



Dry deposition of heavy metals associated with free fall atmospheric dust and its characterization in an industrial city Kota (India) under meteorological influence

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Abstract

Free fall atmospheric dust samples have been collected during months of winter and summer in the period starting from February, 2011 to January, 2013 in Kota City, Rajasthan, India. The analytical results show that heavy metals occurrence in free fall atmospheric dust is found to be highest in fractions strongly bound to organic matter followed by weakly bound (exchangeable, carbonate bound). On the basis of the results of the elemental composition and morphology through scanning electron microscope (SEM) and widely dispersive X-ray fluorescence (WD-XRF), particles have been categorised into two groups: crustal and anthropogenic particles indicating the influence of fly ash emitted from Kota Super Thermal Power Plant (KSTPS) and other industrial activities. Simultaneously meteorological parameters were monitored to evaluate the influence of temperature, relative humidity and wind velocity. Mean concentrations of anthropogenic origin metals such as Cu, Cd, Zn and Pb are observed more at low temperature, high relative humidity and low wind velocity and lower at high temperature, low relative humidity and high wind velocity. On the contrary, crustal origin metals (Fe, Ca and Mg) are found to have a reverse trend under these meteorological conditions. Results of deposition flux (Fd) showed an approximately exponential decay with distance from point source Kota Super Thermal Power Plant (KSTPS).

Keywords: Free fall atmospheric dust, Heavy metals, Scanning electron microscopy, Widely dispersive X-ray fluorescence, Meteorological conditions.

Introduction

Dust fall makes a significant contribution to soil, water and destruction of forest and buildings. The elements from geological sources usually exist in coarse-state while elements originated from anthropogenic activities are fine particles. Settable particles greater than 10µm are the main component of dust fall¹. Chemical studies of the dust indicate that it is primarily composed of natural earth's crust material besides significant amounts of pollutants such as aromatic compounds, biological materials and heavy metals. The elevated heavy metals pollution in ambient air results from industrial and urban activities, escalated traffic burden and excessive and uncontrolled use of fertilizers and pesticides in agriculture². The degree of heavy metal contamination is usually evaluated by the total concentrations of the metals associated with air particulates which pose threat to the environment. Nevertheless, the mobility of metals in the environment depends mainly on the bonding and forms present in the solid phase in which they are found besides their total concentration^{3,4}. These forms can be classified into soluble; exchangeable; carbonate bound; Fe and Mn oxide-bound; organic matter-bound and residual. An in-depth knowledge about the mode of occurrence of metals in air particulates is quite essential for the environmental assessment of this contamination.

The soluble (labile) fraction of the particle-bound metals is considered as the readily available fraction to environmental receptors thereby posing greater environmental risk in comparison to the organic matter-bound (resistant) fraction⁵. Information on the morphology, phase and elemental composition of individual particles is provided with the help of scanning electron microscopy (SEM) and widely dispersive X-ray fluorescence (WD-XRF) methods^{6,7} which give knowledge regarding their origin, atmospheric history besides reaction mechanisms during their formation thereby indicating their influence on atmosphere^{8,4}. The fate and behaviour of heavy metal associated with atmospheric particles are greatly influenced by meteorological parameters such as the temperature, humidity and wind speed⁹. The effect of these parameters on metal levels in ambient air can be understood by understanding their role in dispersion and transport of these metals from the source to the sampling site. After deposition and accumulation, the heavy metals enter the food chain causing harm to the health of all living beings. Earlier reports indicate pulmonary toxicity caused by trace metal contents of airborne particulate matter including the water-soluble fraction^{10,11}.

In Kota city, a major thermal power plant known as Kota Super Thermal Power Station (KSTPS) generates huge amount of fly ash, which is a homogeneous mixture of various metal oxides in

the atmosphere. Various industries (small and large scale) including multiple numbers of Kota stone factories further increase the heavy metal load in the atmosphere.

Therefore, the present study was performed with the main objectives: i. to determine the water soluble, acetic acid and nitric acid soluble part of free fall atmospheric dust in terms of crustal origin (Fe, Ca and Mg) and anthropogenic origin (Cu, Cd, Zn and Pb) metals at various sampling sites of entire study area; ii. to determine physical (size and morphology) and chemical (composition) features of these atmospheric samples by appropriate technique (SEM and WD-XRF) to have accurate information regarding their sources and formation; iii. to monitor the climate effect on the concentrations of these metals as a function of meteorological criteria such as temperature, relative humidity and wind speed; iv. to study the effect of increasing distance from the point source on the deposition flux of heavy metals through exponential regression model.

Materials and methods

Study area: Kota, the main industrial city of Rajasthan state in India, has an area of 527 km². Kota faces semi arid climate with temperature range 6⁰C in winter (January) to 47⁰C in summer (June).

With the help of GPS (Global Positioning System) 50 sampling sites were selected following some specifications given by ASTM D 5111 Standards¹². These specifications were: i. distance from point source KSTPS covering a radius of 12km; ii. the distance from barrier interfering in sampling (double of the altitude of barrier); and iii. logistics (security, access). Table-1 shows the location of all the sampling sites situated in selected zones with reference to the point source and other probable origin of metal under monitoring in the study area.

Sample collection and analysis: All free fall atmospheric dust samples (n = 50 samples x 16 months = 800 samples) were collected in months of both the studied season which are March - May and October (summer) and January- February and November - December (winter) during 2011-12 and 2012-13. In plastic trays of 1m² area situated at a height of 6meter above the roof surface, the samples were collected at the end of month. It should be noted that these samples represent only dry deposition collected during 30 days dry period (excluding any rain event). Following sampling, dry depositions were scraped off the trays according to the washing method used earlier¹³. The dried samples were subjected to digestion process for metal analysis after passing through the sieves with 300BSS (< 53µm).

As the mode of occurrence of metals in these samples depends on their total concentration along with various physico-chemical forms (soluble, exchangeable, carbonate bound, organic metal bound), for proper extractability of these different metal forms, the reagents employed were water, acetic acid and nitric acid. Here it may be noted that the digestion method applied for the

extraction of nitric acid soluble fraction was carried out separately.

Water Soluble Heavy Metal Extraction: This process is performed in beakers by 6 hours agitation of 2gm sieved free fall atmospheric dust with 25mL milli-Q water on a magnetic stirrer. The extract was centrifuged, filtered using Whatman filter paper no. 42 and filtrates are made up in a 50mL polyethylene bottle following acidification.

Acetic Acid Soluble Heavy Metal Extraction: Acetic acid (CH₃COOH) digestion to extract acetic acid (25% v/v) soluble has been carried out¹⁴.

Total Heavy Metal Digestion: Nitric acid (HNO₃) digestion for total metals extraction was carried out¹⁵.

Following digestion the concentrations of 6 selected metals (Fe, Zn, Cu, Cd, Mg and Pb) has been carried out using Direct Air – Acetylene Flame method (AAS-Shimadzu-6300). Flame Photometer (Systronics -128) method was used to determination of Ca metal. Internal standards, certified reference material and quality control blanks were used to monitor precision and accuracy of analysis and the digestion procedure.

Monitoring of meteorological parameters: The meteorological data during the measurement period (February, 2011 to January, 2013) were provided from the Automated Weather Station (model number: DCPAWS02) mounted at the Kota Aerodrome, India. Air sensors are fixed at the top of the roof of the administrative building of Kota Aerodrome. The wind speed, ambient temperature and humidity data were recorded hourly and averaged over the samplers' 24 hours operation time.

SEM Analysis: Scanning electron microscopy (SEM) gives us a clear insight regarding the origin (anthropogenic or the natural activities) and morphology of atmospheric particles. Analysis was performed at Sophisticated Analytical Instrument Facility (SAIF) at STIC, Kochi with the help of computer controlled field emission scanning electron microscope SEM (Jeol 6390LV) equipment at an accelerating voltage of 0.5kV - 30kV with for 4nm resolution and 300,000 magnification.

WD-XRF Analysis: WD-XRF analysis is used for study of composition of atmospheric chemical species. In this study, samples of free fall particulate matter were analyzed by WD-XRF at Sophisticated Analytical Instrument Facility (SAIF) at STIC, Punjab University, Chandigarh. Elemental composition of sample is analyzed using a S8 TIGER Bruker X-ray Fluorescence Spectrometer (WD-XRF).

Results and discussion

Metal analysis: The minimum, average and maximum concentrations (mg/L) of crustal (Ca, Mg and Fe) and anthropogenic (Pb, Zn, Cu and Cd) origin metals in water,

acetic acid and nitric acid soluble (total soluble) phase in free fall atmospheric dust collected (in 16 analysed months) at various sampling sites of Kota city during the selected months of summer and winter season of two sessions 2011-12 and 2012-13 are shown in Table-2.

The highest levels of heavy metals in nitric acid soluble phase justify the separate digestion method used for total extraction of the metals. Further it is evident that metal occurrence is higher in weakly organic matter bound (acetic acid soluble fraction) followed by water soluble phase¹⁶.

It is to be noted that the concentration levels (HNO_3 soluble phase) of crustal metals were found in higher concentrations in comparison to anthropogenic metals as they are abundant in nature being main structural ingredients of soil.

Crustal origin metals (Ca, Mg and Fe) originating mainly from local soil undergo resuspension or mobilization before getting incorporated in the local area. Ca metal concentration is found to be high originating in carbonates resulting from alkaline sources (soil and earth crust)¹⁷. Mg metal results from lime stone earth crust and mining activities carried out at study area while Fe is found to exist as oxides such as goethite, hematite, magnetite etc.

The anthropogenic metals viz. Cu, Cd, Zn and Pb, originate from industrial activities in the area and coal combustion at KSTPS. It is a well known fact that coal combustion processes of power plants foster emission of more volatile metals of lower melting point¹⁸. Being elements of lower melting point, Zn and Pb (relatively more volatile metals) get easily transferred in air and hence known as atmospheric species. Besides emissions from KSTPS, Zn particles in ambient air might have originated from automobiles i.e. lubricating oils, scraps from rubber tires and corrosion of vehicular parts. In our study, Zn concentration was found in highest levels followed by Pb, Cu and Cd respectively in total fraction (nitric acid soluble). This finding of ours is in accordance with the earlier studies showing similar reports¹⁹⁻²¹. Persistent existence of Pb in street

dust is probably from previous vehicular emission (prior to ban of Pb containing fuel) owing to its greater dwelling time in the atmosphere²² along with fly ash emission from KSTPS.

Characterization of Free Fall Atmospheric Dust: SEM Analysis: The SEM image of a representative sample of free fall atmospheric dust (FFAD₁) collected from site closest to KSTPS is shown in Figure-1.

The major particles present in free fall atmospheric dust are classified into 2 groups: crustal and anthropogenic which are further classified into various particles groups as displayed in Table-3.

The results indicate different groups of particles, showing diverse morphology of tubular structure to irregular shape, are both natural and anthropogenic in origin. However, most of the particles show the dominance of crustal matter along with the presence of many other particles existing in fly ash generated from KSTPS. The rod-like particle (as denoted by symbol h) consist of only elemental Fe suggesting its origin from metal wear coming from vehicle tailpipe, breaks etc.²³. The aluminosilicate group are major contributor of crustal class comprising highest percentage of total analyzed particles. This knowledge helped to identify the origin of the particle emission.

WD-XRF Analysis: Though large numbers of free fall atmospheric dust (FFAD) samples have been collected, due to certain limitations WD-XRF analysis of every sample is avoided. For this purpose, samples were collected from five sampling sites lying in selected zones viz. FFAD₁, FFAD₂, FFAD₃, FFAD₄ and FFAD₅ (distinct in external appearance and colour). The sites were selected on the basis of their location with respect to KSTPS.

WD-XRF shown in Table-4 reveal that all these samples contain high % of SiO_2 , CaO, Al_2O_3 , Fe_2O_3 , MgO as they occur in atmosphere as crustal substance as well as in fly ash emission from point source and other industrial activities.

Table-1: Location and features of different zones.

Zone	Situatedness with reference to the point source	Features
Zone-1 (n=10)	In 2 Kilometre radii surrounding point source	Coal dust (as raw material for coal based KSTPS); Fly ash (emitted from KSTPS); Soil dust; densely populated; High traffic density.
Zone-2 (n=10)	In range of 2-10 Kilometre towards North-east from point source	Fly ash (emitted from KSTPS favoured by north-east wind blow mainly during the study period); Soil dust; Traffic dust because of location of several public transport centres.
Zone-3 (n=10)	In range of 2-7 Kilometre towards East from point source	Fly ash (emitted from KSTPS); Soil dust; High traffic dust; Commercial activities (Multimetals Limited, Samtel Glass Limited); densely populated
Zone-4 (n=10)	In range of 2-12 Kilometre towards East-south from point source	Fly ash (emitted from KSTPS); Soil dust; High traffic dust; Presence of industries (DCM Shriram Consolidated Limited, Shriram Fertilizers and Metal India, Shriram Rayons); densely populated
Zone-5 (n=10)	In range of 2-8 Kilometre towards South from point source	Fly ash (emitted from KSTPS); Soil dust; Residential area; Stone mining activities

Table-2: The min., average and max. concentrations (mg/L) of crustal and anthropogenic origin metals in water, acetic acid and nitric acid soluble phase of free fall atmospheric dust during February, 2011 to January, 2013.

Metal	H ₂ O Soluble			AcOH Soluble			HNO ₃ Soluble		
	Min.	Average	Max.	Min.	Average	Max.	Min.	Average	Max.
Ca	56.26	72.34	107.72	528.09	978.78	1180.02	754.75	1822.62	2488.54
Mg	2.5417	3.6612	5.7283	4.2112	5.9993	7.8105	9.8627	13.5110	16.2230
Fe	0.4063	2.5622	7.1764	33.3856	49.8945	58.9998	62.9505	77.0343	89.3064
Pb	0.0248	0.3567	0.7113	0.2269	1.7488	2.6629	0.8447	2.3483	4.9888
Zn	0.0077	0.2565	0.7788	1.4468	2.4688	4.1256	2.9402	4.4888	6.8777
Cu	0.0064	0.0897	0.1070	0.0517	0.2008	0.7344	0.3708	0.7525	1.2647
Cd	0.0007	0.0079	0.0624	0.0071	0.0683	0.0971	0.0409	0.0970	0.1968

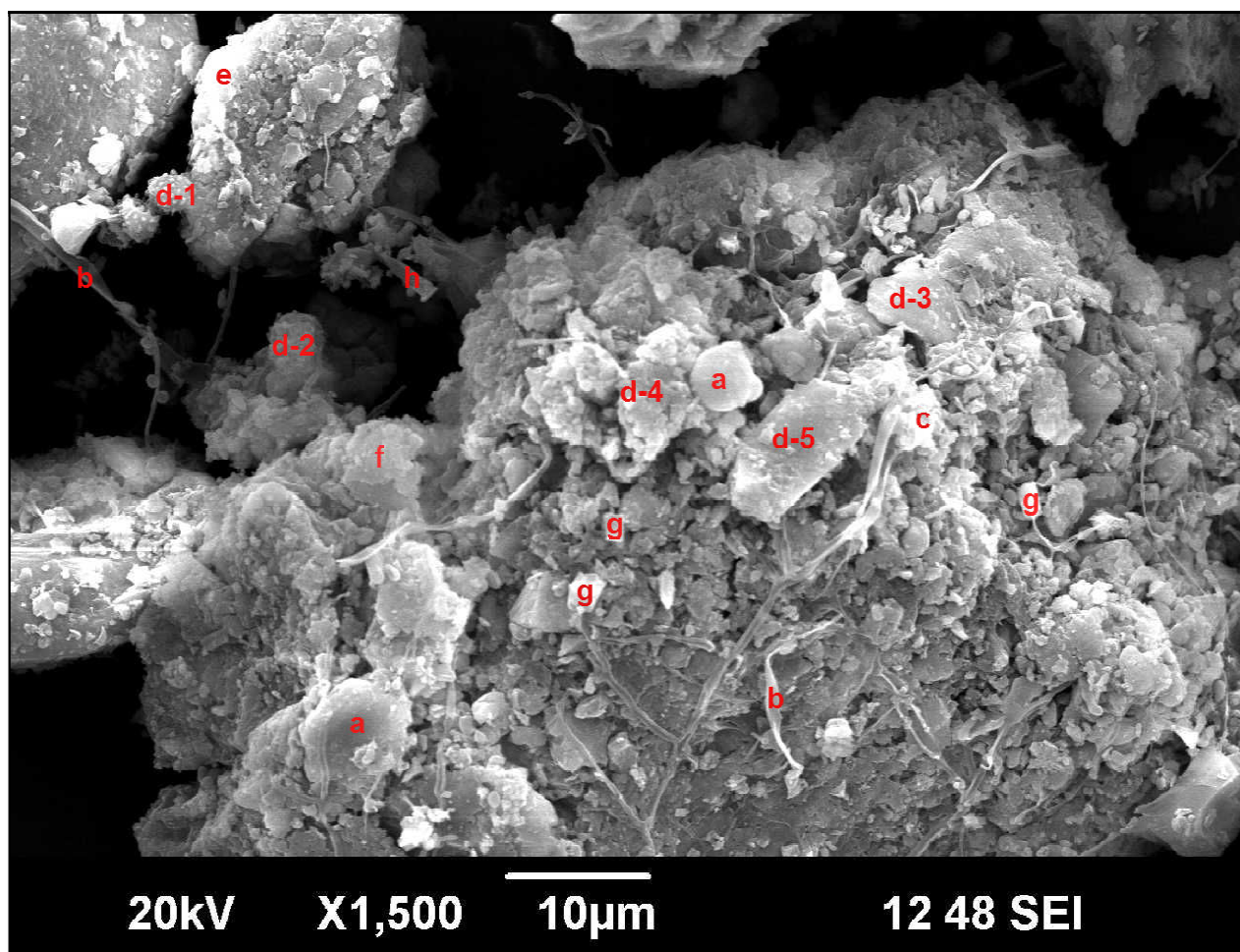


Figure-1: SEM micrograph of FFAD₁ at X1,500 magnification (a=Al-Si-O; b=Si-O; c=Ca-Al-Si-O; d-1=Na-Al-Si-O; d-2=K-Al-Si-O; d-3=Ca-Al-Si-O; d-4=Mg-Fe-Al-Si-O; d-5=Ca-Mg-Al-Si-O; e=Ca-Si-O; f=Fe-Ti-O; g=Cl-Na-K-Mg-Ca; h= Fe rich particle).

Table-3: Classification of particle groups and their major selection criteria.

Particle groups	Possible phase/minerals	Morphology	Symbols in Figure1
Quartz/ Silica	Fly ash/Quartz fibre	Spherical	a
	-	Tubular	b
	Grossular	Irregular	c
Alumino-silicates	Na-feldspar	Irregular	d-1
	K-feldspar	Irregular	d-2
	Ca-feldspar	Irregular	d-3
	Magnesium iron Aluminosilicate	Irregular	d-4
	Calcium magnesium aluminosilicate	Irregular	d-5
Calcium rich Particles	Wollastonite	Irregular	e
Fe/Ti oxide	Iron titanium oxide	Nearly spherical	f
Chloride particles	Chlorides of Na, K, Mg and Ca	Platy	g
Fe rich particle	Elemental Fe	Rod like	h

Other inorganic oxides (of heavy metals viz. Zn, Pb and Cu) present in low % could have originated in emissions from coal based Thermal Power Plant. The main difference in elemental mass composition of samples of free fall atmospheric dust collected from five sampling sites can be seen on the basis of their increasing distance from the point source and other anthropogenic pollution sources.

FFAD₄ and FFAD₅ samples contain more % of CaO as compared to other samples due to dominant crustal source being collected from the distant areas i.e. these sites characterised by mining activities over there mainly along with their far distance from KSTPS. Incidence of anthropogenic particles ZnO and CuO in FFAD₁, FFAD₂ and FFAD₃ samples were found to be higher in comparison to other samples indicating their incorporation from fly ash as these sampling sites were located close to the coal based Thermal Power Plant Station. The concentration of Pb was observed highest in FFAD₃ due to high traffic load of heavy duty vehicles at this sampling site.

Relationship between average metal concentrations in free fall atmospheric dust and meteorological parameters: To ascertain the effect of prevailing meteorological conditions on the dispersion and deposition of heavy metals in free fall atmospheric dust, relationship between monthly average metal

concentrations in free fall particulate matter and average values of temperature, relative humidity (RH) and wind velocity for the analysed 16 months of both measured years are shown in Figure-2 and 3.

The difference in concentration levels of crustal origin metals such as Ca, Fe and Mg and anthropogenic metal species i.e. Pb, Zn, Cu and Cd can be understood by difference in their response to meteorological conditions. The elevated concentration of crustal metal species was found in the temperature range 27⁰C - 35⁰C (Figure-2) while for anthropogenic metal, this range is observed to be 19⁰C - 30⁰C (Figure-3).

The high wind strength at higher temperature range and low relative humidity in summer months is responsible for erosion of earth crust resulting in increased levels of coarse crustal metals as coarse particles get settled down due to gravity. The same meteorological conditions justify the decreased concentration of fine anthropogenic metals as these fine particles are carried far away by powerful winds giving their low values.

On looking at the values of studied metal species at low temperature range and high RH observed in studied months of winter season, we found that anthropogenic metals get increased as stable and cold conditions at this temperature range encourage the long-lasting life of atmospheric particles in the environment resulting in their increased levels. On the other hand, decreased levels of crustal species can be explained as wind velocity required for inflation and erosion of earth crust drop significantly at same temperature range.

The effect of temperature, relative humidity and wind velocity clearly indicate the effect of prevailing meteorological conditions on the concentration of studied metal species though this behaviour may sometimes show diversions probably due to variable or inconsistent wind velocity. Further, intermittent or sporadic episodes of rain event could have caused change in meteorological conditions resulting in distractions from this regular trend.

Exponential Regression Model of Deposition Flux: Since the concentration levels of the analysed anthropogenic origin metals in free fall atmospheric dust samples are closely linked to deposition flux, these are then used to calculate the deposition flux of these metals. The deposition rates were calculated as mass deposited per unit area per unit time as equation²⁴.

$$\text{Deposition flux (Fd)} = \frac{m}{t \times A}$$

Where, m is the mass of the metal deposited (mg), A is the area (m²) and t is the period of sample collection. To get the deposition flux per day (mg m⁻²day⁻¹) a representative sample was taken from a month selected randomly. The mass is calculated by multiplying the concentration (mg L⁻¹) by the total volume (L) of samples measured during sample collection.

Average values of free fall atmospheric dust deposition flux (mg/m²/day) of anthropogenic metals are in the order of 0.43, 0.32, 0.18 and 0.08 for Cu; 0.60, 0.39, 0.31 and 0.24 for Zn; 0.54, 0.43, 0.28 and 0.06 for Pb at four sites lying in one direction with respect to the point source.

Deposition flux for the metals varied only slightly site wise because of location of these four sites which lay within few kilometres apart from each other on the same line of direction

with respect to KSTPS, hence, meteorological conditions remained almost same over these observed sites.

To statistically characterize the relationship between the average deposition flux of each heavy metal (Cu, Zn and Pb) at four sites and their progressive increasing distance from KSTPS, an exponential regression model was applied by plotting regression curves as shown in Figure-4 to 6.

Table-4: Chemical composition of free fall atmospheric dust collected from five sampling sites as determined by WD-XRF.

Metal	FFAD ₁	FFAD ₂	FFAD ₃	FFAD ₄	FFAD ₅
SiO ₂	57.74%	57.54%	58.33%	31.33%	30.54%
CaO	17.34%	13.19%	13.18%	47.42%	49.16%
Al ₂ O ₃	11.77%	12.48%	10.37%	9.00%	9.07%
Fe ₂ O ₃	4.96%	6.03%	5.40%	3.68%	4.08%
MgO	1.87%	2.72%	1.98%	2.21%	2.79%
K ₂ O	1.86%	2.49%	2.02%	1.54%	1.74%
SO ₃	1.75%	2.47%	2.26%	2.23%	2.41%
TiO ₂	0.95%	1.08%	0.85%	0.75%	0.80%
Na ₂ O	0.57%	1.00%	1.13%	0.50%	0.60%
P ₂ O ₅	0.51%	0.37%	0.64%	0.46%	0.39%
Cl	0.23%	0.18%	0.39%	0.57%	0.59%
BaO	0.10%	0.09%	0.07%	0.06%	0.06%
MnO	0.09%	0.12%	0.10%	0.09%	0.09%
SrO	0.07%	0.04%	0.04%	0.04%	0.04%
ZnO	0.05%	0.05%	3.08%	0.03%	0.03%
ZrO ₂	0.04%	0.05%	0.04%	0.02%	0.02%
CuO	0.03%	0.01%	0.02%	0.01%	0.01%
Cr ₂ O ₃	0.03%	0.02%	0.03%	0.02%	0.02%
PbO	0.01%	0.01%	0.04%	78 ppm	75 ppm
NiO	0.01%	93 ppm	0.01%	0.01%	0.01%
Rb ₂ O	83 ppm	0.01%	99 ppm	54 ppm	51 ppm
Y ₂ O ₃	43 ppm	40 ppm	36 ppm	-	-
Ag	42 ppm	-	-	-	-
Ga ₂ O ₃	26 ppm	-	9 ppm	-	-
Nb ₂ O ₅	17 ppm	19 ppm	21 ppm	-	-
Br	14 ppm	-	-	-	-
CoO	4 ppm	-	-	5 ppm	4 ppm
As ₂ O ₃	2 ppm	14 ppm	-	3 ppm	1 ppm
V ₂ O ₅	-	0.03%	-	0.03%	0.02%

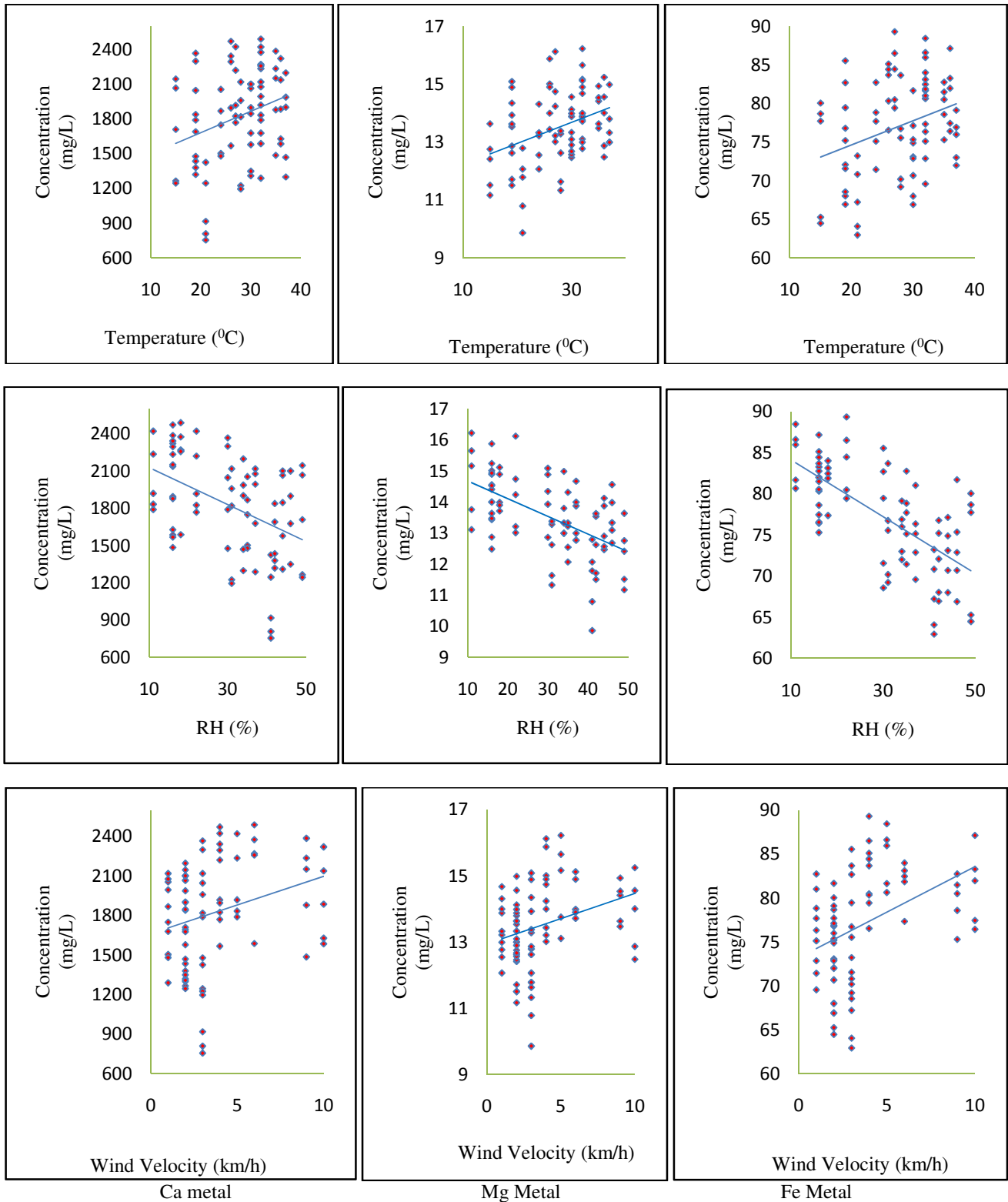


Figure-2: Relationship between average concentrations of Ca, Mg and Fe metals and average temperature, relative humidity and wind velocity in and around Kota city.

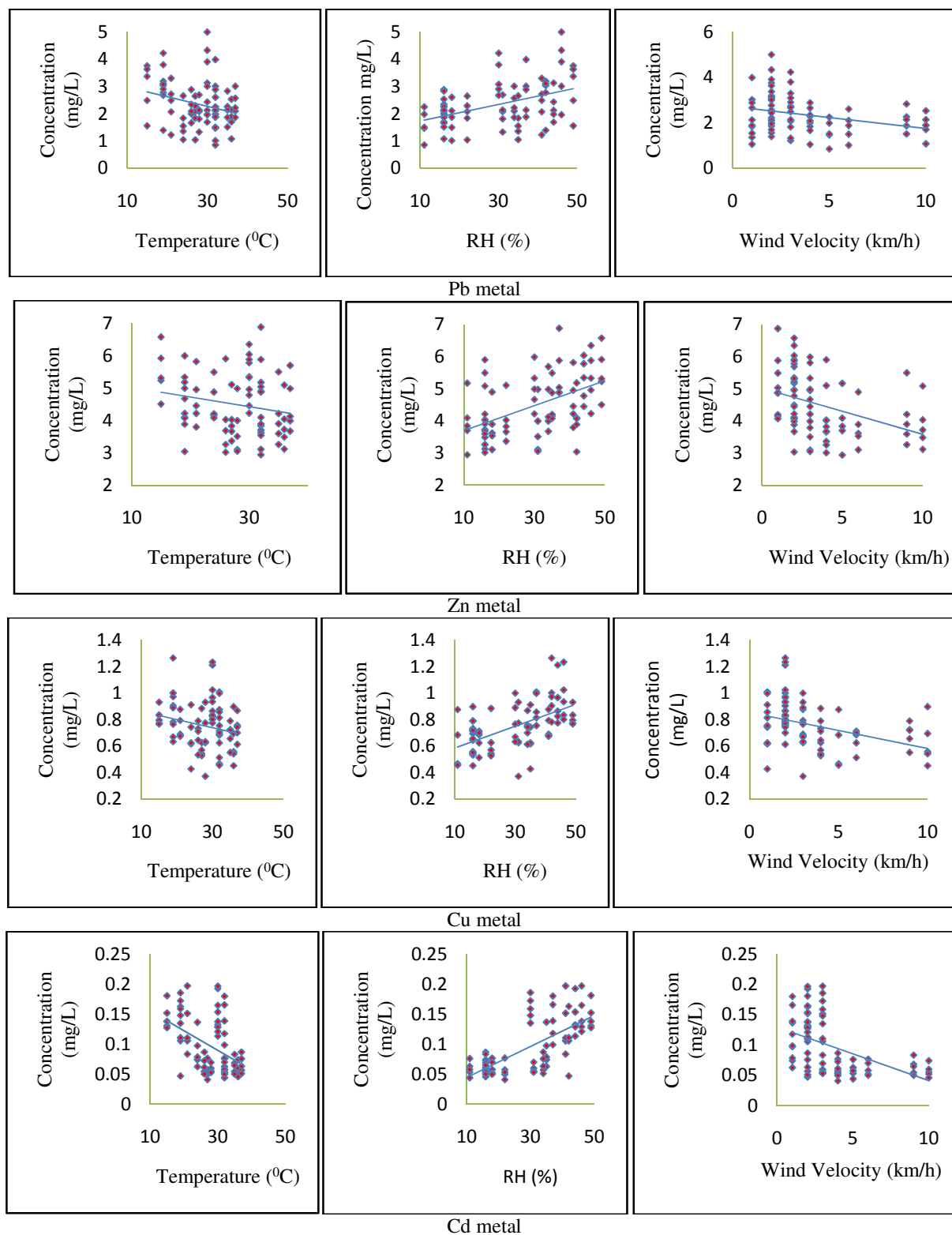


Figure-3: Relationship between average concentrations of Pb, Zn, Cu and Cd metals and average temperature, relative humidity and wind velocity in and around Kota city.

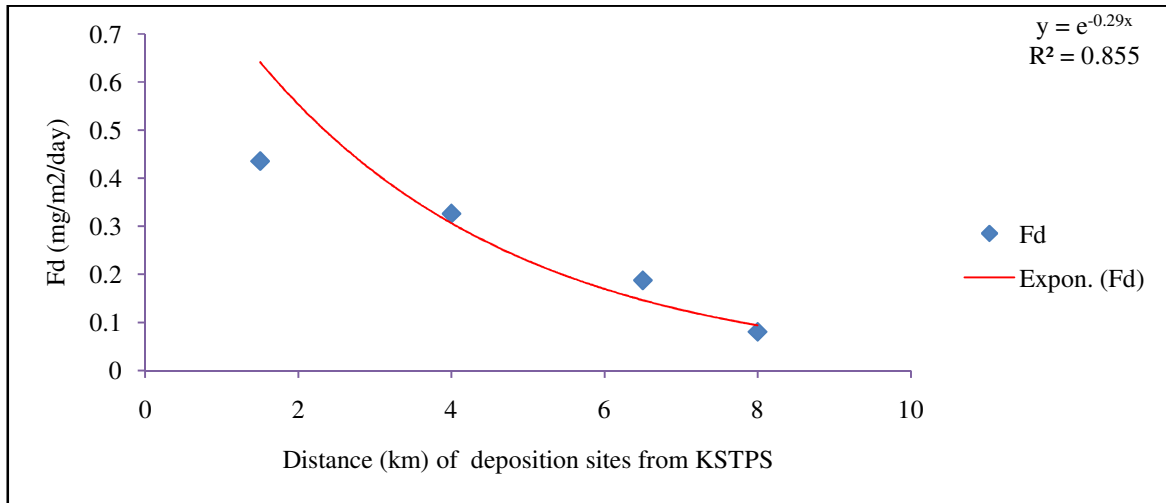


Figure-4: Deposition flux of Cu (mg/m²/day) with respect to distance of deposition sites from point source.

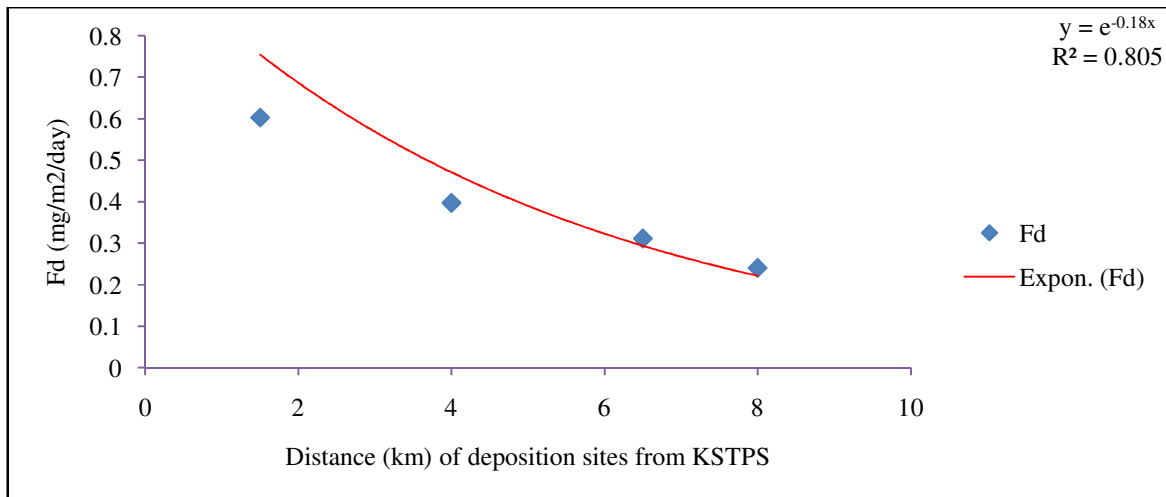


Figure-5: Deposition flux of Zn (mg/m²/day) with respect to distance of deposition sites from point source.

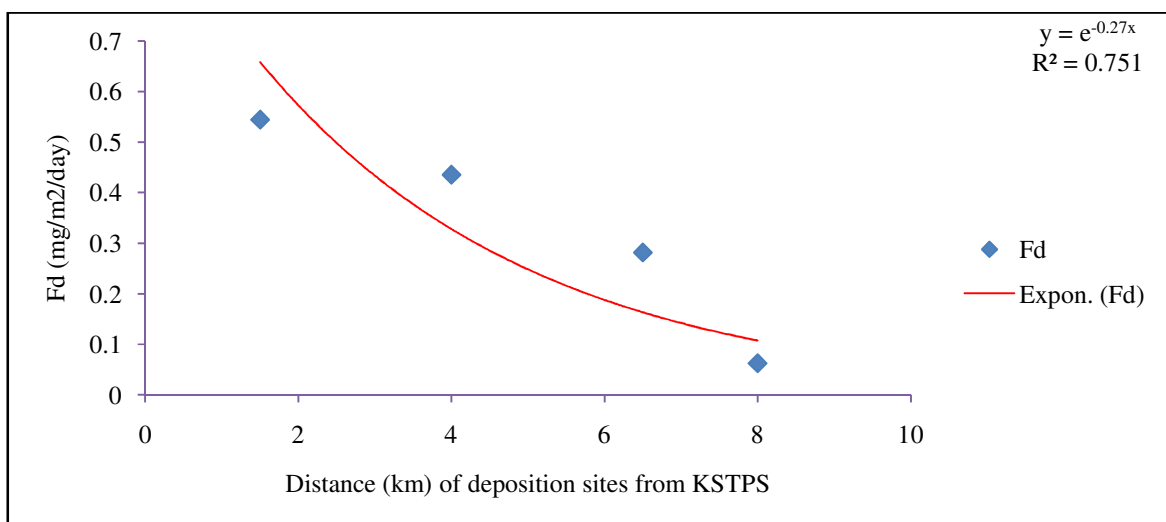


Figure-6: Deposition flux of Pb (mg/m²/day) with respect to distance of deposition sites from point source.

Above Figures indicate a decrease the deposition fluxes of lead, zinc and copper with increase in distance from the point source in an approximately exponential manner. Using a curve-fitting programme, curves of the form $y = a.e^{-kx}$ were applied to each set of data and r (goodness of fit) values are computed for each heavy metal (Table-5). Thus, deposition flux data of copper, zinc and lead depict the closest degree of fit to an exponential decay curve.

Table-5: Goodness for fit (r) values of exponential decay curves to deposition versus distance data (N=16).

Element	R	Significance*
Cu	0.855	$p < 0.001$
Zn	0.805	$p < 0.001$
Pb	0.751	$p < 0.001$

* $p < 0.001$ highly significant.

Conclusion

This research work provides current metal burden associated with dust fall in a rapidly growing industrial city. The average concentrations of seven metal species [heavy metals (Cu, Cd, Zn and Pb) and crustal metals (Fe, Ca and Mg)] in samples of free fall atmospheric dust, collected at selected sampling sites situated in and around Kota city during February, 2011 to January, 2013, was assessed under meteorological influence.

It is concluded that these metals were predominantly present in the residual phase obtained through nitric acid digestion used separately. Analysis result from other two digestion methods depict that metal occurrence is higher in weakly organic matter bound followed by water soluble part.

The morphological and elemental features of particulate matter were analysed through SEM and WD-XRF analysis. The SEM results indicate both crustal and anthropogenic origin for these various diverse groups of dust particles however most of them were crustal matter. Influence of KSTPS and other industrial activities can be observed from the results of WD-XRF which show higher concentration levels of analysed anthropogenic origin metals i.e. Cu, Zn and Pb in the free falling atmospheric dust collected from sampling sites adjacent to point source in comparison to the lower values in the samples from other sites located far away.

Meteorological conditions play a significant role in concentration levels, dispersion and transport of metal species in ambient air. Concentrations of anthropogenic metals were observed to be higher in low temperature, high relative humidity and low wind velocity and lower in high temperature, low relative humidity and high wind velocity while crustal metals were found to have reverse trend.

The correlation between the deposition flux of anthropogenic origin metals and distance of deposition sites from point source has been characterised in an exponential decrease distribution pattern which is useful in evaluating the risk posed by emission of fly ash from KSTPS.

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