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Determination of Particle Size Distribution of Used Black Tea Leaves by Scanning Electron Microscope

Mohammad Abul Hossain[*] and Shigeru Mori[2]

[1]*Department of Chemistry, University of Dhaka, Dhaka-1000, Bangladesh*

[2]*Graduate School of Natural Science and Technology, Kanazawa University, Kakuma-machi, Kanazawa 920-1192, Japan*

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Abstract

The particle size distribution (PSD) of a particulate material is important in understanding its physical and chemical properties. Determination of PSD is essential in many physicochemical processes and production quality depends on the measurement and control of PSD. Scanning electron microscope (SEM), in addition to sieve analysis, has been used to determine the PSD of biomass materials like used black tea leaves (UBTL). Spherical, ellipsoidal and non-spherical particles of used black tea leaves were observed in SEM images. The PSD of four different sizes ranges of spherical and non spherical UBTL particles was determined from the measurement of SEM images of length wise and width wise diameters. The results enabled to determine the mean diameter or representative diameters of particles of wide size ranges.

Key words: Scanning electron microscope, Used black tea leaves (UBTL), Particle size distribution, Geometric mean diameter

I. Introduction

The most important physical property of particulate samples is particle size distribution. Particle size has a direct influence on material properties such as: reactivity of catalysts, dissolution rate of tablets, stability in suspension of sediments and paints, texture and feel of food ingredients, appearance of powder coatings and inks, flowability and handling of granules, packing density and porosity of ceramics, etc. Measuring the of particle size distribution and understanding how it affects the processes are very important for research and development and for quality control in many relevant industries.

The particle-size distribution (PSD) of a powder, or granular material, or particles dispersed in fluid, is a list of values or a mathematical function that defines the relative amounts of particles present, sorted according to sizes[1]. Particle size is a geometric characteristic that is usually assigned to material objects with sizes on scale from nanometers to millimeters. Depending on the nature and size of particles, particle size measurement is routinely carried out across a wide range of techiniques such as sieve analysis, gravitational liquid sedimentation, air elutriation analysis, x- ray diffraction, laser diffraction[2]. Recently developed methods are optical granulometry[3], photoanalysis[4], light scattering[5] ultrasonic attenuation spectroscopy[6], zeta potential measurements[1], etc. There are multiple definitions of the particle size depending on the method of its measurement. Light scattering tends to overestimate contribution of large particles[5]. This is critical in many cases when dilution affects particle size distribution. Zeta potential measurement technique has important applications in a wide range of industries including; ceramics, pharmaceuticals, medicine, mineral processing, electronics, etc. Mastersizer and Zetasizer Nano particle size analysers are new techniques for measurement of rapid and precise particle size distributions of both wet and dry dispersions working over the millimetre, micrometre and nanometre particle size ranges.

Adsorption on activated carbon is one of the important processes for the removal of pollutants from municipal and industrial wastewaters. To minimize costs, recent research efforts have been directed towards the use of biomasses as adsorbent materials. Several authors have reported about the removal of both organic and inorganic pollutants from wastewater by biosorption processes using materials like hazelnut shell, nut shell, leaf mould, used coffee, and others[7]. Recently, we found that used black tea leaves (UBTL) has an excellent adsorptive capacity (q_e> 454 mg/g) for Cr(VI) in aqueous solutions[8, 9]. This is especially interesting for the removal of Cr(VI) from industrial wastewater. The effective utilization of an adsorption process depends on its mechanism, especially on transport mechanism, an important parameter which is influenced by the particle size of the adsorbent[10]. None of the above mentioned methods are applicable for the determination of particle size distribution of a biomass material like used black tea leaves. Scanning electron microscope, in addition to sieve analysis were chosen for the determination of particle size distribution of four different size ranges UBTL.

II. Experimental

Materials and Methods

Fresh black tea leaves were collected from Bangladesh Tea Research Institute, BTRI, Srimongle, Sylhet. Used black tea leaves (UBTL) were obtained after extracting tea liquor from

E-mail: hossainabul@yahoo.com

the fresh ones by boiling with distilled water for 8 hours. The residue of leaves was dried at 105 ^{o}C and ground. Ground UBTL residue was loaded into a series of 8-inch-diameter sieve trays (From top to bottom: 0.850 :: 0.710 :: 0.500 :: 0.250 :: 0.150 mm hole diameter). After shaking the residue in sieves, particles retained on the sieves were collected and classified into four groups as shown in Table 1. The average particle size of each category was determined by arithmetic mean of low and high values of the range. A normal picture of UBTL particles of category UBTL-*a* (0.15-0.25 mm) shown in Fig. 1a.

A high resolution scanning electron microscope (SEM; Hitachi, S-4500) was used to determine the particle size distribution of each of the four categories UBTL based on image analysis. The structural morphology showed that the UBTL particles were not spherical as shown in Fig. 1b. The length (diameter in *x*-axes direction of SEM) and width (diameter in *y*-axes direction of SEM) of 100 individual particles of category UBTL-*a*, sampled randomly, were measured by the SEM. From the above measurements, frequency histograms of the particle size distribution based on both length and width-wise diameters were determined and shown in Fig. 2a. Histogram of a lognormal particle size distribution is shown in Fig. 2b. Similarly, frequency histograms and lognormal histograms of particle size distributions for other three categories UBTL were also determined and shown in Figures 3-5.

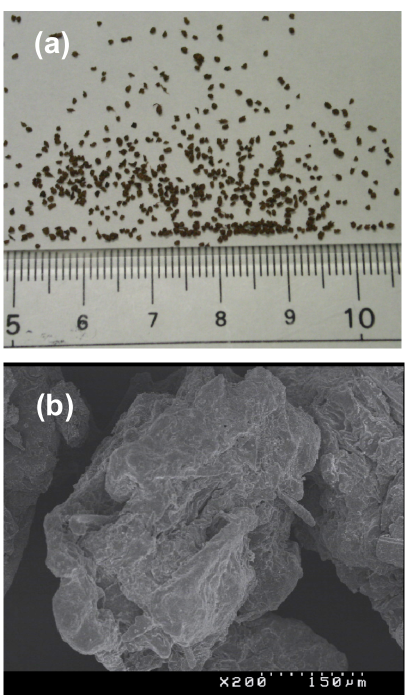

Fig. 1. Used black tea leaves particles at normal (a) and 200× magnification (b).

III. Results and Discussion

The particle size distribution or grain size distribution is one of the most important characteristics of particulate matter.

Sieve analysis of particle size

The PSD is usually defined by the method by which it is determined. The most easily understood method of determination is sieve analysis, where particles are separated on sieves of different sizes. The PSD may be expressed as a "range" analysis, in which the amount in each size range is listed in order. It may also be presented in "cumulative" form, in which the total of all sizes "retained" or "passed" by a single notional "sieve" is given for a range of sizes. Range analysis is suitable when a particular ideal mid-range particle size is being sought, while cumulative analysis is used where the amount of "under-size" or "over-size" needs to be controlled. The way in which "size" is expressed is open to a wide range of interpretations. A simple treatment assumes the particles are spheres that will just pass through a square hole in a "sieve". In the present case particles are irregular, and the way by which such particles are characterized during analysis is very dependent on the method of measurement. From the sieve analysis, we found the particles were of different ranges of sizes (Table-1). The size distribution or mean diameter could not determine.

SEM analysis of particle size distribution

To determine the particle size distribution of UBTL in the range of sizes, the structural morphology and the size of each particle was analyzed by scanning electron microscope. SEM provided information on the area projection particle size and particle shape in wide range of magnification. In Fig. 1b, SEM micrograph at 200× magnifications shows non-spherical shape of different particles in category $UBTL_a$ in the size range 0.15-0.25 mm diameter. In SEM, the particles were counted and width-wise and length-wise diameters in respective directions were measured. The number of particles in a predetermined size range was tabulated and was plotted histogram of particle size distribution (Fig. 2a). The histogram (shown in Figure 2a) is one of the simplest ways to display particle size distribution. It is the particle frequency distribution that shows the percentage of particles found in each size range.

Table. 1. UBTL particles of different size ranges obtained sieve analysis.

Category of UBTL	Ranges in size of UBTL (mm)	Average size of UBTL (mm)
UBTL-*a*	0.15 - 0.25	0.200
UBTL - *b*	0.25 - 0.50	0.325
UBTL - *c*	0.50 - 0.71	0.605
UBTL - *d*	0.71 - 0.85	0.780

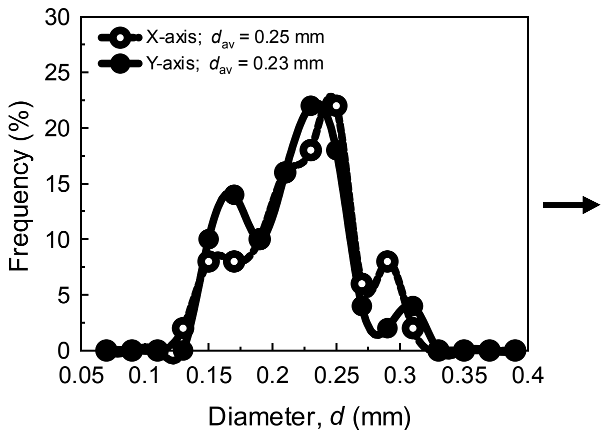

Fig. 2a. Histogram of UBTL-*a* particle size distribution in length and width diameters.

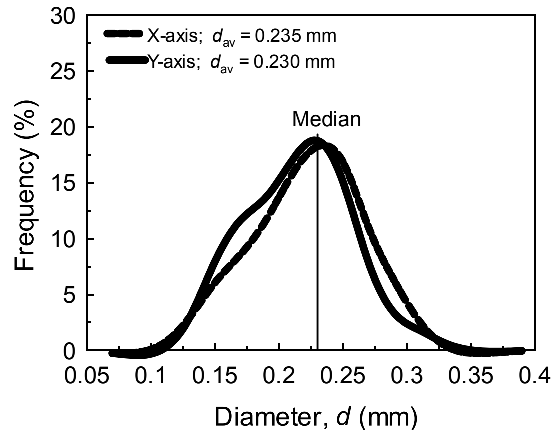

Fig. 2b Histogram of UBTL-*a* lognormal particle size distribution in length and width diameters.

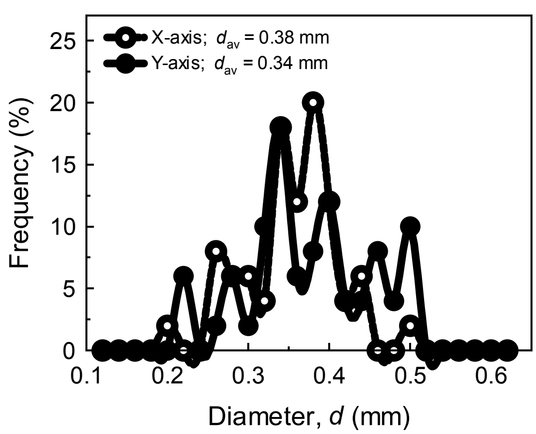

Fig. 3a. Histogram of UBTL-*b* particle size distribution in length and width diameters.

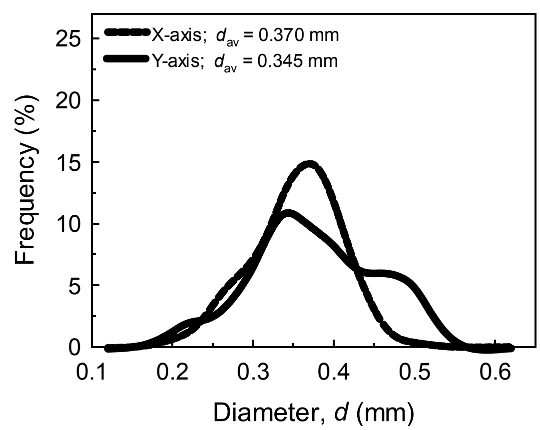

Fig. 3b. Histogram of UBTL-*b* lognormal particle size distribution in length and width diameters.

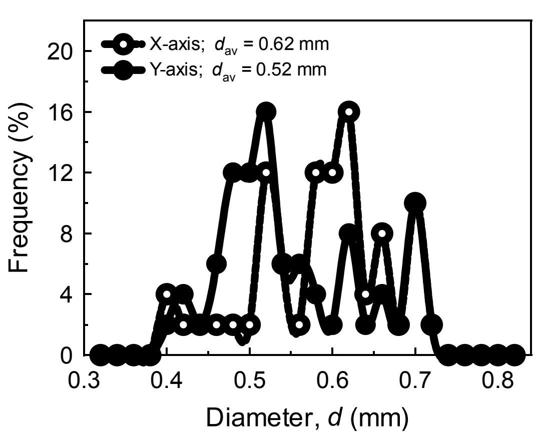

Fig. 4a. Histogram of UBTL-*c* particle size distribution in length and width diameters.

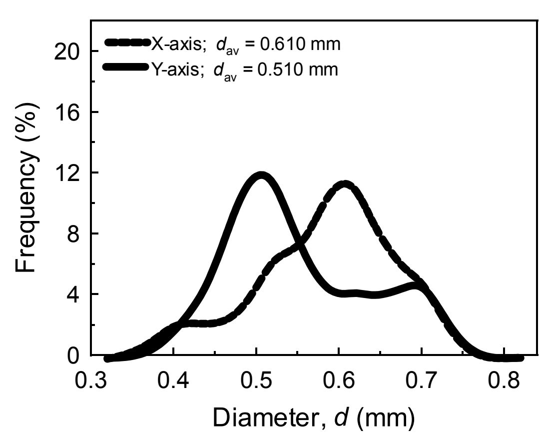

Fig. 4b. Histogram of UBTL-*c* lognormal particle size distribution in length and width diameters.

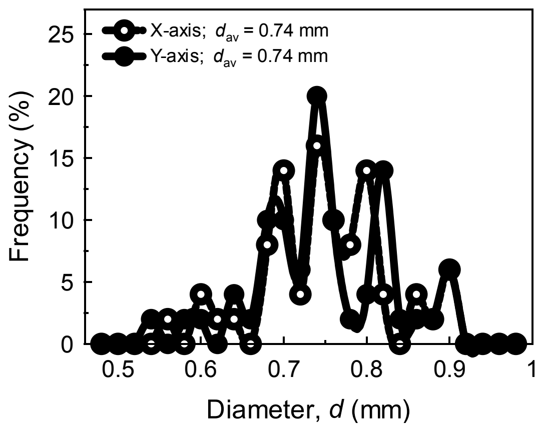

Fig. 5a. Histogram of UBTL-*d* particle size distribution in length and width diameters.

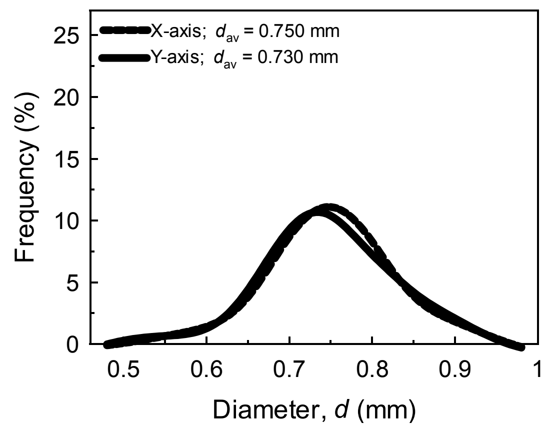

Fig. 5b. Histogram of UBTL-*d* lognormal particle size distribution in length and width diameters.

Table. 2. Diameters of different size ranges UBTL particles obtained from SEM analysis.

Category of UBTL	Ranges in size of UBTL (mm)	Histogram of particle size distribution			Histogram of lognormal particle size distribution		
		Average mean diameter (d_{av})		Rep. mean diameter $d_{rep}=\sqrt[3]{d_{xav}d_{yav}^2}$ (mm)	Average mean diameter (d_{ev})		Rep. mean diameter $d_{rep}=\sqrt[3]{d_{xav}d_{yav}^2}$ (mm)
		x-axis (mm)	*y*-axis (mm)		*x*-axis (mm)	*y*-axis (mm)	
UBTL-*a*	0.15 - 0.25	0.25	0.23	0.236	0.235	0.230	0.232
UBTL - *b*	0.25 - 0.50	0.38	0.34	0.353	0.370	0.345	0.353
UBTL - *c*	0.50 - 0.71	0.62	0.52	0.551	0.610	0.510	0.542
UBTL - *d*	0.71 - 0.85	0.74	0.74	0.740	0.750	0.730	0.737

Lognormal size distribution

When the particle diameters in Figure 2a are curve fitted on a logarithmic scale against the frequency of occurrence, a bell-shaped curve is generated. As shown in Figure 2b, the particle size of category UBTL-*a* is found to be altered and produced equidistant ranges when plotted on a logarithmic basis. This bell-shaped histogram is called a lognormal curve. For many manmade sources, the observed particulate matter distribution approximates a lognormal distribution. Therefore, it is often beneficial to work with particle size distributions on a logarithmic basis.

Mean diameter

The arithmetic mean diameter, usually termed as the mean diameter, is the arithmetic average particle diameter of the distribution. The value of the arithmetic mean is sensitive to the quantities of particulate matter at the extreme lower and upper ends of the distribution. The mode represents the value that occurs most frequently in a distribution. In particle size distributions, the mode is the particle diameter that occurs most frequently. The dimensions of UBTL particles in *x*- and *y*- directions are not equal, i.e. the prepared particles are not spherical. The geometric or equivalent mean diameter or representative particle size diameter in range, d_{rep} ($=\sqrt[3]{d_{xav}d_{yav}^2}$), of category's UBTL-*a* was calculated [11] from average length and width wise (respective figure and Table 2). The representative particle size diameters or geometric mean diameters, obtained from both histograms of particle size distribution (Fig. 2a) and lognormal particle size distribution (Fig. 2b), and are given in Table 2. represents the value that occurs most frequently in a distribution. In particle size distributions, the mode is the particle diameter that occurs most frequently. The dimensions of UBTL particles in *x*- and *y*- directions are not equal, i.e. the prepared particles are not spherical. The geometric or equivalent mean diameter or representative particle size diameter in range, d_{rep} ($=\sqrt[3]{d_{xav}d_{yav}^2}$), of category's UBTL-*a* was calculated [11] from average length and width wise (respective figure and Table 2). The representative particle size diameters or geometric mean diameters, obtained from both histograms of particle size distribution (Fig. 2a) and lognormal particle size distribution (Fig. 2b), and are given in Table 2. Other results are shown in Figures 3-5. The respective representative particle size diameters/geometric mean diameters of other three categories are given in Table 2. The geometric/mean diameters measured by SEM are reliable and can be used for the evaluation of adsorption property or other activity of the biomass materials like used black tea leaves.

IV. Conclusion

SEM is a useful tool for accurately determining size distribution as well as the mean diameters of a range of particles sizes. It is simple in principle, easy to operate and the results directly represent the size distribution of spherical and non spherical particles. The length wise and width wise diameters so obtained lead to the determination of mean diameter of particles.

Acknowledgements

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