EVALUATION OF PHYSICOCHEMICAL PARAMETERS AND HEAVY METALS OF PARTICULATES DUST SAMPLES IN FUNTUA TEXTILE LIMITED KATSINA STATE, NIGERIA

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Abstract: The study investigated twenty-two particulate dust samples from ten different departments and a control site at Funtua textile limited, Katsina, Nigeria for total concentrations of Cd, Cr, Cu, Ni, and Pbusing Atomic Absorption spectrometry. The physicochemical parameters such as pH, Electrical conductivity (EC (μScm^{-1}), ammonium nitrogen (NH_4-N (%), nitrate nitrogen (NO_3-N (%), organic matter (OM (%) and chloride (Cl^-(mg/kg) contents were also investigated in the dust particulate samples using standard analytical methods. The levels of the analysed parameters in the samples were compared with those of world health organization (WHO) in order to draw a meaningful conclusion. The mean concentrations of heavy metals in the particulates dust were in the ranges of; Cd: 0.02 ± 0.00 (S_1& O_1) to 0.15 ± 0.17 (E_1), Cr: BDL (W_2) to 174.30 ± 9.79 (E_1), Cu: 0.18 ± 0.08 (O_1) to 356.68 ± 33.57 (E_1), Ni: 0.09 ± 0.04 (O_1) to 3.44 ± 0.12 (E_1) and Pb: 0.14 ± 0.04 (S_2) to 5.09 ± 0.06 (W_2) $mgkg^{-1}$, respectively. The ranges of 5.235 ± 0.021(O2) to 4.750 ± 0.354(E2), BDL to 0.032 ± 0.004 (S1), BDL (W_2) to 0.011 ± 0.001 (G, S1), 0.640 ± 0.028 (C) to 89.240 ± 1.131 (W_2) and 0.30 ± 0.001 (O_1) to 12.70 ± 0.424 mg/kg (E2) pH, EC (μScm^{-1}), NH_4-N (%), NO_3-N (%),OM (%) and Cl^- (mg/kg), respectively. The levels of the analysed metals in the dust particulate samples within the industry were higher than those in the control site and those of the European commission (2005) tolerable limit which clearly shows that the industry contributes immensely in polluting the immediate environment.

Keywords: Atomic absorption spectrophotometry, Funtua, heavy metals, particulates dust

Introduction

Industrialization is one of the main causes of environmental pollution in many cities in the developing world (UNIDO, 2003). The continuing industrial development has led to a corresponding increase in the level of pollution. Most processes performed in textile mills produce atmospheric emissions. Gaseous pollution has been identified as the second greatest pollution problem (after effluent quality) for the textile industry. Air emissions include dust, oil, acid vapours, odours and boiler exhausts (Modak, 1991).

Heavy metals are natural constituents in nature, usually occurring in low concentration under normal conditions. Anthropogenic activities can cause elevated levels of these metals in various parts of the ecosystem. Environmental pollution by heavy metals may occur via various diffuse and point sources. Heavy metal scattering by traffic is an example of diffuse spread, while the emission of heavy metals by industrial establishments like textile factories, metal smelters and iron works represents point sources. Among the different textile pollution, cotton dust pollution is the most important in terms of health effects (Mehwish *et al*., 2016).

Heavy metals on the other hand refer to any metallic element that has a relatively high density and is toxic even at low concentration (Lenntech, 2004; Duruibe *et al.,* 2007; Uba *et al.,* 2009). The accumulation of heavy metals in soil poses risk to human and the ecosystem's health (Afshin and Masaud, 2008; Nwachukwu *et al.,* 2010; Odoh, 2011). Risks may be passed to human either through the food chain or through direct exposure. Risks arising from pollution of soils by heavy metals are well known (Nriagu, 1990). A range of metals and chemical compounds found in industrial dust environment are harmful (Archer and Barratt, 1976). Pollutants can attack specific sites or organs of the body and disease can develop as a consequence of such exposure (Archer and Barratt, 1976; Ayodele and Gaya, 1998). This paper is therefore aimed at assessing the levels of physicochemical parameters and heavy metals in dust particulate samples of Funtua textile so as to ascertain the levels of these pollutants within the study area which can also serve as data base for future research works.
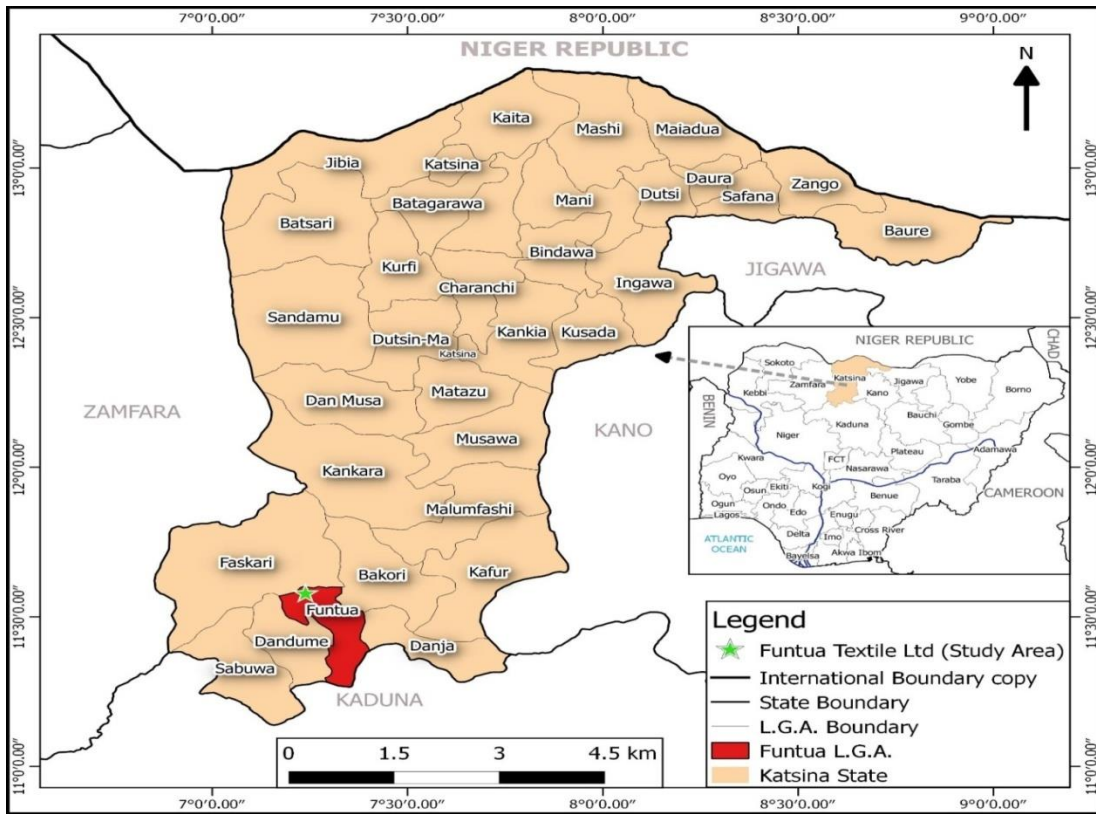
Materials and Methods

Study area

Funtua is located at latitude $11^0 32'$ N and longitude $7^0 19'$ E; it is a Local Government area of Katsina State (Fig. 1). It has an area of 448 km^2 and a population of 420,110 according to census estimate in 2012. The mean annual rainfall in the area is 1024 mm. The annual temperature varies between 21.9 – 29.2^0C (Africa Atlas, 2002). The selected area for this investigation is Funtua Textile Industry which was established to make effective use of cotton grown in large quantity at Funtua town and its environs. The industry is active 24 h a day, five days a week.

The sampling points were selected from each department and two random points within the vicinity: Ginning Department (G), Spinning Department: Blowing & Carding (S_1), Combing, Spinning & roving (S_2). Weaving Department: Doubling & Bleaching (W_1), Weaving knitting (W_2), Dyeing Department: Dyeing & finishing (D), Engineering Department: Maintenance (E_1), Power House (E_2), Ginning surrounding (Outside O_1), Dyeing surrounding (Outside O_2), with a Control area (C) at Dikke Village about 2 km away from the study area (Fig. 2). The metals investigated include Cd, Cr, Cu, Ni, and Pb. These metals were selected because of their toxicity and as an indicator of environmental pollution.

Sample collection and pre-treatment

Twenty-two (22) samples of particulates dust were collected across the sampling points according to the standard method described by Loredo *et al.* (2003); Yeung *et al.* (2003). The collected samples were labelled, stored in clean polythene bags and then transported to the laboratory (Al-Kashman, 2004). The homogenized particulate dust samples were air dried and sieved through a 75 µm stainless steel sieve to remove extraneous matter such as pieces of brick, paving stone and other debris. Care was taken to reduce the disturbance of the fine particles, which could be readily lost by re-suspension (Manta *et al.,* 2002). The temperature range across the sites was 28.39 (W_2) to 32.07(C) °C and 29.50 (W_2) to 32.43 (O_1) °C for morning and evening periods, respectively (Fig. 2).

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875

Source: Modified from the Administrative Map of Katsina State
Fig. 1: Map of Katsina State showing Funtua LGA & Funtua Textile Ltd. (StudyArea)
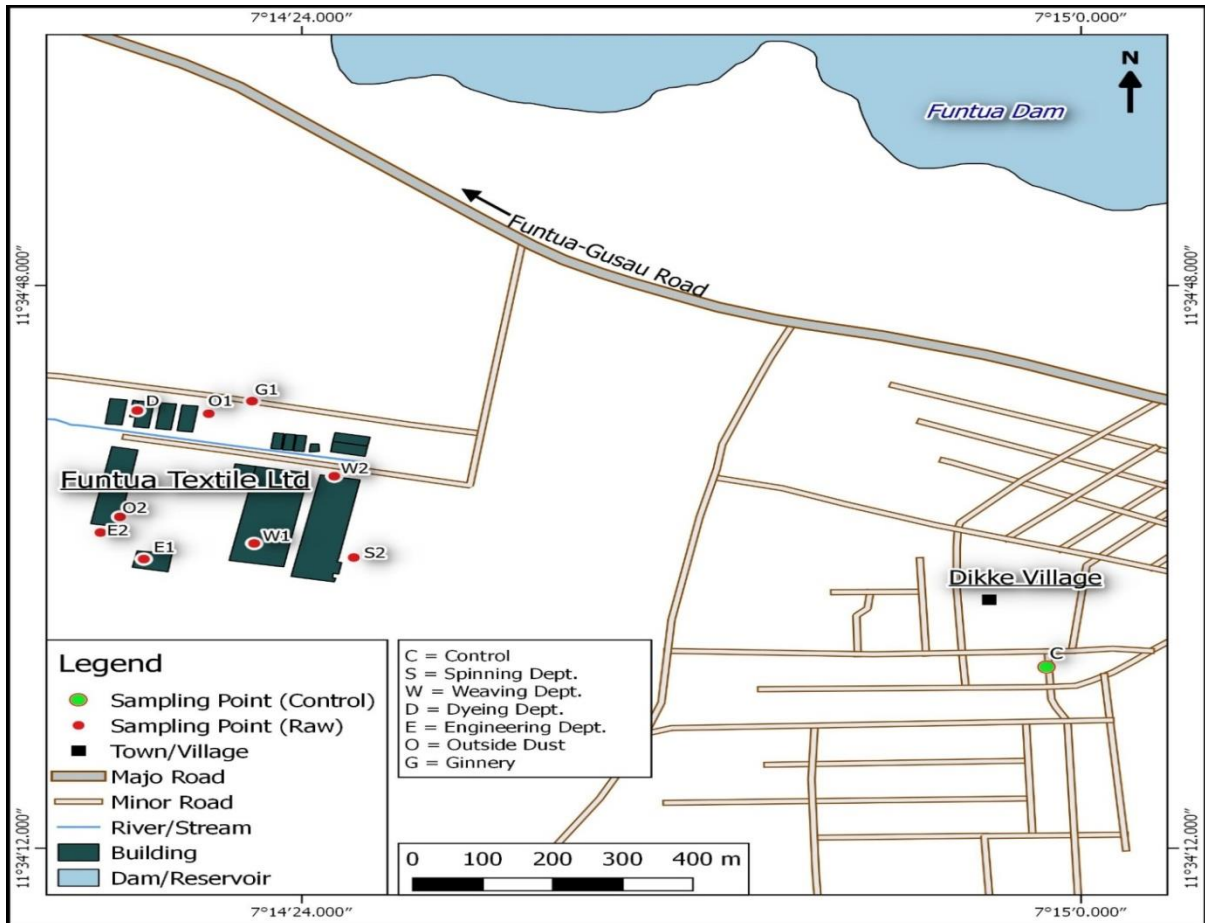
Source: Modified from the Administrative Map of Funtua LGA
Fig. 2: Map of Funtua Textile Ltd. showing sampling point

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876

Quality assurance

All reagents used were of analytical grade, distilled de-ionized water was used. All the glass wares and polythene sample bottles were washed with liquid soap, rinsed with distilled water, soaked in 10% HNO_3 for 24 h and rinsed thoroughly with distilled de-ionized water and thereafter dried (Todorovi *et al*., 2001).

Validation of the technique was conducted on the digested dust sample by spiking the pre-digested sample with multi-element standard solution (5 mg/dm^3 of Cd, Cu, Ni, Pb and 0.5 mg/dm^3 Cr) as reported by Awofolu (2005).

Digestion of dust particulate for elemental analysis

2.0 g of the dried sample was accurately weighed into a conical flask and 10 cm^3 of nitric acid was added and the mixture was heated on a hot plate until the sample is almost dry and then cooled. The procedure was repeated with another 10 cm^3 of concentrated nitric acid followed by the addition of 10 cm^3 of 2 M Hydrochloric acid, the digested dust was warmed with 20 cm^3 of 2 M HCl and filtered into a 50 cm^3 volumetric flask through Whatman filter paper, thereafter, diluted to the mark with double distilled water (Divrikli *et al*., 2006).

Determination of physicochemical parameters

pH (Hendershot et al., 1993)

10 g of the dried sample was weighed into a 50 cm^3 beaker and 20 cm^3 distilled deionized water added. The mixture was left to stand for 30 minutes and stirred occasionally with a glass rod. The electrodes of a pre-calibrated HANNA pH meter (model Grison MicropH 2000) was inserted into the partly settled suspension and pH recorded.
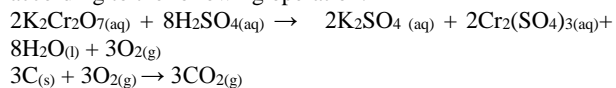
Electrical conductivity (Wilcox, 1995)

25 g of the air-dried sample was placed in a 250 cm^3 beaker. Distilled water was added slowly drop by drop uniformly over the entire sample surface until the sample becomes wet. A stainless steel spatula was used to form a homogenous saturated paste.

The beaker was then covered with a petri-dish. 50 cm^3 distilled water was added and shaken for 1 hour. 40 cm^3 of the extract was transferred into 100 cm^3 beaker and the electrode of the conductivity meter was inserted, the electrical conductivities of the dust samples were thereafter recorded in μScm^{-1}.

Organic matter (Rowell, 1994)

In this method, organic carbon was oxidized by $K_2Cr_2O_7$ in the presence of H_2SO_4 which lead to the formation of CO_2 according to the following operation:

$2K_2Cr_2O_{7(aq)} + 8H_2SO_{4(aq)} \rightarrow 2K_2SO_{4\ (aq)} + 2Cr_2(SO_4)_{3(aq)} + 8H_2O_{(l)} + 3O_{2(g)}$

$3C_{(s)} + 3O_{2(g)} \rightarrow 3CO_{2(g)}$

Exactly 1 g of the particulate dust sample was weighed in triplicates into 250 cm^3 conical flask. 10 cm^3 of $K_2Cr_2O_7$ (0.02 moldm^{-3}) was transferred into each flask and swirled gently to disperse the particulate followed by the addition of 20 cm^3 of H_2SO_4 (R.D 1.84, 98% w/v). The flask is swirled gently until the sample and reagents were thoroughly mixed. The mixture was allowed to stand on a hot plate for 30 min. 100 cm^3 of distilled water was added followed by the addition of 4 drops of ferroin indicator, after which it was titrated with 0.25 moldm^{-3} ammonium ferrous sulphate. Blank titration was carried out in a similar manner. The percentage organic carbon is given by the equation:

% Organic carbon $= \frac{\text{(Blank Titre – Actual Titre) x 0.3 x M x f}}{\text{Weight of air – dried soil taken}}$

Where: f = correction factor = 1.33

M = concentration of ferrous sulphate

Then % organic matter = % organic carbon x 1.729

Chloride (Skoog et al., 1996)

The Mohr's method in which alkaline or alkaline earth chlorides react with silver nitrate in the presence of a few drops of potassium chromate solution as indicator is a simple, direct and accurate method for chloride determination employed.

The soil sample was dried at 110^{0}C for 1 hour and cooled in a desiccator. 2 g of the sample was weighed into 250 cm^3 Erlenmeyer flask and dissolved into 100 cm^3 of distilled water. Small quantities of $NaHCO_3$ were added until effervescence ceases. About 2 cm^3 of K_2CrO_4 was introduced and the solution was titrated to the first permanent appearance of red $Ag_2Cr_2O_4$. The chloride free $CaCO_3$ in 100 cm^3 of distilled water contains 2 cm^3 of $K_2Cr_2O_7$. The percentage of chloride is thus computed using the formula below.

Moles of Cl$^-$ = M_{AgNO3} x V_{AgNO3}

Mass of Cl$^-$ = Moles of Cl$^-$ x Molar mass

% Cl$^-$ $= \frac{Mass\ of\ Cl^-\ \times\ 100}{\text{Mass of sample}}$

Mineral nitrogen (NH_4^+ –N; NO3$^-$–N) (Brady, 1984)

15 g of the dust sample was weighed into a centrifuge tube, to which 30 cm^3 of 1 M KCl was added and agitated for one hour on a shaker. The mixture was centrifuged to obtain a clear solution. 10 cm^3 of the KCl extract was transferred into the distillation flask followed by 2 g of magnesium oxide. The content was distilled over a 2% boric acid with 2 - 3 drops of mix indicator. 30 – 50 cm^3 of the distillate was collected and labeled Ainto the same distillation flask; 1 g of devarda's was added and distilled for nitrate over another 2% boric acid with 2 - 3 drops of mix indicator. 30 – 50 cm^3 of the distillate was collected and labelled B. Both flask A and B were titrated with 0.01 M H_2SO_4 and the titre value from the flask was for NH_4 and NO_3, respectively.

Fig. 3: Particulates dust sampling at Ginning Department (G)

Determination of heavy metal contents

The metals in the final solutions were determined by Atomic Absorption Spectrophotometer (Varian AA650FS).

Results and Discussion

The pH varied from $5.235 \pm 0.092\ (W_1)$ to $6.800 \pm 0.141\ (G)$ which indicates acidic conditions. This is below the range of pH reported by Abechi *et al.* (2010) and Ochigbo (2011) in their studies. The pH serves as useful index for availability of nutrients, the potency of toxic substances present in the soil and the physical properties of the soil. Several studies have shown that availability of heavy metals is pH dependent (Iwegbue*et al.,* 2006; Gonzalex Fernandez *et al.,* 2008). The acidic pH values obtained in this study are indication of availability of these heavy metals in the particulates dust which could be attributed to the nature of

FUW Trends in Science & Technology Journal, www.ftstjournal.com
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877

chemicals used in the various departments of the Funtua textile limited. The highest level of the electrical conductivities recorded at the engineering department (E_2) $(4.750 \pm 0.354$ μScm$^{-1})$ as presented in Table: 1could be attributed to the presence of metallic scraps releasing metals in either sulphate or chloride form (Uba *et al.,* 2008) into the particulates dust as compared with the lowest value of$0.035 \pm 0.011\ \mu Scm^{-1}$ obtained from the control site (C), a village about$2\ km$ away from the textile industry. The dust particulates recorded in this study was found to be non-saline in both the control and the study area ($levels\ of\ electrical\ conductivity < 2000 \mu Scm^{-1}$) based on Boulding 1994 classification of dust/soil electrical conductivity.

The highest concentration of$0.032 \pm 0.004\%\ for\ NH_4 - N$was recorded at blowing and carding section (S1) unlike at the control site (C)where a lowest value of $0.004 \pm 0.0001\%$was recorded. The concentrations of $NH_4 - N$ recorded in this study might be attributed to the reduction of $NO_3 - N$ as reported by Uba *et al.* (2008). Similarly, the highest concentrations of $NO_3 - N$ (%) were obtained at sites G and S₁$(0.011 \pm 0.001\%)$, respectively and this was attributed to decomposition of plants grown at the vicinity of Ginning (G) and spinning (S) departments as it was also observed by Eddy *et al.* (2006). The concentration range of $NO_3 - N$ recorded in this study was lower than the range of 0.056 (RA) to 0.530 mg/kg (SH) reported by Uba *et al.,* (2008). The organic matter contents from this study ranged from0.640 ± 0.028 for the control (C) sample to 89.240 ± 1.131 for the weaving section (W_2) this was not surprising because of the large quantity of organic chemicals and materials used in this section of the factory for weaving as compared to site C that has little or no such activities taken place. The concentrations obtained in these studies were lower

than the values of $3.09 - 5.83\%$ obtained in similar studies by Ochigbo (2011).

The chloride contents of the particulate dust ranged from $0.300 \pm 0.0001 (0_1)$ to $12.700 \pm 0.424\ mg/kg\ (E_2)$. Chloride is found in soil as chloride ion, being an anion, it is fully mobile except when held by soil anion exchange sites (Uba *et al.,* 2008). The high chloride contents of the dust particulates at site E_2 could be due to leaching of heavy metals in their chloride form from the metal scrap waste present within the engineering department. The concentrations recorded in this study are lower than that reported by Uba *et al.* (2008) in a similar study.

The total concentrations of heavy metals for the samples are presented in Table 2. In each case the presented value is a mean observed in three determinations. The students' t-test revealed significant difference in the levels of physicochemical parameters and heavy metal contents at P<0.05 across the studied sites with the exception of Cd as reflected in Table 3. The dust sample in this study showed lower levels of cadmium contamination as compared to $0.75\ mgkg^{-1}$ (Jaradat and Momani, 1999), $2.11\ mgkg^{-1}$ (Amusan *et al.,* 2003) and $1.47\ mgkg^{-1}$ (Ochigbo, 2011) in a similar study. The level of cadmium could be attributed to abrasion from the moving parts of machines from the factory, lubricating oils and/or old vehicular tyres that are frequently used and dumped especially in the engineering section (E_1) of the factory. The concentrations of cadmium in the dust samples were generally lower than the worldwide value $(0.5 - 4.0\ mgkg^{-1})$ (Fergusson, 1991). The levels of chromium from across the sites ranged from $BDL (W_2)$ to $174.300 \pm 9.790\ mgkg^{-1} (E_1)$.

Table 1: Physicochemical parameters of particulate Dust samples of Funtua Textile Limited

Sites	pH	EC (μScm⁻¹)	NH₄–N (%)	NO₃–N (%)	OM (%)	Cl⁻(mg/kg)
G	6.800±0.141	1.950±0.212	0.014±0.000	0.011±0.001	13.925±1.549	2.050±0.071
E₁	6.650±0.212	0.115±0.007	0.009±0.002	0.007±0.000	9.920±0.325	0.450±0.071
E₂	6.550±0.071	4.750±0.354	0.024±0.001	0.009±0.002	3.915±0.219	12.700±0.424
O₁	6.550±0.212	0.140±0.014	0.010±0.001	0.007±0.000	2.470±0.028	0.300±0.000
O₂	6.600±0.141	0.055±0.021	0.007±0.000	0.007±0.000	5.785±0.672	0.400±0.141
S₁	6.550±0.212	0.175±0.035	0.032±0.004	0.011±0.001	30.955±1.761	5.800±0.283
S₂	5.955±0.078	0.210±0.028	0.011±0.000	0.001±0.000	64.685±0.686	1.100±0.141
D	5.300±0.141	0.250±0.014	0.001±0.000	0.001±0.000	78.165±0.290	1.200±0.141
W₁	5.235±0.092	0.380±0.042	0.001±0.000	0.004±0.005	36.725±0.983	2.060±0.339
W₂	5.350±0.071	0.095±0.007	BDL	BDL	89.240±1.131	1.440±0.085
C	6.750±0.212	0.035±0.000	0.004±0.000	0.007±0.008	0.640±0.028	0.900±0.000

Table 2: Total metal concentrations (mg/kg) of particulates Dust in Funtua Textile Limited

Sites	Cd	Cr	Cu	Ni	Pb
G	0.03±0.02	0.59±0.08	0.95±0.45	0.42±0.07	0.42±0.06
E₁	0.15±0.17	174.30±9.79	356.68±33.57	3.44±0.12	3.97±0.11
E₂	0.05±0.01	8.68±5.99	29.19±15.63	1.00±0.39	5.09±0.06
O₁	0.02±0.00	0.05±0.07	0.18±0.08	0.09±0.04	0.41±0.06
O₂	0.05±0.04	1.26±0.60	1.70±0.81	1.00±0.36	1.03±0.09
S₁	0.02±0.00	0.12±0.01	0.58±0.01	0.25±0.01	0.38±0.06
S₂	0.04±0.01	4.12±5.05	0.27±0.02	2.28±2.58	0.14±0.04
D	0.04±0.00	0.19±0.09	1.45±0.31	0.37±0.09	0.59±0.12
W₁	0.03±0.00	0.20±0.14	1.55±0.23	0.41±0.14	0.73±0.14
W₂	0.08±0.03	0.00±0.00	0.55±0.13	0.35±0.02	0.24±0.01
C	0.03±0.00	0.84±0.27	0.30±0.18	0.22±0.09	0.43±0.18
Standard (WHO)	3.00	0.03	140	75	300

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878

Table 3: ANOVA of physicochemical parameters and heavy metal concentrations in dust particulates samples of Funtua Textile Limited

		Sum of Squares	Df	Mean Square	F	Sig.
	Between Groups	7.831	10	0.783	32.568	0.000
pH	Within Groups	0.265	11	0.024		
	Total	8.096	21			
	Between Groups	40.72	10	4.072	254.43	0.000
EC	Within Groups	0.176	11	0.016		
	Total	40.896	21			
	Between Groups	0.002	10	0	99.031	0.000
NH$_4$	Within Groups	0	11	0		
	Total	0.002	21			
	Between Groups	0	10	0	3.7	0.021
NO$_3$	Within Groups	0	11	0		
	Total	0	21			
	Between Groups	21246	10	2124.6	2624.33	0.000
OM	Within Groups	8.905	11	0.81		
	Total	21254.9	21			
	Between Groups		10		85976.2	0.000
P	Within Groups	130.408	11	11.855		
	Total		21			
	Between Groups	272.077	10	27.208	661.549	0.000
Cl	Within Groups	0.452	11	0.041		
	Total	272.53	21			
	Between Groups	0.029	10	0.003	0.988	0.504
Cd	Within Groups	0.032	11	0.003		
	Total	0.061	21			
	Between Groups	54362.9	10	5436.29	379.213	0.000
Cr	Within Groups	157.693	11	14.336		
	Total	54520.6	21			
	Between Groups		10	22802.6	182.76	0.000
Cu	Within Groups	1372.45	11	124.768		
	Total		21			
	Between Groups	22.041	10	2.204	3.464	0.027
Ni	Within Groups	6.998	11	0.636		
	Total	29.039	21			
	Between Groups	55.875	10	5.588	618.338	0.00
Pb	Within Groups	0.099	11	0.009		
	Total	55.975	21			

The concentrations of chromium obtained in this study were above the concentration range of $17.0 - 34.0\ mgkg^{-1}$ reported by Ochigbo (2011) and El–Sayed *et al.* (2010). Although chromium is a major part of automobile bodies, its high concentrations especially at site E_1 is not surprising because vehicular scraps, machine scraps, spent oil and most used machines parts are been deposited in this site. The levels of chromium investigated in some of the sites were generally lower than the set standards by the WHO $(3.00\ mgkg^{-1})$. Similarly the concentration of copper in the dust particulates are presented in Table 2. The highest concentration of Cu was recorded at the site $E_1(356.68 \pm 33.57\ mgkg^{-1})$ while the lowest concentration was recorded at the site $0_1(0.18 \pm 0.08\ mgkg^{-1})$, these values obtained from this study were mostly below the values that are reported in a similar studies by Uba *et al.* (2008). Generally, the highest concentration of Cu in site E_1 could be attributed to metals ($CuCl_2$; $CuSo_4$) present at the Engineering Department which is basically a scraps dump site.

The concentrations of Cu in the dust samples were generally within the worldwide value of $140\ mgkg^{-1}$ in dust. For Nickel (Ni), the values in the dust samples were found to be within the range of $0.09 - 3.44\ mgkg^{-1}$. The highest mean value of Ni was found in the sample from site $E_1(3.44 \pm 0.12)$ while the lowest in site $0_1(0.09 \pm 0.04)$, the mean concentration of Ni was lower than the world-wide value $(50 - 100\ mgkg^{-1})$ (Fergussion and Kim, 1991). The values

obtained in this study were below those reported in a similar study by Ochigbo, 2011. Nickel pollution on a local scale is caused by emission from machines and generator engines that use nickel gasoline and by the abrasion and corrosion of Ni (Al-Shayed and Seaward, 2001). The factory dust lead level ranged from a low concentration of $0.14 \pm 0.04\ mg/kg$ at the spinning section (S2) as compared to high concentrations of $5.09 \pm 0.06\ mgkg^{-1}$ at power section of the textile (siteE_2).

The high concentrations of Pb recorded in the dust particulates was attested to the use of leaded fossil fuel, which consequently settles on the dust this could have a devastating effect on the health of the workers due to bioaccumulation over time. The concentrations obtained in this study were below the concentration range of $20.50 - 120.00\ mgkg^{-1}$ reported by Ochigbo (2011), Caselles *et al.* (2002) and Pouyat *et al.* (1995). However, the concentration of Pb recorded in this study was within the permissible limit of WHO/USEPA tolerable limit without an exception, the residents could be prone to lead poisoning due to bioaccumulation. Irrespective of sampling site, the distribution of total metals in the industrial particulate dust samples generally followed the order: Cu > Cr > Pb > Ni > Cd. Comparing the data of the control site to the studied sites, lower concentrations of metals were obtained at the control site. There were significant differences at P<0.05 for the levels of physicochemical parameters and heavy metal pollution across the sites.

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879

Conclusion
The industrial dust collected from Funtua textile limited shows high concentrations of heavy metals which could lead to serious environmental hazards. Correlation analysis indicates common source of metals in different sitesat p $\leq$ 0.05. This study indicated that industrial particulates dust pollution due to Cu, Cr and Pb may possess serious health risks to the residents in this rapidly developing industry.

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Conflict of Interest
Authors declare that there are no conflicts of interest.

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880

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881