



**DEVELOPMENT OF A METHOD FOR SAMPLING FLY ASH FROM INCINERATED MATERIALS**

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**ABSTRACT**

Forensic analysis of atmospheric fly ash is still an emerging area because of limitations associated with sampling. This project was designed to develop a method for effective generation and sampling of required quantity of fly ash from specific combustible materials for research works. A mini laboratory incinerator was constructed with detachable parts which included: combustion chamber, frustum and chimney. The frustum was built to cap the combustion chamber to accelerate a laminar flow of fly ash into the chimney, deflected by the hood and deposited in the collection tray. To test the efficiency of the incinerator, a kilogram of kernel shells and corn cob were separately incinerated at about 500 °C yielded between 15 – 20 g of fly ash. Characterization indicated total Polycyclic Aromatic Hydrocarbons (PAHs) concentrations of  $78.3 \pm 2.8$  ppm and  $69.9 \pm 0.4$  ppm for palm kernel shell and corn cob, respectively. The concentrations of manganese and zinc were  $156.5 \pm 10.6$  ppm and  $204.4 \pm 2.0$  ppm, and  $125.4 \pm 3.4$  ppm and  $319.0 \pm 11.4$  ppm for palm kernel and corn cob, respectively. Conclusively, with the designed incinerator, it was possible to harvest sufficient quantity of fly ash for characterization.

**Keywords:** Agricultural waste; Combustion; Fly Ash; Incinerator; PAHs; Sampling

## INTRODUCTION

Industrial processes in developing countries due to increased energy demand, transportation and waste management practices have been of concern globally because the combustion emissions produce devastating atmospheric pollution [1]. Fly ash is the combustion emission that becomes entrained in the gas flow through the combustor and is carried downstream with reacting gases in an incinerator [2]. They are particulate matter trapped from flue gas [3, 4], collected by electrostatic or mechanical precipitation [5], fine powder composed of spherical glassy particles of coal combustion products [6].

The relevance and development of various applications of fly ash from different sources as raw materials lie principally on their characterization which reveals compositions, mineralogy, surface chemistry and reactivity [6]. Agricultural wastes fly ash can be utilized as supplementary cementitious materials for partial replacement of cement and concrete production [7-9]. Corncocks and sawdust fly ash were processed and maximized to obtain high surface area due to mesopores volumes and serves as activated carbon for alternative adsorbents to large molecule of organic compounds [10]. Generally, biomass fly ash is composed mainly of inorganic fractions and minor organic carbon fractions which depend on the type of biomass, load and combustion conditions [11].

Incineration is the conversion of toxic waste in a thermal combustion process yielding bottom ash, fly ash and flue gases as products [12], a process of reduction of waste volumes and energy recovery [13]. When agricultural wastes are combusted in the open, PAHs and some Potentially Toxic Elements (PTEs) in fly ash are released [14].

To obtain representative fly ash samples, ash from five selected types of waste incineration plants were combined and homogenized for different analyses [4]. Thorough mixing through repeated coning and quartering method is required to obtain samples for different laboratory analyses [15]. Most equipment for ambient fly ash sampling are costly, difficult to operate, time consuming, designed for industrial purposes, cannot obtain bulk and representative nature of samples [17, 18]. Manually, fly ash sampling are usually done by sweeping fallen samples on surfaces using brush into clean dust pans [17,19, 20], collected from coal or municipal solid waste ash silo incineration power plants, landfill, air pollution control devices and electrostatic precipitators [21]. The basic challenge of these sampling methods is that sample quantity may not be large enough to represent the material or environmental media evaluated

[16]. Because of these limitations most researchers are compelled to studying residual ash instead of fly ash. Moreover, data related to the contributions of individual ambient emissions are limited in literature due to difficulty in sampling fly ash in view of method and techniques. Meanwhile, literature has implicated fly ash as a major polluter of the atmosphere not the residual ash.

The laboratory scale incinerator was therefore designed for incinerating different fuel materials now agricultural waste and developed a method for increasing yield and easy sampling of associated fly ash for characterization.

## **MATERIALS AND METHOD**

Materials include: Laboratory incinerator, thermometer, flint, spatula, wooden brush, stainless metal plates, bolt and nuts, white tile, ash pan.

### **INCINERATOR**

A model laboratory incinerator was designed and constructed in the Mechanical Engineering workshop of Rivers State University, Nkpolu, Port Harcourt, Nigeria, applying basic principles to mimic regular small scale, domestic, municipal and industrial fuel combustion systems [22]. Existing structures of proburn, crate, municipal incinerators were reviewed, [22-24], and modified. This incinerator was designed to perfect sampling of fly ash from specific combustible materials and ambient atmospheric particulate matter. It was made of six detachable parts namely, combustion chamber, mobile drawer, feedstock pot, frustum, chimney and fly ash collection tray.

#### **Design of Incinerator**

Other factors considered in the design and fabrication included:

**Types of fuels:** The design was based on liquid and solid fuels (domestic and industrial) that are regularly combusted at temperature up to 500 °C and above.

**Mass/Volume of Fuels:** The capacity of the combustion chamber was to contain not less than 1 kg of solid fuels or 10 L for liquid fuel.

**Residence Time and Air/Fuel Ratio:** The residence time of combustion varies according to the material being burned and samples are left to burn at a steady temperature of 500 °C until the fire

goes off. A proportionate theoretical air/fuel mixture was applied, given that one mole of carbon and sulphur in fuels, requires 4.76 mole of air and for one kilogram atom of available hydrogen in the fuel, 2.38 mole of air is needed [22, 26].

### **Construction and Assembly**

Mild steel plate and angle iron and flat bar were used in construction. The material belongs to the relatively cheap group of low carbon steels and possesses good welding properties [25]. According to the dimensions listed on Table 1, parts of the incinerator were fabricated separately before it was coupled according to the design by welding.

**Combustion Chamber:** The combustion chamber is a rectangular box fitted with mobile feedstock drawer that roll on a rail inside the chamber. The feedstock drawer is internally insulated from the wall of the combustion chamber to reduce heat loss from burning fuels. The combustion chamber also contains mini vents on both sides for aeration. The average temperature of the combustion chamber lies between 500°C and 600°C depending on the type of fuel. The mobile inbuilt drawer contains two separated aluminum pots where liquid feedstock materials are loaded for incineration and bottom ash remains after incineration.

**Frustum:** The structure of the frustum is squared at the bottom, railed with metal flat bar and fixed on the combustion chamber below and connected to the chimney at the upper circle. The combustion chamber is attached to a frustum to enhance laminar flow of combustion products and flue gases into the chimney before emission into the atmosphere.

**Chimney and Cap:** The chimney is a cylindrical structure measuring one meter high. It was fastened to the frustum with bolts and nuts and the cone-shaped cap was fixed at the top. The height of the chimney was made one meter in order to reduce the distance travelled by the combustion emissions to improve the quantity of samples generated.

**Ash Tray:** The ash tray is an aluminum pan that contains small white tiles and is situated at the chimney top below the hood. The fly ashes are gradually deposited on the white tiles in the ash tray. This model promotes laminar flow of fly ash samples and flue gas that travels from the combustion chamber into the chimney and sharply deflected from the hood and finally deposit on the white tiles in the collection tray.

Table 1.0: Parts and Dimensions of Laboratory Incinerator for Fly Ash Sampling

S/No	Parts of Incinerator	Dimensions (cm)
1	Combustion Chamber	L=63, W=63, H=92
2	Mobile Drawer	L=62, W=50, H=30
3	Frustum	L=63, W=63, H=39
4	Chimney	H=62, $\phi=15$
5	Hood	H=15, $\phi=20$
6	Fly Ash Tray	L50, W=50
7	Vent	L=16, W=16

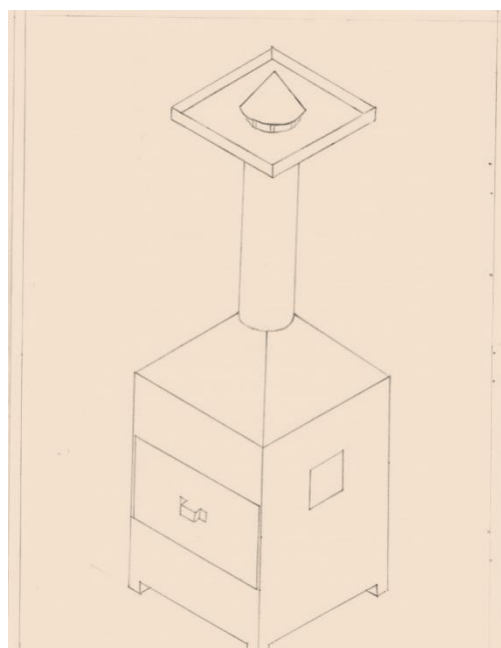


Figure 1: Sketch of the Incinerator

### Sample Source, Preparation and Incineration

To test run the efficiency of the constructed incinerator, the incineration and harvesting procedure, two agricultural waste materials (palm kernel shell and corncobs) were employed.

Palm kernel shell were sourced from Koroma town in an oil mill factory in Tai Local Government Area, Rivers State, while the corncobs were bought from Igbo-Etche market in Etche Local Government Area in Rivers State, Nigeria. The fruits were removed to generate the corncobs. Recovered samples were sun-dried for 6 weeks before incineration. About 1 kg of each material were placed as feedstock in the aluminum pot inside the combustion chamber and directly ignited with the aid of a splinter. Generated fly ash samples were carried along in the flue gases during combustion to the chimney and settled in the ash tray padded with white tiles while some escaped into the atmosphere and bottom ash were deposited in the aluminum pots.

### **Sampling of Fly Ash**

To obtain representative samples and generate sufficient quantity of fly ash for laboratory analysis, each material was combusted for five days and samples were collected immediately after combustion daily. At the end of burning of each fuel, fly ash were deposited on the white tiles on the ash tray directly below the hood and by adsorption on the internal walls of the frustum, chimney and hood of the incinerator. With the aid of a clean short brush, deposited samples on the white tiles were swept onto clean white sheet of A4 papers before it was carefully transferred into 250 ml amber specimen bottle and firmly corked. Various proportions of fly ash deposited inside the hood, chimney and frustum were recovered by dismantling and sweeping with long and short brushes as may be applicable yielded about 15 – 20 g of fly ash. Samples were then transferred to the PostGraduate Laboratory and stored in cool dry box at room temperature.



Plate 1: Deposition of Fly Ash on Tray during Incineration of Kernel Shell



Plate 2: Collection of Fly Ash using Brush



Plate 3: Kernel Shell Fly Ash Sample

Figure 2: Field Pictures Showing Harvest and Sampling of Fly Ashes.

**Gas Chromatography-Flame Ionization Detector (GC-FID) Analysis:** PAHs concentration was determined using Varian 3800 chromatography as outlined in USEPA 8720D. Sample detection and identification were carried out using Flame Ionization Detector (FID) by taking advantage of the differences in retention time of the components while quantification was obtained from the corresponding areas of the respective chromatograms. Solvent blank was analyzed and quantified with no PAHs found in these blanks. The GC was calibrated using dichloromethane-based standards. The coefficient of determination values ( $R^2$ ) was greater than 0.95. Surrogate standards were introduced to the sample to attenuate abnormal matrix effect [27].

**Atomic Absorption Spectroscopy (AAS) Analysis:** The fly ash were recovered for the different samples and transferred into sample bottle and stored under laboratory conditions. Triplicates of each samples weighing 0.5 g was placed in boiling tubes. *Aqua regia* preparation was made by mixing concentrated hydrochloric acid and concentrated nitric acid in ratio of 3:1. To the measured samples in the boiling tube was added 15 ml of *aqua regia*. The mixture was placed on hot plate, heated for one hour in the fume box and allowed to cool. It was then filtered into a 100 ml volumetric flask with the aid of Whatman filter paper and marked up with distilled water. The

filtrate were transferred into sample bottles and sent to the laboratory for heavy metals analysis using Atomic Absorption Spectrophotometer, GBC XplorAA, Australia.

## RESULTS AND DISCUSSION

The combustion efficiency ( $\eta$ ) of the incinerator was 83%, slightly lower than 86.5% reported by Olisa *et al* [23], which may be attributed to the reduction in air supply due to vent sizes. The process of incineration of agricultural waste generated fly ash carried in flue gas as shown on Plate 1, which was deposited on white tiles in the ash tray. Deposited fly ash samples were manually harvested and transferred to the laboratory and collected with the aid of a small hand brush as demonstrated on Plate 2. Finally, Plate 3 showed kernel shell fly ash as a product of this laboratory incinerator which was simple to sample and indicated a high yield and set for characterization.

The results for Polycyclic Aromatic Hydrocarbon (PAHs) in the fly ash samples are presented in Tables 1. The total mean concentration of individual PAHs in kernel shell fly ash and corncobs is  $78.32 \pm 2.8$  ppm and  $69.9 \pm 0.4$  ppm, respectively. Kernel shell indicated lower concentrations of the individual PAHs dominant among were 3 and some 4-member-rings (except PYR, BAA) and also a 5-member-ring (DHA), and a 6-member-ring ((IPY). Similarly, 75% of the HMW PAHs (BBF, BKF, BAP) have slightly high concentrations with BPE (a 6-rings-member) having the highest mean concentration of 6.7 ppm. Low Molecular Weight (LMW) and Medium Molecular Weight (MMW) PAHs were dominant among recorded PAHs and can be attributed to the geogenic origin of kernel shell which is a non-fossil fuel. Though there are limited literatures on palm kernel PAHs, this result is similar to that reported by Matamba *et al* [28], indicating increased generation of PAHs through elevated pressure and temperature during pyrolysis of kernel shell. Corncobs indicated high concentrations of the individual PAHs including 3, 4 and 5-member-rings (except FLN, BAP) while the 6-member-ring have low concentrations (except DHA). Corncob indicated high concentrations among the LMW and MMW individual PAHs and dominant above the HMW PAHs. Comparatively, this result is contrary to that reported by Masto *et al* [29], where total PAH content was higher for fly ash than bottom ash (193 mg/kg) derived from wood combustion. The increased concentration of PAH in corncobs may be attributed to the burning temperature [30].



Table 2.0: Concentration of PAHs in Palm Kernel Shell and Corncobs Fly Ash

PAHs (ppm)	NAP	ACY	ACE	FLN	ANT	PHE	FLT	PYR	BAA	CHR	BBF	BKF	BAP	DHA	IPY	BPE	Total
Kernel Shell	5.7±0.21	0.13±0.01	0.05±0.01	0.05±0.01	0.12±0.01	0.06±0.01	0.18±0.01	1.44±0.09	6.97±0.14	0.29±0.02	28.9±0.10	18.5±0.26	8.42±0.29	0.41±0.4	0.4±0.02	6.7±0.16	78.3±2.8
Corn cobs	8.9±2.02	8.2±0.01	9.2±0.02	1.64±0.03	3.1±0.04	3.2±0.01	5.1±0.01	4.7±0.01	8.0±0.01	2.3±0.01	3.0±0.01	3.0±0.01	1.0±0.01	6.0±0.01	1.0±0.01	1.0±0.1	69.9±0.4

Naphtalene-NAP, Acenaphtylene-ACY, Anthracene-ANT, Pyrene-PYR, Acenaphthene-ACE, Fluorene-FLN, Fluoranthene-FLT, Phenanthrene-PHE, Chrysene-CHR, Benz(a)anthracene-BAA, Benzo(a)pyrene-BAP, Benzo(b)fluoranthene-BBF, Benzo(k)fluoranthene-BKF, Dibenz(a,h)anthracene-DHA, Benzo(g,h,i)perylene-BPE, Indino(1,2,3-c,d)pyrene-IPY.

The result for the heavy metals as contained in palm kernel fly ash and corncobs shown on Table 2.0 indicated manganese (156.6 mg/kg) and zinc (204.0 mg/kg) and manganese (125.4 mg/kg) and zinc (319 mg/kg), respectively. This result for palm kernel was similarly reported by Evbuomwan *et al* [31], in the comparative analysis of the physico-chemical properties of activated carbon from palm oil waste. Also, the result for heavy metals is similar to the findings of Garcia *et al* [19], where zinc has relatively high value of 400 mg/kg.

Table 3.0: Concentration of Heavy Metals in Palm Kernel Shell and Corn cobs

Heavy Metals	Manganese	Zinc
Kernel Shell	156±10.6	204±2.0
Corncobs	125.4±3.5	319.0±4.2

## CONCLUSION

With the aid of the constructed laboratory scale incinerator, it was possible to harvest sufficient fly ash matrix from farm waste. The design was easy to apply by students and researchers since it was cheaper to construct, easy to operate and provides potentials for sampling fly ash.

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