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Original article

Magnetic properties of air suspended particles in thirty eight cities from south India

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ABSTRACT

Air pollution is a basic problem nowadays and it requires special concern. In India, the air pollution is a growing problem because of the enhanced anthropogenic activities such as burning fossil fuels involving industrial processes and motor vehicles. We study airborne dust particles collected at the height of 7 m in roadside and land area from thirty-eight cities in the state of Tamil Nadu. The collection involves a total of 111 samples concerning vehicular, industrial and residential areas, and allows us to assess the spatial distribution of magnetic particles produced and emitted on a short period of time (about one month). Magnetic properties of these air suspended particles were determined by techniques of environmental magnetism, revealing the presence of magnetite and hematite. We found the overall average of mass-specific magnetic susceptibility χ of 589.0×10^{-8} m^3 kg^{-1} and saturation of remanent magnetization SIRM of 68.1×10^{-3} A m^2 kg^{-1}; as well as χ and SIRM values higher than 900.0×10^{-8} m^3 kg^{-1} and 700.0×10^{-3} A m^2 kg^{-1}, respectively, corresponding to the most impacted zones in industrial/vehicular areas and in cities located in the central/eastern region respectively (e.g.: Hosur, Krishnagiri, Salem, Dharapuram, Ranipet, Ayanavaram, Cuddalore and Chidambaram). We analyzed the relationship between magnetic parameters, between areas and possible grouping of cities using multivariate statistical analysis. The SEM-EDS observations and grain size estimations reveal the presence of trace elements (Sb, Zn, Co, Ni, As and V) and fine particles (1–5 μm) that can be inhaled and therefore are dangerous to human health.

1. Introduction

In South and Southeast Asia, pollutants are highly emitted to the atmosphere due to growing economies, particularly India (World Bank, 2000). In India, pollution has become a great topic of debate at all levels and especially the air pollution because of the enhanced anthropogenic activities such as burning fossil fuels, i.e. natural gas, coal and oil-to power industrial processes and motor vehicles. Smoke from traditional household solid fuel combustion commonly contains a range of incomplete combustion products, including both fine and coarse particulate matter (PM) and a variety of organic air pollutants (Harrison, 2004).

Particulate matter is one of the most important factors contributing to air pollution. Despite the predominant contribution of vehicular traffic to PM levels in terms of mass, this is not the only emission source of PM present in the urban environment (e.g., industrial emissions, shipping contributions, residential heating systems, etc. Karagulian et al., 2015). The physical–chemical, morphological and dimensional determinations of the complex mixture of organic and inorganic particulates are one of the major aspects for their characterization and for identification of emission sources that contribute to particulate air concentrations (Contini et al., 2010). In fact particles have different shapes, sizes and chemical composition in relation to emission sources (Grassi et al.,

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2003; Pipal et al., 2011). Vehicular traffic is known to be one of the major, if not the largest, sources of atmospheric PM in the urban environment (Almeida et al., 2005; Amato et al., 2009; Gietl et al., 2010). Airborne traffic-related PM is emitted mainly by tailpipe exhaust from gasoline and diesel engines (exhaust emissions); wear from brake linings and tires and re-suspension of road dust (non-exhaust emissions) by moving vehicles (Lawrence et al., 2013; Rogge et al., 1993). The small size PM particles can penetrate the deepest parts of the lungs and the smallest ones even the alveoli, the gas exchange cavities of the lungs. Exposure to traffic-derived PM poses adverse effects on human health and increases the risk of respiratory illness, cardiovascular diseases, and asthma (Brauer et al., 2002), resulting in increased mortality (Nel, 2005). PM exposure may also create oxidative stress and inflammation in the brain directly after the entry of the particles into the brain, or indirectly via the stimulation of peripheral/systemic inflammation (Block and Calderón-Garcidueñas, 2009; Genc et al., 2012; Mills et al., 2009).

Since the 1980's, magnetic measurements have been done in order to understand natural and anthropogenic processes occurring in different ecosystems (Evans and Heller, 2003). Thompson and Oldfield (1986) reported pioneering research on magnetic properties, postulating a linkage between iron oxides and heavy metals. According to Petrovský and Elwood (1999), atmospheric pollution is identified as one of the most harmful factors for ecosystems. Often, industrial and urban fly ashes included toxic elements and heavy metals; such airborne pollutants are diffused due to atmospheric circulation. The connection for particles derived from anthropogenic emissions may be due to either the incorporation of heavy metals into the lattice structure of Fe-rich particles and/or the adsorption of heavy metals onto the surface of such particles. Although magnetic methods only identify iron oxides, the relationship between magnetic minerals and toxic elements such as heavy metals allows studying pollution of inorganic compounds through Environmental Magnetism methods. Therefore, magnetic parameters may be suitable proxies of pollution and a number of authors have proved their usefulness for mapping anthropogenic pollution in different environments, including soils, sediments and biological species (e.g., Blundell et al., 2009; Chaparro et al., 2007, 2008, 2012, 2013; Desenfant et al., 2004; Fabian et al., 2011; Flanders, 1994; Hunt et al., 1984; Jordanova et al., 2014; Kapicka et al., 1999; Kim et al., 2007; Knab et al., 2006; Lehndorff et al., 2006,; Magiera et al., 2002; Petrovský et al., 1998, 2013; Sandeep et al., 2011; Zhang et al., 2011).

Magnetic monitoring constitutes a complementary or an alternative to traditional PM monitoring methods. It is based, as discussed, on the fact of atmospheric particulate matter contains Fe-oxides (ferrimagnetic and antiferromagnetic minerals). A limited number of investigations have focused on the magnetic studies in atmospheric particulate matter, among others, Castañeda-Miranda et al. (2014); Górka-Kostrubiec et al. (2012); Maher et al. (2013); Muxworthy et al. (2003); Petrovský et al. (2013); Sagnotti et al. (2006); Salo et al. (2012); Shu et al. (2001).

As mentioned, India has experienced serious air pollution problems due to its rapid economic growth and urbanization in the past decade. It is regarded as a major source region of the Indo-Asian haze, due to significant industrial emissions, coal burning, vehicular exhaust emission, and waste incineration (Lelieveld et al., 2001). At a regional scale in the study area, Senthil Kumar and Rajkumar (2014) reported the mineralogical compositions existing in air dust particles in various districts of Tamil Nadu, India. They report the presence of different minerals in the air that causes great concern regarding public health issues.

In this contribution, continuing the previous studies, we focus on the magnetic properties of Fe-oxides present in particulate matter collected with passive samplers located in residential, vehicular and industrial areas from 38 cities (Tamil Nadu, India). This study aims at (a) the characterization of magnetic particles present in suspended PM; (b) the identification of relevant magnetic parameters as potential pollution indicators using multivariate statistical methods; (c) the identification of more impacted areas.

2. Materials and methods

2.1. Sampling

The air suspended particles were collected in containers on shiny wrapping tissue papers (A4 size), located at the height of 7 m in roadside and land area. The deposition time of collecting samples was 20–25 days, and this work was carried out on summer season (February to May 2011) when the weather conditions were normal cloudy days. These tissue papers were washed in distilled water after collection in order to remove and collect deposited particles completely from the tissue papers, following a proposed methodology from Ramasamy and Ponnusamy (2009). In this way, around 20 g of deposit particles were obtained. The collected dust particles were then dried and used for analysis. The samples collected from 38 towns and cities in Tamil Nadu (South India) were labeled as 1 to 111. The samples were mainly collected from vehicular (i.e., bus stand, main traffic areas, main junctions, etc., V), industrial (I) and residential (R) areas which cover almost all major districts of Tamil Nadu. Sample areas are visualized in Fig. 1 and are listed out with their latitude and longitude in Table 1.

Each sample was sub sampled for magnetic studies using plastic containers (2.3 cm^3). Dry samples were packed, weighted (about 1 g), and labeled. After that, all samples were fixed using sodium silicate to prevent unwanted movements in studies of remanence magnetization. Each sample was impregnated and saturated with a sodium silicate (diluted at 5%wt) solution, and dried at room temperature until its consolidation.

2.2. Magnetic measurements

Magnetic measurements were carried out in the laboratory of the CIFICEN (Tandil, Argentina). Parameters related to magnetic concentration, mineralogy (antiferromagnetic and ferrimagnetic minerals) and grain size were calculated from these measurements.

Magnetic susceptibility measurements were performed using a magnetic susceptibility meter MS2, Bartington Instruments Ltd., linked to MS2B dual frequency sensor (0.47 and 4.7 KHz). The volumetric susceptibility (κ), $\kappa_{FD}\%$ frequency-dependence ($\kappa_{FD}\% = 100 * [\kappa_{0.47} - \kappa_{4.7}]/\kappa_{0.47}$) and mass-specific magnetic susceptibility (χ) were computed. The mass-specific susceptibility values were corrected by sample mass, assuming a standard volume of 10 cm^3 (Bartington, 1994). The χ is, perhaps, the best and the most used parameter for assessing magnetic concentration in environmental samples, assuming uniform mineralogy and consideration of paramagnetic and diamagnetic components (Peters and Dekkers, 2003). The $\kappa_{FD}\%$ parameter is sensible to the presence of superparamagnetic (SP) grains. According to Bartington Instruments Ltd (1994); values of $\kappa_{FD}\%$ <2.0% indicate virtually no SP grains; between 2.0 and 10.0% indicate admixture of SP and coarser grains.

The anhysteretic remanent magnetization (ARM) was imparted using a device attached to a shielded demagnetizer Molspin Ltd., superimposing a DC bias field of 50 and 90 μT to a peak alternating field (AF) of 100 mT in the AF interval 100–2.5-mT, and an AF decay rate of 17 μT per cycle. The remanence magnetization was measured by a spinner fluxgate

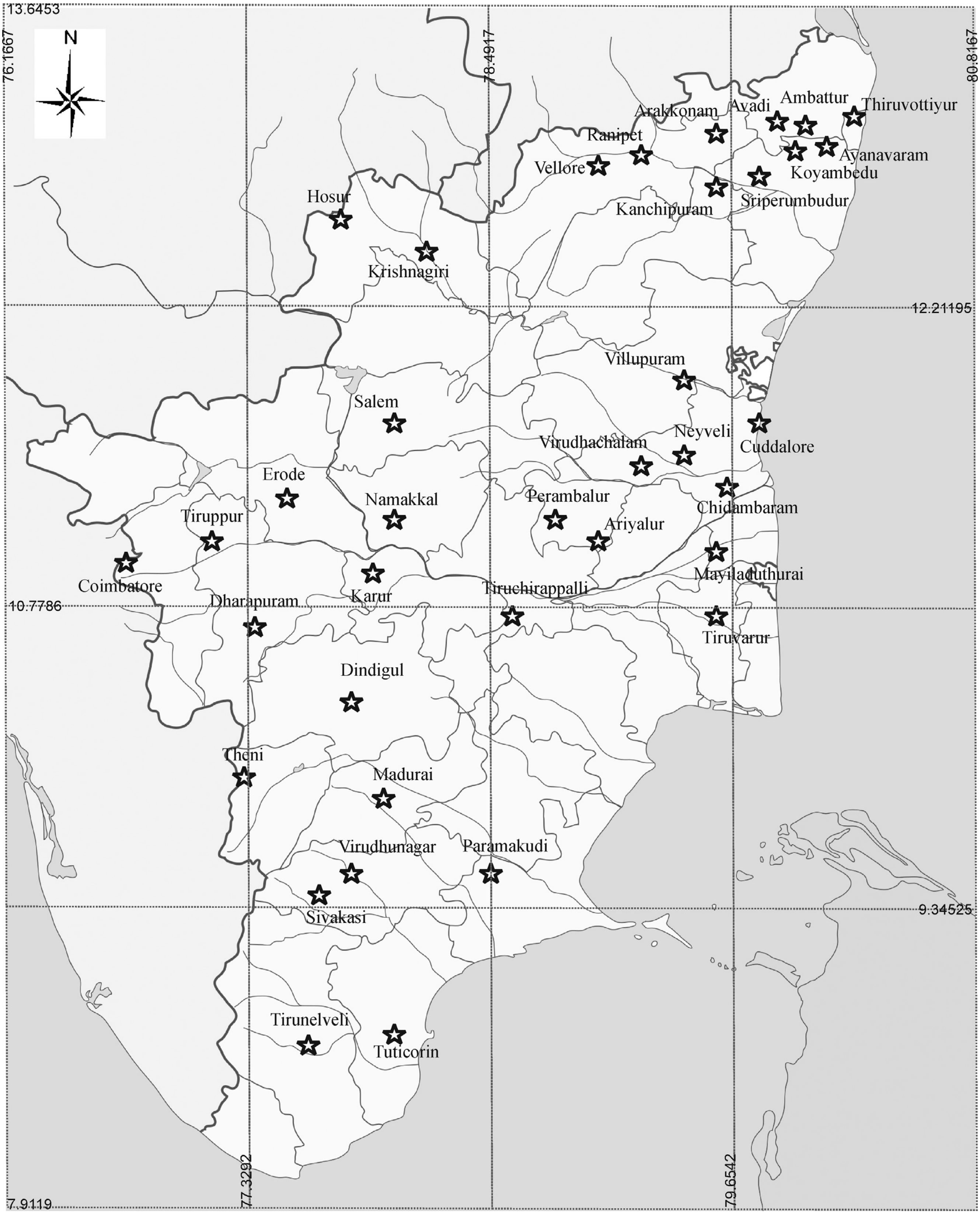


Fig. 1. Cities and collection sites in Tamil Nadu state, South India. Air suspended particles were collected in three areas (residential, vehicular and industrial) from each city.

Table 1
Summary of sample sites and each sample label.

City/Town	Latitude (°)	Longitude (°)	Population	District	Sample numbers		
					Vehicular	Residential	Industrial
Kanchipuram	N 12,83	E 79,70	497,000	Kanchipuram	1	2	3
Arakkonam	N 13,08	E 79,67	508,000	Vellore		4,5,6	
Villupuram	N 11,94	E 79,50	715,000	Villupuram	7	8	9
Cuddalore	N 11,75	E 79,75	426,000	Cuddalore	10	11,12	13
Ranipet	N 12,93	E 79,33	262,000	Vellore	14	15,16	17
Krishnagiri	N 12,52	E 78,21	600,000	Krishnagiri	18	19	20
Vellore	N 12,92	E 79,13	686,000	Vellore		21	22
Hosur	N 12,73	E 77,83	540,000	Krishnagiri	23	24,25	26
Dharmapuri	N 12,13	E 78,16	440,000	Dharmapuri	27	28	29
Salem	N 11,66	E 78,15	1,274,000	Salem	30	31	32
Namakkal	N 11,22	E 78,17	540,000	Namakkal	33	34	35
Erode	N 11,34	E 77,73	820,000	Erode	36	37	38
Tiruppur	N 11,11	E 77,35	980,000	Tiruppur	39	40,41	42
Coimbatore	N 11,02	E 76,96	2,250,000	Coimbatore	43	44	45
Dharapuram	N 10,74	E 77,53	283,000	Tiruppur	46	47	48
Karur	N 10,96	E 78,08	450,000	Karur	49	50	51
Tiruchirappalli	N 10,79	E 78,70	850,000	Tiruchirappalli	52	53	
Ariyalur	N 11,14	E 79,08	250,000	Ariyalur	54	55	56
Neyveli	N 11,53	E 79,48	105,000	Cuddalore	57	58,59	60
Perambalur	N 11,23	E 78,88	162,000	Perambalur	61	62,63	64
Dindigul	N 10,37	E 77,98	643,000	Dindigul	65	66	67
Theni	N 10,01	E 77,48	200,000	Theni	68	69	
Madurai	N 9,93	E 78,12	1,800,000	Madurai	70,71	72	73
Virudhunagar	N 9,58	E 77,96	250,000	Virudhunagar	74	75	76
Paramakudi	N 9,55	E 78,59	265,000	Ramanathapuram	77		78
Sivakasi	N 9,45	E 77,80	427,000	Virudhunagar	79	80,81	103
Tuticorin	N 8,76	E 78,13	480,000	Tuticorin	82	83,84	85
Tirunelveli	N 8,73	E 77,70	643,000	Tirunelveli	86	87,88	89
Chidambaram	N 11,40	E 79,70	469,000	Cuddalore	90	91	92
Virudhachalam	N 11,51	E 79,33	250,000	Cuddalore	93		94
Tiruvarur	N 10,77	E 79,64	152,000	Thiruvarur	95		96
Mayiladuthurai	N 11,10	E 79,65	259,000	Nagapattinam	97		98
Thiruvottiyur	N 13,16	E 80,30	250,000	Thiruvallur	99	100	101
Avadi	N 13,12	E 80,10	230,000	Thiruvallur	102		104
Sriperumbudur	N 12,97	E 79,94	427,000	Kanchipuram		1,05,106	
Ambattur	N 13,10	E 80,16	230,000	Thiruvallur			107
Ayanavaram	N 13,10	E 80,23	112,000	Chennai	108	110	109
Koyambedu	N 13,07	E 80,19	65,000	Chennai	111		

magnetometer Minispin, Molspin Ltd. The ARM (and χ_{ARM}) is a magnetic concentration dependent parameter, only sensitive to concentration of ferromagnetic minerals; however, ARM is also grain size dependent, varying one order of magnitude greater in fine magnetite of 0.1 μm than in magnetite of 1 μm (Dunlop and Özdemir, 1997). Anhysteretic susceptibility (κ_{ARM}) was estimated using linear regression for ARM acquired at different DC bias fields, i.e.: $ARM_{50\mu T}$ and $ARM_{90\mu T}$. Related parameters, such as, χ_{ARM}, King's plot (χ_{ARM} versus χ, King et al., 1982) and the κ_{ARM}/κ-ratio were also calculated. In particular, the κ_{ARM}/κ-ratio is a magnetic grain size sensitive parameter (Peters and Dekkers, 2003), for example, values of this parameter greater than 5 are indicative of the presence of very small magnetite grains.

The isothermal remanent magnetization acquisition (IRM) studies were carried out by using a pulse magnetizer model IM-10-30 ASC Scientific. Each sample was magnetized by exposing it to grow stepwise DC fields, 27 forward steps from 1.7 mT to 2470 mT. The remanent magnetization after each step was measured using the above-mentioned magnetometer Minispin. In these measurements, IRM acquisition curves and SIRM (IRM@2470 mT) were found using forward DC fields. The SIRM is a magnetic concentration dependent parameter, only sensitive to concentration of ferromagnetic minerals. SIRM values vary for antiferromagnetic and ferrimagnetic materials. Remanent acquisition coercivity ($H_{1/2}$, the field required to reach the SIRM/2), remanent coercivity (H_{CR}, the backfield required to remove the SIRM, or IRM = 0) and S-ratio ($= -IRM_{-300}/SIRM$, where IRM_{-300} is the acquired IRM@-300 mT) were also calculated from IRM measurement. In addition, the numerical method IRM-CLG (Kruiver et al., 2001) was performed in order to get a component analysis on IRM acquisition curves. The S-ratio is a dimensionless parameter that indicates content of ferrimagnetic (e.g. magnetite, titanomagnetite or maghemite) versus antiferromagnetic materials (e.g. hematite or goethite). Values close to +1 correspond to the predominance of ferrimagnetic minerals; on the other hand, values close to −1 correspond to antiferromagnetic minerals. H_{CR} values from 8.0 to 69.5 mT are characteristic of (titano)magnetite minerals, on the other hand, values of antiferromagnetic minerals such as hematite are above 100 mT (up to 900 mT) (Peters and Dekkers, 2003).

2.3. Microscopy

Particles were examined by scanning electron microscopy (SEM), using a JEOL JSM-6100 microscope (Tokyo, Japan) in the Nanotechnology Research Center at the SRM University (Chennai, Tamil Nadu, India). It has a large specimen chamber that allows observation of the entire surface of a specimen up to 150 mm and a tilt of −5° to 90°. SEM facilitates the observation of very fine details with higher resolution (4.0 nm at 8 mm working distance) of powder materials and good focus over a wide range of specimen surface. The elemental composition was analyzed by X-ray energy dispersive spectroscopy (EDS). Samples were coated with an

Table 2
Univariate statistics and multiple comparison Test.

	χ [10^{-8}m^{3}kg-1]	ARM [10^{-6}Am^{2}kg-1]	SIRM [10^{-3} Am^{2}kg-1]	$χ_{ARM}$ [10^{-8}m^{3}kg-1]	KARMK [dimensionless]	H_{CR} [mT]	S-ratio [dimensionless]	SIRM/χ [kAm-1]	ARM/SIRM [dimensionless]
N	111	111	111	111	111	111	111	111	111
Mean	589.0	731.4	68.1	834.9	1.79	35.3		13.3	0.012
S.D.	998.9	571.0	74.4	689.9	0.64	5.22		3.4	0.006
Min-Max	14.2-9533.3	42.5-4517.5	3.1-700.0	57.3-5246.4	0.60-4.70	7.5-45.1	0.62-0.99	4.3-26.1	0.036

Multiple comparison test (Conover-Inman test) after Kruskal-Wallis (p-value:0.5)

	χ I	χ R	χ V	ARM I	ARM R	ARM V	SIRM I	SIRM R	SIRM V	$χ_{ARM}$ I	$χ_{ARM}$ R	$χ_{ARM}$ V	KARMK I	KARMK R	KARMK V	H_{CR} I	H_{CR} R	H_{CR} V	S-ratio I	S-ratio R	S-ratio V	SIRM/χ I	SIRM/χ R	SIRM/χ V	ARM/SIRM I	ARM/SIRM R	ARM/SIRM V
(median by group)	444.9	324.7	563.1	654.9	556.3	846.1	58.3	46.3	70.3	751.5	621.6	1060	1.7	1.9	1.5	35.9	36.8	34.3	0.91	0.92	0.94	12.8	13.2	11.7	0.011	0.013	0.011
I-R		TRUE			FALSE			FALSE			FALSE			TRUE			FALSE			FALSE			FALSE			TRUE	
I-V		TRUE			TRUE			FALSE			TRUE			FALSE			TRUE			TRUE			TRUE			FALSE	
R-V		TRUE			TRUE			TRUE			TRUE			TRUE			TRUE			TRUE			TRUE			TRUE	

ultra-thin (about 100–1000 Å) layer of electrically-conducting material like carbon, aluminum, gold, or Au–Pd alloy (Schatten and Pawley, 2007).

2.4. Statistical techniques

Descriptive statistics and correlation Pearson's coefficients for all variables and data were applied as a first step. A Kruskal–Wallis and Conover–Inman test (KW test and CIn test; Conover, 1999; Siegel and Castellan, 1988) were performed in order to analyze the differences between types of source pollution (industrial-vehicular-residential) for each magnetic parameter. The CIn test was applied when the KW test was significant (i.e., if at least one of groups is significant different from at least one of the others). It is an alternative method of multiple comparisons that helps to determine which groups are different with pairwise comparisons adjusted appropriately.

The relationship of all magnetic parameters was analyzed by principal component analysis (PCA) with matrix correlation. A non-hierarchical k-means clustering (CA) was performed in order to build clusters of cities/samples with similar magnetic features. Each of the clusters is characterized by magnetic variables. For each cluster and for each quantitative variable, the v-test (a test-value) is calculated as follows:

$$v - test = \frac{\overline{x_q} - \overline{x}}{\sqrt{\frac{s^2}{I_q}\left(\frac{I - I_q}{I - 1}\right)}} \sim N\left(\overline{x}, \frac{s}{\sqrt{I_q}}\sqrt{\frac{I - I_q}{I - 1}}\right)$$

where x_q is the average of variable X for the individuals of cluster q, x is the average of X for all individuals, and I_q is the number of individuals carrying the cluster q. This value is used to test the following null hypothesis: "*the average of X for category q is equal to the general average*". In order to eliminate the dimensions which are related to "noise" the coordinated of the rows on the principal components (obtained from PCA) were used to build the clusters. A very high percentage of the inertia (above 80%) should be retained by the components selected to obtain hierarchy stable and clearer. The main use of the hierarchy obtained is therefore to help in interpreting the results of the principal component method. Then when applying the cluster analysis, in order to analyze the relationship between the clusters and the source pollution (industrial-residential-vehicular), a Chi-squared Test of Independence (Fisher and van Belle, 1993) was performed. The statistical analyses was performed using the R free software (R version 3.1.3, 2015).

3. Results and discussion

Magnetic characterization (concentration, magneto-mineralogy and magnetic size grain) of 111 dust samples from Tamil Nadu State, India are reported. Three samples out of the total were identified as outliers and studied separately because they present anomalous magnetic values. Samples were analyzed taking into account the possible pollution contribution and hence the sampling sites categories, i.e., R, V and I, which are detailed in Table 1.

3.1. Magnetic mineralogy

The main magnetic mineral contributions were analyzed using different parameters and analysis. It is observed from H_{CR} values (Quartile 1 = 33.2 mT and Quartile 3 = 38.2 mT), that ferrimagnetic minerals (magnetite or titano-magnetite) dominate the overall magnetic signal; therefore they seem to be the main magnetic carriers in these dust samples. The H_{CR} tends to increase from

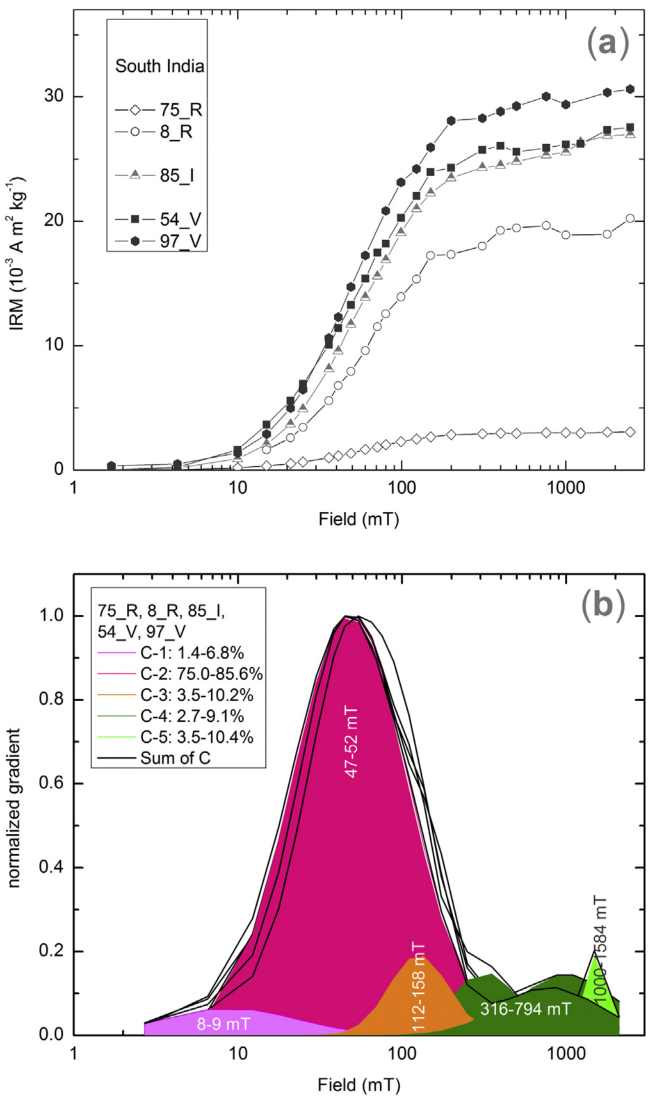


Fig. 2. Measurements of acquisition IRM for selected samples. (a) IRM and (b) gradient of IRM.

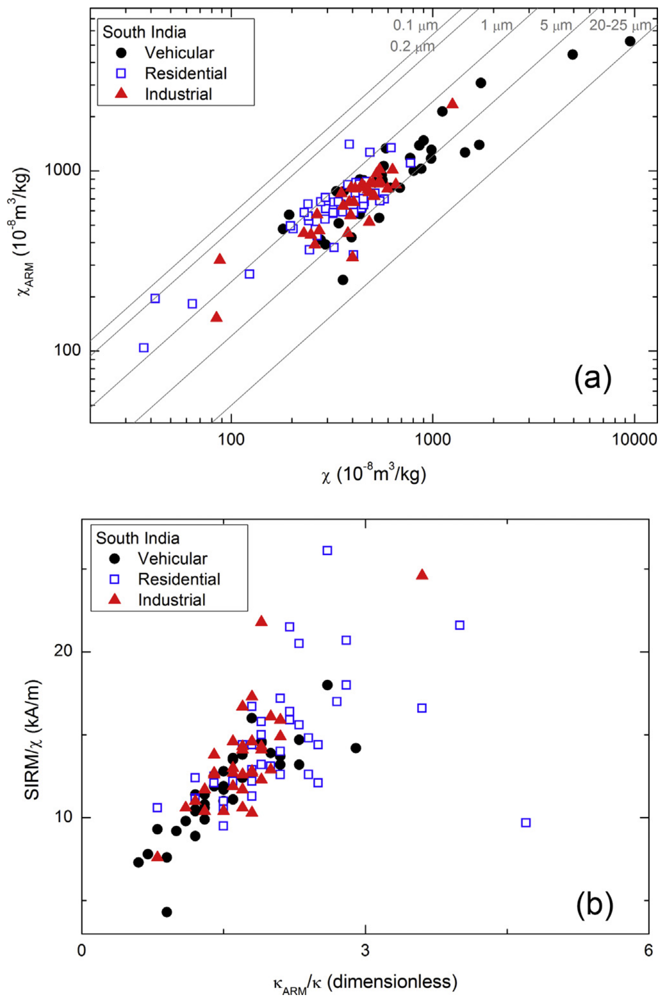


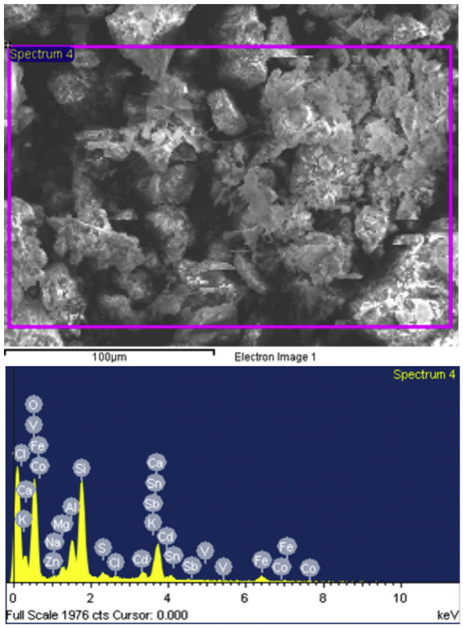
Fig. 3. (a) King's Plot (χ_{ARM} versus χ) for all samples. Most of the samples are between 1 and 5 μm. (b) Biplot of magnetic grain size dependent parameters (SIRM/χ versus κ_{ARM}/κ) for all samples.

vehicular (median = 34.3 mT) to industrial (median = 35.9 mT) and residential areas (median = 36.8 mT, Table 2), the S-ratio takes the opposite trend, decreasing from vehicular to industrial areas. Thus, the industrial and residential dusts seem to have a distinctive antiferromagnetic component.

The IRM studies revealed the dominance of ferrimagnetic minerals as observed in curves of acquisition IRM (Fig. 2a). Most of these curves reached 90% of their SIRM at field of about 300 mT; however, it is observed that these curves evidence an increase from this field, hence another magnetic phase corresponding to antiferromagnetic (high-coercivity) minerals seems to be present. The component analysis on IRM acquisition curves (Kruiver et al., 2001) yields the dominant magnetic mineral contribution, of magnetite (component C-2; contribution to the SIRM = 75.0–85.6%; $H_{1/2}$ = 47–52 mT; Fig. 2b) and other additional high-coercivity magnetic mineral contribution of hematite (component C-4; contribution to the SIRM = 2.7–9.1%; $H_{1/2}$ = 316–794 mT). Both magnetic phases correspond to ferrimagnetic and antiferromagnetic minerals (according the $H_{1/2}$ values reported by Peters and Dekkers, 2003), which were also reported by Senthil Kumar and Rajkumar (2014) using FTIR and XRD analyses. From FTIR analysis, the authors identified hematite in most of samples (110 samples) and magnetite in 60 samples.

Following the same analysis, in term of main contribution pollution areas and taking into account that magnetite like minerals dominate the magnetic signal, several variables and associated graph were used to identify the magnetic grain sizes. The κ_{FD}% mean values indicate a slight tend to increase from industrial (median = 2.12%) to vehicular area (median = 2.82%), however, these differences are statistically non-significant (at p = 0.68). According to κ_{FD}% range of values, all groups may reveal the presence of admixtures of SP (superparamagnetic) grains with larger PSD (pseudo-single domain) grains.

In general, for all samples, the King's plot (Fig. 3a) shows predominance of magnetic grains of 1–5 μm. These grains and finer were observed by SEM analysis as reported by Senthil Kumar and Rajkumar (2014). The magnetic grain distribution evidences no clear differences between industrial, vehicular and residential areas, and no cluster association between groups can be observed. According to Peters and Dekkers (2003), the magnetic parameters SIRM/χ, κ_{ARM}/κ and ARM/SIRM can be interpreted in term of magnetite grain sizes, these ratios show higher values for finer magnetic particles and SIRM/χ and κ_{ARM}/κ can be observed in Fig. 3b. In this study case, values of these parameters indicate the presence of finer particles in dust samples from



	Vehicular	Residential	Industrial	
	#82	#83	#73	#85
Element	Weight%	Weight%	Weight%	Weight%
O K	36.65	59.58	52.42	49.03
Na K	0.47	0.30	0.58	0.13
Mg K	1.30	1.66	2.83	2.01
Al K	6.77	4.93	5.02	6.17
Si K	21.45	13.80	23.47	17.76
S K	1.39	1.05	0.56	1.11
Cl K	0.91	0.54	0.42	0.52
K K	4.61	1.87	2.31	2.15
Ca K	13.10	10.48	3.61	11.00
Ti K	0.41		0.54	
V K	0.11	0.02		0.18
Fe K	9.58	3.05	6.43	5.20
Co K			0.37	0.35
Ni K	0.27			
Ge L		0.18		
Zn L		0.98	0.94	0.94
As L	0.26	0.03		
Mn K	0.04		0.15	
Pd L	0.58	0.23		
Sn L				1.23
Cd L	0.17	0.25		0.57
Sb L	1.91	0.96	0.34	1.65
Pb M		0.09		
Totals	100.00	100.00	100.00	100.00

Fig. 4. SEM image and corresponding EDS spectrum of the sample number 85 (industrial area). Elemental composition is also detailed for four samples corresponding to residential, industrial and vehicular areas.

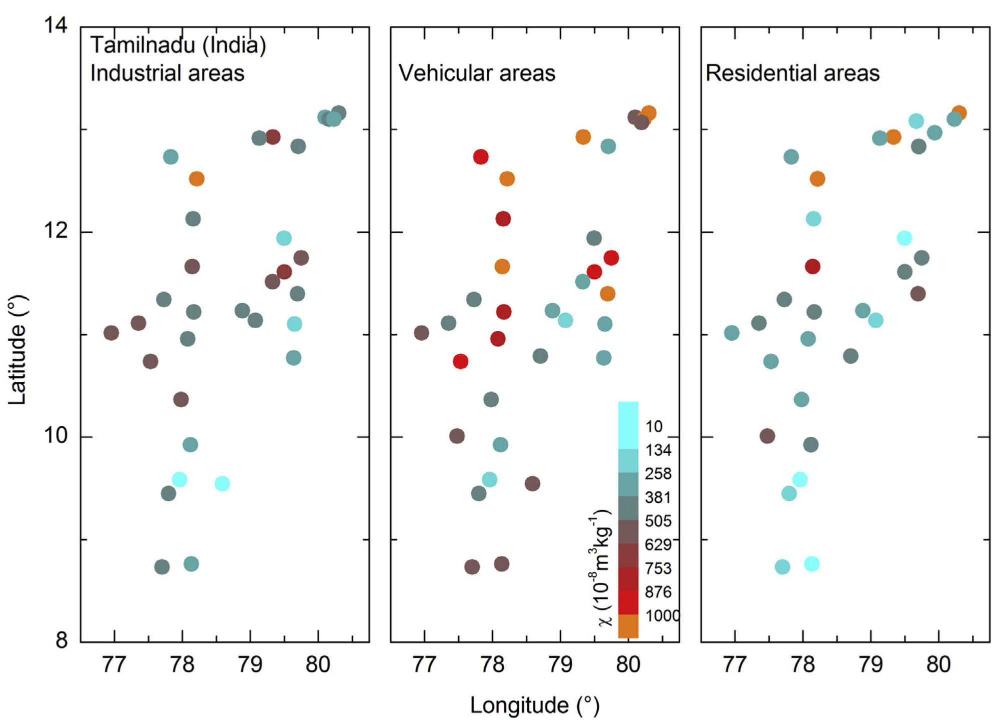


Fig. 5. Spatial distribution of χ (magnetic concentration-dependent parameter) for each area (residential, vehicular and industrial).

residential areas (median SIRM/χ = 13.2 kA/m, median κ_{ARM}/κ = 1.86 and median ARM/SIRM = 0.013) than from vehicular areas (median SIRM/χ = 11.7 kA/m, median κ_{ARM}/κ = 1.52 and median ARM/SIRM = 0.011), and the values are statistically different (Table 2).

Senthil Kumar and Rajkumar (2014) found irregular aggregates mostly, and seldom even nearly spherical particles; in general, particle shapes vary from compact and rounded to thin flakes, fibers, angular and aggregates. They also concluded that vehicular areas are characterized by more irregular shapes than industrial and residential areas. In this work, the composition analysis by EDS revealed the presence of the following elements: O, Si, Ca, Fe, Al, S and Mg, as well as contents of toxic elements: Sb, Pd, Zn, Co, Ni, As and V (Fig. 4), which can be a product of vehicular/industrial pollution (Chaparro et al., 2010; Kukier et al., 2003). The spherical shape is typical for particles originated from fast cooling of melts, or as results from combustion of fossil fuel (Flanders, 1994; Petrovský et al., 2013).

3.2. Magnetic concentration

The magnetic concentration dependent parameters (χ, ARM and SIRM) were analyzed for all samples and their different pollution loadings (residential, vehicular and industrial; see Table 2). Differences in χ between areas and sites can be appreciated in Fig. 5, showing that the most impacted areas are observed in industrial/vehicular areas and in the central (Hosur, Krishnagiri, Dharmapuri, Salem, Namakkal, Karur, Dindigul and Dharapuram), eastern (Ranipet, Ayanavaram, Thiruvottiyur, Avadi, Cuddalore, Chidambaram, Virudhachalam and Neyveli) and western (Tiruppur, Coimbatore and Dharapuram) geographical sites.

These results of concentration dependent parameters are similar to those reported on dust samples by Warrier et al. (2014) in the Karnataka state that is located at the western of Tamil Nadu state. The authors obtained χ values ranging between 220 and 2050 $\times$ 10^{-8} m^3 kg^{-1} on collected samples after one month and they attributed the high magnetic values to atmospheric pollution from heavy vehicular traffic and industrial activities. On the other hand, lower magnetic concentration values were reported on roadside dust samples from Visakhapatnam (located at the north-eastern of Tamil Nadu, Goddu et al., 2004) and on dust samples from the north-western of Tamil Nadu state (Gudadhe et al., 2012). Although both studies found lower χ values ranging between 13.9 and 101.4 $\times$ 10^{-8} m^3 kg^{-1} and 43.6 $\times$ 10^{-8} m^3 kg^{-1} respectively, they also identified pollution sources as vehicular traffic and industrial activities and polluted areas associated with higher magnetic concentration.

According to the variables χ, ARM and SIRM, the order of amount of magnetic concentration are vehicular areas > industrial areas > residential areas. The Kruskal–Wallis test was performed in order to compare the medians between groups. Hence, χ values have shown significant differences (at $p < 0.05$) between residential (324.7 $\times$ 10^{-8} m^3 kg^{-1}) and industrial areas (444.9 $\times$ 10^{-8} m^3 kg^{-1}) and between residential and vehicular areas (563.1 $\times$ 10^{-8} m^3 kg^{-1}, Table 2).

3.3. Extreme magnetic values

Samples 6, 30 and 99 presented anomalous magnetic values and correspond to the cities/towns: Arakkonam (residential area), Salem (vehicular area) and Thiruvottiyur (vehicular area). These anomalous values may be due to number of vehicles and small scale industries, which are high around the sampling sites. Furthermore, there is a narrow road and a main route for the industrial area in the sample 99. Although most of these samples show extreme values of concentration dependent magnetic parameters (e.g. χ values higher than 4930 $\times$ 10^{-8} m^3 kg^{-1}), they are also characterized by distinctive values of mineralogy dependent magnetic parameters, which may be interpreted as softer magnetic minerals with coarser grain size. Parameters H_{CR} and SIRM/χ show, in general, lower values (7.5–32.9 mT and 4.3–9.7 kA/m, Fig. 3b; respectively) than the corresponding overall medians (35.5 mT; 13.4 kA/m; respectively).

3.4. Multivariate analysis

The relevant magnetic parameters as potential pollution indicators were identified using biplots and multivariate statistical techniques.

The hypothesis of normality of Shapiro–Wilks test was rejected ($p < 0.000001$) for all magnetic parameters. Therefore, in order to analyze the differences between types of pollution source, two non-parametric tests (KW and CIn tests) were applied. The KW test revealed that most of magnetic parameters have significant differences ($p < 0.01$) between collection areas, i.e., industrial, vehicular and residential areas.

For each parameter, it was applied the CIn test and the results are summarized in Table 2. Such results showed a significant difference between residential and vehicular areas from χ, ARM and SIRM (magnetic concentration); however, ARM does not show differences between industrial and residential samples and SIRM for industrial-residential and industrial-vehicular areas. In spite of such differences, these three parameters ever show the same incremental behavior: residential < industrial < vehicular samples. The parameters H_{CR} and S-ratio (magnetic mineralogy) show no statistical differences between industrial and residential samples. The highest concentration of ferrimagnetic (lower H_{CR} and higher S-ratio) minerals in vehicular samples seems to produce these mineralogical differences. The magnetic grain size dependent parameters, i.e. κ_{ARM}/κ and SIRM/χ, show significant difference between residential and vehicular areas. Statistical differences are not observed between industrial and vehicular samples for κ_{ARM}/κ, and between industrial and residential samples for SIRM/χ (Table 2).

In order to estimate the dataset adequacy for the PCA, the value of Kaiser-Meyer-Olkin index (KMO, Pérez, 2004) was calculated. The KMO value of the dataset was 0.63 and it indicates that is appropriate to apply the PCA for the dataset. Therefore, PCA with correlation matrix was performed considering all magnetic parameters (χ, ARM, SIRM, κ_{FD}%, χ_{ARM}, κ_{ARM}/κ, H_{CR}, S-ratio, SIRM/χ and ARM/SIRM) and setting the main collection areas (R, I and V areas) as supplementary variables. Three samples (6, 30 and 99, see Table 1) were not used for multivariate analysis because they were identified as outliers (Sect. 3.3). The results and graphical representations are based on the 3 first principal components (PC1-2 and PC1-3) because 78.1% of the inertia (of dataset) was accumulated and explained by them showing a strong relationship. The correlation and loadings values of each parameter for PC1, PC2 and PC3 are shown in Table 3. The cloud of parameters (variables) is represented in Fig. 6. Concentration dependent parameters χ, SIRM, ARM and χ_{ARM} show high positive correlation and contribute to the construction the first PC (PC1). The magnetic grain size-dependent parameters (κ_{FD}% and κ_{ARM}/κ) contribute to the second component PC2 and mineralogy magnetic parameters (H_{CR}, S-ratio and ARM/SIRM) to the PC3. Notice that most of the correlation values with corresponding PCs are positive.

After this analysis, the CA was made to visualize possible city (samples) groupings. The CA was made with 4 principal components because they retain the 86.4% of inertia of data set. This value ensures a cluster hierarchy stable and clearer. The data set was partitioned into 3 groups made of 24, 72 and 12 samples, respectively (Fig. 7). Although each group is composed by samples corresponding to different areas, clusters C1, C2 and C3 are mainly made of samples from R, I and V areas, respectively. There is a significant statistical

Table 3
Correlations (and loadings) of magnetic parameters with each principal components.

Parameters	PC1	PC2	PC3
χ	**0.95 (20.19)**	−0.04 (0.06)	0.01 (0.01)
SIRM	**0.94 (19.72)**	0.27 (3.41)	−0.15 (1.81)
ARM	**0.92 (19.03)**	0.31 (4.52)	0.01 (0.01)
χ_{ARM}	**0.91 (18.64)**	0.34 (5.34)	0 (0)
κ_{ARM}/κ	−0.42 (3.89)	**0.86 (34.22)**	0.11 (1.13)
κ_{FD}%	−0.38 (3.20)	**0.54 (13.22)**	−0.01 (0.01)
ARM/SIRM	−0.57 (7.38)	0.32 (4.73)	**0.59 (29.40)**
SIRM/χ	−0.12 (0.35)	**0.86 (34.26)**	−0.32 (8.82)
H_{CR}	−0.45 (4.52)	−0.07 (0.25)	−0.42 (15.33)
S-ratio	0.37 (3.08)	0 (0)	**0.71 (43.49)**

Values in bold correspond to the best association obtained between each parameter and the PCs.

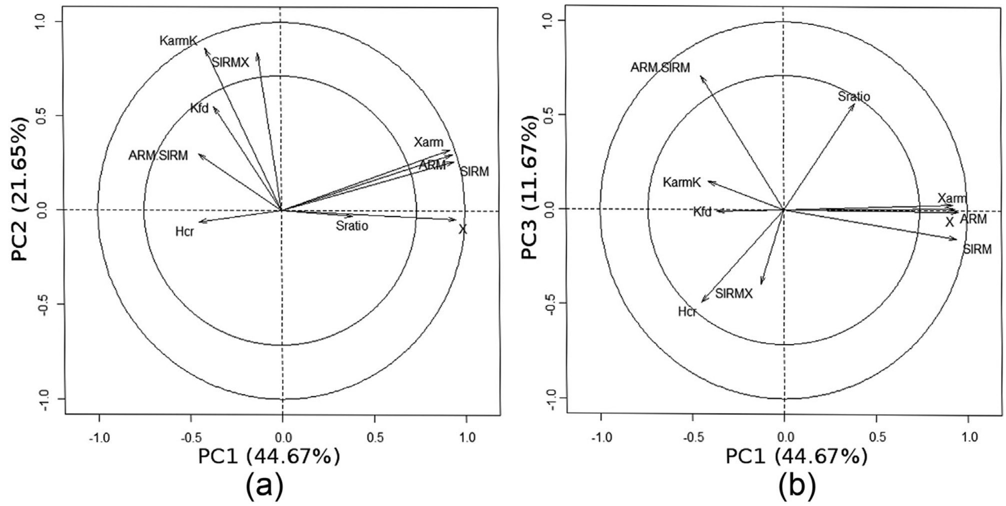


Fig. 6. Principal component analysis (PCA), representation of the variables (magnetic concentration-dependent parameter: χ, ARM and SIRM; magnetic mineralogy-dependent parameters: H_{CR} and S-ratio; and magnetic grain size-dependent parameters: κ_{ARM}/κ-ratio, κ_{FD}%, SIRM/χ, ARM/SIRM) in the coordinate planes PC1-2 (a) PC1-3 (b). The inner circle define the 50% of reconstruction of the variable (variables within the circle are not considered reconstructed).

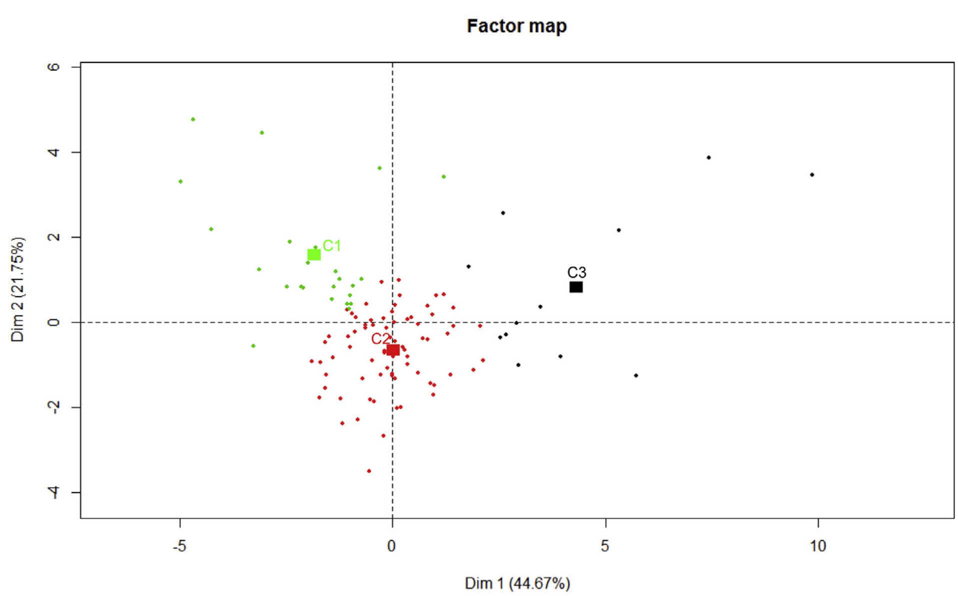


Fig. 7. Representation of the CA on the principal component map.

Table 4
Cluster centroids obtained by non-hierarchical classification (k-means clustering).

Feature	Variable	Overall average	C1 (n = 24)	C2 (n = 72)	C3 (n = 12)
			71% residential	40% industrial	75% vehicular
Concentration	χ [10^{-8} m^3 kg^{-1}]	471.0	231.2	449.4	1080.2
	χ_{ARM} [10^{-8} m^3 kg^{-1}]	766.7	572.0	690.9	1611.0
	ARM [10^{-6} A m^2 kg^{-1}]	675.1	498.0	622.1	1347.5
	SIRM [10^{-3} A m^2 kg^{-1}]	61.5	38.1	55.7	143.5
Mineralogy	H_{CR} [mT]	35.5	37.8	35.9	31.4
	SIRM/χ [kA/m]	13.4	16.7	12.4	13.6
	S-ratio [dimensionless]	0.91	0.90	0.91	0.96
Grainsize	κ_{ARM}/κ [dimensionless]	1.79	2.55	1.56	1.58
	κ_{FD}% [%]	2.8	4.9	2.2	2.6
	ARM/SIRM [dimensionless]	0.0116	0.0098	0.0119	0.0125

Table 5
Grouping of cities obtained by cluster analysis.

C1 (n = 24)		C2 (n = 72)				C3 (n = 12)	
Arakkonam	R	Ambattur	I	Perambalur	R,V,I	Avadi	V
Ariyalur	R,V	Arakkonam	R	Ranipet	R(2),I	Ayanavaram	V
Avadi	I	Ariyalur	I	Salem	I	Chidambaram	R,V
Ayanavaram	R	Ayanavaram	I	Sivakasi	R,V,I	Cuddalore	V
Cuddalore	R	Chidambaram	I	Sriperumbudur	R	Dharapuram	V
Dharapuram	R	Coimbatore	R,V,I	Theni	R,V	Hosur	V
Dharmapuri	R	Cuddalore	R,I	Thiruvottiyur	R,I	Krishnagiri	V,I
Hosur	R	Dharapuram	I	Tiruchirappalli	R,V	Namakkal	V
kancipuram	V	Dharmapuri	V,I	Tirunelveli	R,V,I	Ranipet	V
Krishnagiri	R	Dindigul	R,V,I	Tiruppur	R(2),V,I	Salem	R
Paramakudi	I	Erode	R,V,I	Tiruvarur	V,I		
Perambalur	R	Hosur	R,I	Tuticorin	V,I		
Sivakasi	R	kancipuram	R,I	Vellore	I		
Sriperumbudur	R	Karur	R,V,I	Villupuram	V,I		
Tirunelveli	R	Koyambedu	V	Virudhachalam	I		
Tuticorin	R(2)	Madurai	R,V(2),I				
Vellore	R	Mayiladuthurai	V,I				
Villupuram	R	Namakkal	R,I				
Virudhachalam	V	Neyveli	R(2),V,I				
Virudhunagar	R,V,I	Paramakudi	V				

dependence ($p = 0.073$, at 90% of confidence) between clusters and sampling areas (residential–industrial–vehicular). From residual analysis, the C1 is related to residential samples (at $p < 0.1$); the C2 is related to industrial samples (at $p < 0.1$) and the C3 is related to vehicular samples (at $p < 0.01$). The variable centroids and cities clustering of each group are detailed in Tables 4 and 5.

The cluster C1 is characterized by lower values of magnetic concentration, finer grain size and higher coercivity magnetic minerals (i.e.: higher values of κ_{ARM}/κ, $\kappa_{FD}\%$, H_{CR} and SIRM/χ and lower values of S-ratio) than the overall average (OA), respectively. This cluster has 17 samples from R areas, which represent 71% of all residential samples. The 40% ($n = 26$) of all industrial samples were grouped in the cluster C2. This cluster is made of 72 samples (out of 108) and is characterized by similar magnetic values to the OA without important differences. The cluster C3 has 12 samples (Table 5), where 75% ($n = 9$) of them are vehicular samples (V areas). Samples in this cluster show very high values of magnetic concentration, surpassing twice-three times the OA. Both magnetic mineral dependent parameters have indicative values (i.e.: $H_{CR} = 31.4$ mT and S-ratio = 0.96) of ferrimagnetic mineral with lower coercivity than the OA.

4. Conclusions

The combination of magnetic, SEM analysis and multivariate statistical techniques provides rapid and low cost method in studies of environmental monitoring of atmospheric pollution. According to the results, it is concluded that.

1) Collected dust samples present a strong magnetic signal taking into account the short time of collection (20–25 days). The mean values of χ, ARM and SIRM are 589.0×10^{-8} m^3 kg^{-1}, and 731.4×10^{-6} A m^2 kg^{-1} and 68.1×10^{-3} A m^2 kg^{-1}; respectively.
2) The order of amount of magnetic concentration (according to concentration dependent magnetic parameters) are vehicular areas > industrial areas > residential areas. The Kruskal–Wallis test shows significant differences between vehicular ($\chi = 563.1 \times 10^{-8}$ m^3 kg^{-1}) and residential areas ($\chi = 324.7 \times 10^{-8}$ m^3 kg^{-1}).
3) Ferrimagnetic minerals (magnetite) dominate the overall magnetic signal; therefore they seem to be the main magnetic carriers in these dust samples. The IRM studies revealed the dominance of ferrimagnetic minerals (e.g. from the IRM analysis, the contribution to the SIRM is 75.0–85.6%) but it is also observed evidence of another magnetic phase corresponding to antiferromagnetic (hematite) minerals.
4) The predominant magnetic grains are 1–5 μm in size and the SEM-EDS observations reveal the presence of trace elements (e.g.: Sb, Zn, Co, Ni, As and V). Such fine particles are commonly emitted by vehicles and industries, being potentially dangerous to human health because they can be inhaled and hence hosted in human tissues.
5) Three clusters of cities were identified from multivariate analysis, which involve groups of cities with different pollution impact. Each of these clusters is characterized with different magnetic characteristics. The first cluster C1 (most of samples from R area) has lower values of magnetic concentration dependent parameters, finer grain size and higher coercivity magnetic minerals. The second group (C2, most of samples from I area) is characterized by similar magnetic values to the overall average. The last one (C3, most of samples from V area) has very high values of magnetic concentration dependent parameters, surpassing twice-three times the OA, and magnetic mineral dependent parameters have indicative values of ferrimagnetic mineral with lower coercivity than the OA.

Conflict of interest

There is no conflict of interest.

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