

Particle size analysis of amalgam powder and handpiece generated specimens

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Abstract

Objectives: The increasing interest in the elimination of amalgam particles from the dental waste (DW) stream, requires efficient devices to remove these particles. The major objective of this project was to perform a comparative evaluation of five basic methods of particle size analysis in terms of the instrument's ability to quantify the size distribution of the various components within the DW stream.

Methods: The analytical techniques chosen were image analysis via scanning electron microscopy, standard wire mesh sieves, X-ray sedimentation, laser diffraction, and electrozone analysis. The DW particle stream components were represented by amalgam powders and handpiece/diamond bur generated specimens of enamel; dentin, whole tooth, and condensed amalgam.

Results: Each analytical method quantified the examined DW particle stream components. However, X-ray sedimentation, electrozone, and laser diffraction particle analyses provided similar results for determining particle distributions of DW samples. These three methods were able to more clearly quantify the properties of the examined powder and condensed amalgam samples. Furthermore, these methods indicated that a significant fraction of the DW stream contains particles less than 20 μm .

Significance: The findings of this study indicated that the electrozone method is likely to be the most effective technique for quantifying the particle size distribution in the DW particle stream. This method required a relative small volume of sample, was not affected by density, shape factors or optical properties, and measured a sufficient number of particles to provide a reliable representation of the particle size distribution curve. © 2001 Academy of Dental Materials. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Amalgam powder; Particle size analysis

1. Introduction

1.1. Dental waste-water discharge studies

Over the past 150 years, amalgam has been used extensively as a tooth filling material. Currently, amalgam accounts for 70–80% of all direct tooth restorations [1]. In the United States it is estimated that 90–100 tons of amalgam are used yearly in dental restorative work [1]. Until recently, the lack of reliable information about the mercury in the dental waste (DW) stream and a lenient regulatory framework for small quantity generators (such as dental offices) helped to obscure the significance of this waste stream. The current conditions and perceptions of the environmental significance of mercury are rapidly changing

leading to mandatory treatment requirements in Europe and more stringent regulations in the United States.

Waste from dental offices contains amalgam particles with sizes ranging from large visible particles to a sub-micron colloidal suspension. Silver and mercury are the two major constituents in dental amalgam. In the DW stream, the dental amalgam may be mixed with water, oral fluids, dentin, enamel, blood, mouthwash, and disinfectants. The in-line vacuum trap retains a substantial amount of the DW particles. The remaining portion in the DW stream may be collected for disposal or may eventually end up in the sewer system. Treatment at the source of this waste stream is not yet mandated in the USA. Nevertheless, the discharge of the DW stream into the sewer system from many individual sources (i.e., dental clinics) may influence the performance of wastewater treatment facilities and their ability to meet their National Pollution Discharge Elimination System (NPDES) permit requirements. Dental clinics are suspected to be contributing a

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significant percentage of the total mercury load to local wastewater treatment facilities [2–4]. In recent years, many sanitary districts have had difficulties complying with existing or anticipated requirements of their NPDES permits concerning priority pollutants such as mercury. Unregulated small quantity generators, such as dental clinics, may be one of the sources causing these compliance problems.

Arenholt-Bindslev and Larsen [5] have studied the effect of amalgam particle separators installed in dental clinics on the mercury load to WW treatment facilities. Ten clinics with amalgam particle separation equipment and ten clinics without treatment equipment were investigated. The study included 40 full-time and 3 part-time dentists. The amalgam particle separators which were used met the German and Dutch removal criteria (i.e., at least 95% amalgam particle retention) [6,7]. The ten clinics without particle separation equipment had an average output of 250 mg Hg/day per dentist; whereas, in the ten clinics with the separation equipment installed the mercury output was significantly reduced (i.e., output of 35 mg Hg/day per dentist). A second study [4] of mercury levels in the sludge from WW treatment facilities after the installation of amalgam particle separators in contributing dental clinics resulted in over a 50% reduction of the mercury content. This study was conducted in an area where there were no known sources of mercury other than the dental clinics. A report by Fan et al. [8] reinforced this concept i.e., ‘in locations where other sources of mercury discharge have been substantially reduced or are virtually eliminated, reduction of mercury discharge from dental offices may make a notable difference.’

1.2. Particle size distribution analysis

The current ISO Standard 11143, Dental Equipment-Amalgam Separators, is focused on the evaluation of devices that will reduce the number of amalgam particles before the dental wastewater stream enters the sewer system [9]. The test sample consists of triturated amalgam particles, ground such, that by weight 6 g are between 3150 and 500 μm in diameter, 1 g between 500 and 100 μm in diameter, and 3 g less than 100 μm in diameter. This sample is based on a number of investigations on actual dental treatment centers (American, Dutch, and German); however no American references are provided and limited data on the actual determinations are lacking.

A number of DW particle separation devices (centrifugal, sedimentation, filter, and combinations of these types) are commercially available to remove mercury-laden particles from this waste stream. A critical factor for assessing the efficiency of such devices is the particle size distribution curve of the DW particle stream before and after treatment (i.e., removal). The aim of this study was to perform a comparative evaluation of five basic particle size analysis methods, using uncondensed amalgam powders in addition

to simulated (lab generated) DW particle stream components. A critical parameter was the potential of each analytical technique to quantify the low-end range of each sample’s particle size distribution curve. The low particle size range is likely to be the determining factor of removal efficiency since the large end of the size distribution of the DW reaching the separator will already have been removed by the in-line screen and associated plumbing of the vacuum waste system.

Scanning electron microscopy (SEM) with manual sizing techniques, sieves, X-ray sedimentography, laser diffraction and electrozone analyses were selected as the basic methods for determining the various particle size distribution curves. Each technique belongs to one of two basic classes of sizing instrumentation i.e., ensemble analyzers or stream counters. Ensemble devices such as the laser diffraction analyzer, sieves and X-ray sedimentography can process grams of suspension (millions of particles) in a single analysis. SEM coupled image analysis and the electrozone technique belong to the class of stream counters. These two devices analyze individual particles and therefore they are also capable of determining the concentration of particles per ml of suspension fluid.

2. Analytical techniques

2.1. SEM/image analysis

The techniques associated with SEM are well known and do not need to be mentioned here. Modern SEM devices can be fitted with image analysis capability in order to conduct particle size distribution analysis. However, the methodology requires some very specific considerations on sample loading and dispersion. Ideally, all particles must be separated from each other so as to avoid coincidence effects, which result in undercounting and over sizing. The dynamic range of the sample must not be large or the concept of Area Normalization must be used to blend data from many fields of view at different magnifications. Generally, particle sizing via SEM should be limited to rather narrow distributions of particulate or the count data can easily be biased towards the fines. On a wide distribution sample, i.e. 0.2–100 microns, one simply cannot count enough particles to adequately define the distribution curve on a Mass basis. Statistically missing one or two large particles from some number of random fields of view will seriously bias the mass distribution towards the fines.

2.2. Sieves

Sieving is the oldest type of technique selected for this work. It is the reference method by which other techniques are often judged (including electrozone). Since it is so common, it is readily available, easy to use and very inexpensive compared to the other analytical methods. Data can be generated by a wet or dry sieving routine. Generally, wire

mesh sieves are available from stock over a range of 50,000 microns down through about 38 microns. The manufacturing quality is consistent through the application of numerous ASTM sieve standards [10].

Normally, one can dry sieve down to the 38-micron level without excessive difficulty. Below this level, electrostatic forces limit one's ability to dry sieve properly. However, in this case study, the particles were capable of effectively being dry sieved at the 20-micron level, presumably due to the lack of electrostatic forces associated with these sample types. One advantage of this method is that large sample sizes can be utilized, thereby increasing representation of the bulk material. Normally, on 8-inch diameter sieves, 100 g of sample can be utilized. As sample size drops, smaller diameter sieves can be used to increase powder recovery and weighing accuracy. When sample size is very small, plastic frame sieves with stainless steel mesh sizes can be weighed directly on an analytical balance to eliminate sample transfer errors.

The major disadvantage of sieves is that the data is sensitive to particle shape. The square mesh opening can easily and efficiently retain spherical particles, but as the aspect ratio (length/diameter) increases, data bias increases. In a similar fashion, as a particle system becomes more plate like, data bias increases, because plate-like particles can pass through a given mesh size on the diagonal dimension of the square opening.

2.3. X-ray sedimentography

Among the various methods for measuring particle size distributions, sedimentation using Stoke's Law, is a long established and reliable technique. This measurement yields particle diameters, which are equivalent to spheres settling in a fluid at the same rate as the measured particles. These devices employ a narrow, low energy X-ray beam that scans the particles as they settle in the sample cell. By scanning from the bottom to the top of the sample cell at optimum rates, analysis times are reduced, usually to a few minutes. The attenuation of the X-ray beam determines the percent mass of the various size particles while the beam position and time defines the particle diameter. Particles ranging from 300 through about 0.1 μm are automatically measured with this class of instrumentation [11].

2.4. Laser diffraction

A laser diffraction particle size analyzer comprises a helium neon laser beam, which has been spatially filtered and collimated to produce a clean parallel beam of light. The beam is then focused down by a Fourier or reverse Fourier lens to a point at the center of a detector which consists of a large number of photosensitive segments radiating outward from the center, increasing in size as they do so. If no particles pass through the laser beam, all the light falls on to a detector known as an obscuration detector. However, as soon as a particle enters the beam it scatters light at an

angle inversely proportional to its size. A particular property of the Fourier lens is that it collects the scattering light from an ensemble of particles and overlays the common angles of scattering on the detector array. This overlaid data is known as a 'light energy distribution'. There is a direct relationship between this and the particle size distribution, which gave rise to it. The range for a majority of laser diffraction analyses is generally about 0.1–1000 microns diameter. Therefore, the technology is well qualified to span the analytical range of DW stream components.

2.5. Electrozone

The above mentioned classic analytical techniques were chosen for their diversity of physical characterization principles. The electrozone is the only technique that determines the envelope volume of a particle in three dimensions. The measurement is not dependent upon the color, transparency, refractive index, density, conductivity or to a large degree, particle shape. The technique determines the displaced volume of a particle suspended in a conductive fluid, as it travels through a cylindrical aperture. The displaced conductive electrolyte results in a change in resistance across the aperture upon which is impressed a constant current. Since the particle traverses the aperture in about 100 μs , a voltage pulse is produced. The magnitude of the pulse is proportional to particle volume. Normally, the analytical range of the technique is 0.5–400 microns. At least 100,000 particles are counted and sized, thereby producing a Frequency Distribution analysis. The resulting distribution is then converted to a Mass Distribution and reported on the basis of 'Equivalent Spherical Diameter'.

3. Materials and methods

To perform a comparative evaluation of five basic particle size analysis methods, amalgam powders and components of the DW particle stream (bur-ground samples of enamel, dentin, whole tooth, and condensed amalgam in teeth) were examined (see Table 1 for more details). This experimental approach was selected in order to avoid potential interferences from certain actual DW sample components (e.g., microbiological, saliva, proteins, disinfectants, mouthwash, etc.) and concurrently, determine the base distribution properties of the DW particle stream components. This approach allowed for a simulated sample generated under clinical conditions (using a high speed handpiece to removed condensed amalgam in extracted teeth) to be generated and then divided to have essentially the same sample analyzed by each of the particle analysis methods. Actual calibration of particle size data is based on spherical particles. Unfortunately specimens from dental wastewater are not spherical. This approach utilized the same prepared samples for each particle size analyzer technique to allow comparison of the five techniques.

A second approach was to use amalgam particles from

Table 1
Materials analyzed

Code	Material	Description
V	Valiant	A spherical amalgam powder
VP	Valiant PhD	An admixed amalgam powder
VT	Valiant	Condensed, set amalgam removed from extracted teeth using a diamond bur in a high speed handpiece
VPT	Valiant PhD	Condensed, set amalgam removed from extracted teeth using a diamond bur in a high speed handpiece
D	Dentin	Dentin removed from extracted teeth using a diamond bur in a high speed handpiece
E	Enamel	Enamel removed from extracted teeth using a diamond bur in a high speed handpiece
T	Whole tooth	Enamel and dentin removed from extracted teeth using a diamond bur in a high speed handpiece

double spill amalgam capsules as an empirical approach to evaluate the analysis methods. The assumption was made that the particle distribution of the amalgam powders would be fairly uniform and their particle size distribution curve expected to have a narrow range near the peak of the curve; i.e., majority of particles in a narrow range near the mode of the curve. Since one of the amalgams was a spherical amalgam and the second amalgam powder a mixture of spherical and admixed amalgam, this approach would allow a more theoretical comparison of the particle analysis techniques.

Each of these selected methods has an optimum analytical particle size range, and all were relatively good at measuring particles in the size range of the alloys (i.e., approximately 0.5–100 μm). Two amalgam alloys were investigated (V) Valiant (Kerr Dental, Romulus, MI), a spherical alloy; and (VP) Valiant PhD (Kerr Dental, Romulus, MI), a phase-dispersed alloy of the same composition as Valiant with the addition of lathe cut particles. For the present study, the two different types of dental amalgam powders, V and VP, were condensed into extracted third molars to produce two bur-ground samples of admixed and spherical dental amalgam, VT and VPT (see Table 1). Bur-ground samples of dentin (D), enamel (E), and a mixture of enamel and dentin (T) were also examined to provide greater detail of the DW particle stream components. More specifically the sample preparation procedures and instrument analysis methods were as follows.

4. Sample preparation

4.1. Amalgam powders (V and VP)

Double capsule amalgam powders were selected at random from a stock supply and opened just prior to starting each type of particle size analysis. The entire contents of each capsule was placed into a glass vial and mixed well by tumbling to promote a homogeneous state. Each analytical technique required a different quantity of the blended

powder base to be scoop sampled just prior to the final dispersion step and analysis.

4.2. Tooth preparation (E, D, and T)

To evaluate the size of tooth particles generated by typical preparation methods, approximately 90 extracted third molar teeth were prepared using a medium grit diamond chamfer bur (Diatech, Inc., New York, NY) in a conventional high speed handpiece (Midwest Quiet Air, Chicago, Illinois USA) at 350,000 rpm with water spray. Burs were replaced whenever they became clinically dull. The three individual tooth layers were collected directly into a container, bypassing the normal vacuum system of the dental unit.

The enamel particles were removed from the teeth, without intrusion, into the dentin-enamel junction. The teeth previously ground for enamel collection were further processed into dentin particulate. A separate group of teeth were prepared as close to the pulp chamber as was practical, collecting both the dentin and the enamel fraction. These prepared samples were pooled within in each group (enamel, dentin, and whole tooth) and then divided for analysis by each of the five methods. The division of the sample consisted of placing the collecting container into an ultrasonic bath for 5 min and then immediately pouring the contents, with stirring, into five separate containers.

5. Amalgamated sample preparation (VT and VPT)

To mimic clinical conditions as much as possible, approximately 80 extracted third molars were prepared with large occlusal, Class I, amalgam preparations, following standard operative procedures. Half of the teeth were restored with V, and the other half with VP, according to manufacturer's instructions. Resin bonding agents were not used, and the samples were aged for 6 weeks. The amalgam restorations were removed using the same bur type as above. Every effort was made to avoid removing additional tooth structure. During removal, occasionally large pieces of amalgam fractured off. These particles were weighed following the sieving, however they were removed prior to instrumental analysis for two reasons: (1) to avoid instrument damage, and, (2) normally these fractured off large particles would be trapped on the in-line screen filter (trap). These large particles accounted for 10–12% of the total weight of amalgam (i.e., particle size greater than 500 μm). This approach of removing the particles greater than 500 μm was based on a simple assumption. Since the same handpiece and bur type was used for each amalgam removal, the distribution of these particles would be essentially the same for all amalgam removal specimens. The removal technique therefore allows for a relative comparison for the particle distributions under 500 μm . These prepared samples were pooled within each amalgam and then divided for analysis by each of the five methods.

Table 2
Analysis as weight percent less than or equal to indicated size of material analyzed

Instrumental method	Materials analyzed						
	V	VP	D	E	T	VT	VPT
<i>Electrozone</i>							
Range (μm)	0.5–100.1	0.5–100.1	0.2–90.6	0.1–33.4	0.2–80.0	1.9–50.0	1.0–50.0
10th percentile (μm)	5.3	4.7	1.5	1.3	1.5	2.7	2.3
50th percentile (μm)	15.1	16.3	7.3	4.1	5.9	8.4	7.1
90th percentile (μm)	30.7	31.5	23.0	10.6	17.7	18.8	16.6
Span	1.7	1.6	2.9	2.3	2.8	1.9	2.0
<i>Laser diffraction</i>							
Range (μm)	0.1–130.0	0.1–130.0	0.1–80.0	0.1–80.0	0.1–80.0	0.2–68.5	1.4–77.6
10th percentile (μm)	6.8	7.2	1.7	0.5	0.7	2.4	2.1
50th percentile (μm)	15.6	17.3	7.5	3.0	4.6	11.6	9.2
90th percentile (μm)	29.3	35.4	26.1	8.7	15.5	35.7	30.6
Span	1.4	1.6	3.3	2.7	3.2	2.9	3.1
<i>X-ray sedimentography</i>							
Range (μm)	4.8–75.4	0.4–94.9	0.4–37.8	0.2–18.9	0.4–119.4	0.4–119.4	0.7–92.7
10th percentile (μm)	6.9	3.3	0.4	0.3	0.4	2.1	2.1
50th percentile (μm)	12.5	12.2	17.3	2.1	4.2	8.1	7.2
90th percentile (μm)	28.1	25.6	23.0	7.5	15.0	18.0	13.4
Span	1.7	1.8	1.3	3.4	3.5	2.0	1.6
<i>Sieves</i>							
Range (μm)	< 20– > 212	< 20– > 500	< 20	< 20	< 20	< 20– > 500	< 20– > 500
10th percentile (μm)	13.0	12.0	N/A	N/A	N/A	12.5	15.0
50th percentile (μm)	22.0	20.5	N/A	N/A	N/A	23.0	27.0
90th percentile (μm)	38.2	35.0	N/A	N/A	N/A	800	1400
Span	1.1	1.1				34.2	51.2
<i>Scanning Electron Microscopy</i>							
Range (μm)	0.1–17.0	0.1–17.0					
10th percentile (μm)	7.9	6.6					
50th percentile (μm)	12.0	11.1					
90th percentile (μm)	15.0	18.9					
Span	0.5	1.1					

6. Instrumental analysis

6.1. SEM

To assure proper field calibration and aid in counting and size discrimination of particles, an internal standard was prepared for each sample by attaching small squares of Mylar diffraction grating to the SEM stub with carbon tape. Depending on the size distribution of the sample, two diffraction gratings were used: (a) an interference-type grating with a d-spacing of 4.2 μm (6000 lines/inch, Edmund Scientific, NJ), and (b) a replica grating with a d-spacing of 1.1 μm (23,000 lines/inch, Edmund Scientific, NJ). A single, non-triturated capsule of Valiant as well as a single capsule of Valiant PhD amalgam powder was opened and each emptied into a separate screw cap vial. A small amount of particulate from each vial was sprinkled on the stub mount and then sputter-coated with gold-palladium alloy for approximately 100 s.

Four photographs of Valiant (V) alloy were taken at a magnification of 1100 \times and two photographs were taken of Valiant PhD alloy at 1100 \times using a JEOL 35C SEM (JOEL USA, Peabody, MA) at 10 kV. Low accelerating voltages were necessary to prevent ablation of the Mylar

film. Each set of photographs was examined by two skilled observers using a millimeter grid to size classify the particles. Approximately 500 particles were evaluated for the longest dimension and tabulated as a Frequency of Occurrence versus Size. Next, the midpoint of each size range was raised to the third power and multiplied by the number of particles in each class. A Cumulative Mass Distribution for each sample was generated and plotted on log probability paper. The data at the 10, 50 and 90th percentile levels are presented in Table 2.

6.2. Sieve analysis

Seven stainless steel 8-inch diameter sieves corresponding to 500, 300, 212, 106, 53, 32 and 20 μm , and a pan to catch fines smaller than 20 μm were used in this test series. Dry sieving is not normally used below 20 microns. The alloy contents of approximately 30 double capsules of V and VP were weighed, and gently sieved by hand. An anti-static brush was used as necessary to control static. Weights of sample residue left on each screen were recorded and the resulting data was plotted as a Cumulative Weight Percent Greater Than Or Equal To the Indicated Size. Data

at the 10, 50 and 90th percentile was determined from this plot.

6.3. X-ray sedimentography

All analyses were performed using a Microscan II X-ray Sedigrapher (Quantachrome, Boynton Beach, FL). Samples were preconditioned by sieving through a 300 μm sieve since material larger than this size cannot be analyzed by sedimentation. The minus 300-micron fraction was wetted in a small quantity of 50% glycerol in distilled water using a few drops of 0.1% aqueous Calgon (Sigma Chemical Co., St. Louis, MO) as a wetting and dispersing agent. The suspension was then ultrasonicated for a timed 2 min in a standard laboratory ultrasonic bath to achieve a dispersed state. Data was collected as percent transmission versus time over the range from 300 to 0.3 μm using an appropriate suspension media, and converted by the software package to a Differential Percentage Histogram as well as to a Cumulative Weight Percent versus Size. Literature values or manufacturers' specified data were used for all particulate densities.

6.4. Laser diffraction

A commercial Federal Drug Administration (FDA) registered fine particle laboratory (Particle Technology Labs, Downers Grove, IL) was used for this analysis method. The instrument was a Malvern Mastersizer S Laser Diffractometer (Malvern Inc. Burrough, MA), with an analytical range of 0.1–800 μm . Amalgam and alloy samples were dispersed in a Niaproof-4 (an anionic surfactant) solution, diluted with water and ultrasonicated for 2 min in a standard laboratory ultrasonic bath. Tooth samples were dispersed in 1% aqueous Daxad-11 and Niaproof-4 solution, once again using the ultrasonic bath to complete the dispersion just prior to analysis.

6.5. Electrozone analysis

A commercial FDA registered fine particle laboratory (Particle Technology Labs) was used also for this analysis method. Samples were analyzed using an Elzone computerized particle size analyzer (Elzone 112PC, Micromeritics, Norcross, GA). Each sample was wetted in a mix of 1% Daxad -11 and Niaproof-4 solutions. The resulting suspensions were ultrasonicated for 2 min in a standard laboratory ultrasonic bath to complete the dispersions. Following the dispersion step, an aliquot portion of each sample was added to 0.2 micron filtered, 2% by weight saline solution (electrolyte) just prior to each analysis. The aliquot samples were added to electrolyte in such a manner so as to achieve a particle coincidence level at the detector of 1–2% for each analysis. The resulting suspensions were analyzed in accordance with standard operating procedures for broad range samples with this instrumentation type. Data was collected over a population count of approximately 250,000 particles

and was then formatted by the software package to yield mass distribution statistics on the basis of equivalent spherical diameter.

The electrozone (Coulter Counter) analytical technique has been used since the late 1950s as industries leading instrumental methodology and it is well known that metal particles behave as non-conductors. It is possible to render metal particles as well as blood cells conductive, but specific analytical conditions need to be present (very high current density in aperture). If an aperture over voltage situation does occur, the results are dramatic. Particles traversing the detection aperture in an over voltage situation are undersized not oversized.

7. Instrumental comments

The approach employed in this project was to take essentially identical specimens and compare the outputs from five different methods of particle analysis. There was no standard used since the only means to obtain the same result from different instrument types is to use opaque spheres, a condition not found in dental wastewater. ISO standard 11143 uses triturated amalgam particles, ground such that by weight 6 g are between 3150 and 500 μm in diameter, 1 g between 500 and 100 μm in diameter, and 3 g less than 100 μm in diameter. Since the grinding will probably be different in each laboratory, the only consistency in these samples is the weight. There is no consistency between particle size, shape, or weight of the particles or number of particles of the same size or weight. This project used powder amalgams and handpiece generated specimens to mimic the dental wastewater stream. The approach utilized in this project allowed a comparison between a known shape, the amalgam powder, and the same material ground by a handpiece. The techniques were evaluated on completeness of the sample analysis and the representation of the analyzed specimen.

8. Results

Descriptive statistics from the results of the analyses by each instrument are presented in Table 2. For most techniques seven sample types were analyzed. Data is presented on a Mass Percent Less Than or Equal To The Indicated Size at the 10, 50 and 90th percent levels along with the Span. The span is defined as the difference between the particle diameter at the 90th percentile minus the diameter at the 10th percentile divided by the particle diameter at the 50th percentile. This value gives a relative span of the sizes of the particles in the sample.

The SEM analysis was only completed for the two amalgam alloy powders. A visual representation of the results, except for SEM, is provided in Figs. 1–7. In these figures, the distribution of the particle sizes is plotted against weight percent providing a comparison between the amalgam

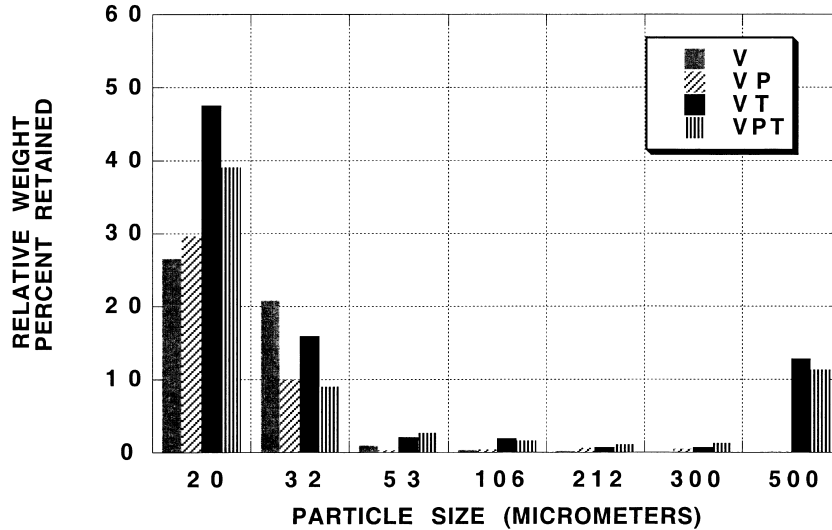


Fig. 1. Sieve analysis of amalgam powder (V and VP) and condensed amalgam (VT and VPT).

powders, the removed amalgams, and the tooth components. Fig. 1 represents the results from the sieve determination of the amalgam powder, V and VP, and removed amalgam (VT and VPT). The extracted tooth specimens, E, D and T did not yield a meaningful distribution curve because all particles were less than 20 µm. The X-ray sedigraphy analysis results are depicted in Figs. 2 and 3, laser diffraction, in Figs. 4 and 5, and the electrozone analysis results, in Figs. 6 and 7.

9. Discussion

For each analytical method, a substantial portion of each aliquot sample was used in the analysis, except for the SEM approach. Despite the efforts to standardize field magnification and various parameters associated with a well dispersed, uniform layer of particulate matter, SEM proved

to be the least suitable method for determining particle size distribution curves of the DW stream components. All the other methods examined a much larger and broader portion of the DW sample. SEM, on the other hand, counted less than 500 particles per sample at a given magnification. Since the field of view is very small at 1100×, (lower detection limit of about 1.0 µm) large particles that contain all the mass are easily missed in a half dozen or so random fields of view. Since it takes 1,000,000 one micron diameter particles to equal the same mass as one particle 100 microns diameter, missing a few large particles by count seriously biases the mass distribution towards the fines. Further, errors are introduced because small particles tended to settle under or on top of other particles. For the spherical alloys, it was relatively easy to determine the edges of adjacent particles. However, samples with rough and fractured

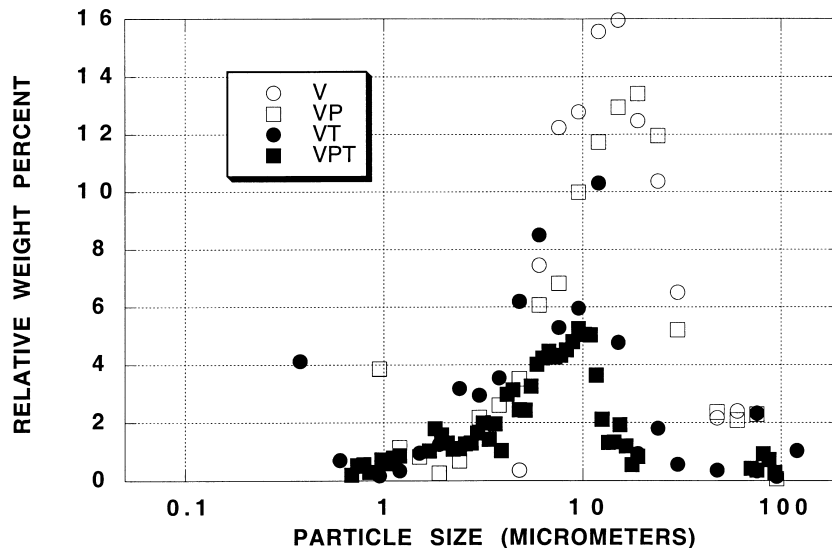


Fig. 2. X-ray sedigraphy analysis of amalgam powder (V and VP) and condensed amalgam (VT and VPT).

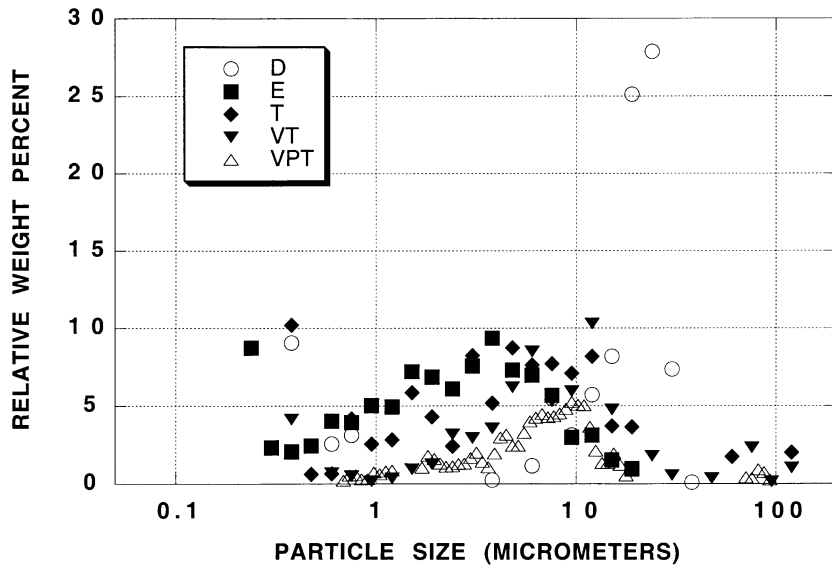


Fig. 3. X-ray sedigraphy analysis of ground specimens of enamel (E), dentin (D), whole tooth (T), and amalgam (VT and VPT).

edges, render this determination very difficult. SEM appears to be only good for well defined specimens that have a particle size distribution with a relatively narrow range. In addition to the above mentioned statistical sampling and counting problems, the work is very labor intensive, tedious and expensive.

Our data reveals that a 20 μm sieve is larger than the median size for the DW samples tested by the various instrumental techniques. Therefore, it would seem impractical to examine the DW particle stream components using sieves. A powder sample large enough to leave a significant quantity retained on sieves larger than 20 μm , would encompass hundreds of teeth. The sieve method, like the SEM, provided primarily qualitative data. One advantage of the sieve method is its ability to quantify the distribution

of gross particulate collected at a typical dental clinic waste drain during a series of routine dental procedures.

In general, electrozone analysis, X-ray sedigraphy, and laser diffraction all gave relatively similar estimates (see Table 2). However, the electrozone and laser diffraction analysis methods were able to demonstrate the expected size distribution properties of the amalgam powders (i.e., narrow range near the mode of the distribution curve). These instruments also generated the expected distinct curves for the condensed amalgam samples removed with a diamond bur (i.e., VT and VPT; Figs. 4–7). The X-ray sedigraphy results failed to manifest such characteristics (see Figs. 2 and 3). All three instrumental methods use different physical characterization/detection methods along with individually unique data reduction algorithms

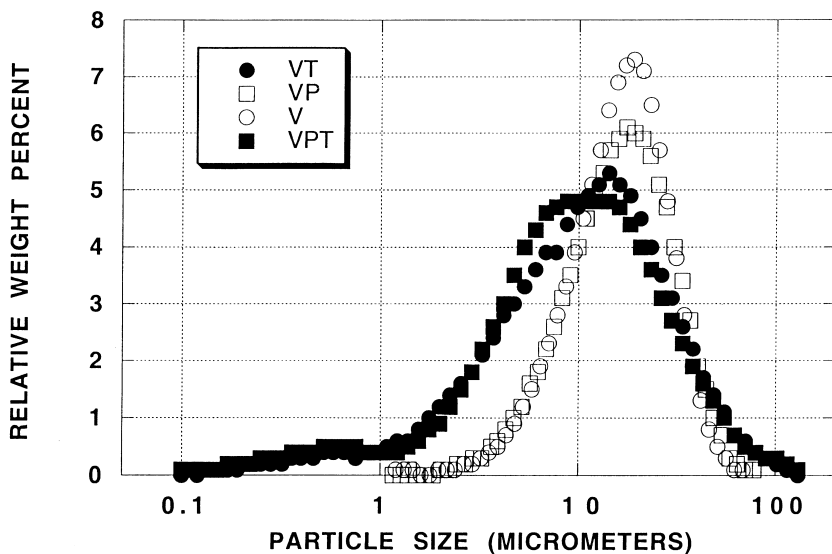


Fig. 4. Laser diffraction analysis of amalgam powder (V and VP) and condensed amalgam (VT and VPT).

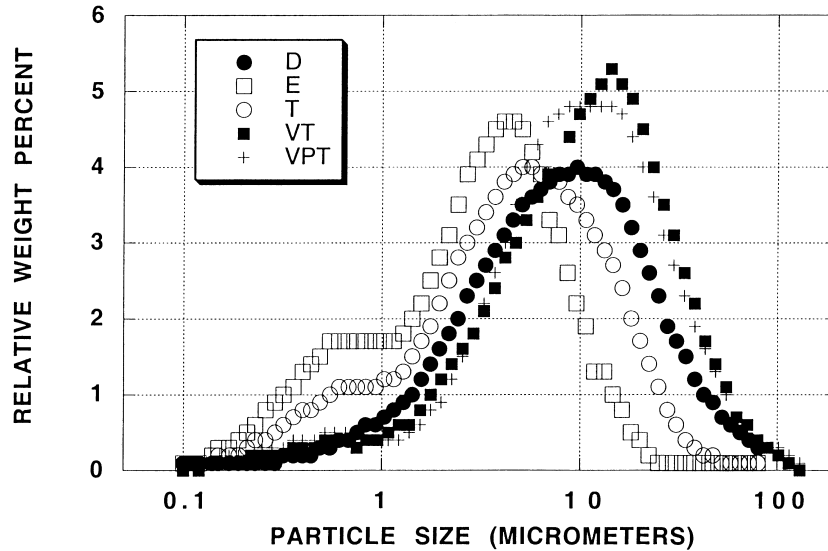


Fig. 5. Laser diffraction analysis of ground specimens of enamel (E), dentin (D), whole tooth (T), and amalgam (VT and VPT).

to determine the particle size distribution curve. X-ray sedimentography determines the particle size distribution by monitoring a change in the transmission rate of an X-ray beam as particles settle through the beam with time. Particles with irregular surfaces will tend to be undersized relative to the other techniques because of decreased settling speed due to drag. Hence, the size distribution is based upon the ‘hydrodynamic settling velocity’ of the suspension. Needles or shards may be overestimated in size due to streaming, with more rapid settling taking place. By contrast, laser diffraction measures particles based on their area and then converts the generated diffraction pattern to that of a size distribution based on smooth spheres of equivalent spherical diameter. The technique is also parameter sensitive; that is, the refractive index of the sample and carrier medium need to be known, it is not sensitive to low concentrations of large

particles, and the data reduction algorithm is dependent on the manufacturer’s mathematical model, no two of which are alike. Electrozone technology simply counts and measures particles based on their individual volumes. Data is also reported as either a number or a mass distribution based upon the concept of equivalent spherical diameter.

Letzel et al. [12] used sieves and SEM to determine the particle size distribution of a dental waste water stream. Their results showed no particles in the 30–200 μm range; whereas, in the present study it was found that a significant fraction of particles existed in this range. This difference is likely to be attributed to their analysis methods: SEM and sieves. The bimodal distribution observed by Letzel et al. was not seen in this study. This is attributed to the fact that only particles that passed through the 300 μm sieve were analyzed in this study. The second distribution

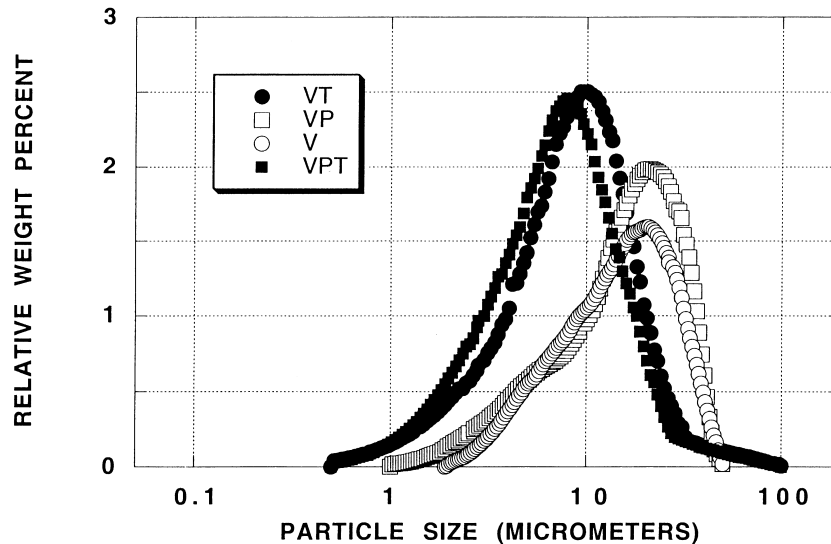


Fig. 6. Electrozone analysis of amalgam powder (V and VP) and condensed amalgam (VT and VPT).

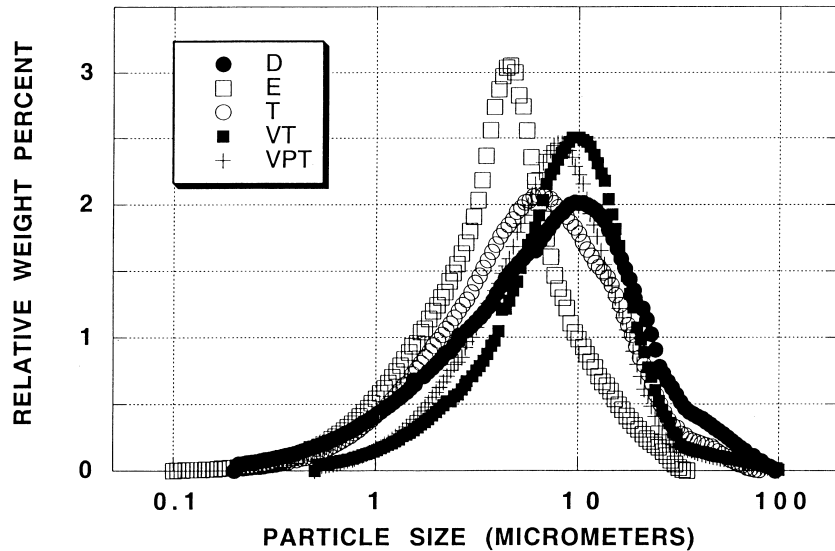


Fig. 7. Electrozone analysis of ground specimens of enamel (E), dentin (D), whole tooth (T), and amalgam (VT and VPT).

observed by Letzel et al. was seen in specimens greater than 300 μm .

A continuum of measurements across particle size ranges is likely to be another requirement for a proper definition of the properties of the DW particle stream. This is seen by comparing the particle size distribution curves in Figs. 1–7. The sieve analysis method provides a ‘discreet’ representation of the data with a severe limitation at the lower range of the curve, since this method can not easily determine particles less than 20 μm . The X-ray sedigraphy is also a somewhat discreet analysis method. Generally analyzing particles in the 100 micron and smaller size range. The laser diffraction, X-ray sedigraphy, and electrozone analysis methods provided a better, ‘continuous’, representation of the particle size distribution curve. From these three methods, the electrozone analysis method is likely to provide a more comprehensive representation of the curve, since more than 250,000 particles are generally counted and sized, free of artifacts which affect sedimentation and laser diffraction. This analysis, Fig. 7, clearly illustrates the narrow range for the small enamel particles, E, the much wider range for dentin, D, and the whole tooth curve falling in between these two curves. The distinction between the curves of the powder and the condensed removed amalgam samples was better defined as well (see Fig. 6). With this method, the mode of each distribution was well separated (i.e., approximately 10 μm for V and VP, in comparison to approximately 20 μm for VPT and VT).

A critical parameter for assessing the applicability of these particle size analysis methods is their ability to quantify the low percentile range of the particle size distribution curve. Under normal operating conditions, particle removal techniques such as sedimentation, filtration, or centrifugation, will remove the majority of large particles of the incoming DW stream. The critical range, in terms of defining removal efficiency of DW treatment devices, is likely to

be the range of the distribution curve below 20 μm , since only a fraction of the particles within this range will be removed. Assessment of the efficiency of DW treatment devices can be defined in terms of particle size removal. However, this may not be equivalent to mercury removal since the particles can be enamel, dentine, cement, polishing compounds, etc., or colloidal particles of amalgam and/or mercury. This study showed that enamel and dentin particles are the smallest of the components of the DW stream. These particles have the potential, along with other organic components, to interfere with the performance of removal devices, such as clogging or blocking of filtration devices. In addition, the particles below 20 μm are less efficiently removed by devices based on centrifugation (i.e. low mass/weight), or sedimentation with a low retention time.

Taking into account all of the operational parameters, the electrozone analysis method is likely to be the most appropriate method for the determination of the particle size distribution curve of the DW stream, since it required a small sample, was not affected by density, shape factors or optical properties, and yet individually measured a sufficient number of particles to provide a reliable representation of the curve.

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