

# Existence of Nano-Organic Carbon Particles below the Size Range of 10 nm in the Indoor Air Environment

Bireswar Paul, Amitava Datta

**Abstract**—Indoor air environment is a big concern in the last few decades in the developing countries, with increased focus on monitoring the air quality. In this work, an experimental study has been conducted to establish the existence of carbon nanoparticles below the size range of 10 nm in the non-sooting zone of a LPG/air partially premixed flame. Mainly, four optical techniques, UV absorption spectroscopy, fluorescence spectroscopy, dynamic light scattering and TEM have been used to characterize and measure the size of carbon nanoparticles in the sampled materials collected from the inner surface of the flame front. The existence of the carbon nanoparticles in the sampled material has been confirmed with the typical nature of the absorption and fluorescence spectra already reported in the literature. The band gap energy shows that the particles are made up of three to six aromatic rings. The size measurement by DLS technique also shows that the particles below the size range of 10 nm. The results of DLS are also corroborated by the TEM image of the same material.

**Keywords**—Indoor air, carbon nanoparticles, LPG, partially premixed flame, optical techniques.

## I. INTRODUCTION

INDOOR air pollution from the combustion sources used for cooking purposes is a big concern nowadays in the developing countries. The fuel used for cooking purposes is different in various regions depending on the socioeconomic status of the people. In the rural areas of developing countries wood, agri-residue, and dung cakes etc. are commonly used for domestic cooking; whereas in urban areas, the use of coal, kerosene and liquid petroleum gas (LPG) is quite common [1]. The combustion of such fuels in the domestic environment gives rise to the emission of volatile and semi-volatile compounds and oxides of different elements such as carbon, nitrogen and sulfur. Besides these gaseous pollutants, there are also emissions of respirable particulate matter in the indoor air environment from the combustion sources [2]-[4]. Among these pollutants poly-aromatic hydrocarbon (PAH) and particulate matter below the size range of 10 nm poses a high risk to human health. Several respiratory and chronic obstructive pulmonary diseases, cancers, and other illnesses in developing countries have been attributed to the emission of such toxic pollutants in the indoor air environment [5], [6].

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According to a WHO report [7], nearly half the world continues to cook with solid fuels. This includes more than 75% of people in India, China and nearby countries, and 50% – 75% of people in parts of South America and Africa. Limited ventilation is common in many developing countries and increases exposure, particularly for women and young children, who spend much of their time indoors. In India alone, it has been estimated that the use of solid fuels causes 400,000 – 550,000 premature deaths annually among women and children below five years of age [8]. Exposures have been measured to be many times higher than WHO guidelines and national standards, and thus, can be substantially greater than outdoors in cities with the most severe air pollution. The solid fuel used in rural areas is burned in homemade clay-stoves or chulhas having efficiency as low as 8-12% [9]. The inefficiency of the chulhas results in incomplete combustion of the solid fuels leading to the formation of PAH and other pollutants in an uncontrolled manner [10]. However, modern devices used in the urban areas e.g., a kerosene cook stove, has an efficiency of 50% and a LPG has close to about 60% [11], [12]. These modern energy conversion devices are much cleaner fuel than the solid fuels, emitting less quantity of gaseous and particulate phase pollutants [10], [11], [13].

There have been a number of research works [2]-[4], [9]-[20] undertaken until now to characterize and estimate the emission of pollutants in the indoor air environment from the combustion sources used for cooking and heating purposes. In rural areas biomass combustion has been identified as the main source of indoor air pollutants [1], [10]. While in urban areas liquefied petroleum gas or kerosene is used as the main fuel for cooking purposes [1]. In most of the studies, the focus is concentrated on the measurement of PAH and respirable particulate matter present in the indoor air environment. The PAH present in the indoor environment has been distributed between gaseous and particulate phase [3], [4], [10], [14]. Zhu et al. [14] has investigated the PAH in the indoor and outdoor environment. They found a high indoor-to-outdoor ratio ( $>3$ ) of PAH, indicating the certain generation of PAHs from the indoor sources. Zhu et al. [14] along with others [3], [10], [15] have also concluded that the PAHs can remain in gaseous (2 to 3 ring), particulate ( $>5$  ring) and in between gaseous and particulate phase (3 - 4 ring). Particle emission from the combustion sources in the indoor air environment has also been studied because of the adverse effect of these particles on the human health [13], [16]-[20]. The existence of ultrafine particles ( $<100$  nm) in the indoor environment has been

reported by many researchers using different types of techniques [4], [16]-[20]. Dennekamp et al. [18] has reported a particle diameter in the range of 15 - 40 nm in the indoor environment produced from gas burning. Monkkonen et al. [4] measured the number and mass concentration of fine particulate matter ( $D_p > 10$  nm) in urban households, mainly using LPG or kerosene as the cooking fuel. The maximum concentration of the particles has been reported during the cooking period. Wallace et al. [19] has conducted an experimental study to measure the size distribution and number concentration of ultrafine particles generated in the gas and electric stoves. Peak concentration of the particles was observed corresponding to a particle diameter of approximately 10 nm in diameter. The formation of ultrafine nano particle in the blue burning flame of a domestic gas cooker consuming natural gas has been established recently by Minutolo et al. [16], Wallace et al. [20], and Wagner et al. [13]. The particle size has been found out to be below 10 nm for these partially premixed flames established on the gas burners. The mean particle size corresponding to the peak number concentration reported by Wallace et al. [19] and Wagner et al. [13] are approximately 5 and 7 nm. While Minutolo et al. [16] have measured even very small particles with a mean size of about 3 nm. Minutolo et al. [16] and D'Anna [21] has divided the particles into two groups based on their sizes. Particles with sizes up to about 10 nm were termed molecular particle precursors and they are detectable by UV light absorption and laser-induced fluorescence (LIF). Particles larger than 10 nm were termed soot, and these particles are typically detected by visible light absorption and by laser-induced incandescence (LII) techniques. The results of Minutolo et al. [16] indicate that the particles formed by the gas cooker having a mean size of 2 - 3 nm are molecular precursor particles, rather than soot particles. These molecular precursors of soot particles are formed in the early stage of the soot formation process.

The precursor particles of soot show considerable differences in their physical, morphological and chemical characteristics from the fully grown soot particles. Unlike soot particles, which absorb over a wide range of electromagnetic spectrum from UV to visible, the soot precursor particles absorb only in the UV range of the spectrum below a wavelength of 400 nm with a small hump in the region of 250-300 nm [22]-[24]. Optical band gap energy of the particles also shows that these particles consist of two to five member aromatic rings [23], [25]. The fluorescence spectra of these particles show an emission peak in the region of 310 nm. Moreover, these particles are hydrophilic and readily form a colloidal suspension in water [26]. The TEM images reveal that these particles mostly occur as singular spheroids, while soot particles remain in agglomerated form [27], [28]. The precursor particles also show a liquid like consistency for which they spread when dropped on the TEM grid. As a result, the precursor particles detected by TEM exhibits a larger diameter in comparison to that detected by other methods [28].

Literature review of the emission of pollutant (both in the gaseous and particulate phase) in the indoor air environment

shows that majority of the work focused on the emission of PAH in the indoor environment and its harmful effects on the occupants. Biomass burning has been considered in these studies as sources of PAH. While few works are also available where the study of particulate matter emitted from indoor combustion sources are also available. The particle size in most of the cases has been reported to be above 10 nm, with the exception of a few works by Minutolo et al. [16], Wallace et al. [19] and Wagner et al. [13], where they have successfully detected particles below the size range of 10 nm. Natural gas and methane has been used as a fuel in these cases. Few works using LPG and kerosene have also been carried out, but they were unable to detect particles below 10 nm. However, LPG is the largest consuming cooking fuel in urban areas of the developing countries. Thus, the study of the formation of precursor particle below the size range of 10 nm in the LPG combustion also needs to be carried in order to assess their existence in the indoor environment. Because it is a well known fact that as the particle size decreases, the particle number concentration increases for the emission of the same amount of mass of that particle. It has been observed in recent studies that the number concentration could be a better predictor and indicator of the health risks than mass concentration [29]-[31]. In this experimental work, the main objective was to show the formation and existence precursor soot particles below the size range of even 5 nm in a partially premixed LPG/air laboratory flame, which is a representation of the flame that actually establishes on the LPG gas cooking burner.

## II. EXPERIMENTAL

In this experimental work, an LPG/air partially premixed flame has been established on a co-axial Bunsen type burner. The burner has an inner diameter of 12.2 mm and thickness of 0.8 mm; while the inner diameter of the outer tube is 51 mm. Arrangement has also been made in the burner to confirm flow uniformity at the exit of the burner and to avoid any flash back. The LPG/primary air mixture pass through the inner tube and the secondary air flows through the annular space. The air is supplied from a compressor after removing the moisture by passing it through a container of silica gel. The domestic gas burners also operate on a Bunsen burner principle and produce a premixed or partially premixed flame. For our experimental work, the flow rate of fuel, primary air and secondary air has been maintained at 0.132 l/min, 0.82 l/min and 19.6 l/min, respectively, using three digital mass flow controllers (make: Aalborg, USA). The corresponding flow velocities of the primary and secondary jets are 13.6 cm/s and 17.25 cm/s. The equivalence ratio for the primary fuel-air mixture is 4.64.

The burner is placed on a traversing mechanism, which can be moved in two directions in the horizontal plane. The burner is surrounded in three directions by Perspex sheet to prevent the flame from being disturbed by external air disturbances, while one side is kept open for collecting the sample. The bottom and upper side is kept open for fresh air flow and exhaust removal. The sample from the flame has been

collected corresponding to an axial and radial location of 6 mm and 4mm, respectively, from the burner tip. A quartz microprobe having a tip opening of 250 micrometer is used to isokinetically sampling the combustion product. A vacuum pump is used to draw the sample from the sampling location of the flame and passed through 6 ml of de-ionized (DI) water kept in a glass test tube and after that is exhausted to the atmosphere. The sampling rate has been kept very low so that the precursor particles get sufficient time to disperse in to the DI water. The sampling has been done for a period of 1.5 hours. This type of sampling acts as a sort of fractionation collecting only the hydrophilic precursor soot particles, while the hydrophobic soot particles and gases pass through. This type of sampling procedure has been used previously by other researchers to sample combustion generated nanoparticles from flames [21]-[24].

After collection, the sample has been analyzed by using UV-visible absorption and fluorescence spectroscopy, dynamic light scattering (DLS) and transmission electron microscope (TEM) techniques. UV-visible absorption has been carried out in a UV-visible spectrophotometer (Shimadzu UV-1601PC). Two light sources, viz. deuterium and halogen lamps, have been used to provide the energy in the spectral range of 190-1100 nm. The fluorescence spectra of the same samples are taken in a luminescence spectrofluorometer (Perkin Elmer LS-55) by exciting the samples at 266 nm. The size distributions of the sampled materials have been detected by dynamic light scattering (Model DLS—Zetasizer Nano ZS, Malvern Instruments) after filtering the samples several times through a 0.22  $\mu\text{m}$  Millipore membrane filter. Another method, transmission electron microscopy (TEM), has also been used to estimate the average particle size of the precursor soot particles collected from the flame. A JEOL JEM 2010F electron microscope operating at an acceleration voltage of 200 kV has been used for this purpose. Samples were prepared by placing and drying a drop of hydrosol on a 300 mesh copper grid coated with a thin amorphous carbon film. The TEM image has been taken at a magnification of 60000.

### III. RESULTS AND DISCUSSIONS

Fig. 1 shows the LPG/air partially premixed flame established on a co-flow burner. The flame shows a double flame structure having inner rich premixed flame and outer non-premixed flame. The fuel air mixture comes out of the inner tube first burns in the inner rich premixed flame and the remaining product of incomplete combustion burns in the outer non-premixed flame. The radiation emitted from the flame also shows that the flame has two distinct regions. The blue/green appearance of the lower part of the flame is due to the chemiluminescence from the local radicals, while the upper yellow/orange radiation is from the grown soot particles. This kind of flame is a representative of the flame that is produced in the LPG gas stoves. In LPG gas stoves, the LPG from the gas cylinder comes through a pipe and expands through a nozzle before going to the mixing tube under the burners. The jet of fuel issued from the nozzle entrains

primary air from the atmosphere and a fuel air mixture is formed in the mixing tube. Finally, the fuel-air mixture comes out of the ports of the burner cap and burns in the atmosphere. The additional air required for complete combustion comes up from the atmosphere through the central hole of the burner and the small gap between the reflector plate and the burner. So, the individual jet of fuel-air mixture coming out of the ports of the burner can be considered as a partially premixed flame that has been established in the laboratory co-flow burner.

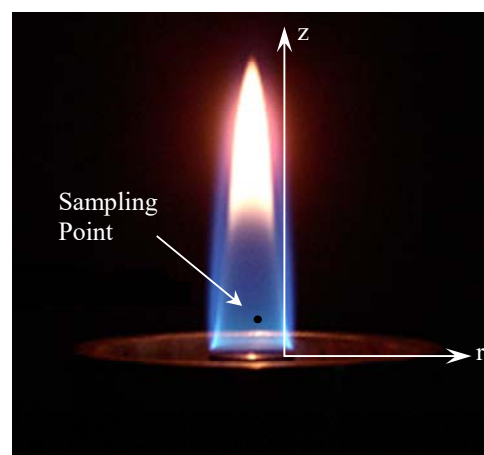


Fig. 1 Partially premixed flame of LPG/air mixture

The sample for our experimental work has been collected from slightly inner side of the inner flame front in order to exclude any possibility of sampling of particles other than the combustion generated particles. The sampling location is 6 mm above the burner tip and 4 mm away from the flame axis in the radial direction. Fig. 2 shows the combined absorption and fluorescence spectra of the hydrosol sample collected from the LPG/air flame. The absorbance of the sample first increases from its value at 200 nm to its maximum value at around 210 nm and after that it decreases monotonically and becomes negligible beyond 350 nm with a small hump in the region of 250-300 nm. The lack of absorbance value in the visible range confirms that the sample is devoid of soot particles. The fluorescence spectra of the sample have been obtained by exciting the sample with an excitation wavelength of 266 nm. There are two sharp peaks, at wavelengths of 310 nm and 330 nm, respectively, in the fluorescence spectra. As the fluorescence peaks generally occur as the mirror image of the absorbance peaks, so the fluorescence peaks at around 310 nm and 330 nm are due to the emission from the substances that has absorbed in the region of 250 – 300 nm and 210 nm. The similar nature of absorption and fluorescence spectra of the hydrosol sample collected from ethylene/air flame has also been reported by D'Alessio et al. [24] and Bruno et al. [22]. The material sampled by them has been reported to be nano-organic carbon (NOC) particles, which is nothing but the precursor soot particles. Thus, the absorption and fluorescence spectra of our sample clearly show the existence of precursor soot particles in the hydrosol. However, there is a slight qualitative difference between the spectra of the sampled



material and that of the spectra of the NOC particles reported by D'Alessio and Bruno. The absorption and fluorescence peaks at around 210 nm and 330 nm have not been observed by D'Alessio and Bruno. While, Paul et al. [25] has conducted a similar type of experiment, where they have reported that the additional peaks from the sample is due to the presence of some lighter gaseous phase PAH (naphthalene) in the colloidal solution along with the precursor soot particles. This type of lighter gaseous PAH having 2 to 3 aromatic ring has also been found in the indoor air environment [14], [15]. Viau et al. [15] have measured the PAHs in the indoor air environment. A high concentration of naphthalene, fluorine, Phenanthrene and Acenaphthylene along with a moderate amount of benzo(a)pyrene, in the indoor environment has been reported by them. The high concentration of naphthalene, fluorine and phenanthrene has also been reported by Viau et al. [15] along with Zhu et al. [14], who has reported that the two and three-ring PAHs were present mainly in the gaseous phase while five- and six-ring PAHs appeared mostly in the solid phase. The four-ring PAHs, fluoranthene and pyrene, appeared almost equally in both phases.

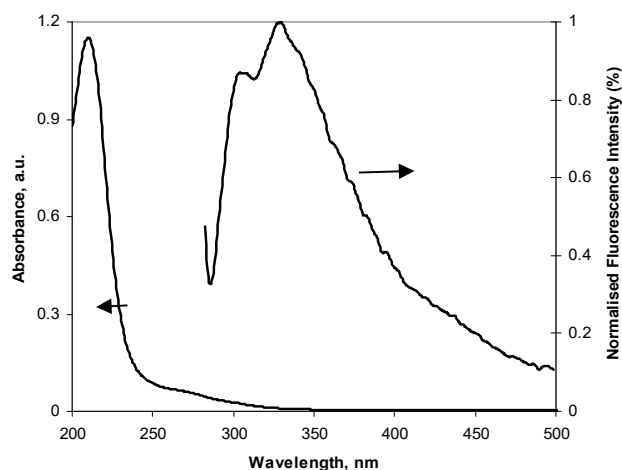


Fig. 2 Absorption and fluorescence spectra of the sample

Now, in order to find out the chemical structure and the number of aromatic rings in the precursor soot particle, we have analyzed the optical band gap energy using the absorption result. The band gap energy is a powerful parameter for the characterization of aromatic structures [23], [32] because of the information it gives on the size of the aromatic clusters inside the structure. The band gap energy is the difference between the energy levels of the valance band and the conduction band. In solid state physics the  $\pi$  states form the valance band and the  $\pi^*$  states form the conduction band. The energy gap between the  $\pi$  -  $\pi^*$  molecular states in aromatic molecules decreases with the number of carbon atoms. The absorbance ( $\alpha$ ) can be correlated with the optical band gap energy ( $E_g$ ) using the Tauc equation [22], [23], as given below:

$$\sqrt{\alpha E} = B(E - E_g) \quad (1)$$

where,  $E$  is the energy of the incident photon and  $B$  is a constant. The photon energy can be computed from the wavelength of the incident photon using the velocity of light in the medium (in this case water). The optical band gap energy can be obtained as the intercept on the abscissa by fitting a linear plot between  $E$  (on the abscissa) and  $\sqrt{\alpha E}$  (on the ordinate). When different kinds of particles are present in the sample, the above plot does not identify a single band gap energy. In such cases, different band gap energy is evaluated from the plot [23]

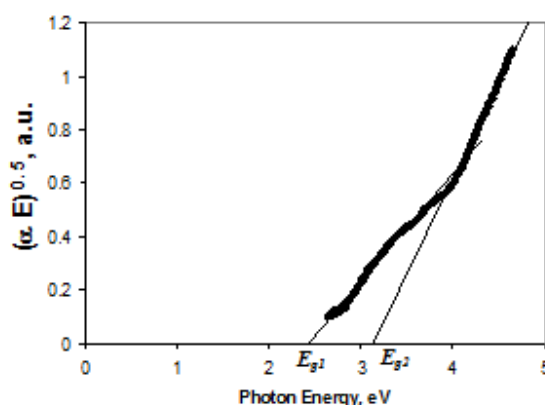


Fig. 3 Tauc plot of the sampled material

Fig. 3 shows the Tauc plot of the sampled material collected from the flame. The Tauc plot has been obtained by plotting the data up to 350 nm only, because of the lack of absorbance thereafter. Two band gap energy mainly 2.42 eV ( $E_{g1}$ ) and 3.13 eV ( $E_{g2}$ ) has been obtained by fitting the data of the absorption spectrum. The higher value of band gap energy of our sample indicates towards the presence of only precursor soot particles and the absence of grown soot particles ( $E_g \sim 0.6$  eV). Moreover, Robertson and O'Reilly [32] have reported that the band gap energy ( $E_g$ ) varies more or less inversely with the cluster size as:

$$E_g = \frac{6}{M^{1/2}}, \quad (2)$$

where,  $M$  is the number of aromatic rings in the cluster. According to (2) the band gap energies of 3.13 eV and 2.42 eV correspond to aromatic structures having three and six aromatic rings.

So, the results of the band gap energy study show that there is an existence of poly aromatic hydrocarbon having higher aromatic structure (six rings) in the sample. Viau et al. [15] and Zhu et al. [14] have already reported that the higher ring PAHs remain in the solid or particle phase. Now in order to find out the size distribution of the particle phase PAHs or precursor soot particles in our sample, we have performed the dynamic light scattering technique. Dynamic light scattering technique is based on the time-resolved measurement of the scattered light intensity from a sample cell that contains the

particles in a solvent. The scattered light intensity fluctuation due to the erratic motion (Brownian motion) of the particles contains the information about the diffusion co-efficient of the particles, which in turn is size dependent. A volume mean particle size of 1.4 nm has been obtained by DLS. However, the maximum particle size has been found out to be 2.7 nm.

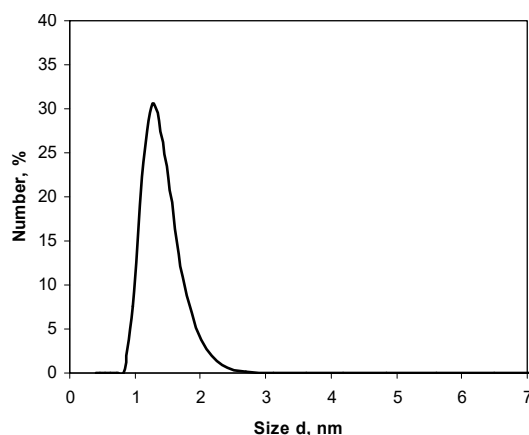


Fig. 4 Size distribution of the precursor soot particles

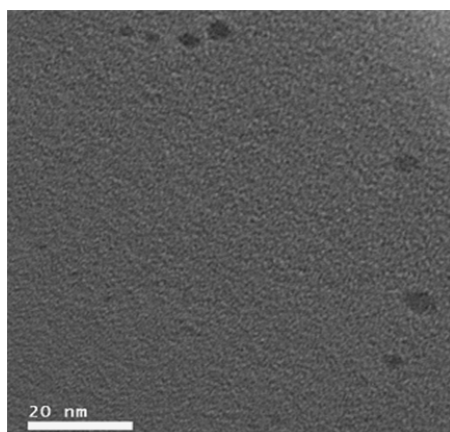


Fig. 5 TEM image of the precursor soot particles

The precursor soot particle size has also been found out using transmission electron microscopy. In this method, a direct image of the sampled material has been obtained. Fig. 5 shows the TEM image of the precursor soot particles. From the direct size measurement of the sampled material by TEM it is clear that the particles below the size range of 10 nm are generated in the LPG/air combustion. The figure also shows that these particles are semi-transparent to the electron beam, spherical in shape and are not in the agglomerated form. But there is distinguishable difference between the size distribution by DLS and TEM. The difference in size distribution between the techniques may be due to the liquid like characteristics of the precursor soot particles for which they spread over the TEM grid and form a spherical segment, as reported by Zhao et al. (2007) [33]. Zhao et al. (2007) have reported a ratio of height to projected diameter of 0.1 as a result of the spread. Hence, the actual sizes of the particle are lower than that can be observed from the TEM image. Thus,

the size distribution of the particles by these two techniques show that there is an existence of precursor soot particles below the size range of 10 nm in the LPG/air partially premixed flame. The results of the study also show that these precursor soot particles are consist of three to six number of aromatic rings.

#### IV. CONCLUSION

Existence of nano organic carbon particles has been confirmed in LPG/air partially premixed flame using UV absorption and fluorescence spectroscopy. It has been found that the particles consist of three to six number of aromatic rings. The particle size has been found to be below the size range of 10 nm by DLS. The DLS results are further supported by TEM of the sampled material from the same location. Thus it can be ascertained that though we are approaching towards cleaner cooking fuel by choosing LPG still there is existence of these ultrafine particle. These ultrafine particles are much more carcinogenic in nature than the fully grown soot particles, due to the presence of activated aromatic rings in them.

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