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ELEMENTAL COMPOSITION, SIZE DISTRIBUTION AND IMAGE INTERPRETATION OF FINE PARTICULATE MATTER IN URBAN CITY ROAD SIDES, MYSORE (KARNATKA) INDIA

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ABSTRACT

The study was carried out using vacuum air pump sampler to collect particulate matter in the urban city roadsides. Elemental composition, size distribution and image interpretation of particles was analyzed using the methods of Energy Dispersive X-Ray (EDX), Dynamic Light Scattering (DLS) and Scanning Electron Microscope (SEM), respectively. Irwin road, the highly dense traffic area in Mysore city, has been selected for study purpose due to its high vehicular emissions. EDX analysis found that roadside particulate matter was dominated by black carbon (C) about 56% affected mostly by tail end pipe emissions. The samples were also rich in crustal elements like silicon (Si), iron (Fe), calcium (Ca), aluminium (Al), sodium (Na) and potassium (K) either in single elements or as chemical compounds. The results from DLS and SEM image interpretation showed that almost 90% of ambient particulate matter collected in the sampling site was in the size of fine particles ($PM_{2.5}$) and around 74% of them have degree of roundness or circularity above 0.75.

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KEY WORDS : Air pollution, DLS, EDX, Particulate matter, SEM, Urban roadways, Vehicular emission

Introduction

Air pollution in urban areas is serious concern in recent times, particularly for developing countries such as China and India and it may further extend from local to global scale[5,6,14]. Awareness of health problems related to air pollution arising from industrialization and urbanization has increased, especially during the last two centuries. Different health effects attributable to particulate matter (PM) have been documented[2,26].

The PM consists of different size, shape and composition. PM_{10} refers to particulate matter comprising particles less than 10 ìm in diameter. PM more than 2.5 ìm in diameter are generally referred to as coarse particles, while PM less than 2.5 ìm and 100 nm referred to as fine and ultrafine particles, respectively. The majority of recent health studies suggest that fine particles ($PM_{2.5}$) that arising mainly from man-made sources are more harmful than coarse particles[23]. Several efforts have also been specifically aimed at studying concentrations and potential health effects of the so-called ultrafine particles in the size range below 0.1 ìm[19,20].

The main sources of PM are vehicles, industries, combustion of fossil fuels and biomass[25]. Vehicular PM emissions are result of many processes like fuel and oil combustion; tires, bearings, brake linings and road surface wear products; and re-suspension of road and soil dust[12].

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Fig. 1: Irwin Road (shown by an arrow) as location of the sampling site

A recent study shows that common constituent emitted from motor vehicles is black or elemental carbon, which often referred to as soot and mostly generated from heavy-duty diesel engines, primarily diesel trucks[3]. Other elements that have often been associated with vehicular emissions include Cu, Zn, Pb, Br, Fe, Ca and Ba[9,11,24].

A number of approaches to characterize the shape or texture of particles have been introduced. A comprehensive work describing the size and shape of wear debris is presented[21]. Workers[22] used scanning electron microscopy techniques to examine particles produced in a vertebral disc prosthesis wear simulator. Whereas many other studies provide precise size details, morphology descriptions, quantifiable shape and texture estimations of many different objects[1].

Analysis of particle size has been conducted in light[3] and electron microscopes[15]. Size of particulates can be measured in terms of radius, length or diameter, area and perimeter by using latest technology in laboratory instruments and computational applications. Therefore, the objective of this study is to analyze fine particulate matter collected in near-roadway environment in Mysore city including their elemental composition, size distribution and image shape interpretation.

Materials and Methods

The Study Area:

Mysore city is located at 135 km south of Bangalore metropolitan city and lies at 12° 18' 25" N latitude and 76° 38' 58" E longitude. Mean sea level or altitude of the city is 765 m. Mysore city is the second biggest city of the state and well known for its important tourist and heritage centre. According to the 2011 national population census of India, the population of Mysore was 918,225. The city is well connected to the neighbouring states of Kerala and Tamil Nadu through road transport and rail network. The district is one of the southern most districts of the state and is bordered by four districts of the Karnataka state and one district of the other state, Kerala. The Mysore city has a warm, cool and salubrious climate throughout the year. The minimum temperature in winter is 15°C and the maximum temperature in summer is 35°C. Mysore gets most of its rain during the monsoon between June and September with an annual average of 782 mm[4]. Like many other Indian cities, Mysore city

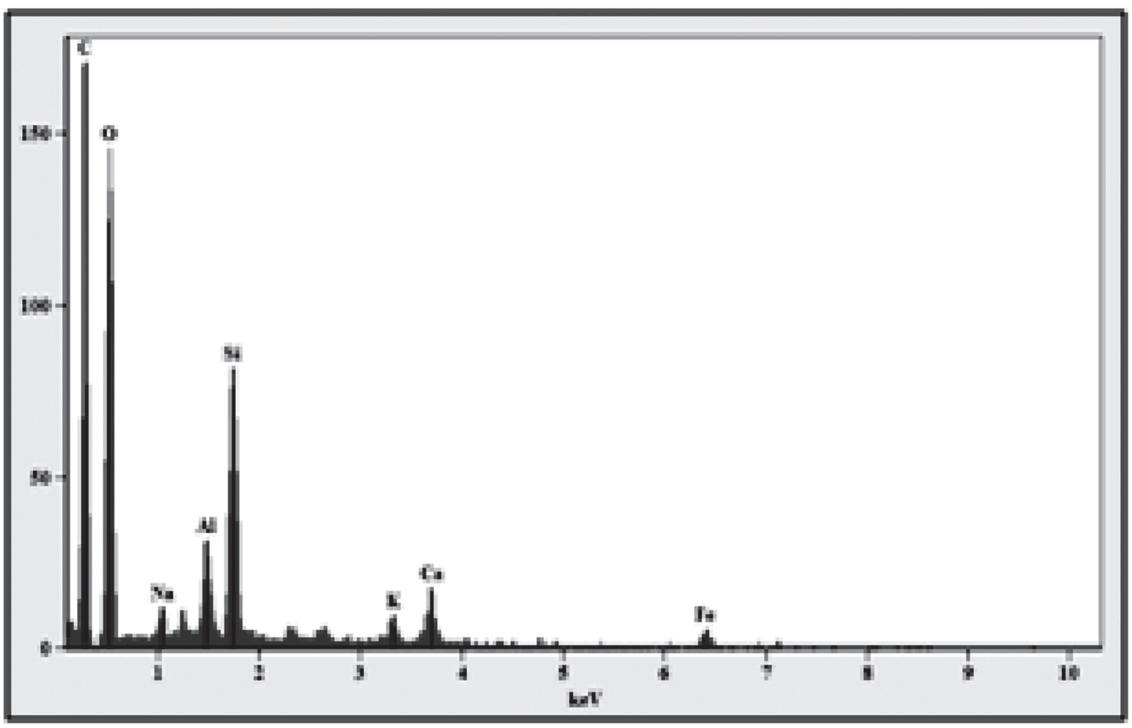

Fig. 2: EDX spectra showing peaks corresponding to the elements present in particulate matter

has also high vehicular growth and emissions problem, particularly PM. It has over 523 thousand vehicles registered in the city in 2015 and is projected to expand about 120% in 2020[7]. Irwin road is the highly narrow and congested traffic lane in Mysore city and has been perfectly selected as study site for PM collection (Fig. 1).

Experimental Set Up:

Vacuum air pump sampler was used for collection of particulate matter. The experimental set up consisted of cubical chamber measured 37 cm (length) x 26 cm (width) x 23 cm (height). It was attached to vacuum pump containing auto adjustable speed of one inlet and outlet with three types of exchangeable filters. One end supply the air from the pump and the pump pushed the air to the other end into the chamber with a flow rate of 25 L/min. The sampling chamber was located on the first floor building in Irwin road, Mysore city. The inlet prevented the admission of ambient particles, whereas the other received atmospheric air filtering system (5-6 hours/day). The temperature in the room and in the chamber was recorded (25±2°C). The sampling was carried out in one month period during late summer 2014. Particulate matter entrapped in the vacuum pump filters were then taken out and stored to laboratory for further analysis.

TABLE-1: Particle size distribution in nm

Percentile	Size above (nm)
10	377.9
20	335.7
30	311.9
40	293.6
50	277.7
60	263.1
70	248.6
80	232.5
90	211.8
95	197.8

Analysis of Elemental Composition:

Elemental composition of PM was analyzed using Energy Dispersive X-Ray (EDX), also referred to as Energy Dispersive Spectroscopy (EDS) method. The technique detects X-rays emitted from the sample during bombardment by an electron beam to balance the energy. The data generated by EDX instrument consists of spectra showing peaks corresponding to the elements. The count number of emitted X-ray (X-axis) versus their energy (Y-axis) is evaluated to determine the true elemental composition of the sampled volume being analyzed[10,16]. The X-ray energy was converted to voltage pulse and recorded in voltage units (usually in keV). The height of the peaks represents relative abundance of X-rays emitted by the elements. The higher the peak, the more X-rays emitted by a specific element, thus the abundance of that element presents in the sample was also higher. After calculating by atomic weight of each element, the data was then converted into elemental composition units in weight percentages.

Analysis of Particle Size Distribution:

Particle size distribution can be determined by measuring the random changes in the intensity of light scattered from a suspension or solution. The technique is commonly known as Dynamic Light Scattering (DLS) method. Particles can be dispersed in a variety of liquids. Small particles in suspension undergo random thermal motion known as Brownian motion. Light from the laser source illuminates the sample in the cell. The scattered light signal is collected with one of two detectors. The provision of both detectors allows more flexibility in choosing measurement conditions. The variations in the signal arise due to random Brownian motion of the particles. The "noise" from particle motion will be used to extract particle size[8].

Analysis of PM Images:

A scanning electron microscope (SEM) can capture an image of a sample by scanning it with a focused electron beam over its surface. The

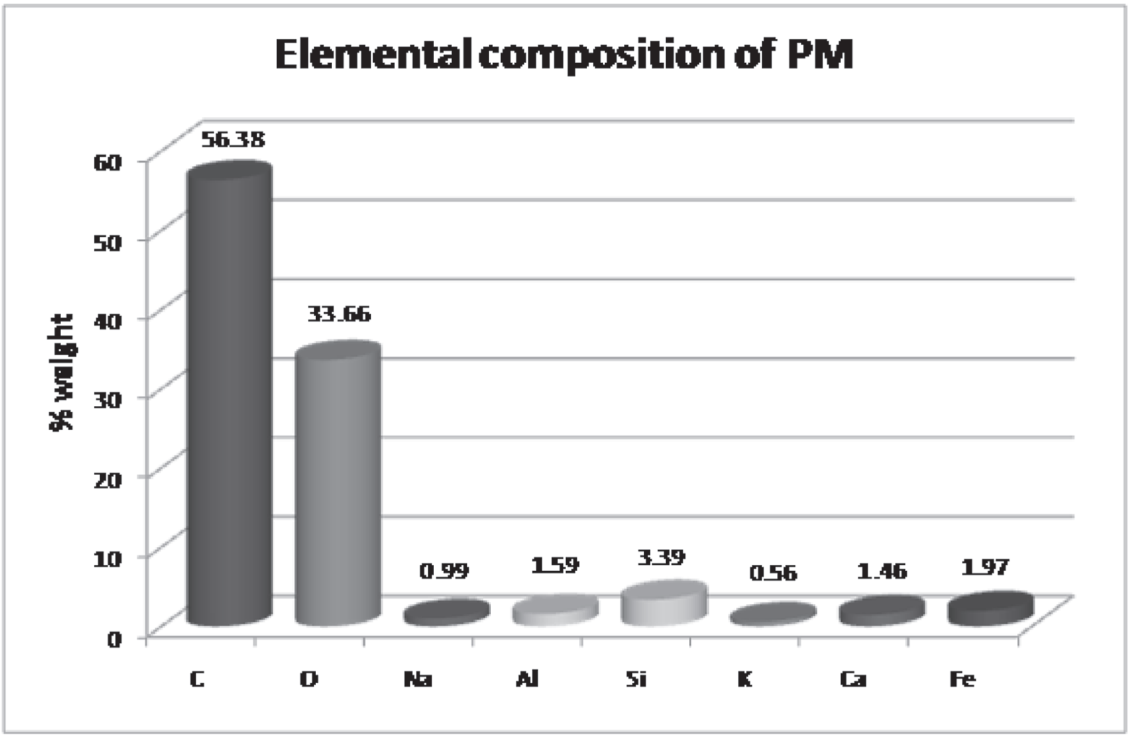

Fig. 3: Weight percentage of elements present in particulate matter

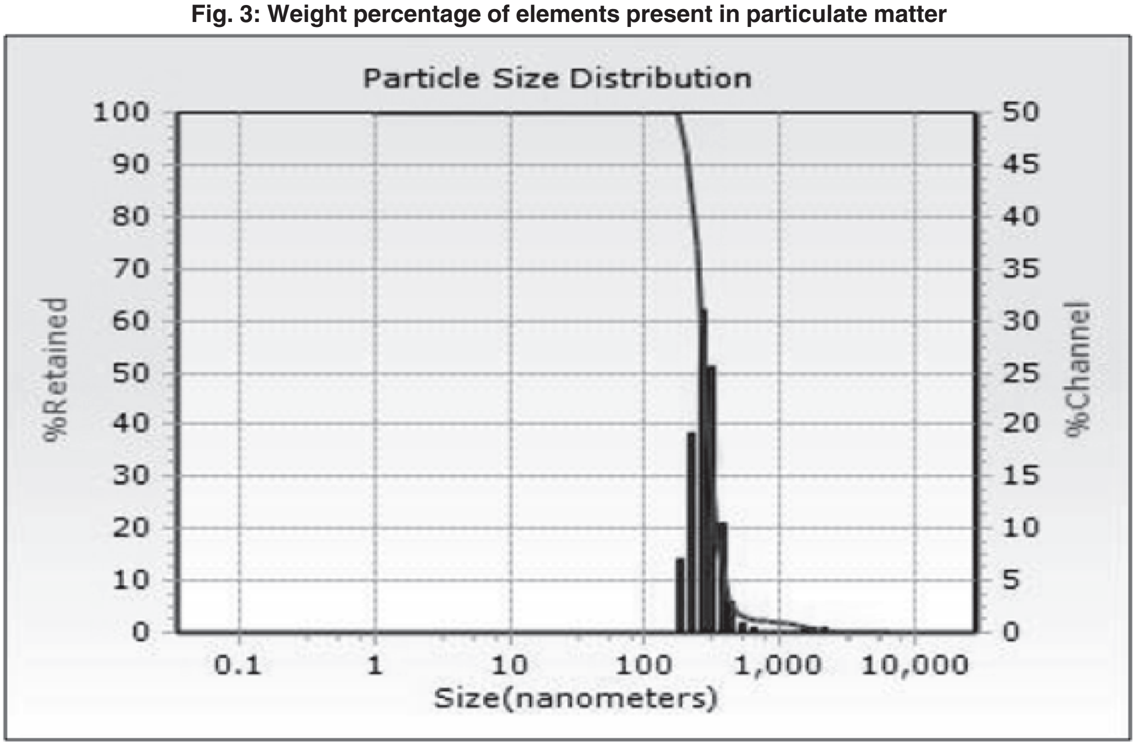

Fig. 4: Particle size distribution from DLS analysis

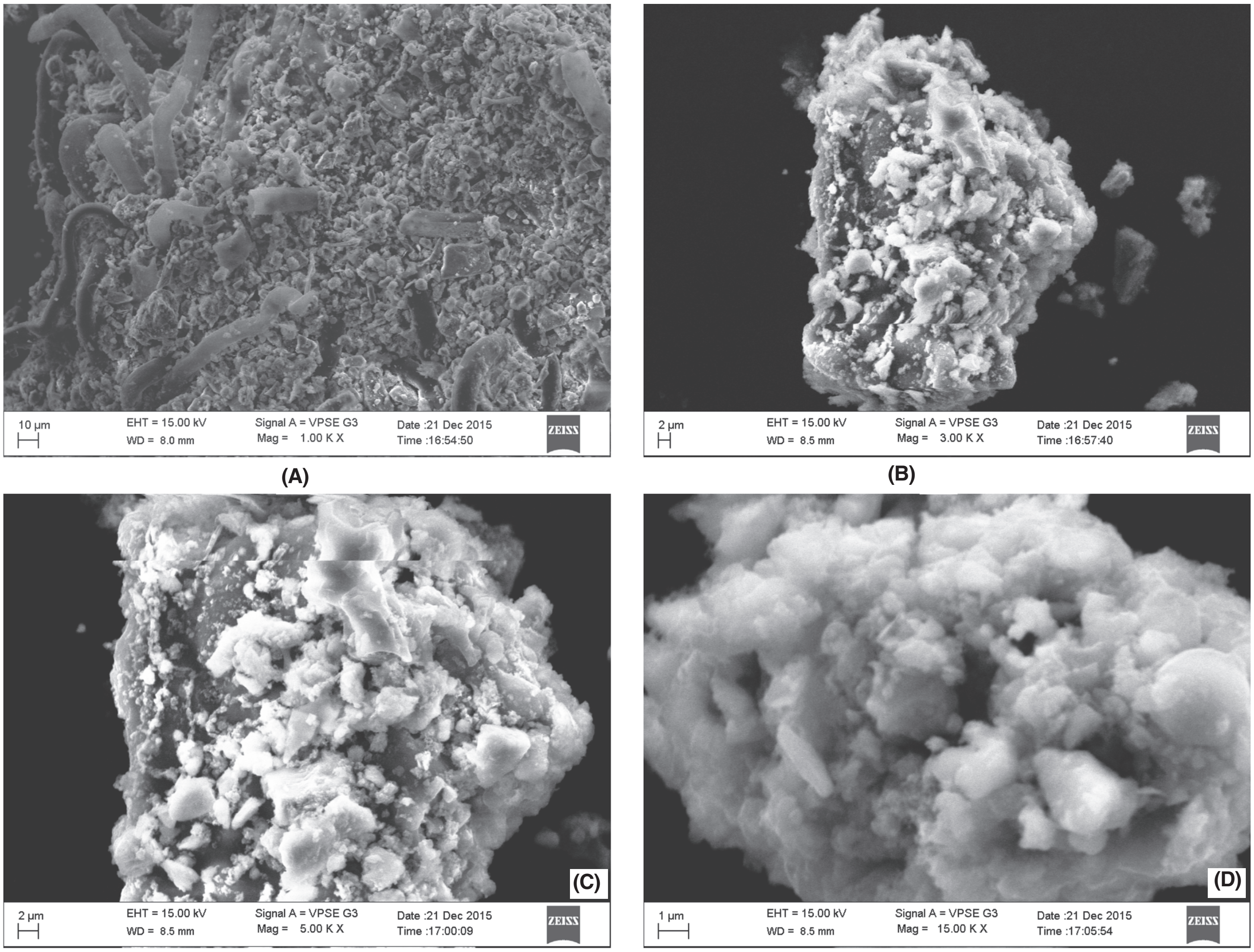

Fig. 5: Scanning Electron Microscope images of PM by magnification of (a) 1,000X (b) 3,000X (c) 5,000X and (d) 15,000X

electrons in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and composition[18]. SEM can produce very high-resolution images of a sample surface to about 250 times the magnification limit of the best light microscopes. Images taken from microscopes are needed to be stored into a digital format for analysis. The digital images can then be processed in a computer to adjust their contrast and brightness, and to quantify their size (length or diameter), area, perimeter, and circularity. The circularity, also known as degree of roundness that represents the shape or form factor of particles is dimensionless[1,17]:

By using this formula, a perfectly rounded particle sphere should have circularity value of 1. More irregular shape of particles will have values lesser than that. Length, area and perimeter of particles were precisely measured by employing specific image processing software (Digimizer version 4.6.0 from MedCalc) before exported to Microsoft Excel to calculate their size distribution and circularity values.

Results and Discussion

Elemental Composition of PM

Samples of particulate matter were analyzed by EDX instrument. The results obtained describe a spectrum chart showing a number of peaks that correspond to the chemical elements presented in the particulates (Fig. 2). The results showed that ambient particulate matter collected from roadside sampling site consists of 56.38% carbon (C), 33.66% oxygen (O), 3.39% silicon (Si), 1.97% iron (Fe), 1.59% aluminium (Al), 1.46% calcium (Ca), 0.99% sodium (Na), 0.56% potassium (K), and other elements in smaller fraction (Fig. 3).

In nature, these elements may present as single elements like carbon, iron, and aluminium, or as chemical compounds joined with other elements, such as silicon presents as silica (SiO_2), iron as iron oxides (FeO or Fe_2O_3), aluminium as alumina (Al_2O_3), calcium as CaO, $CaCO_3$ or $CaCl_2$, sodium and potassium present as chloride salts ($NaCl$ and KCl, respectively) or other possible forms of compound like sulphate, phosphate, etc.

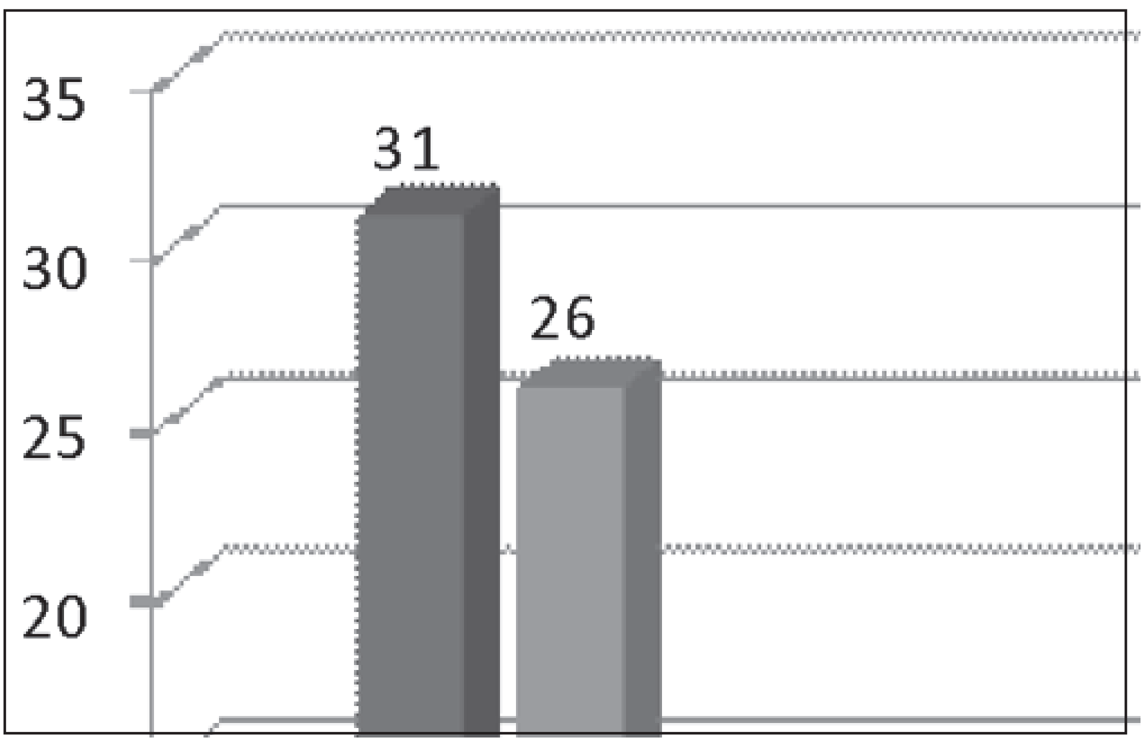

Fig. 6: Size distribution of roadway particles analyzed from SEM digital image

Particle Size Distribution:

The dynamic light scattering (DLS) method was used to identify the size variation of particles taken from the sampling site. The samples were suspended in a specific liquid before taken into the instrument. Viscosity of the solution after mixing with the samples was measured 0.819 cp at temperature 28.76°C. Conductivity of the solution was 888 ìS/cm, while the pH was relatively neutral (7). Laser light then illuminates the sample solution from its container. The scattered light signal collected by the DLS detectors was analyzed in the computer and presented as particle size distribution graph (Fig. 4).

The result showed that ambient particulates entrapped in the sampling chamber were fine particles ($PM_{2.5}$) with size less than 2.5 ìm or 2500 nm. About 95, 90, 80, 70, 60, 50, 40, 30, 20, and 10 percent of particles have size above 197.8; 211.8; 232.5; 248.6; 263.1; 277.7; 293.6; 311.9; 335.7; and 377.9 nm, respectively (Table-1). The larger the size, the less percentage of particles was identified. The size was certainly higher for particles in ultrafine category or $PM_{0.1}$ (size <100 nm) and much lower for PM_{10} category (size 2.5 to 10 ìm).

SEM Images Interpretation:

Images of PM were captured by SEM manufactured by Zeiss. The instrument provided black and white, TIFF-format images at resolution of 1024x768 pixels that varied from 1,000X to 15,000X magnifications (Fig. 5). These digital images were then processed by Digimizer software to measure particle length (or diameter) for size distribution analysis and to identify area and perimeter of particles for circularity measurement.

Analysis of SEM digital images has resulted in particle size distribution (Fig. 6) and circularity values of the particles (Fig. 7). It was found that under scanning electron microscope, the mean size of the particles collected in the sampling site was 1.66 ìm. In details, they were distributed in a variety of size, mainly <1 ìm (31%), <1.5 ìm (26%), <2 ìm (11%) and <2.5 ìm (10%). It indicated that majority of roadsides PM in Mysore city belongs to $PM_{2.5}$ category, even though a few of them size above 2.5 microns, but also due to resolution constraint,

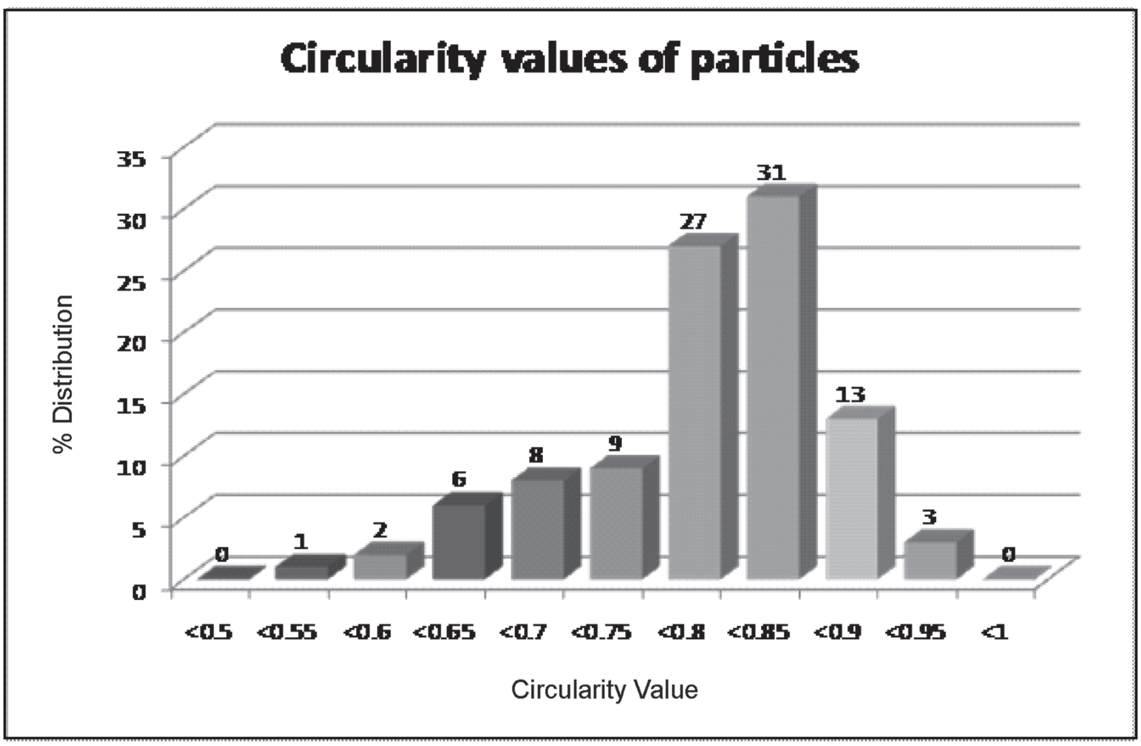

Fig. 7: Classification data for the circularity values of particulate matter

images of particles size below 0.5 microns should be more than they have identified in the analysis.

The shape of urban roadsides particles was found close to circular. Majority of particles have circularity values or degree of roundness up to 0.85 (31%), 0.8 (27%) and 0.9 (13%). However, the results could be possibly higher than these if using higher resolution images or considering only larger size particles for shape evaluation to avoid difficulty while digitizing the images in the computer program[1].

Conclusion

Particulates released in Mysore city are mainly due to vehicular emissions. Irwin road with high number of motorized vehicles, poor traffic control and daily congestion has become perfect site for study area. Elemental composition of particulate matter collected on the urban roadsides environment has been identified using energy dispersive X-ray analysis and it was dominated by black or elemental carbon (C) about 56% and some crustal elements or compounds constitute of silicon (Si), iron (Fe), calcium (Ca), aluminium (Al), sodium (Na), potassium (K), and other elements from earth and road dust particles. Size of the particulates was measured around 200 to 300 nm using dynamic light scattering method, hence categorized as fine particles ($PM_{2.5}$). Interpretation of scanning electron microscope images also provides the same result, in which 83% of particles measured belong to the size below 2.5 microns in diameter. About 74% of roadside particles identified from the SEM digital images have degree of roundness or circularity values above 0.75.

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