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An Investigation of Size Deposition Upon Individual Stages of a Cascade Impactor*

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ABSTRACT

The particle size deposition characteristics upon each stage of an inertial impactor have been investigated. Disodium fluorescein and kaolin samples of approximately the same median diameter and distribution were collected by inertial impaction. The Delron DCI-6 cascade impactor was used conventionally, with 38 mm diameter glass slides, and a modified arrangement, with 11 mm diameter glass slides placed concentrically within the larger ones. Samples could thus be separated into those depositing at the center of the slide and those depositing at the outer edge. Quantitative and qualitative analyses were performed. It was concluded that size fractionation upon a stage , that is large particles at the center and smaller ones at the outer edge,

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does occur, however this is complicated by the incidence of bounce and blow off. Further investigations employing monodispersed powders would enable the contribution of the latter phenomenon to be assessed.

INTRODUCTION

The inertial impaction of aerosol particles and droplets has been employed in their characterization for many years [1-3]. The problems associated with the interpretation of data obtained from devices which employ impaction have been considered in some detail [4,5]. The issues which have been of most concern are the incidence of bounce and blow off at each stage and wall losses between stages, which contribute to errors in size classification.

Inertial impaction sampling devices employ surfaces placed in a moving air stream to collect aerosol particles and droplets. The phenomenon of impaction arises as the persistence of an aerosol particle or droplet to move in an initial direction of flow overcomes the obstacle of a change in the direction of air flow. The change in air flow direction may be the result of a difference in the position of the walls of the tube, through which it is being channeled, or the insertion of an obstacle (e.g.plate, sphere or cylinder baffle) in the path of the airflow. There may be a size dependency of these deposits depending on their position on the stage.

It was the purpose of these studies to investigate deposition upon each stage with a view to improving the precision of the data collected and its usefulness in deriving the parameters describing the particle size and distribution.

METHODS

Preparation of Aerosol Powders

Two model substances were employed in these studies. Disodium fluorescein (DF; Fisher Scientific, Springfield, NJ) was micronized, to 3-5 µm particles, using an air jet mill (Trost Gem-T research jet mill, Garlock Inc., Plastomer Products, Newtown, PA). Kaolin (Aluminum silicate hydroxide, Fisher scientific, Springfield, NJ) was used as it was supplied by the manufacturer. Both of these materials were dried in a vacuum oven, DF at 40 ^{o}C and Kaolin at 120 ^{o}C, and subsequently stored over dessicant (Silica gel, Fisher Scientific, Springfield, NJ) prior to preparation or generation.

A quantity of 0.15g of DF was levigated with 0.21g of sorbitan trioleate (Span 85, Fluka AG, Ronkonkoma, NY,[7]). 0.24g of paste were weighed into a plastic-coated glass pressure resistant bottle (Wheaton Glass, Mays Landing, NJ) and a mixture of chlorofluorocarbon propellants 11, 12 and 114 (Dymels, Dupont, Wilmington, DE, in a 1:2:1 ratio respectively) were added, up to a total mass of 10g. The final product was, therefore, a 1% suspension of DF [7]. Propellants were packed under pressure using small scale aerosol pressure packaging equipment (Pamasol, Pfaffikon, Switzerland [7,8]) for filling.

DF and kaolin were employed to assess the quantitative and qualitative performance, respectively, of the cascade impactor .

Generation of Aerosol Powders

Figure 1a shows the arrangement of the pressure packed DF suspension container with respect to the actuator through which it is delivered. A metering chamber within the 25µl inverted metering valve (Valois DF30, BLM Packaging Inc., Greenwich, CT) isolates a known volume of propellant and suspended particles. Upon actuation this chamber is opened to the atmosphere where the vapor rapidly equilibrates to ambient pressure projecting the suspended particles, associated surfactant and unevaporated propellant through the jet in the direction of the actuator outlet. A single sample consisted of 4 actuations into the cascade impactor.

Figure 1b shows the arrangement of a dry powder sieving system for generating kaolin powder. The kaolin was a free flowing fine powder following drying. The position of the sieves slowed the flow of the powder and enabled deaggregation under the influence of a vibration source.

Collection by Inertial Impaction

Figure 2 shows a schematic diagram of a Delron DCI-6 cascade impactor (Delron Products, Powell, OH).

This device consists of six jets and an absolute filter. The glass inlet at the top of the impactor was a modification [8] employed for sampling metered doses of DF.

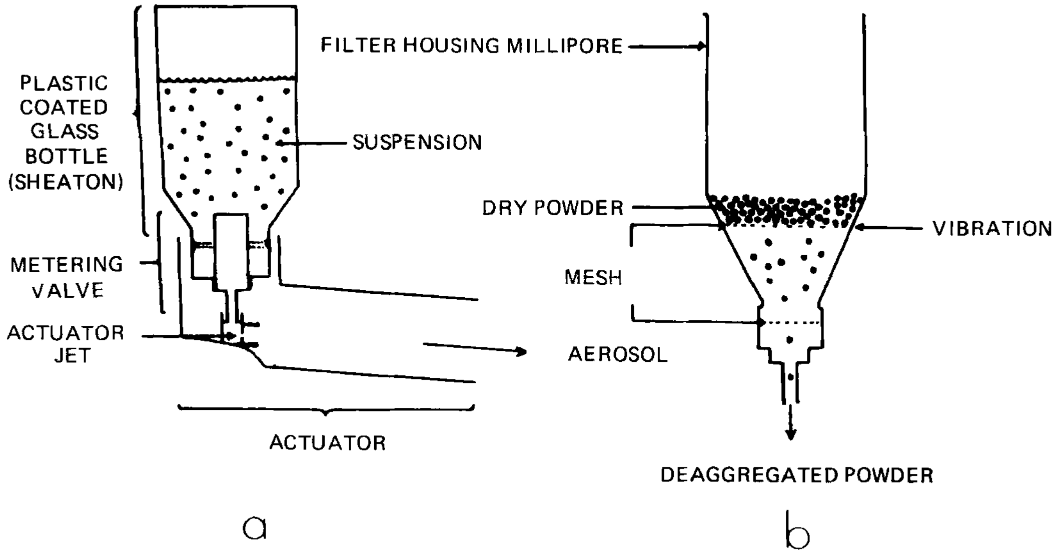

Figure 1: Diagram of a) metered dose inhaler containing disodium fluorescein suspension, indicating the container, valve and actuator and; b) the hopper and sieves employed for the generation of a dry kaolin powder aerosol.

Kaolin was generated directly into the open cascade impactor.

The schematic illustrates the conventional positioning of 38mm diameter circular glass slides immediately beneath air jets. The jet sizes decrease in diameter as the air passes through successive stages of the impactor. Similarly the distance to the impaction surface, the slide, becomes smaller.

It was the purpose of these studies to investigate the behavior of particles upon each stage of the impactor. Each stage from 2 to 5 was modified by placing an 11mm diameter circular thin glass slide concentrically upon each 38mm

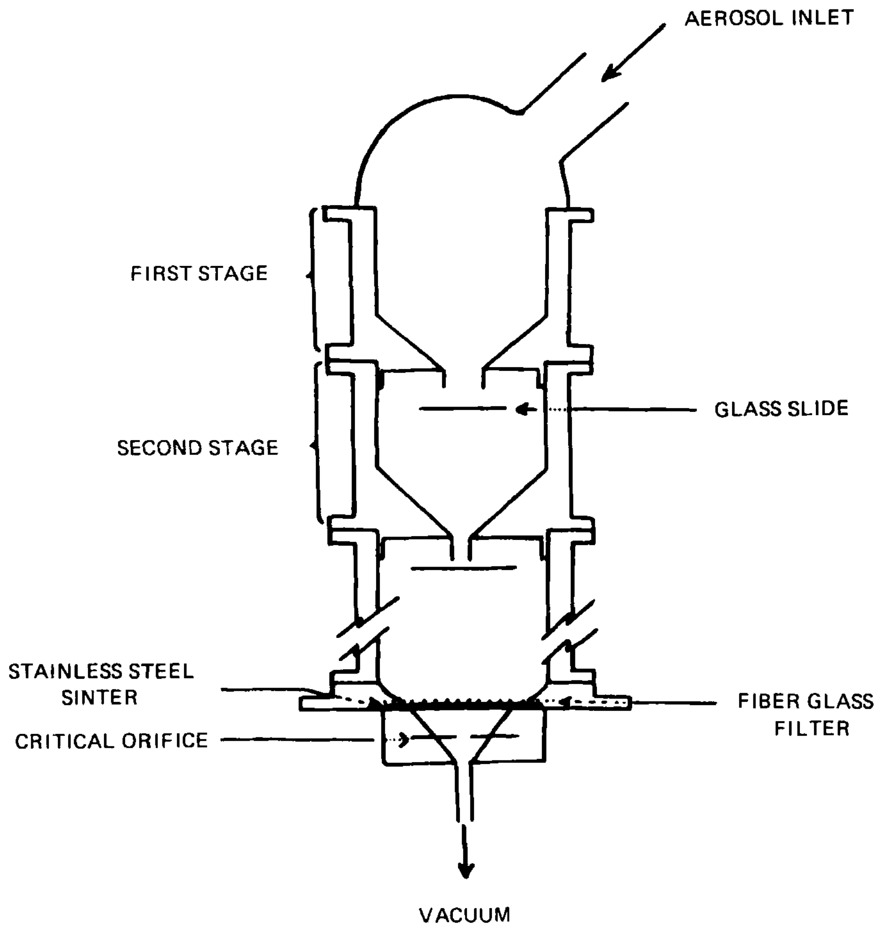

Figure 2: Diagram of the Delron DCI-6 cascade impactor

slide at each stage. Upon completion of sampling of the aerosols the small slide was removed from the centre of the large slide and the material collected was analyzed separately.

Spectroscopic Analysis of DF

DF was dissolved in phosphate buffer pH 7.4 and analyzed spectrophotometrically at 488.5 nm (Cary 118, Varian

Instument Co., Sunnyvale, CA, [9]). Thus a quantitative analysis was performed.

Electron Microscopy

Scanning electron microscopy was performed on gold coated samples at 20kV (Hitachi Scientific Instruments, Mountain View, CA). Kaolin samples were employed for these qualitative studies.

Laser Light Diffraction

Laser light diffraction (Malvern Instruments, Malvern, U.K.) was performed on samples of kaolin dispersed in water using a small quantity of sodium dodecyl sulfate (Fisher Scientific, Springfield, NJ). The selection of kaolin for the qualitative analyses was influenced by the quantitative similarity of its particle size characteristics to those of DF. As an inert material, in the absence of a spectroscopic method, gravimetric analysis would have been the option for quantitative assessment of this material using cascade impaction. This method requires greater sensitivity than may be obtained using conventional laboratory balances. Laser light diffraction was an independent method of accurately assessing the particle size characteristics of the kaolin sample.

RESULTS

Generation of Powders

Both the generation of DF and kaolin resulted in large losses in the first stage of the impactor. Table 1 shows the

TABLE 1: Total deposition (µg) in the actuator,glass housing and impactor, conventional and modified arrangement, expressed as mean and standard deviation (n=3).

SITE OF DEPOSITION	TOTAL DEPOSITION (µg) IN IMPACTOR	
	CONVENTIONAL	MODIFIED
Actuator	586.0 ± 23.6	639.9 ± 146.8
Glass housing	559.9 ± 59.4	597.1 ± 53.8
Impactor	427.6 ± 27.1	440.0 ± 26.7
TOTAL	1573.4 ± 58.4	1677.0 ± 177.8

total deposition in the actuator, glass housing and the impactor of DF.

The overall amount recovered was approximately 80% (1600µg/2000µg) of the mass administered based on the original formulation . This may be attributed most notably to losses in the first stage of the impactor which acts as an impaction surface.

Wall losses occur throughout the impactor and contribute to the shortfall in aerosol powder collected [10]. A significant proportion, approximately 75% of the total aerosol output collected was lost prior to entering the impactor. This may be caused by aggregation and/or by the high inertia of droplets upon leaving the actuator jet. The results of these phenomena are premature deposition in the actuator, the glass housing and the upper stages of the impactor.

Collection by Inertial Impaction

Figure 3 shows the appearance of the slides following the collection of DF samples. This illustrates the minor influence which the presence of the small slides have upon the overall deposition upon each stage. Table 2 shows that there was no significant effect ($p<0.05$, t-test [11]) of the extra slides upon the amount deposited on each stage or on the distribution between stages.

Table 3 shows the percentage of the total amount deposited in the impactor appearing at each stage. Stage 2 was modified, as illustrated in Figure 3. The data from this stage has not been divided into the amount deposited on each of the concentric slides. The large number of particles depositing on this stage did not allow individual discrimination upon microscopic examination.

The mass median aerodynamic diameter and the geometric standard deviation of the DF aerosol powder were 6.49 μm and 2.01 respectively, as estimated from conventional impactor data, and 6.38 μm and 2.00 from the modified impactor data, shown in Table 2. Transforming the data by expressing the cumulative percentage undersize in probits (standard deviations) and the natural logarithms of the diameter enables linear regression analysis to be performed. The correlation coefficient for the regression analysis of the conventional and modified data were 0.972 and 0.976 respectively. Using the modified impactor data in its entirety, as shown in Table 3, a similar result was obtained to that indicated above. The median diameter was 6.1 μm

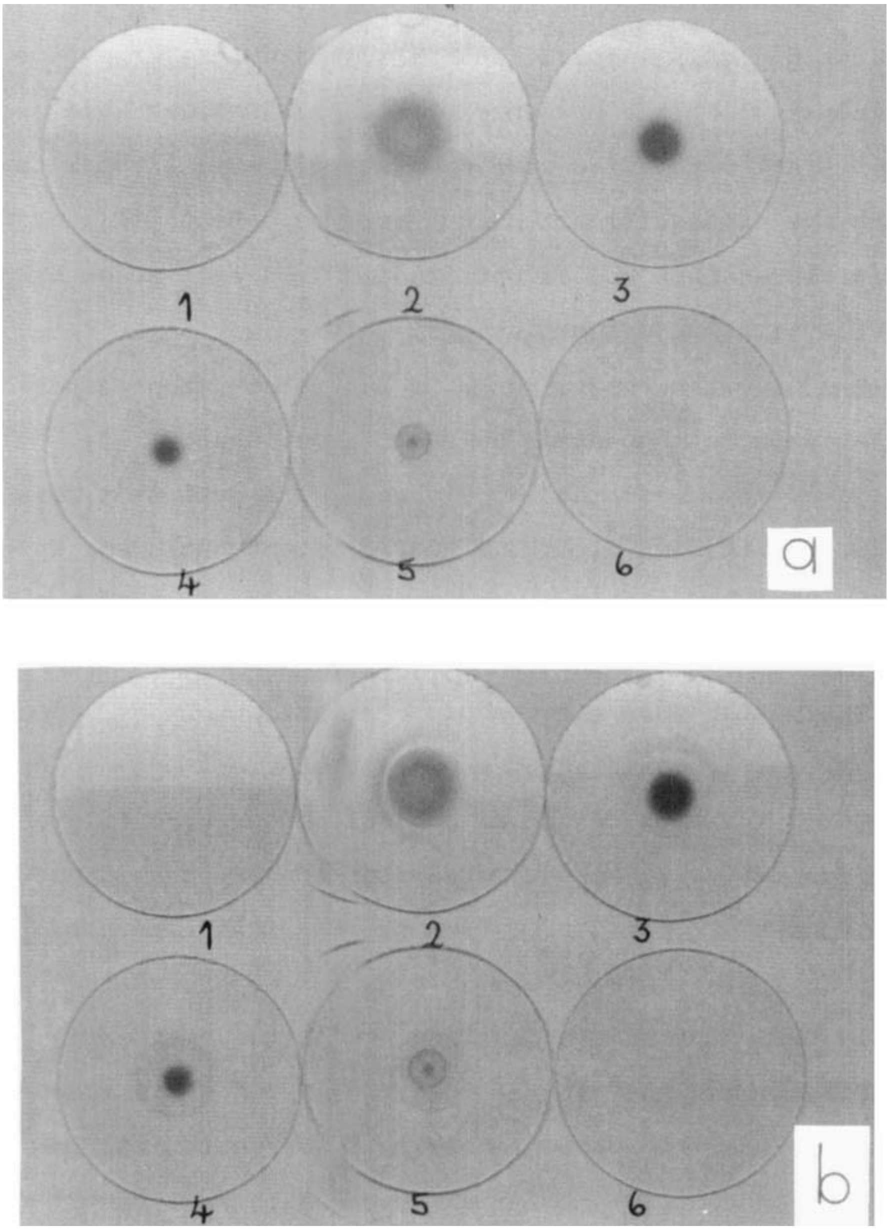

Figure 3: Photographs of the collection slides from the cascade impactor in the a) conventional and b) modified arrangement.

TABLE 2: Total deposition (µg) on each stage of the impactor in both conventional and modified arrangement, expressed as mean and standard deviation (n=3).

STAGE	*D_{50} (µm)	TOTAL DEPOSITION (µg) IN IMPACTOR	
		CONVENTIONAL	MODIFIED
1	11.2	68.4 ± 5.7	64.4 ± 8.2
2	5.5	173.6 ± 13.7	187.8 ± 13.1
3	3.3	155.9 ± 10.1	157.0 ± 7.9
4	2.0	22.6 ± 0.6	22.7 ± 1.2
5	1.1	4.7 ± 0.2	6.6 ± 1.0
6	0.5	2.1 ± 0.5	1.3 ± 0.4
Filter	0.2	0.3 ± 0.1	0.2 ± 0.2
TOTAL	-	427.6 ± 27.1	440.0 ± 26.7

*Median diameter, Gonda et al., 1981, [12].

Spectroscopic Analysis of DF

TABLE 3: Percentage undersize for the total deposition of DF upon each slide (conventional or modified) in the cascade impactor expressed as mean and standard deviation (n=3).

STAGE	PARTICLE SIZE (µm) [1]D	PERCENTAGE UNDERSIZE	
		CONVENTIONAL	MODIFIED
1	>10.5	16.01 ± 1.01	14.61 ± 1.3
2	10.5	40.56 ± 1.43	42.66 ± 0.48
3	5.1	36.46 ± 0.44	-
3.1(IN)	*5.1	-	31.62 ± 1.67
3.2(OUT)	*3.2	-	4.12 ± 0.33
4	3.0	5.31 ± 0.3	-
4.1(I)	*2.1	-	4.84 ± 0.15
4.2(O)	*1.9	-	0.31 ± 0.19
5	2.0	1.1 ± 0.04	-
5.1(I)	*1.3	-	1.32 ± 0.24
5.2(O)	*0.8	-	0.17 ± 0.08
6	0.7	0.48 ± 0.1	0.3 ± 0.07
Filter	0.5	0.07 ± 0.02	0.04 ± 0.04

[1]Gonda et al., 1981 [12] ;
*Estimated microscopically using kaolin data and converted to aerodynamic diameter using a density of 3.6g/cm^3.

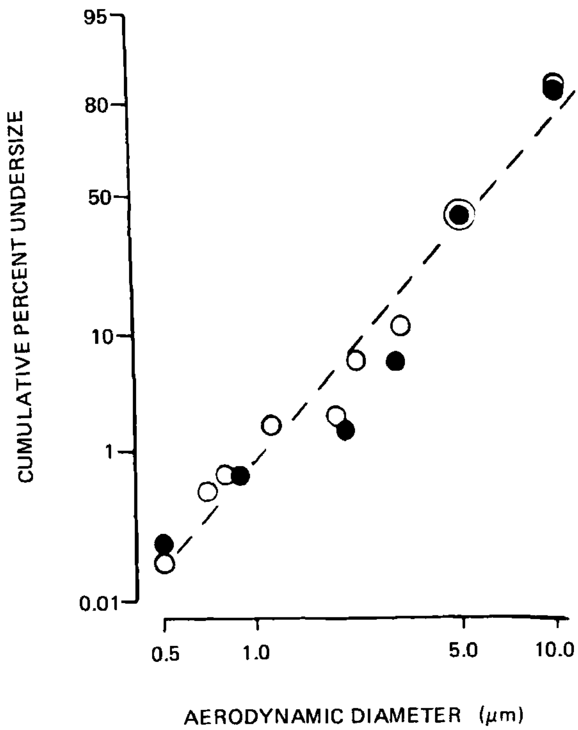

Figure 4: A logarithmic-probability plot of cumulative percentage undersize against particle size (aerodynamic diameter, μm), for conventional [●] and modified [-o-] impactor data.

while the geometric standard deviation is 2.08. The correlation coefficient for the linear regression analysis of the transformed data was 0.984.

The line fit to the modified data obtained from the impactor as expressed by the correlation coefficient appears to be more precise than that to the conventional impactor data. This is illustrated in Figure 4.

Electron Microscopy

Figures 5a and 5b show scanning electron micrographs of the appearance of kaolin particles on the inner and outer edge of the slide at stage 4 of the cascade impactor. The particles on the inner stage were 2-4μm in diameter while those on the outer edge were 1-2μm in diameter. Figures 6a and 6b show scanning electron micrographs of the appearance of kaolin particles on the inner and outer edge of the slide at stage 5 of the cascade impactor. A greater proportion of the particles on the inner stage were <1μm than those on the outer edge.

Laser Light Diffraction

The particle size distribution of the kaolin powder was expressed in volume diameters and converted to aerodynamic diameters using the density of kaolin ($Da = \sqrt{\rho}\ Dv$; ρ = 3.6 g/cm^3 [13]). The correlation coefficient to the log-probability transformed data was 0.999 indicating linearity and thus a log-normal particle size distribution. The mass median aerodynamic diameter of kaolin was 5.81 um and the geometric standard deviation was 2.06 which was similar to the same parameters reported for DF.

DISCUSSION

In the original reports of secondary deposits upon cascade impactor slides [6] it was suggested that the most likely causes of this phenomenon were particle size fractionation and bounce and blow off effects. In the present studies dry powder aerosols have been collected upon uncoated glass

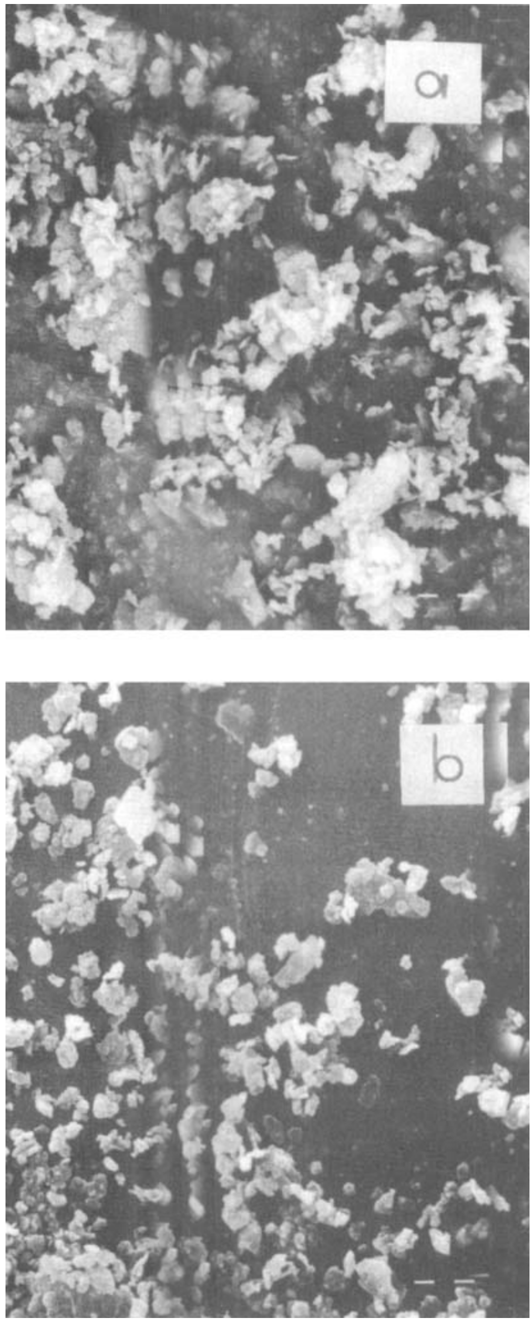

Figure 5: Scanning electron micrographs of kaolin particles collected at a) the centre and b) the outer edge of the collection slide at stage 4 of the cascade impactor. Each bar, lower right, represents a measure of 1 um.

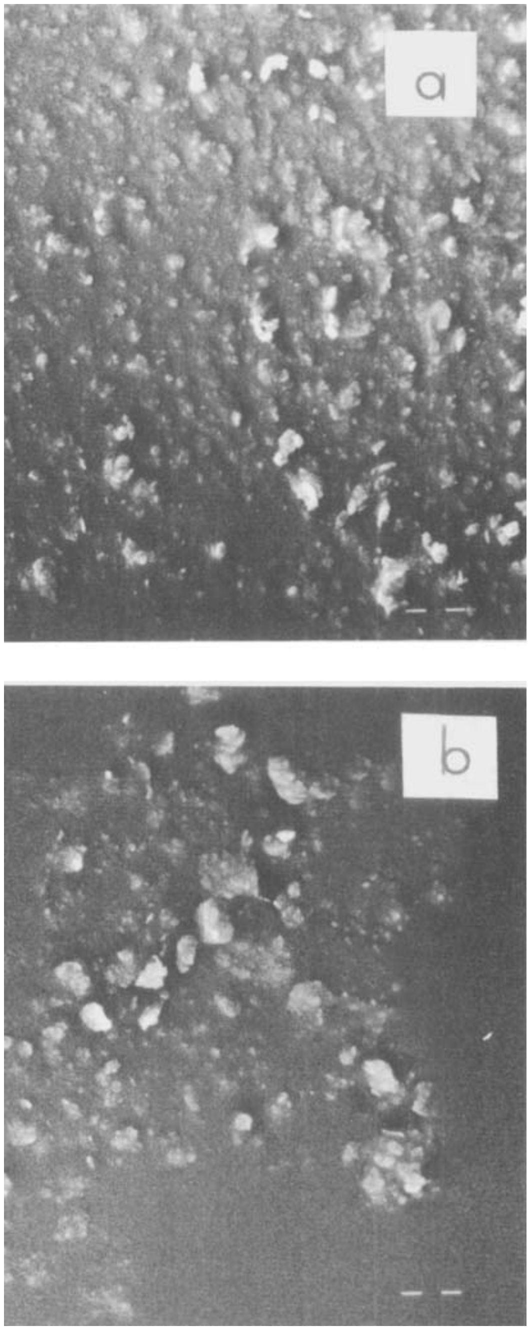

Figure 6: Scanning electron micrographs of kaolin particles collected at a) the centre and b) the outer edge of the collection slide at stage 5 of the cascade impactor. Each bar, lower right, represents a measure of 1 µm.

surfaces. Coated collection surfaces have been shown to reduce the effects of bounce and blow off [14,15]. Thus each of the effects leading to secondary deposits may express themselves in the arrangement chosen for the present studies. The presence of an additional collection surface does not appear to disrupt the flow characteristics over the surface of the slides enough to alter the particulate deposition.

The plot of the data obtained from the cascade impactor, assuming a log-normal distribution, indicates that extra data points for the calculation of the particle size parameters may be derived from the modified impactor. Examining the scanning electron micrographs of kaolin, Figures 5 and 6, the pattern of size fractionation, large particles at the centre and small particles at the outer edge of the slide, is indicated as shown on stage 4. Stage 5, however, shows a reversal of the size deposition indicating the prevalence of bounce and blow off.

It is likely that the effect of bounce and blow off contributed to deposition in the upper stages but were minor in comparison with size effects.

In order to investigate this phenomenon further monodisperse aerosols [16] may be employed to calibrate the system.

CONCLUSIONS

Studies of the behavior of particles upon each stage of a cascade impactor have shown that size fractionation does occur, however, this was complicated by bounce and blow off effects. These observations confirm previous reports [6]. It may be possible to obtain a greater number of data points within the sampling range of a single jet cascade impactor using the technique suggested. Thus, more precise estimates of the particle size parameters may be obtained.

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