSSION

The CKD has comparable chemical, physical and mechanical properties to that of cement in most ramifications. More so, the TCLP test proven that CKD is safe to be used for construction purposes with the obtained limits well below the set limits. by X-ray fluorescence (XRF), X-ray Diffraction (XRD), toxicity characteristics leaching procedure (TCLP), specific gravity and Blaine fineness tests. Result suggest that CKD has desirable characteristics for use as filler in asphalt in place of cement.

From the gradation result presented in Fig. 3., CKD has met the fineness modulus test requirement of cement of having a minimum percentage of 10% passing the 90 μm test sieve. The high specific surface area of CKD as revealed by the Blaine fineness test and presented in Table 1 which nearly doubles that of cement and a lower loss on Ignition than cement suggests that CKD can adequately serve as a filler in asphalt. The function of a filler in asphalt is to increase stiffness, reduce voids and prevent asphalt bleeding in asphalt.

CONCLUSION

The microstructural characteristics of CKD was assessed in this study and based the outcome shown that the particular CKD can safely be employed for construction purposes as the toxic heavy metals are discovered to be within acceptable limits set by EPA. Moreover, substances, it has the requisite specific gravity, fineness and desired chemical composition needed to serve as a filler in asphalt. Furthermore, the OPC cement has high crystallinity, specific gravity and active chemical constituents which satisfies the ASTM C150 requirement for Type II & Type IIA cements necessary for strong bond in asphalt. It is therefore concluded that the CKD from Obajana cement factory can satisfactorily be incorporated in asphalt production.

REFERENCES

Adebayo Mujedu, K., Ab-Kadir, M. A., & Ismail, M. (2020). A review on self-compacting concrete incorporating palm oil fuel ash as a cement replacement. In *Construction and Building Materials* (Vol. 258, p. 119541). Elsevier Ltd. https://doi.org/10.1016/j.conbuildmat.2020.119541

Ali Jattak, Z., Abdul Hassan, N., Athma Mohd Shukry, N., Khairul Idham Mohd Satar, M., Naqiuddin Mohd Warid, M., Md Nor, H., & Zurairahetty Mohd Yunus, N. (2019). Characterization of industrial by-products as asphalt paving material. *IOP Conference Series: Earth and Environmental Science*, *220*(1). https://doi.org/10.1088/1755-1315/220/1/012012

Amadi, A. A., & Lubem, S. (2014). Assessing Stabilization Effectiveness of Combined Cement Kiln Dust and Quarry Fines on Pavement Subgrades Dominated by Black Cotton Soil. *Geotechnical and Geological Engineering*, *32*(5), 1231–1238. https://doi.org/10.1007/s10706-014-9793-0

Arulrajah, A., Mohammadinia, A., D'Amico, A., & Horpibulsuk, S. (2017). Effect of lime kiln dust as an alternative binder in the stabilization of construction and demolition materials. *Construction and Building Materials*. https://doi.org/10.1016/j.conbuildmat.2017.07.070

ASTM. (2011). *C188-16, Standard Test Method for Density of Hydraulic Cement*. *95*(Reapproved 2003), 37–39. https://doi.org/10.1520/C0188-09.2

ASTM C136. (2010). ASTM C 136 Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates. In *Annual Book of ASTM Standards* (Vol. 04, Issue C, pp. 1–5). https://doi.org/10.1520/C0136-06.2

Gautam, P. K., Kalla, P., Jethoo, A. S., Agrawal, R., & Singh, H. (2018). Sustainable use of waste in flexible pavement: A review. *Construction and Building Materials*, *180*, 239–253. https://doi.org/10.1016/j.conbuildmat.2018.04.067

Hainin, M. R., Abdulridha, A. A., Yaacob, H., Haron, Z., & Yusoff, N. I. (2013). Fatigue life of Malaysia hot mix asphalt mixtures. *Malaysia Journal of Civil Engineering*, *25*(1), 94–105.

Ismail, A. I. M., & Belal, Z. L. (2016). Use of Cement Kiln Dust on the Engineering Modification of Soil Materials, Nile Delta, Egypt. *Geotechnical and Geological Engineering*, *34*(2), 463–469. https://doi.org/10.1007/s10706-015-9957-6

Kalaitzaki, E., Engineer, C., Kollaros, G., & Athanasopoulou, A. (2017). *SIGNIFICANCE OF FINES IN HOT MIX ASPHALT*. *6*(3), 30–42. https://doi.org/10.1515/rjti-2017-0052

Kunal, Siddique, R., & Rajor, A. (2012). Use of cement kiln dust in cement concrete and its leachate characteristics. *Resources, Conservation and Recycling*, *61*, 59–68. https://doi.org/10.1016/j.resconrec.2012.01.006

M. Kassim, M. (2014). The durability of sulfate resisting mortar with partial replacement by cement kiln dust. *Anti-Corrosion Methods and Materials*, *61*(3), 182–190. https://doi.org/10.1108/acmm-02-2013-1243

Mackie, A., Boilard, S., Walsh, M. E., & Lake, C. B. (2010). Physicochemical characterization of cement kiln dust for potential reuse in acidic wastewater treatment. *Journal of Hazardous Materials*. https://doi.org/10.1016/j.jhazmat.2009.08.081

Najim, K. B., Mahmod, Z. S., & Atea, A. K. M. (2014). Experimental investigation on using Cement Kiln Dust (CKD) as a cement replacement material in producing modified cement mortar. *Construction and Building Materials*, *55*, 5–12. https://doi.org/10.1016/j.conbuildmat.2014.01.015

Peploe, R. J., & Zealand, N. (2006). Environmental Impact of Industrial By-products in Road Construction – a Literature Review. In *Land Transport New Zealand Research Report 308*.

Sorvari, J., & Wahlström, M. (2014). Industrial By-products. In *Handbook of Recycling* (pp. 231–253). Elsevier. https://doi.org/10.1016/B978-0-12-396459-5.00017-9

Sultan, M. E., Abo-El-Enein, S. A., Sayed, A. Z.,

EL-Sokkary, T. M., & Hammad, H. A. (2018). Incorporation of cement bypass flue dust in fly ash and blast furnace slag-based geopolymer. *Case Studies in Construction Materials*, *8*(December 2017), 315–322. https://doi.org/10.1016/j.cscm.2018.02.009

Usman K. R., Ahmad Z., Musa R., Zailani J., Abdulkadir M. S. Shamsuddeen U., S. F. U. (2023). Physico-mechanical and chemical characterisation of cement kiln dust as compared to normal set portland cement as a mineral filler in recycled asphalt. *International Journal of Engineering Research and Technology*, *13*(5), 77–88.

Usman, K. R., Shuaibu, I., Zubairu, G., & Sani, A. (2022). Nuhu Bamalli Polytechnic ,. *15th Multidisciplinary National Conference – 5-6 October, 2022 – Zaria, Nigeria*, 1–4.