

Residual mercury content and leaching of mercury and silver from used amalgam capsules[☆]

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Abstract

Objective: The objective of this investigation was to carry out residual mercury (Hg) determinations and toxicity characteristic leaching procedure (TCLP) analysis of used amalgam capsules.

Methods: For residual Hg analysis, 25 capsules (20 capsules for one brand) from each of 10 different brands of amalgam were analyzed. Total residual Hg levels per capsule were determined using United States Environmental Protection Agency (USEPA) Method 7471. For TCLP analysis, 25 amalgam capsules for each of 10 brands were extracted using a modification of USEPA Method 1311. Hg analysis of the TCLP extracts was done with USEPA Method 7470A. Analysis of silver (Ag) concentrations in the TCLP extract was done with USEPA Method 6010B.

Results: Analysis of the residual Hg data resulted in the segregation of brands into three groups: Dispersalloy[™] capsules, Group A, retained the most Hg (1.225 mg/capsule). These capsules were the only ones to include a pestle. Group B capsules, Valliant PhD[™], Optaloy[™] II, Megalloy[™] and Valliant Snap Set[™], retained the next highest amount of Hg (0.534–0.770 mg/capsule), and were characterized by a groove in the inside of the capsule. Group C, Tytin[™] regular set double-spill, Tytin FC[™], Contour[™], Sybraloy[™] regular set, and Tytin[™] regular set single-spill retained the least amount of Hg (0.125–0.266 mg/capsule). TCLP analysis of the triturated capsules showed Sybraloy[™] and Contour[™] leached Hg at greater than the 0.2 mg/l Resource Conservation and Recovery Act (RCRA) limit.

Significance: This study demonstrated that residual mercury may be related to capsule design features and that TCLP extracts from these capsules could, in some brands, exceed RCRA Hg limits, making their disposal problematic. At current RCRA limits, the leaching of Ag is not a problem. © 2002 Academy of Dental Materials. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Dental materials; Amalgam; Amalgam capsules; Mercury; Silver; Residual mercury; Toxicity characteristic leaching procedure (TCLP)

1. Introduction

Environmental aspects of mercury (Hg) have recently become an important issue for dentistry [1–6]. While environmental concerns continue to center on the heavy-metal content of dental-unit wastewater, issues involving solid-waste disposal have the potential to become equally significant. Two reports are available that describe the residual Hg content of disposable amalgam capsules [7,8] with the latter reporting values as high as 33.89 mg of Hg per capsule [8].

The policy that governs the disposal of solid waste in the United States is promulgated in the Resource Conservation

and Recovery Act (RCRA) [9]. Through this act Congress provided the United States Environmental Protection Agency (USEPA) with the foundation to develop regulatory programs to manage solid waste, hazardous waste, medical waste, and underground storage tanks. In 1965, Congress enacted the Solid Waste Disposal Act (SWDA), which provided the first federal statutory requirements intended to improve solid-waste disposal practices. The SWDA was modified in 1970 by the Resource Recovery Act, and modified again in 1976 by RCRA. RCRA established a system for controlling hazardous waste from its point of generation to its final disposal (cradle-to-grave). RCRA has been amended many times since 1976, most notably by the Hazardous and Solid Waste Amendments (HSWA) of 1984. HSWA was fashioned principally in response to citizen concerns that existing methods of hazardous waste disposal were neither safe nor sufficient. The Federal Facilities Compliance Act modified RCRA again in 1992. This act makes the federal government part of the regulated

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Table 1
Amalgam brands, Hg content, spill size, trituration times and speed

| Brand of amalgam | Manufacture | Mercury/capsule (mg) ^a | Spill size ^b | Trituration time (s) | Cycle speed (cpm) ^c |
|---------------------------|----------------------------------|-----------------------------------|-------------------------|----------------------|--------------------------------|
| Dispersalloy™ Regular Set | Dentsply/Caulk, Milford, DE USA | 396 | Single | 14 | 3600 |
| Valliant PhD™ | Vivadent Ivoclar, Ontario Canada | 471 | Double | 10 | 4200 |
| Optaloy™ II | Dentsply/Caulk, Milford, DE USA | 296 | Single | 12 | 3600 |
| Megalloy™ | Dentsply/Caulk, Milford, DE USA | 443 | Double | 12 | 4200 |
| Valliant Snap Set™ | Vivadent, Ontario Canada | 314 | Single | 10 | 4200 |
| Tytin™ Regular Set | Kerr, Romulus, MI USA | 447 | Double | 6 | 3600 |
| Tytin FC™ | Kerr, Romulus, MI USA | 453 | Double | 10 | 4200 |
| Contour™ Self Activating | Kerr, Romulus, MI USA | 355 | Single | 10 | 4200 |
| Sybraloy™ Regular Set | Kerr, Romulus, MI USA | 321 | Single | 10 | 4200 |
| Tytin™ Regular Set | Kerr, Romulus, MI USA | 300 | Single | 6 | 3600 |

^a This data was obtained from the manufacturer and may vary from actual mercury present in capsules.

^b Spill size: single spill 400 mg alloy; double spill 600 mg alloy.

^c Cycle speed in cycles/min.

community, and it holds government facilities and managers to the same array of enforcement measures, including fines and penalties, as the rest of the nation. The latest legislative change to RCRA was the Land Disposal Program Flexibility Act of 1996, which provided regulatory flexibility for the land disposal of certain hazardous wastes.

To help determine if a waste is hazardous, the USEPA designed a laboratory analysis called Toxicity Characteristic Leaching Procedure (TCLP) [10,11], which determines the mobility of analytes in an acetic acid buffer solution. The concentration of regulated analytes in the extract determines the toxicity characteristic of a sample, and therefore whether it is subject to disposal regulations under RCRA. The test was designed to predict whether landfill wastes might leach dangerous levels of chemicals into ground water. TCLP regulatory levels exist for 40 different toxic chemicals. The TCLP limits for Hg and Ag are 0.2 and 5.0 mg/l, respectively [10,11].

Facilities that generate less than 100 kg (220 lb) of hazardous waste per month or less than 1 kg (2.2 lb) per month of acutely hazardous waste are excused from most federal RCRA requirements and are classified as 'Conditionally Exempt Small Quantity Generators' (CESQGs) [9]. An essential caveat to this exemption is that some states prohibit the disposal of *any* hazardous waste into municipal solid-waste landfills, even from CESQGs. Facilities that produce solid waste are well warned to contact local, regional, and state solid-waste regulators to confirm regulatory requirements.

The present study was undertaken to assess residual Hg levels from currently available dental-amalgam capsules and to obtain information on the potential leaching of mercury (Hg) and silver (Ag) from these capsules. Leaching of these metals above regulatory levels could make the disposal of these capsules a vexing issue for the clinical dentist and for environmental regulators. It was hypothesized that certain capsule design features might provide opportunities for Hg and/or amalgam retention. These

features included the presence of Hg-containing packets, mixing pestles, and internal grooves associated with the ultrasonically sealed capsules. In addition, it was reasonable to expect that residual Hg might be greater in double-spill than in single-spill amalgam capsules.

2. Materials and methods

2.1. Residual mercury

Amalgam capsules were purchased from dental supply companies or acquired from the Naval Dental Center, Great Lakes, IL, USA. The amalgam used in this study included the following 10 brands (Table 1): Tytin™ regular set single-spill operator activated capsules (Kerr Manufacturing, Romulus, MI, USA), Tytin™ regular set double-spill operator activated capsules (Kerr), Sybraloy™ regular set single-spill self-activating capsules (Kerr), Megalloy™ double-spill self-activating capsules (Dentsply/Caulk, Dentsply International, Milford, DE, USA), Optaloy™ II single-spill self-activating capsules (Dentsply/Caulk), Dispersalloy™ regular set single-spill self-activating capsules (Dentsply/Caulk), Contour™ regular set single-spill self-activating capsules (Kerr), Valliant Snap Set™ single-spill self-activating capsules (Vivadent, Ivoclar North America, Inc., Ontario, Canada), Valliant PhD™ double-spill self-activating capsules (Vivadent), and Tytin FC™ double-spill self-activating capsules (Kerr). Twenty-five capsules of each brand (20 capsules in the case of the Optaloy™ II alloy) were triturated in a ProMix™ (Dentsply/Caulk) amalgamator at the recommended speeds and times (Table 1). The capsules were weighed before and after trituration. The amalgamated pellet was removed and the empty capsule was weighed. Each capsule was then placed into a separate 50-ml screw-top plastic test tube.

Analysis of residual Hg was accomplished using USEPA Method 7471 [12,13]. Capsules were digested in 10 ml of

Table 2
Hg retained as a function of capsule features

| Capsule | Mercury packet | Pestle | Groove | Spill | Mean (SD) residual Hg ^a | Duncan ^b ($P \leq 0.05$) |
|---------------------------|----------------|--------|--------|--------|------------------------------------|---------------------------------------|
| Dispersalloy™ Regular Set | Yes | Yes | No | Single | 1.255 (0.793) | A |
| Valliant PhD™ | Yes | No | Yes | Double | 0.770 (0.330) | B |
| Optaloy™ II | Yes | No | Yes | Single | 0.650 (0.644) | B |
| Megalloy™ | Yes | No | Yes | Double | 0.590 (0.504) | B |
| Valliant Snap Set™ | Yes | No | Yes | Single | 0.534 (0.398) | B |
| Tylin™ Regular Set | No | No | No | Double | 0.266 (0.184) | C |
| Tylin FC™ | Yes | No | No | Double | 0.223 (0.120) | C |
| Contour™ Self Activating | Yes | No | No | Single | 0.181 (0.121) | C |
| Sybraloy™ Regular Set | Yes | No | No | Single | 0.142 (0.163) | C |
| Tylin™ Regular Set | No | No | No | Single | 0.125 (0.063) | C |

^a Units in mg Hg per capsule.

^b Brands with the same letter comprise homogeneous subsets.

aqua regia (3 volumes of concentrated HCL to 1 volume of concentrated HNO₃) at 95°C for 2 min. After cooling, 15 ml of potassium permanganate solution and 50 ml of reagent water were added, and each sample was heated to 95°C for an additional 30 min. After sample cooling, an additional 50 ml of reagent water was added and 6 ml of sodium chloride-hydroxylamine sulfate was added to reduce the excess permanganate. Five ml of stannous chloride was added to each sample to reduce Hg⁺² to Hg⁰. The volatile Hg⁰ was carried to a Varian Model 600 atomic absorption spectrometer (Varian Analytical Instruments, Sugar Land TX, USA) by nitrogen gas (N₂) at a rate of 1 l/min. Hg levels were determined by cold vapor atomic absorption spectroscopy with Zeeman Correction from a calibration curve. Quality Assurance/Quality Control with duplicates and spiked samples was completed as required by USEPA Method 7471.

2.2. Toxicity characteristic leaching procedure (TCLP) analysis

For TCLP analysis, 25 amalgam capsules for each of 10 brands (250 total capsules) were triturated at the recommended time and speed (Table 1). The mixed amalgam was removed and used capsules were extracted with an adaptation of USEPA Method 1311 [10,11]. Each batch of 25 capsules was weighed and processed with TCLP extraction fluid #1 at a volume 20 times the weight of the capsules [11]. For extraction fluid #1, glacial acetic acid (11.4 ml) and 1 N sodium hydroxide (128.6 ml) were combined with reagent water to a final volume of 2 l (pH 4.93 ± 0.05). The sample vessels containing the capsules and extraction fluid were rotated end-over-end at 30 RPM for 18 h. The extraction fluid was then decanted into a filtration apparatus and filtered under pressure through an acid washed and deionized-distilled water rinsed Whatman™ glass microfiber filter (grade GF/F, 14.2 cm diameter, 0.7 μm pore size). The filters were again acid washed and the pH of the extract corrected to < 2.0 with concentrated nitric acid. Analysis of the TCLP extracts for Hg was completed using USEPA

Method 7470A employing cold vapor atomic absorption spectroscopy [12,13]. Analysis of TCLP extracts for Ag was accomplished using USEPA Method 6010B, Induction Coupled Plasma Atomic Emission Spectroscopy, ICP-AES [12,13]. The detection limits for these methods were 0.0002 mg/l for Hg and 0.0500 mg/l for Ag.

The adaptation of USEPA Method 1311 employed in this investigation involved the use of a standardized number of capsules (25) rather than a standard 100-g sample. However, the proportion of extraction fluid used to the weight of the capsules was kept constant, with the volume of extraction fluid always 20 times the sample weight, as required by the method.

3. Results

3.1. Residual mercury

Twenty-five specimens from each of the 10 capsule types were analyzed, except for Optaloy™ II for which only 20 specimens were available. The 245 Hg scores ranged from 0.025 to 29.500 mg/capsule. However, the second largest score was only 3.480 mg/capsule, establishing the 29.500 mg/capsule score as a statistical outlier. Both of these scores were for Dispersalloy™ and, in fact, five of the 10 largest scores were for this group. Nevertheless, the 29.500 mg/capsule score was so extreme that it could be considered a singular event that might not consistently reoccur. For this reason, a score of 3.5 mg/capsule (the approximate value of the next highest score) was substituted for this data point. Substituting the next highest value was considered a superior ad hoc strategy since it appropriately retained relevant information, while dropping the data point would have biased the mean estimate downwards to a greater degree. It will be seen that this substitution did not affect the statistical conclusions, because the Dispersalloy™ group differed significantly from all other groups.

Table 2 shows mean residual Hg values for each capsule design. A one-way analysis of variance was statistically

significant ($P < 0.001$) and a Duncan multiple range test established three groupings among the capsules with respect to Hg retention. It should be noted that the Duncan test does not control for experiment-wise error, which can be substantial for multiple comparisons among 10 groups. When the data was subjected to Tukey post-hoc comparisons, where experiment-wise error is fixed at $P \leq 0.05$, the results remain essentially the same with the exceptions that Optaloy™ II and Megalloy™ did not differ from Tytin™ regular set double-spill; and Valliant™ Snap Set did not differ from Tytin™ regular set double-spill, Tytin FC™, or Contour™ Self Activating.

Table 2 shows that Dispersalloy™ capsules retain more Hg than any other capsule, possibly due to the unique use of a pestle. Because the pestle effect would overwhelm the influence of other design features, other attributes of Dispersalloy™ capsules were not considered in further evaluations. Only two capsules did not use an Hg packet, and it did not appear from the available comparisons that the packets influenced Hg retention. A dramatic effect was associated with the presence of a groove on the inside of the capsule. There was no overlap in means between capsules that had and those that did not have a groove, and this grouping was confirmed by the Duncan test. Within the groove and no groove groups, more Hg was retained by double- than by single-spill capsules, but these effects were not statistically significant using these post-hoc testing procedures.

3.2. Toxicity characteristic leaching procedure analysis

The data from TCLP analysis are presented in Table 3. Two groups of batched capsules leached Hg above the RCRA limits: Contour™ and Sybraloy™. The Contour™ extract had an Hg concentration of 0.4120 mg/l and the Sybraloy™ extract had an Hg concentration of 0.2530 mg/l. None of the capsules leached Ag above RCRA limits (5 mg/l): all extracts were at non-detectable levels for Ag (< 0.0500 mg/l).

4. Discussion

While Hg contamination of dental-unit wastewater is one contributor to the environmental burden of clinical dentistry, the disposal of solid wastes is another major concern. This study examined both the residual Hg in used amalgam capsules and the leaching of Hg and Ag from used capsules. While it is known that used amalgam capsules contain residual Hg [7,8], this is the first published study to demonstrate the leaching of metals from used capsules.

The residual Hg content of the 10 brands of capsules used in this study was less than the levels reported in two previous studies [7,8]. Barkmeier et al. [7] reported a mean Hg per capsule of 12.1 mg. Cheuk et al. [8] evaluated six different amalgams and found residual Hg to range from 2.75 mg/capsule for Valiant (Vivadent) to 15.55 mg/capsule for Dispersalloy (Dentsply/Caulk). The mean residual Hg in

Table 3
Toxicity characteristic leaching procedure results

| Capsule | Hg (mg/l) | Exceeds RCRA Hg limits ^a |
|---------------------------|---------------|-------------------------------------|
| Dispersalloy™ Regular Set | 0.0477 | No |
| Valliant PhD™ | 0.0261 | No |
| Optaloy™ II | 0.0340 | No |
| Megalloy™ | 0.0399 | No |
| Valliant Snap Set™ | 0.0425 | No |
| Tytin™ Regular Set | 0.0104 | No |
| Tytin FC™ | 0.1640 | No |
| Contour™ Self Activating | 0.4120 | Yes |
| Sybraloy™ Regular Set | 0.2530 | Yes |
| Tytin™ Regular Set | 0.0110 | No |

^a RCRA limit for Hg is 0.2 mg/l.

this study ranged from 0.125 to 1.255 mg. The lower levels of residual Hg seen in this study may reflect the different digestion procedures used or the use of single-spill capsules in six of the 10 brands studied. The segregation of capsules into three groups appears to be a function of the capsule design. However, the definitive influence of capsule design on Hg retention could not be determined because of the overlapping design features of capsules used in the current study. More complicated capsule designs may tend to retain more amalgam and therefore more Hg. In the case of Dispersalloy, the pestle adds increased surface area where amalgam and Hg can adhere. In addition, forces generated in pestle containing-capsules during trituration may cause amalgam to adhere more tightly to insides of the capsules. As a group, the ultrasonically fused capsules retained the next highest amount of Hg. These capsules have a groove that tends to retain amalgam in an annular pattern (Fig. 1). The third group had the simplest capsule design and retained the least amount of Hg.

Fan et al. [14] and the United States Air Force Dental



Fig. 1. Photograph of one ultrasonically sealed Megalloy™ capsule and two capsules triturated and opened. Arrow identifies an annular pattern of retained amalgam in the top portion of one of the amalgam capsules. This pattern of amalgam retention was seen in virtually all the ultrasonically sealed capsules used in this study.

Investigation Service [15] have completed TCLP studies of amalgam scrap. However, to our knowledge, this is the first published report that applied this procedure to disposable amalgam capsules. The TCLP data from 10 batches of 25 capsules revealed two brands that exceeded the 0.2 mg/l RCRA limit for Hg (highlighted in Table 3). In a prior preliminary TCLP study [16] done with pooled 100-g samples across several different brands of amalgam capsules, Hg levels of TCLP extracts averaged 0.1291 mg/l with one sample exceeding the RCRA limit for Hg ($N = 12$, range = 0.0369–0.6700, $SD = 0.1733$), and Ag levels were non-detectable (< 0.0500 mg/l) except for one measurement of 0.0503 mg/l. It is of interest to note that the brands that retained the most Hg did not have the highest Hg levels in the TCLP extracts. This surprising and unanticipated result is now the subject of further research.

In several cases, capsules that contained Hg packets did not mix due to failure of the packets to break open during trituration. This phenomenon was seen across many brands that used Hg packet technology and was the reason that only 20 capsules were used for the retained Hg analysis of the Optaloy™ II capsules (five Optaloy™ II capsules did not mix). Anecdotal information from several Navy dental clinics confirms this problem, which can be particularly severe in some lots of the same amalgam brand. Unruptured Hg packets can create a serious disposal dilemma as they contain a sizeable amount of elemental Hg (Table 1).

Discarding capsules in municipal-solid-waste landfills is not ideal and could, in some cases, violate state solid-waste-discharge statutes, since some states regulate all generators of hazardous waste, even from CESQGs. Recovery of heavy metals through the recycling processes is environmentally more responsible than disposal in landfills where the potential exists for metals to leach into ground water. Reclaimed metals can be reused in the manufacturing of dental amalgam.

Incineration of used amalgam capsules must be avoided to prevent volatilization of Hg to the atmosphere. Deposition of atmospheric Hg to land, lakes, rivers, and streams can be substantial [17] and lead to the bioaccumulation of organic Hg in fish and other aquatic organisms [17–26]. Nearly 100% of the Hg that concentrates in fish tissue is methylmercury [17,26]. High Hg levels in remote pristine lakes, where atmospheric deposition appears to be the key instrument of contamination, is further evidence of the importance of this pathway [17,24].

5. Conclusion

Capsule design features can influence retention levels of amalgam and Hg in used capsules. Retained Hg in used amalgam capsules can leach in amounts that make their disposal exigent and problematic. The leaching of Ag does not appear to be a subject for concern at current RCRA levels.

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