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Non Destructive Methods (XRF and XRD) For Estimation of Impure Carbon and Heavy Metals in Printer Toner Ink Powder

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Abstract

Air pollution constitutes the largest among all of the environmental risks. Dust and soot fragments forms components of air particulates, which are released into the air as extremely small particles or liquid droplets. The basis of this research is to characterize toner ink powder and wood soot samples and the detection of metallic pollutants in wood soot (WS) and printer toner ink (PIS) for their physicochemical properties (pH, conductivity, bulk density and moisture content) and instrumental analysis using scanning electron microscopy (SEM) and Fourier Transform Infrared (FTIR). Two non- destructive analytical techniques; Dispersive X-ray Fluorescence Spectrometry (ED-XRF) and X-ray Diffraction (XRD) were adopted for heavy metals (elemental) composition and mineralogy respectively. The pH of printer ink and wood soot shows higher pH value which indicates that they are alkaline. Low conductivity values were reported with low moisture, indicating easy fragmentation and spreading. The bulk density values for samples shows that the soot can be easily spread by air current to the environment. The EDS analysis indicates that the soot particles to be composed of primarily impure carbon, thus pointing at potential organic pollutants. The IR spectra show characteristics signals at 749.2 cm^{-1}, 745.5 cm^{-1}, 738.0 cm^{-1} and 745.5 cm^{-1} for wood soot and printer ink which correspond to C-H of aromatic group, 1703.4 cm^{-1}, 1699.7 cm^{-1}. The XRF analysis reveals high concentration of Chromium and other toxic metals. The mineralogical components of the soot and printer ink samples revealed the presence of associated minerals. Generally, levels of toxic metal exceed the permissible legislative limit for air samples.

Keywords: Printer ink; Soot; Air pollution; Carbon; Toxic metals; Particates.

1. Introduction

According to the World Health organization [1], air pollution constitutes the largest among all of the environmental risks: 3 million annual deaths are associated with outdoor air pollution exposure. In 2012 alone, 11.6 percent of global deaths equivalent to 6.5 million deaths were outdoor air pollution-related.

1.1. Toner Powder

Toners are complex mixtures with a particle size of approximately 8–12 μm [2], polymer-based powder, widely used in laser printers, photocopiers and fax machines, to form texts and images on the paper by electrophotographic technology. The conventional toner are produced from: styrene-acrylate copolymer and (ii) polyester resin or (iii) combination of styrene-acrylate and polyester. Hence, the major components of toners are polymers, resins, pigments or dyes, iron oxide, amorphous silica, charge control agents, paraffin wax, surfactants and other inorganic/organic additives [2]. As for the black toner, carbon black is mainly used. Important properties of carbon black are their dispersibility in resin in hot melt mixing and their tendency to charge either positive or negative.

Other than carbon black, magnetite is often used in toners to impart magnetic properties to the toner. Some charge control additives such as nigrosine are good black pigments, and their use in a toner can lead to reduction or elimination of the carbon black. Generally, a toner comprises of a binder resin (toner resin), a colorant, a magnetic oxide, a charge control agent and other additives, such as small amount of one or more additives, usually added to adjust the performance of toner in various aspects, including flow control, charge control, cleaning, conductivity control and decrease humidity sensitivity. Surface additives such as fumed silicas and titanias are added to the surface of the toner particles to improve flow characteristics and to prevent agglomeration [3].

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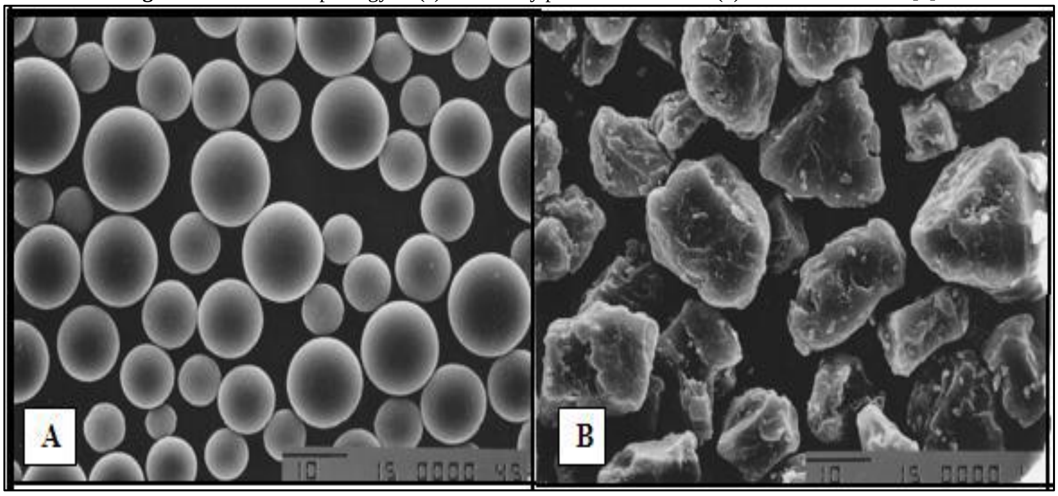
Figure-1. Surface Morphology of (a) chemically produced toner and (b) conventional toner **[3]**



In this present study, we report an insight into the available impure carbon and other pollutants in physico-chemically characterized toner powder used as printer ink. Characterization of soot was carried out using the X-Ray Fluorescence (XRF) spectrometry, X- Ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Fourier Transform Infra-red (FTIR) Spectroscopic techniques.

2. Materials and Methods

Non-destructive analytical techniques were adopted in the chemical and physicochemical characterization adopted in this studies. The instruments employed for this research work include, energy dispersive x-ray florescence spectrometer (Mini PAL4), scanning electron microscopy (Q150R), X-Ray Diffraction Instrument (X-SUPREME8000) and Fourier Transform Infra-Red (Agilent tech.Cary630).

Sampling: Printer ink (PIS) was obtained from various printing press and ink refilling points within business center of Bukuru metropolis, Jos Nigeria. Reported sample handling method [4] was adopted. Samples from the same sources were screened, harmonized and stored for analysis. High analytical grade purity chemical reagents and distilled water were used throughout the experiments.

Physiochemical Parameters Toner Ink: The physiochemical parameters of toner ink samples such as pH, conductivity, bulk density and moisture content were carried out using standard laboratory procedures [4].

2.1. Characterization of Samples

The characterization method reported by the National Metallurgical Development Centre; NMDC Jos, Nigeria was adopted with slight modification in mass- volume measurements.

Elemental and chemical composition of toner powder was based on XRF analysis. The Scanning Electron Microscopy (SEM) was performed to examine the surface morphology, X-ray powder diffraction is most widely used for the identification of unknown crystalline materials (e.g. minerals, inorganic compounds), crystalline sizes and diffraction pattern. Results are presented as peak positions at 2ϴ and X-ray counts (intensity) in the form of a table. The crystalline size was computed using the Debye-Scherrer equation given as Eqn. 1

$$D = \frac{K\lambda}{\beta Cos\Theta} \quad \textbf{(1)}$$

where D is the Crysalline size, K is Scherrer Constant with value 0.9, λ is the wavelength of X-ray (1.540598), β is full width at half maximum and Θ *is* the differential angle [5]. The functional groups present in the soot and printer ink samples were investigated using the Fourier Transform Infrared Spectrometer.

3. Results and Discussion

3.1. Visual Inspection of Samples

Plate 1 is pictorial representation of printer toner ink powder (a) compared with wood soot (b). Apparent colour of samples were examined with naked eye and were found to be fine black particles, chiefly composed of carbon, they are extremely tiny about 2.5 micro meters or smaller in diameter. It is irritating to the eyes, nose and throat, and it's odor may be nauseating. Due to its color and texture, soot tends to darken or stain surfaces. It stains ventilations, walls, ceiling, floors, clothes, and even skin. It is slightly sticky and tends to cling to surfaces. It was reported that soot is a powdery mass of fine black particles [6].

3.2. Physiochemical Parameters of Soot Samples and Printer Ink

Table 1 presents the physiochemical parameters such as pH, conductivity, moisture content and bulk density of kerosene based soot, diesel based soot, printer ink and wood soot used as the control experiment.

pH – Results in Table 1 shows that the pH range for printer ink and wood soot falls within the alkaline range, for printer ink which is 8.47 and for wood soot which is 7.51. It is an indication that trace metals are present in the samples.

Conductivity –printer ink and wood soot are low (0.00-0.78). This is an indication that the compound of the samples are mostly covalent with high carbon content [4].

Moisture Content- The results obtained in this analysis indicate the level of clumsiness or ease of dissociation of the soot samples. Low moisture content could also be linked to high persistence and low microbial action (degradation) [7].

Bulk Density - All the values in this analysis shows values of less than1. This is an indication that the soot can be easily spread by air current to the environment [4].

Plate-1. (a) Printer Toner Ink powder and (b) Wood Soot

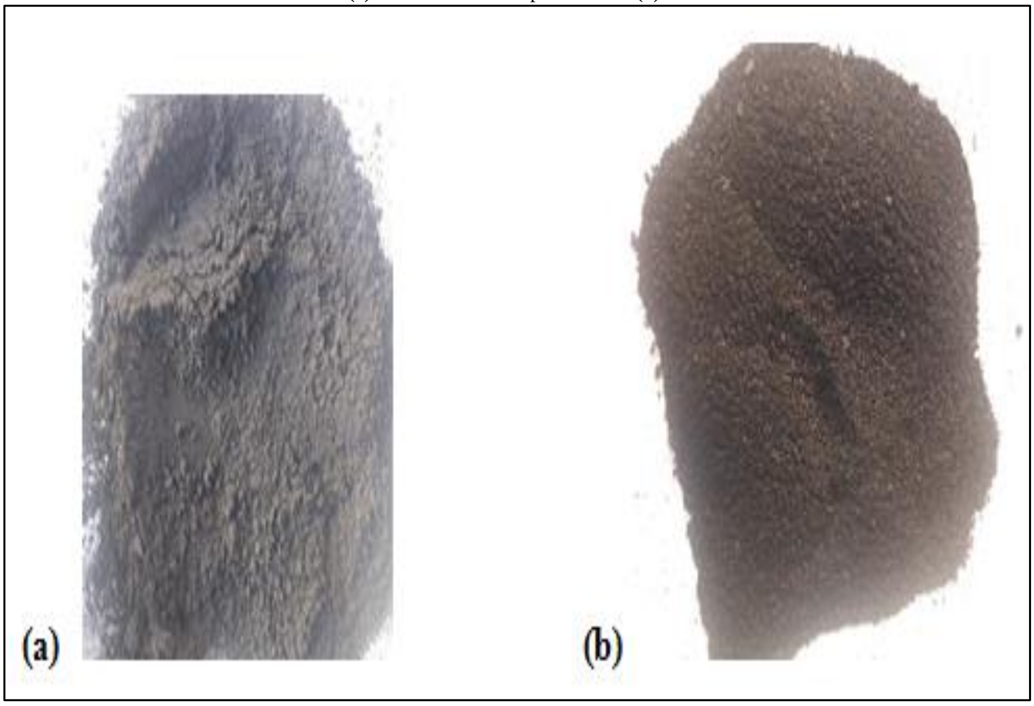


Table-1. Physiochemical Parameters Toner Powder Ink and Wood Soot Samples

S/N	Parameters	PIS	WS
1.	pH	8.47	7.51
2.	Conductivity (µʃ/cm)	0.07	0.78
3.	Moisture content (%)	20	5
4.	Bulk density (g/cm^3)	0.6166	0.4736

3.3. SEM Characterization of Toner Powder and Wood Soot

The morphology of printer ink and wood soot was performed and their respective images displayed as plate 2. Surface morphology of the kerosene based soot, diesel based soot, printer ink and wood soot were examined using the scanning electron microscopy. Several grains with irregular sizes and shapes were observed. The printer ink particles are extremely small. The SEM image of the printer ink show particles of Carbon which are chain-like agglomerations

Energy dispersive spectroscopy (EDS) of printer ink is presented in Figure 1. The spectra show high percentage weight of Carbon and Oxygen. The composition of the printer ink aggregates from the EDS analysis indicates the sample to consist of about 57.62% weight of Carbon, 31.44% weight of Oxygen, 8.64% weight of Nitrogen, 1.29% weight of Silicon, 0.37% weight of Sulfur, 0.33% weight of Phosphorus, and 0.31% of Aluminium. The wood soot consists of 66.26% Carbon, 25.86% Oxygen and 5.25% Nitrogen. From the result, the percentage weight of Carbon in wood soot is higher than that of the printer ink. This may be the reason why soot is simply in a quasi-graphic form of nearly pure elemental carbon that is distinguished by its very low quantities of extractable organic compounds and total inorganics [8].

3.4. Morphology of Wood Soot

The SEM micrograph of wood soot is presented in plate 2. The surface morphology of the wood soot through the use of scanning electron microscopy shows compact aggregates that are irregular in shapes. There are several grains with what looks like carbon. Energy dispersive spectroscopy (EDS) of wood soot is presented in Figure 2. The spectrum shows the presence of Carbon and Oxygen as the combustion product of wood. The composition of the soot aggregates from the EDS analysis indicates the soot to consist of about 66.26% weight carbon, 25.86% weight oxygen, 5.59% weight Nitrogen, 1.01% weight Chlorine, 0.56% weight Sulfur, 0.15% weight Aluminium, 0.14% weight Sodium, 0.12% weight Silicon and 0.12% weight Phosphorus. The results show that wood soot composed mainly of Carbon and Oxygen with a low level percentage of Nitrogen, Chlorine, Sulfur, Magnesium, Aluminium, Sodium, Silicon and Phosphorus.

Plate-2. SEM Image of (a) Toner Ink and (b) Wood Soot

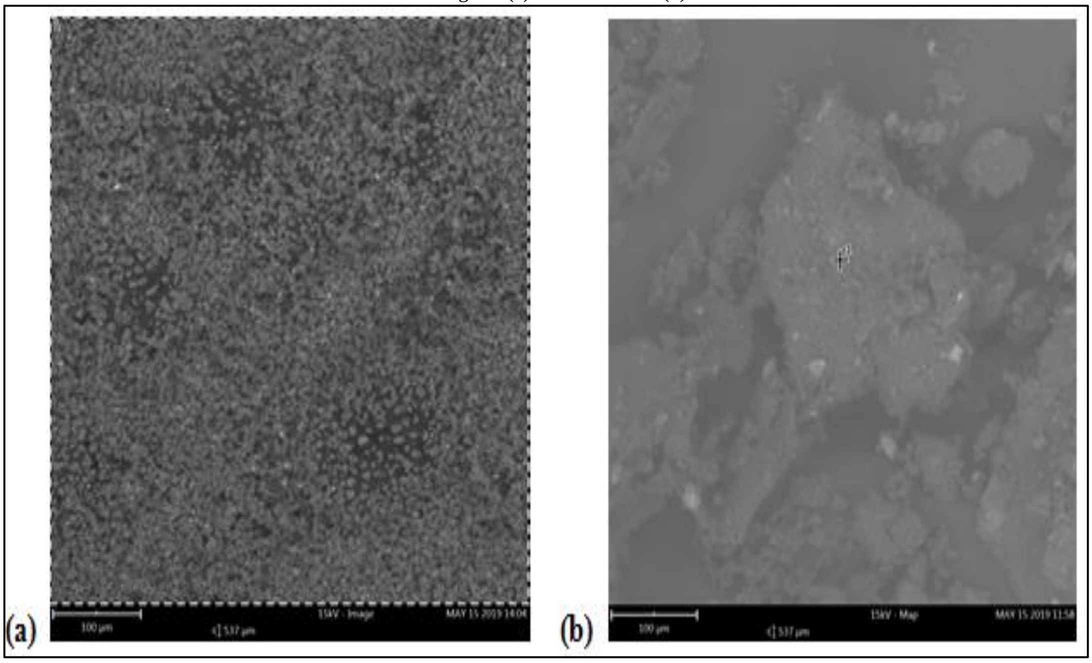


Figure-2. EDS spectrum of (a) Printer Toner Powder and (b) Wood Soot

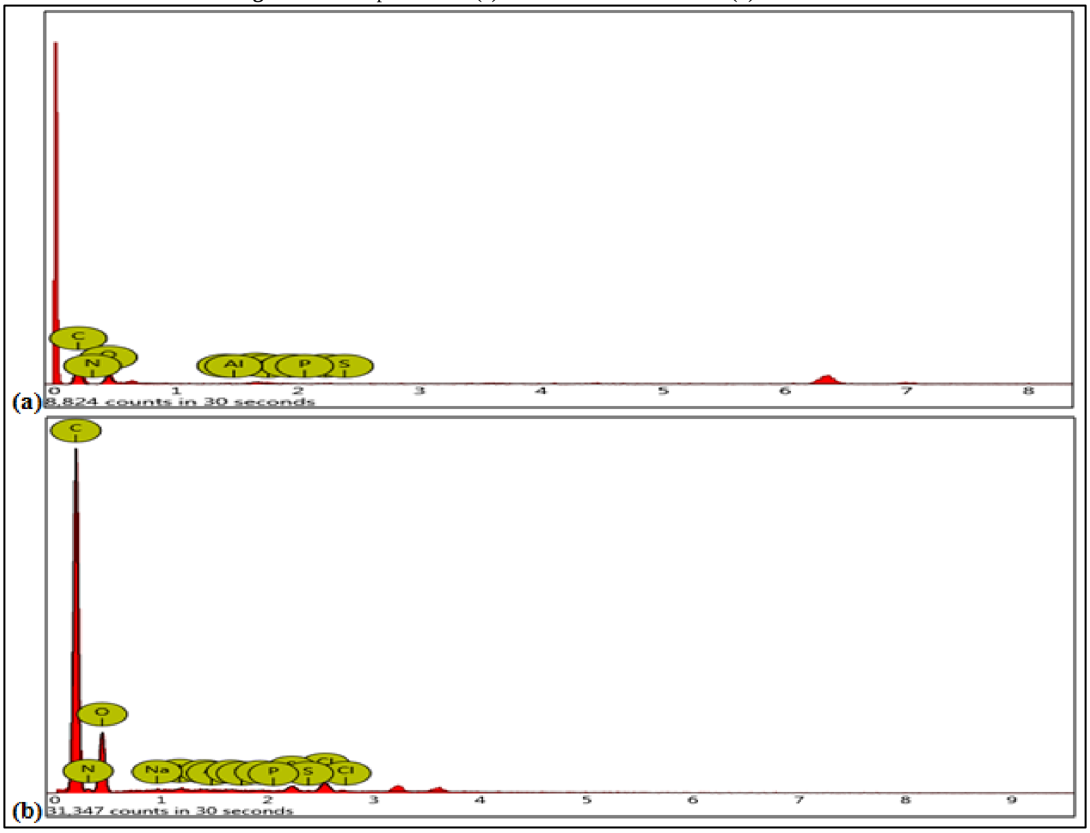


Table-2. Elemental Composition (wt %) of Toner Ink by PIS-SEM

Atomic No.	Element Name	Weight Concentration (%)	
		PIS	WS
6	Carbon	57.62	66.26
8	Oxygen	31.44	25.86
7	Nitrogen	8.64	5.59
14	Silicon	1.29	0.12
16	Sulfur	0.37	0.56
15	Phosphorus	0.33	0.12
13	Aluminium	0.31	0.15
17	Chlorine	-	1.01
12	Magnesium	-	0.20
11	Sodium	-	0.14

3.5. Fourier Transform Infra-red Results of Printer Ink and Wood Soot Samples

The quantitative and functional groups of Printer ink powder and wood soot were determined by FTIR technique. This chemical analysis revealed various spectral characteristics for each sample as presented in Figures 3 (a) and (b) respectively. The peak at 693.3cm^{-1}, 1446.2cm^{-1}, 1722.0cm^{-1}, 2914.8cm^{-1} and 3022.9cm^{-1} in Printer Ink toner (PIS) comes mainly from C-Cl stretch in alky halides, C-H bend in alkanes, C=O stretch in esters, saturated aliphatic, C-H stretch alkanes and C-H aromatic respectively. The 29.22.2cm^{-1}, 1036.2cm^{-1} and 1110.7cm^{-1} for WS are also attributed to C-H stretch alkanes, C-N stretch alcohols carboxylic acid, esters, ethers respectively. In a published research [9], the extractable material of young soot obtained in the inception region of an ethylene premixed flame was chemically characterized using FTIR analyses. The results indicated that the extractable fraction of the soot demonstrated not only aromatic but also aliphatic characteristics. PAHs are a group of semi-volatile organic compounds composed of 2 or more aromatic rings, generated during incomplete combustion of organic matter. During this process molecules and radical fragment are combined, thus creating these substance [10]. These compounds are major environmental pollutants because they are considered to be potentially carcinogenic and mutagenic, hence considered "air quality markers" in terms of the health risks their presence represents [11]. In the presence of light, amines released in the liquid or gaseous form react with atmospheric oxidants including oxidized Nitrogen compounds (photo-oxidation). As a result, new compounds are formed such as nitrosamines, nitraamines and amides. The formation of nitrosamines is dangerous because these substances are toxic and carcinogenic even at very low concentrations [12]. Formaldehyde, one of the most abundant carbonyls in air, has been classified as a human carcinogen by the International Agency for Research on Cancer (IARC) due to its carcinogenicity [13]. The peak at 1543.1cm^{-1} and 1509.6cm^{-1} for DES and DVS respectively are attributed to N-O asymmetric stretch in nitro compound, are not present in wood soot. Nitro compounds are group of the organic substances containing one or more nitro (-NO_2) groups within their aromatic ring or aliphatic chain. They are characterized by, especially those aromatic characters high toxicity [14], carcinogenicity [15] resistance to degradation and tendency to accumulate in the environment.

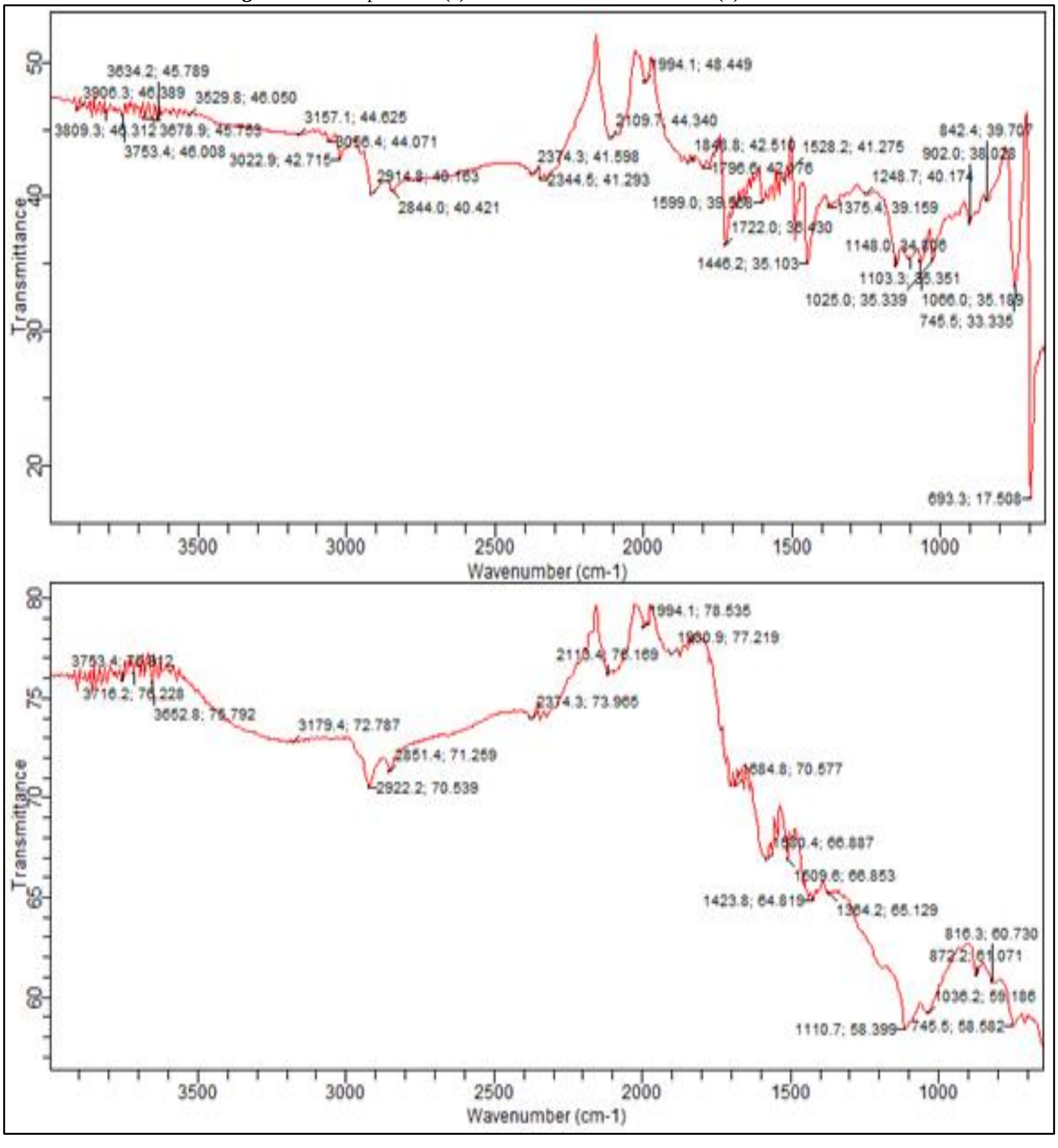


Figure-3. FTIR Spectra of (a) Printer Toner Ink Powder and (b) Wood Soot

Table-3. FTIR Spectral Information of Printer Ink and Wood Soot

S/NO	Group frequencies (cm^{-1})	Functional group	Observed frequencies (cm^{-1}) PIS	WS	Assignment
1	3100-3000	Aromatics	3022.9	-	C –H stretch
2	3000-2850	Alkanes	2914.8	2922.2	C – H stretch
3	2830-2695	Aldehydes	-	-	H-C=O:C-H stretch
4	2260.-2100	Alkynes	2109.7	2113.4	-C (triple bond)C-Stretch
5	1750-1705	Esters, saturated aliphatic	1722.0	-	C = O stretch
6	1470-1450	Alkanes	1446.2	-	C-H bend
7	1320-1000	Alcohols, carboxylic acids, esters, ethers	-	1110.7	C-O stretch
8	1250-1020	Aliphatic amines	-	1036.2	C-N stretch
9	900-675	Aromatics	745.5	745.5	C-H "OOP"
10	850-550	Alkyl halides	693.3	-	C-Cl stretch

3.6. XRF Characterization of Toner Printer Ink Powder and Wood Soot

The comparative study of elements in printer ink and wood soot were carried out. This chemical characterization of the samples to determine the elemental composition using energy dispersive X-ray fluorescence spectrometer (ED-XFR) revealed the results presented in Table 4. The XRF spectra for samples and printer ink are presented in Figures 4 (a) and (b). Table 4 presents the results of elemental composition (mg/kg) of printer ink and wood soot sample using electron dispersive XRF. The result reveal that printer ink contained Mn 573.746, Cr 91.092, Cu 20.940, Zn 54.305, Ti 4982.871, Fe 115899.265, Sn 2606.545, Al 333.529, S 476.948, Si 727.633, Cl 301.121. Compared with wood soot, Mn 29.611, Cu 12.795, Zn 32.631, Ti 136.350, Fe 332.824, Pb 22.755, Sn 19440.988, Al 361.691, P 879.469, S 3478.185, Si 154.876, Cl 7374.882, Br 18.523, Ca 6717.88. Wood soot presents high value for phosphorus and bromine but no value in printer ink. Very high value of Chromium was detected in printer ink but wood soot presents no value for chromium. Manganese concentrations as recorded from the analyses are generally high. Although manganese is an essential nutrient, exposure to high levels via inhalation or ingestion may cause some adverse health effects. It has been suggested that these adverse health effects, especially neurologic effects, are occurring on "continuous of dysfunction' that is dose-related [16]. In other words, mild or unnoticeable effects may be caused by law, but physiologically excessive, amounts of manganese, and these exposure level or duration of exposure increases. Chronic exposure to manganese at very high levels results in permanent neurological damage, chronic exposure to much lower levels of manganese (as with occupational exposures) has been linked to deficits in the ability to perform rapid hand movements and some loss of coordination and balance, along with an increase in reporting mild symptoms such as forgetfulness, anxiety, or insomnia [17].

Figure-4. XRF of (a) Printer Toner Ink Powder and (b)Wood Soot

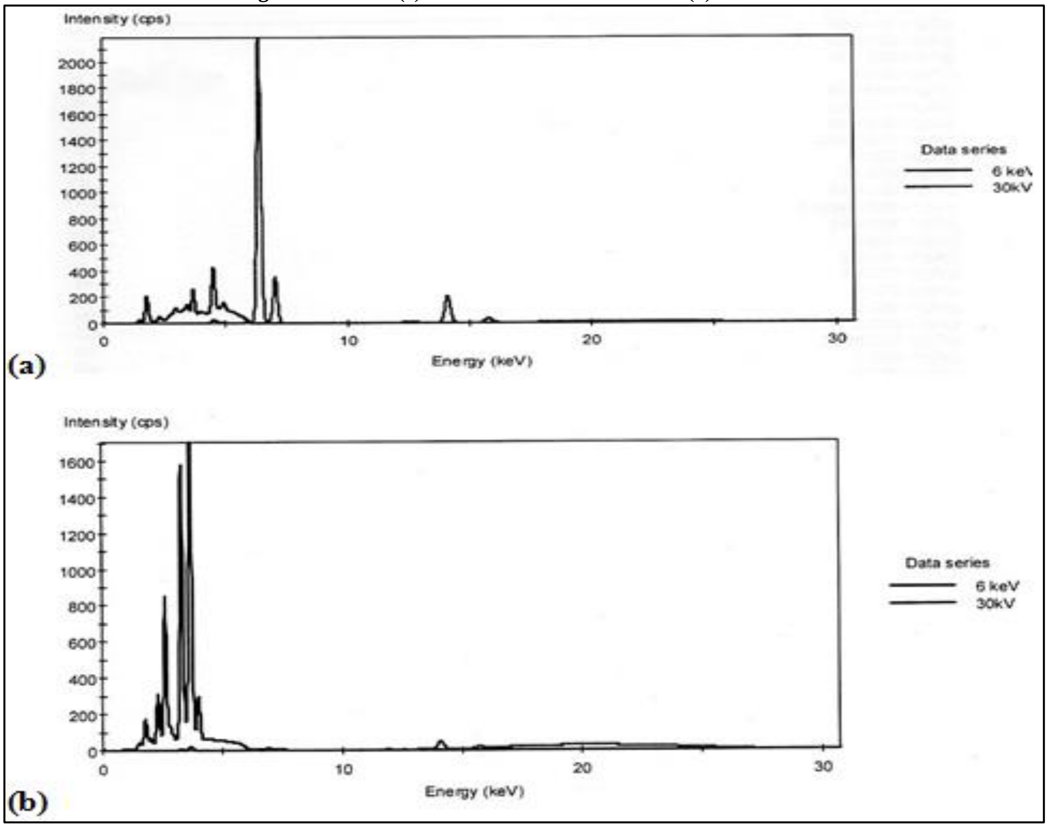


Table-4. Elemental Composition (mg/kg) of Printer Ink, Wood Soot Samples Obtained from XRF Analysis

			Concentration (mg/kg)	
S/N	Class	Element	PIS	WS
1.	Heavy metals	Manganese	573.746	29.611
		Chromium	91.029	ND
		Copper	20.940	12.795
		Zinc	54.305	32.631
		Titanium	4982.871	136.350
		Iron	115899.265	332.824
		Lead	ND	22.755
		Tin	2606.545	19440.988
		Calcium	685.879	6717.887
		Aluminium	333.529	367.691
2.	Non-metal	Phosphorus	ND	879.469
		Sulphur	476.948	3478.185
		Silicon	727.633	154.876
		Chlorine	301.121	7374.882
		Bromine	ND	18.523

3.7. XRD Characterization of Printer Ink Toner Powder and Wood Soot Samples

Tables 5 and Figure 5 presents the crystallographic parameters for printer ink and wood soot analyzed, using X-ray diffraction technique.

3.8. Mineralogy of Printer Ink

The mineralogical component of printer ink was carried out by X-ray diffraction technique. The main minerals found in the printer ink sample were magnetite and magnesite respectively. With each of this compound having a phase information from the XRD pattern. The result for this shows that 2ϴ values for magnetite in printer ink produce high intensities at 35.439 and 62.546 regions respectively. This like other compounds present from XRD analysis shows that broadening of reflections beyond (25%) intensity due to instrumental factors is attributed to crystalline size effects. Magnetite with the chemical formula $Fe_{24}O_{32}$ has cubic crystal system. Magnetite is discussed in regards to its potential applications in environmental engineering, biomedical/medical, microfluidic, and mechano-electrical fields [18]. Environmental, chemical and biological engineers consider magnetite nanoparticles to be used effectively in environmental contaminant removal and cell separation. Small amount of magnetite are used as a toner in electrophotography, as a micro-nutrients in fertilizers, as a pigment in paints and as an aggregate in high density concretes.

Magnesite is a magnesium carbonate mineral with a chemical fomular $Mg_6C_6O_{18}$. It is named after the presence of magnesium in its composition. As observed in this research magnesite as one of the component of XRD characterization of printer ink reveals that the 2ϴ values shows high intensity at 32.660 and 43.005 regions, respectively. This means that broadening of reflections beyond 25% intensity due to instrumental factors is predominantly attributed to crystallite size effects. The crystal system of magnesite is hexagonal. Magnesite usually forms during the alteration of magnesium-rich rocks or carbonate rocks by metamorphism or chemical weathering. Magnesite is used to produce Magnesium Oxide (MgO), which serves as a refractory material for the steel industry and as a raw material for the chemical industry. small amounts of magnesite are also used as a gem and lapidary material [19].

3.9. Mineralogy of Wood Soot

The mineralogical component of wood soot sample was carried out by X-ray diffraction technique. The main minerals found in the sample were Calcite, Sylvite and Quartz. Each of these compounds have a phase information from XRD pattern. The chemical analysis of the Calcite in wood soot sample reveal that high intensity observed for 2ϴ value is only at 29.390. Broadening of reflection attributed to crystallite size is less. Calcite is widely distributed in earth's crust; it appears most commonly within sedimentary rocks, where it occurs as the principal mineral of limestone, and as a natural cementing agent in many siliceous sand, stone and shale units that were deposited under marine conditions. Calcite also dominates some metamorphic rocks such as marble and calcareous gneiss; occurs widely in hydrothermal system, where it forms extensive vein networks; and is common in some unusual carbonate-rich igneous rocks such as carbonatites. Calcite is used as an acid neutralizer in the chemical industry in areas where streams are plagued with acid mine drainage, crushed limestone is dispensed into the streams to neutralize their waters [20].

Quartz also reveals a single high intensity at 2ϴ value 26.624, other intensities reveals lower values below 25% intensity. Broadening of these reflections beyond that due to instrumental factors is attributed to crystallite size effects. Quartz is one of the most famous mineral on the earth, it occurs in essentially all mineral environments, and is the crucial constituent of many rocks. It is likewise the maximum varied of all minerals, taking place in all distinct bureaucracy, habits and colouring. Quartz is highly resistant to both mechanical and chemical weathering. This durability makes it the dominant mineral of mountain tops and the primary constituent of sea side, river, and wilderness sand. It is ubiquitous, ample and durable. Mineral deposits are determined at some stage in the world. The weathering of any quartz bearing rocks creates sand: igneous, sedimentary or metamorphic rock rocks. It is a continuous cycle of rocks formation and erosion that started with the earth's formation and continues today. Quartz is used in a great variety of products and the term "Quartz sand in its finest form, as micro-silica is used as an essential raw material for the glass and foundry casting industries, as well as in other industries such as ceramics, chemical manufacture and for water filtration purposes. This result revealed that the 2ϴ values for Sylvite between 28.314 and 40.472 shows high intensities. This means that broadening of reflections beyond 25% due to instrumental factors is predominant for Sylvite in wood soot sample and this is attributed to crystallite size effects. Sylvite is an economically important mineral and is extensively mined, though it is rarely presented in mineral collections as attractive specimens. It is the most significant form of potash, or potassium bearing compounds. Sylvite is the main source of potash, which is an important ingredient in fertilizers. It has other important industrial uses, including aluminium recycling, metal electroplating, and in oil drilling fluids.

Figure-5. X-Ray Diffractogram of (a)Toner Powder Ink and (b) Wood Soot

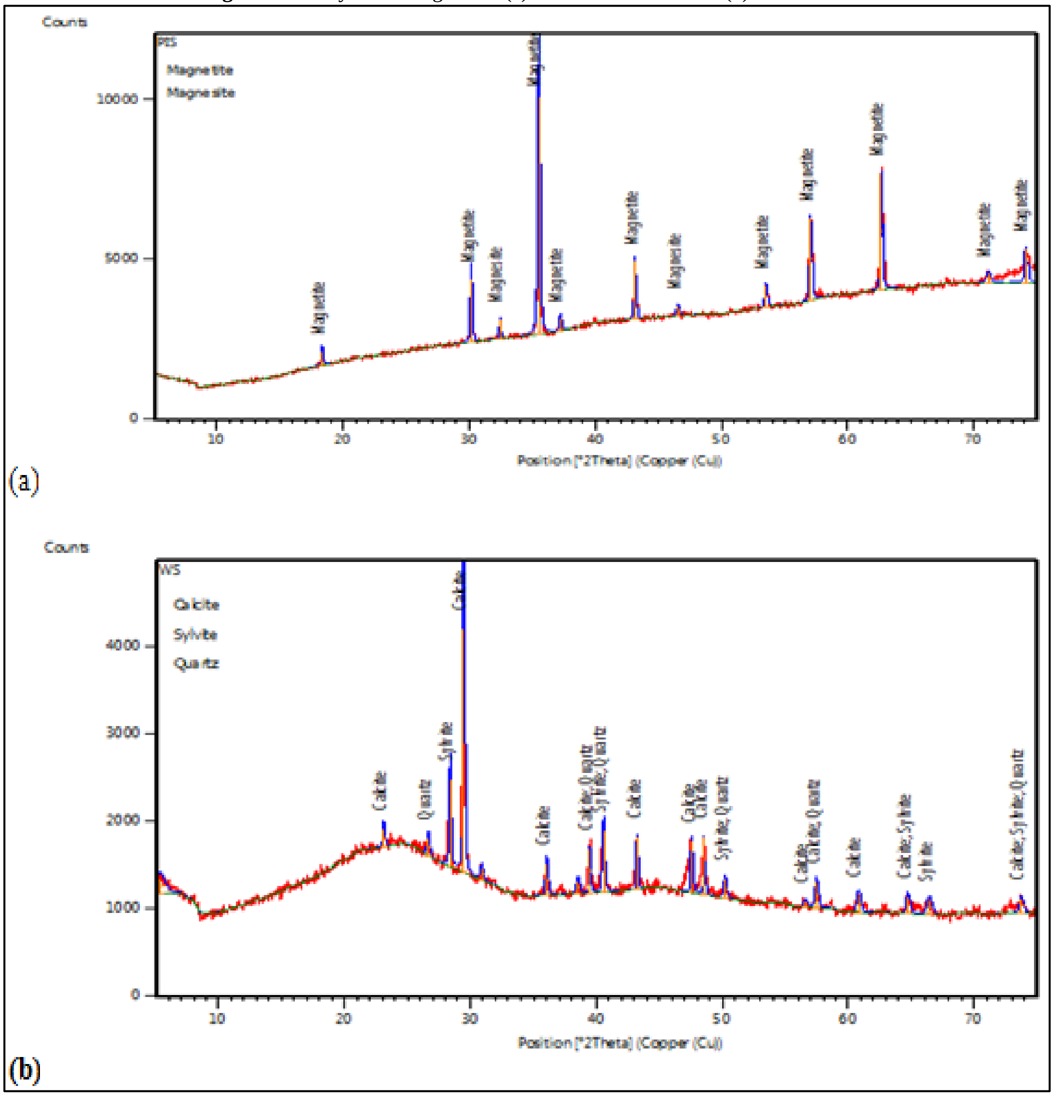


Table-5. Crystallographic Parameters of Printer Ink

Pos. [^{0}2Th.]	FWHMLeft [^{0}2Th.]	d-spacing [Å]	Crystalline size (nm)
18.3170	0.1535	4.84359	9.497
30.1401	0.1535	2.96515	10.425
32.4099	0.1023	2.76248	16.122
35.4842	0.1791	2.52988	9.497
37.1940	0.2047	2.41741	8.506
43.1155	0.1279	2.09814	14.909
46.4967	0.2047	1.95314	9.904
53.4744	0.1791	1.71358	12.958
57.0145	0.1535	1.61531	16.705
62.6112	0.1279	1.48370	23.500
71.1127	0.3070	1.32577	14.00
74.0779	0.2047	1.27987	24.759
Average crystalline size			**14.23**

Table-6. Crystallographic Parameters of Wood Soot

Pos. [^{0}2Th.]	FWHMLeft [^{0}2Th.]	d-spacing [Å]	Crystalline size (nm)
5.3797	0.8187	16.42750	1.701
23.1074	0.2047	3.84918	7.371
26.6446	0.2047	3.32567	7.589
28.3881	0.1535	3.14403	10.278
29.4583	0.1535	3.03221	10.386
30.8807	0.3070	2..89570	5.271
36.0381	0.2047	2.49226	8.352
38.5149	0.3070	2.33750	5.777
39.4586	0.1023	2.28374	17.550
40.5433	0.1791	2.22511	10.270
43.2187	0.1023	2.09336	18.487
47.5592	0.1535	1.91195	13.461
48.5223	0.1023	1.87623	20.694
50.1903	0.2558	1.81772	8.454
56.5598	0.3070	1.62721	8.204
57.4441	0.2558	1.60424	10.047
60.7967	0.3070	1.52356	9.305
64.7126	0.3070	1.44051	10.584
66.4047	0.4093	1.40785	8.454
73.6843	0.4070	1.28573	16.122
Average crystalline size			**10.418**

Table-7. Associated Compounds in Soot and Printer Ink Samples

			Crystallographic parameters		
2	**Samples**	**Associated compounds**	**Crystal system**	**Calculated density**	**Volume of cell**
1.	WS	Calcite	Hexagonal	2.71	368.20
		Sylvite	Cubic	1.98	249.93
		Quartz	Hexagonal	2.64	113.21
2.	PIS	Magnetite	Cubic	5.20	591.43
		Magnesite	Hexagonal	3.01	278.75

4. Conclusion

Highlights of the studies revealed high concentration of impure carbon in both toner ink powder and wood soot, with crystallographic sizes at the nano range, marking the possibilities of easy fragmentation into air current. The FTIR spectral analysis revealed that aromatic, primary amines, carbonyl, and alkynes were present. These compounds are major environmental pollutants and are considered to be potentially carcinogenic and mutagenic.

Acknowledgement

Special thanks to the original and unreached author of Fig. 1, whose adopted SEM image was cited in International Agency for Research on Cancer IARC [13].

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