



# An experimental design for the investigation of water repellent property of candle soot particles



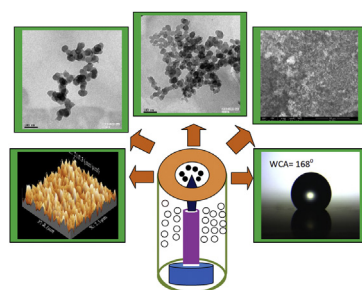
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## HIGHLIGHTS

- Mechanistic aspect for hydrophobicity of candle soot is demonstrated.
- Hydrophobicity of soot particles at different exposure time is described.
- Agglomeration of soot particles related to fractal dimension is reported.
- Mechanism of formation of soot particles in the candle flame is also described.

## GRAPHICAL ABSTRACT



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## ABSTRACT

The mechanistic aspect of candle soot particles under controlled atmosphere has been reported. The soot particles were characterized using Fourier transformation Infrared Spectroscopy, Raman spectroscopy, Transmission electron microscopy and X-ray diffraction. Hydrophobicity of the candle soot particles was confirmed from the presence of C–H group which enhances water repellency and can be used as filler material for fabrication of superhydrophobic coatings. The layered soot particle on the glass slide exhibits maximum water contact angle of 168°. Roughness of soot particle and various hydrophobic groups involved for obtaining superhydrophobicity were exposed. The Raman spectrum of soot particles revealed the presence of disorder graphene which was confirmed from appearance of D1 band. The agglomeration of candle soot particles has been discussed by measuring fractal dimension ( $D_f$ ) of the particles. The in-depth investigation for bringing the mechanism of formation of soot particle inside the flame reveals the inception of the first particles, growth of soot particles, particle coalescence, agglomeration and oxidation. Here, we have found that the mechanism of particle formation in candle flame involves various steps, in which the sintering as well as coalescence/collision process plays a major role.

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## 1. Introduction

Amongst all elements present in nature, carbon is the most abundant. The availability and application of different forms of

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carbon such as carbon black, coke, carbon nanotubes, carbon nanofibres, glassy carbon, active carbon, diamonds, fullerene etc. have stimulated the materials scientists to fabricate artificial carbon materials [1]. Soot is technically defined as the solid product of incomplete combustion of hydrocarbons in controlled atmosphere. Since, it acts as filler and pigments, now-a-days various paint industries actively involve it for manufacturing coatings for large scale applications [2]. On the other hand, soot particles are solid carbon rich materials and condense from the vapour phase during

combustion [3,4]. Carbon soot particles derived from various hydrocarbons and fuels are most elegant materials among all the carbon based materials. Earlier, it is noted that aerosol made soot particles derived from fossil or biomass burnt are chain like agglomerated particles, which are composed of graphitic layers of concentric nano onion like structure [5,6].

In general, aerosol made soot particles are ubiquitous in environment and comprise ~10–70% of the total atmospheric particulate matter. The importance of soot's morphology, structure, chemical analysis on atmosphere has been discussed by many authors to gather knowledge, which has become an area of immense interest in materials research. Similarly, the physicochemical properties of soot and different particles arising along with the soot particles in flame in the process of soot formation have been essence copious investigation for more than 100 years and such investigation have not vanished their significance to the present days [7].

Soot particles are being produced as industrial products in large scale (more than 100 tons per year) [7]. Till date, a large number of experiments have been proposed on the process of soot formation and various models have been projected to provide mechanism for the formation of soot particles. However, the exact mechanism for soot formation is yet improperly stated hence it needs to be understood. Earlier, authors have revealed some unique techniques for producing carbon soot particles by in-situ combustion of carbon rich materials. Veranth et al. have identified the un-burnt ash samples as the main source of production of soot particles [8]. Transformation of coal derived soot particles have been reported by Rigby et al. [9]. Brown and Fletcher have suggested model for predicting coal derived soot particles [10]. Similarly, soot particles derived from diesel engine have been proposed as the main object for industrial applications [11]. Kim et al. have addressed the comparative study of oxidative properties between flame and diesel soot particles and its interaction with metal particles [12]. Morphological properties and changes in chemical structure of in-situ diesel exhausts soot particles after treatment have been reported by Muller et al. [13,14]. Tian et al. reported production of soot particles using combustion process of natural gas [15].

Candle is a highly rich hydrocarbon materials derived from crude petroleum oil. Crude oil based paraffin wax materials with carbons range from C<sub>16</sub>–C<sub>30</sub> have been used as object for combustion to produce carbon soot particles. Study of the emission of carbon particle from combustion of burnt candle was reported by Li and co-authors [16–19]. Similarly, Liu et al. have reported the preparation of fluorescent carbon particle from candle soot [20]. Ray et al. have reported fluorescent carbon nanoparticles derived from candle wax which have been used for Bio imaging applications [21].

Herein, we adopt a new scientific technique to study the mechanism of formation of candle soot particle under controlled combustion. The physical properties of soot particles in terms of Fractal dimension, projected length to aggregation length of soot particle involved in agglomeration were quantified by using Transmission Electron microscopy. Similarly, the mechanism involving formation of coagulation and agglomerations of soot particles have been well established. Functional groups actively involved in providing hydrophobicity and surface roughness of soot particles were characterised using FT-IR and AFM respectively. This study aims at in-sights on the mechanistic aspects of superhydrophobicity of soot particles.

## 2. Experimental

### 2.1. Chemicals

Acetone (99.8%, Sigma Aldrich), Ethanol (99.9%, Sigma Aldrich), were all used as received. Candle of size of (15 × 2 cm, (L × D)) was

used as source for carbon soot particles without any further purification. Glass substrates (75 × 25 × 3 mm) were received from Fisher Scientific (India). Water was supplied by a Barnstead Nanopure Water System (18.3 M3 cm).

### 2.2. Procedure for cleaning glass substrates

Glass substrates were ultrasonicated (Sonicator Model- EI-6LH-SP, Sl. No- 1209-122, India) at 20 kHz and 20 W in 20 mL ethanol for 15 min followed by ultrasonication using deionised water for 10 min. These cleaned glass substrates were then used for collection of soot particles.

### 2.3. Synthesis of carbon soot particles

A candle of size of (15 × 2 cm (L × D)) was placed in a silica crucible and the entire assembly was kept inside a perforated polycarbonate chamber. The candle soot particles were collected on a glass substrate placed just above the flame for 5–10 s. The schematic for collection of soot particles is shown in Fig. 1. As shown in Fig. 1, a candle of size of (15 × 2 cm, (LXD)) is placed on a silica crucible.

Earlier, we have revealed a novel platform for the synthesis of catalyst free photo luminescent camphor soot particles for superhydrophobic application [22]. However, in this paper, we have proposed to examine the agglomeration/coagulation study of irregular nano spike structured candle soot particles for superhydrophobic applications. In order to exhibit the mechanistic studies of candle soot particle, the investigation was carried out in a perforated polycarbonate chamber. A candle of size of 15 × 2 cm (LXD) is placed in a silica crucible (25 mL) which is enclosed inside a perforated polycarbonate (PC) chamber with pore dimensions of 5 mm. The PC setup was then placed inside Haier Laminar Flow (Model: HR30:IIA2) during the soot collection process. The constant down flow velocity of 0.30 m s<sup>-1</sup> in the laminar, ensures controlled air atmosphere around the PC chamber. The perforations in the PC chamber enhance the air availability to facilitating lean combustion. Since the entire soot collection process is carried out in a designed PC chamber and in a controlled combustion environment (laminar), the sampling process is highly repeatable.

### 2.4. Characterization

Functional group analysis was carried out using Fourier transformation infrared (FTIR) spectrophotometer (Nicolet™-380) in the wavelength range 500–3000 cm<sup>-1</sup>. The morphology and particle size of the candle soot were examined using FESEM (JSM-6700F) and HR-TEM (FEI Tecnai TF20 operated at 200 kV). The HR-TEM

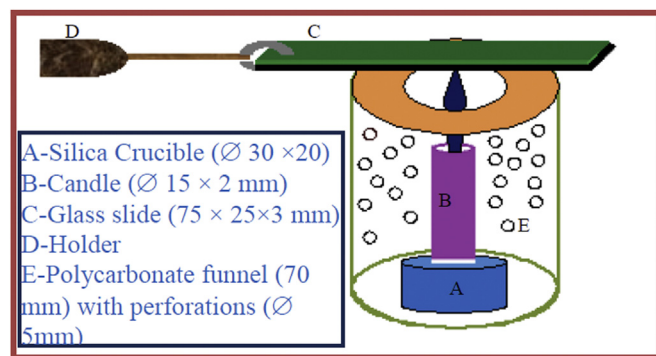


Fig. 1. Schematic shows the collection of soot particles from candle flame.

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