CHAPTER 4

SYNTHESIS AND CHARACTERISTICS OF DIESEL SOOT

4.1 Preparation of Diesel Soot

The real soot was prepared by partial combustion of locally available commercial diesel (HP) in a lamp with limited supply of air, and collected on the inner walls of an inverted beaker kept over the lamp as shown in figure 4.1. The soot was collected from the recipient walls of the beaker and then dried in an oven for overnight at 120^{o}C to remove moisture and volatile components. The dried soot was stored in an air tight bottle for further use in the experiments.

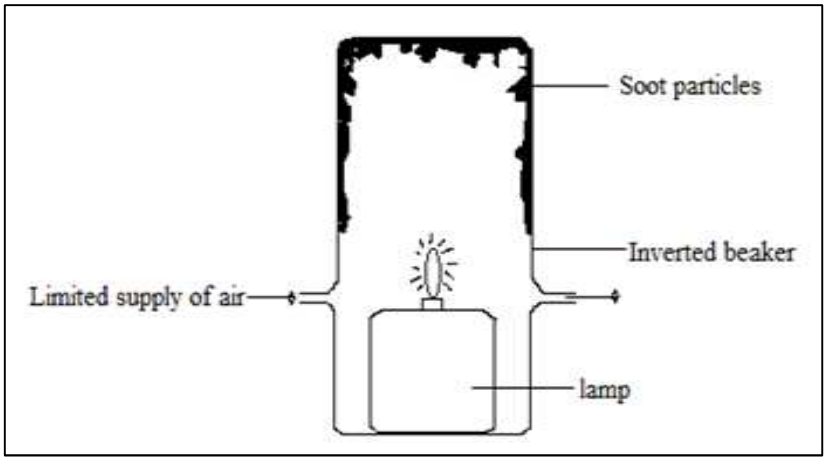

Figure 4.1 Schematic diagram of preparation of diesel soot

4.2 Diesel Soot Characterization

The prepared diesel soot was characterized by the following techniques:

4.2.1 Proximate Analysis

Proximate analysis of the laboratory prepared soot is reported in Table 4.1. The fixed carbon/VM ratio in the present study was found higher as compared to the value reported in the published paper for carbon/H_2 [Neri et al. 1997]. This difference is obvious because the techniques used to characterize the soot were different, further we have heated the prepared soot at 200^{o}C before the performing the proximate analysis, so some of the VM is already removed from the sample. The ash content in the prepared soot was 0.1wt%, which is far less than 7.1wt% reported in the literature [Neri et al. 1997]. This may be due to absence of lube-oil in the prepared soot. this shows that the main source of the ash is lube oil additives such as Ca, Zn, Mg, etc. ash plugs DPF hence it is a necessary evil added in the lube oil.

Table 4.1 Proximate analysis of soot

Constituents	wt%
Fixed carbon	77.9
VM	21.8
Moisture	0.2
Ash	0.1

4.2.2 Calorific Value

Calorific Value (CV) is a measure of heating power and is dependent upon the composition of the gas. The CV refers to the amount of energy released when a known weight of fuel is completely combusted under specified conditions. The calorific value of the soot was calculated using a bomb calorimeter under the following conditions:

Wt. of soot taken = 0.9902 gm, Water taken in the calorimeter =1750 mL, Pressure of O_2 in the bomb = 25 kg/cm^2. Room Temperature = 28.4 ^{o}C

Table 4.2 Data obtained from bomb calorimeter

Time (min)	0	1	3	4	5	6	7	8	9	10	11
ΔT (^oC)	0.01	0.41	0.96	1.22	1.34	1.42	1.45	1.48	1.48	1.52	1.52

The calorific value of the soot was calculated using the equation (4.1)

$$\mathbf{C_v = \{W * \Delta T - (C_{vt} + C_{vw})\}/ m} \qquad (4.1)$$

Where;

W (water equivalent) = 2248 cal/ ^oC

Heat produced by combustion of thread used in the experiment (C_{vt}) = 21 Cal./gm

Heat produced by burning of fuse wire used in the experiment (C_{vw}) = 9.31 Cal./gm

Substituting the corresponding values in the equation (4.1), the calorific value of the soot determined was = 3397.47 Cal./gm

4.2.3 Particle Size Analysis

Particle size analysis of the soot gives mean diameter of 1.30 μm based on particle number density as shown in figure (4.2).

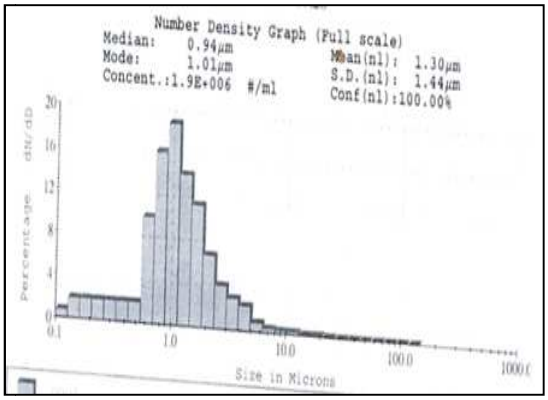

Figure 4.2 Particle size analysis based on particle number density

Whereas particles of 15.25 μm occupy maximum volume based on volume density as shown in figure (4.3). This result is obvious as discussed earlier section 3.4.1 that

number concentration of smaller particle is more while larger particle occupy more mass. Detailed analysis is given in appendix-A.

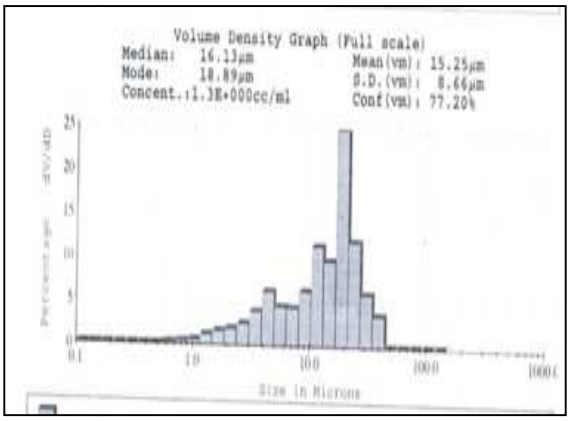

Figure 4.3 Particle size analysis based on particle volume density

4.2.4 X- ray Diffraction Analysis

The X-ray diffraction (XRD) pattern of diesel soot is shown in figure 4.4 The Bragg diffraction broad peaks at $2\theta = 23.68^{o}$ and 42.01^{o} are the only peaks obtained in the X-ray diffraction analysis. These two peaks correspond to hexagonal graphite lattice of multi-walled carbon nano-tubes [Malek Abbaslou et al. 2010, Mi et al. 2010].

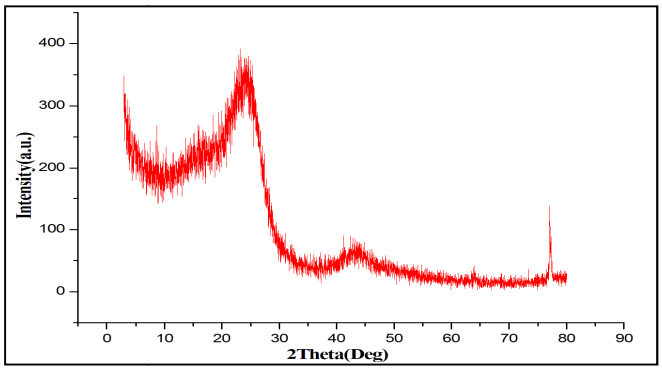

Figure 4.4 X-Ray pattern of the laboratory prepared diesel soot

The peaks at $2\theta = 23.68^{o}$ is a moderately high intensity broad peak which indicates the presence of large amounts of amorphous material in association with nano-tubes. The

low intensity broad peak at $2\theta = 42.01^{o}$, is an indication of the amorphous carbon nano-material present in the soot. The crystallite size of the soot was estimated to be 93.00 nm using the Scherrer equation (3.9). The crystallite size of 93.00 nm found in the present study was comparable with reported values in the literature (70-100 nm) [Kostoglou et al. 2003].

4.2.5 Scanning Electron Microscopy

The SEM micrograph of real diesel soot is presented in Figure 4.5. The surface morphology of the soot is seen to be non-uniform. There are several grains which look like carbon nano-spheres. These sphere join together to form chains of spheres. This chain like structure is seen throughout the surface. The SEM analyses show that the majority of the particle is about 0.3μm and less.

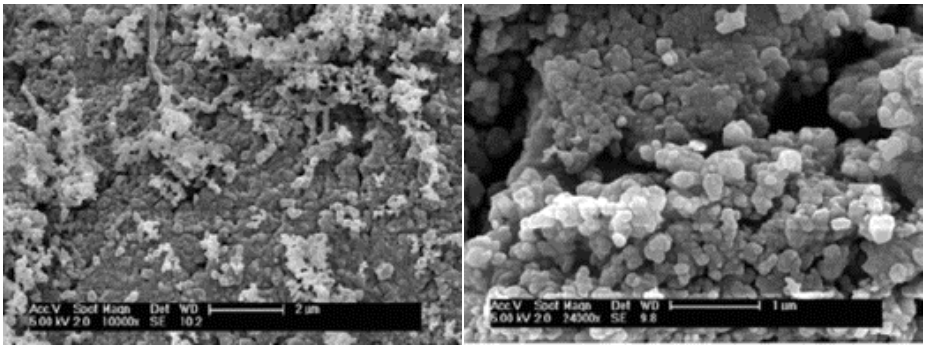

Figure 4.5 SEM images of the laboratory prepared diesel soot