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The Removal of Mercury from Dental-operator Wastewater by Polymer Treatment

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Abstract

The mercury (Hg) content of dental-operator wastewater has become an issue in many localities, and Hg removal is rapidly becoming a matter of concern for all dental clinics. This preliminary study tested the efficacy of polymers for the removal of Hg contaminants from the dental-unit wastewater stream. Two commercially available polymers were used to treat dental-operator wastewater. Used separately, each polymer removed from 74.9% to 88.4% of the Hg from dental-wastewater supernatant. The polymers used in combination, within the recommended pH range, removed up to 99.9% of the total Hg from dental-wastewater supernatant. The estimated optimal concentration of the two polymers is approximately 2.33 ml of each per liter of waste, and more than 90% of the Hg may be removed with 0.13 ml/l. Results indicate that a combination of the two polymers may sufficiently reduce Hg levels to allow discharge of clarified supernatants into public sewer systems. **Key words:** amalgam, dental, mercury, polymer, wastewater. **Environ Health Perspect** 107:3-8 (1999). [Online 4 December 1998]

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Introduction

The heavy-metal content of wastewater is under scrutiny in several states, and regulations governing heavy-metal discharge into the environment are becoming more stringent, (e.g., permits required by local wastewater-treatment plants under the National Pollution Discharge Elimination System). Mercury (Hg) remains among the top 20 hazardous substances listed on the Agency for Toxic Substances and Disease Registry/EPA priority list. A recent EPA conference on Mercury in the Midwest (1), held 22-23 October 1996, highlighted the need for keeping Hg out of medical waste and out of the wastewater stream. Heavy metals including Hg are present in Great Lakes water and fish (2), and the consumption of fish contaminated with heavy metals represents an important source of human exposure (2-4). The recently implemented Great Lakes Water Quality Guidance criteria (5) call for an ambient Hg water level of 1.3 ng/l for the protection of wildlife. Such guidelines have become a driving force for lowering the permitted release of pollutants into public sewage treatment facilities.

Small-quantity generators such as dental offices contribute to the Hg load of sewage treatment facilities. In 1990 it was estimated that dental facilities in the United States used 44 metric tons of Hg (6). The Seattle Metro Study (7) and a later study by Barruci et al. (8) reported that about 12-14% of the Hg load to local sanitary districts originated from dental clinics. There have been few studies investigating the environmental aspects of the metals released from dental amalgam (9). Recent collaborative studies by Naleway et al. (10) and Cailas et al. (11) were the first to rigorously define the dental amalgam-wastewater stream. A later study (12) demonstrated a significant level of soluble (13) Hg (<0.45 μ m) in the liquid portion of dental wastewater.

This study was initiated as part of the overall plan to remove significant amounts of Hg from

the wastewater of a dental clinic housing 45 dental units. Elevated Hg discharge levels of 0.7 and 0.6 $\mu\text{g Hg/l}$ detected downstream from this clinic about 1 month apart resulted in warnings from the local sanitary district, which mandated a Hg discharge limit of 0.5 $\mu\text{g/l}$. Hg concentrations in grab samples from this clinic were found to average 2.9 $\mu\text{g/l}$. The local sanitary district required the development of a plan to ensure that there were no further Hg releases in excess of the established limit. Therefore, the environmental office of the clinic's public works center requested that our institute aid in developing a method for Hg removal.

Industrial wastewater-treatment technologies have been developed to address specific manufacturing applications (14). However the use, development, and research on waste-treatment technologies for dental-operator wastewater are in their infancy (15,16). Due to the relatively small quantity of dental-operator wastewater generated and its heterogeneous nature, developing effective, nontoxic, and cost-effective treatments has been difficult (10,11,17). Commercially available polymers, which are currently used in industrial waste treatment, may provide an effective treatment option for dental facilities. The objective of this investigation was to test the ability of two such polymers, individually and in combination, to remove Hg from dental-operator wastewater.

Materials and Methods

The two polymers selected for use in this study were an aqueous 20-40% solution of aluminum hydroxychloride and polyquaternary amine (18) at pH 4.0 (Nalco polymer N8186, Nalco Chemical Company, Naperville, IL) and an aqueous solution of polymeric precipitant and salt at pH 11.5-13.0, which had metal chelating molecules bound to a polymer backbone (19,20) (Nalco polymer N8702, Nalco Chemical Company). The company recommends a pH range of 6.0-9.0 as optimal for the use of these polymers.

All operator wastewater samples were collected from the air/water separator tanks, collecting waste from active dental treatment centers housing 45 or more operatories. The samples were collected in clean polypropylene containers during lunch breaks or at the end of the work day when the evacuation system could be shut down.

For test 1, 4 liters of dental-unit wastewater were mixed with a magnetic stir bar at a continuous rate, and five consecutive 125-ml aliquots were removed at a constant sampling depth. A second 4-liter sample was mixed, allowed to settle overnight, and the following morning five consecutive 125-ml samples of supernatant were removed with a pipette at a constant depth. EPA test method 245.1 (21) and Perkin Elmer method 245.1A (22) were used to determine the total Hg content of the aliquots from both procedures. The cold-vapor test method 245.1 (21), performed at an independent EPA-approved laboratory, employed the use of a Varion SpectrAA model 600 atomic absorption spectrophotometer (Varion Analytical Instruments, Sugar Land, TX); the cold-vapor test method 245.1A (22), performed at our own Illinois state EPA-approved laboratory, employed the use of a Perkin-Elmer model 5100PC atomic absorption spectrophotometer (Perkin-Elmer Corporation, Norwalk, CT). The Hg

method detection limits were 0.2 and 0.07 µg/l, respectively. This experiment was done to identify possible sampling errors incurred during aliquoting procedures and to ensure that supernatants of aliquots from a single source were similar in Hg content.

Test 2 was designed to compare the Hg removal effectiveness of the polymers individually, and test 2a was designed to test their effectiveness when used in combination at equal volumes. No sample pH adjustment was made because the pH was within the range recommended by the manufacturer. A large dental-office wastewater sample was aliquoted into three sets of six 200-ml samples. The polymers were added by micropipette, individually and together in equal amounts, to achieve milliliter per liter concentrations of 0.0 (control), 0.01, 0.07, 0.13, 0.33, and 3.33 for each polymer. Following the addition of the polymers, each treatment sample and control sample was immediately mixed by gentle swirling for 2 min and allowed to settle overnight (15-16 hr). The clarified supernatants from each of the aliquots were tested for total Hg by test methods 245.1 and 245.1A.

Further tests of the polymers used in combination were performed on two additional wastewater samples. In test 3, aliquoted samples were treated with both polymers in combination at concentrations of 0.0 (control), 0.13, 0.33, 0.67, 1.33, 3.33, 5.0, and 10.0 ml/l for each. The dental wastewater samples for test 3 were found to be in the recommended pH range and no adjustments were made. In test 4, both polymers were again tested in combination at the following concentrations: 0.0, 0.13, 0.33, 1.33, 2.33, and 3.33 ml/l. The test 4 sample was found to be at pH 3.3 and was adjusted with sodium hydroxide to pH 7.5 before aliquoting and polymer addition. The low pH of this wastewater sample was due to the use of a commercial acidic line cleanser product. The supernatants from test 3 and test 4 samples were again analyzed for Hg by methods 245.1 and 245.1A.

Test 5 was designed to compare the effectiveness of the polymers when used within and below the recommended pH range. An additional sample of dental wastewater at pH 3.2 was collected and divided into four 900-ml portions while undergoing continuous mixing. For quality assurance, two of the 900-ml aliquots were spiked with an additional 4,500 µg (5,000 µg/l) of Hg by adding a commercially prepared mercury standard. The addition of the Hg standard lowered the pH from 3.2 to 2.2. One nonspiked portion was adjusted from pH 3.2 to pH 7.4 and one spiked portion was adjusted from pH 2.2 to pH 7.5. All four portions were then further aliquoted with mixing. One aliquot from each portion served as a control, and equal volumes of the polymers were added in combination at 0.33, 1.33, 3.33, and 5.00 ml/l. The aliquots were gently mixed by swirling for 2 min and allowed to settle overnight prior to supernatant sampling. The supernatants from these test 5 samples were also tested for total Hg content by methods 245.1 and 245.1A.

The quality control requirements of EPA Method 245.1 were met (i.e., laboratory reagent blanks, laboratory-fortified blanks, laboratory duplicates for every 10 samples, laboratory-fortified matrix, and initial, continuing, and final curve blanks with verifications). All supernatants from treated samples and control samples were preserved with nitric acid and stored at 4°C prior to testing for total Hg. All analyses were performed within 28 days of

sample collection.

The percent of Hg removed following treatment was calculated as follows:

$$\left(\frac{\text{Hg Concentration of Control} - \text{Hg Concentration of Sample Supernatant}}{\text{Hg Concentration of Control}} \right) \times 100$$

All of the Hg-containing waste material generated during the course of this study was disposed of as hazardous waste in accordance with regulatory requirements.

Results

The results of test 1, done to evaluate sampling error resulting from the aliquoting process used in this study, are shown in Table 1. The five samples from the continuously mixed waste and the five samples of the supernatant from the settled operatory waste were assayed for Hg. Total Hg from the five continuously mixed samples ranged from 5.3 to 12.0 mg/l [mean = 7.3 mg/l, standard deviation (SD) = 2.74]. All five supernatant aliquots taken from the sample allowed to settle overnight were found to contain 1.8 mg/l of total Hg. These data validate the use of supernatant from settled samples to evaluate the efficacy of the polymers for Hg removal.

Table 1. Test 1: sample error resulting from consecutive aliquoting of continuously mixed and settled dental wastewater supernatant samples

| Sample number | Continuously mixed | | Settled supernatant | |
|---------------|--------------------|------------------------------|---------------------|------------------------------|
| | Total Hg (mg/l) | Percent difference from mean | Total Hg (mg/l) | Percent difference from mean |
| 1 | 5.3 | 27.4 | 1.8 | 0.0 |
| 2 | 5.5 | 24.7 | 1.8 | 0.0 |
| 3 | 6.5 | 11.0 | 1.8 | 0.0 |
| 4 | 12.0 | 64.5 | 1.8 | 0.0 |
| 5 | 7.2 | 14 | 1.8 | 0.0 |
| Mean ± SD | 7.3 ± 2.74 | | 1.8 ± 0.00 | |

SD, standard deviation.

The results of tests 2 and 2a to assess the ability of the polymers to remove Hg from the dental amalgam wastewater supernatant are shown in Table 2 and shown graphically in Figure 1. These compare the effectiveness of the polymers to remove Hg when used individually and in combination at equal concentrations. The maximum Hg removal efficiencies for N8186 and N8702 used singly were 74.9% and 88.4%, respectively. The maximum Hg removal for the polymers used in combination was 97.5%.

Table 3 and Figure 2 show the efficiency of the polymers when used in equal volumes to remove Hg from dental-office wastewater supernatant for three different wastewater samples (tests 2a, 3, and 4). Concentrations ≥ 0.13 ml/l of both polymers removed greater than 90% of the total Hg from all wastewater supernatant samples. Hg removal efficiencies ranged from 97.5 to 99.9% at polymer concentrations of 3.33 ml/l. Nearly 100% of the Hg was removed from test 3 and test 4 samples. These samples contained about 8 and 17 times the Hg as the test 2a samples, respectively. About 18% of the Hg precipitated when the pH was adjusted to near neutral in the test 4 control samples.

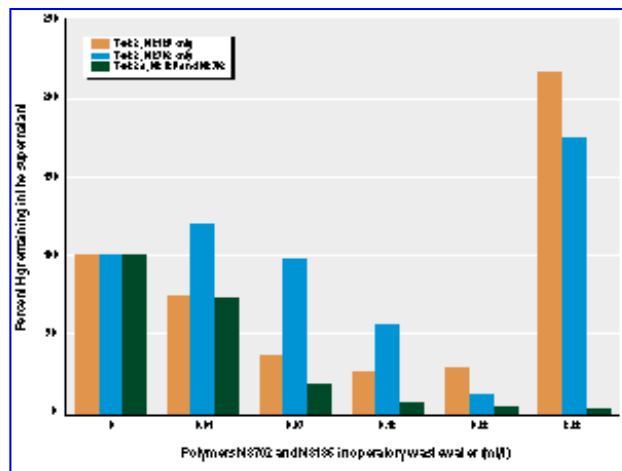


Figure 1. Tests 2 and 2a: percent Hg remaining with polymers used individually and together in equal volumes in three separate tests.

Table 2. Tests 2 and 2a: Hg removal results with polymers N8186 and N8702 used individually (Test 2) and both polymers (Test 2a) in equal volumes

| Polymer in wastewater (µg/l) | Test 2: polymers used individually | | Test 2a: both polymers | |
|------------------------------|-------------------------------------|----------------------------------|-------------------------------------|----------------------------------|
| | Hg remaining with N8186 only (µg/l) | Percent Hg removed by N8186 only | Hg remaining with N8702 only (µg/l) | Percent Hg removed by N8702 only |
| 0.00 (Control) | 557 (100.0) | 0.0 | 557 (100.0) | 0.0 |
| 0.01 | 439 (77.54) | 26.46 | ND | ND |
| 0.07 | 185 (33.21) | 62.41 | ND | ND |
| 0.13 | 31 (5.58) | 94.41 | 393 (7.23) | 92.77 |
| 0.33 | 19 (3.41) | 96.82 | 190 (3.41) | 96.22 |
| 0.67 | ND | ND | 115 (2.08) | 97.71 |
| 1.33 | ND | ND | 38 (6.82) | 93.24 |
| 2.33 | ND | ND | ND | 9 (1.61) |
| 3.33 | 15 (2.71) | 97.49 | 1.1 (0.20) | 99.98 |
| 5.00 | ND | ND | 43 (7.71) | 93.14 |
| 10.00 | ND | ND | 188.2 (33.79) | 96.65 |

ND, not determined.
 *Percent Hg remaining in the supernatant after treatment and precipitation is shown in parentheses.
 †pH 2.2 sample, when pH adjusted to 7.4, Hg content was 0.200 µg/l, an 18.3% decrease.

Table 3. Tests 2a, 3, and 4: Hg removal results with polymers N8186 and N8702 used concurrently in equal volumes

| Both polymers in wastewater (µg/l) | Test 2a: N8186 and N8702 | | Test 3: N8186 and N8702 | | Test 4: N8186 and N8702 | |
|------------------------------------|--------------------------|--------------------|-------------------------|--------------------|-------------------------|--------------------|
| | Hg remaining (µg/l) | Percent Hg removed | Hg remaining (µg/l) | Percent Hg removed | Hg remaining (µg/l) | Percent Hg removed |
| 0.00 (Control) | 557 (100.0)* | 0.0 | 5,026 (100.0) | 0.0 | 10,046* (100.0) | 0.0 |
| 0.01 | 439 (77.54) | 26.46 | ND | ND | ND | ND |
| 0.07 | 185 (33.21) | 62.41 | ND | ND | ND | ND |
| 0.13 | 31 (5.58) | 94.41 | 393 (7.23) | 92.77 | 66 (0.66) | 93.94 |
| 0.33 | 19 (3.41) | 96.82 | 190 (3.78) | 96.22 | 25 (0.25) | 99.75 |
| 0.67 | ND | ND | 115 (2.29) | 97.71 | ND | ND |
| 1.33 | ND | ND | 38 (0.76) | 93.24 | 10 (0.10) | 99.90 |
| 2.33 | ND | ND | ND | ND | 9 (0.09) | 99.91 |
| 3.33 | 15 (2.71) | 97.49 | 1.1 (0.02) | 99.98 | 9 (0.09) | 99.91 |
| 5.00 | ND | ND | 43 (0.86) | 93.14 | ND | ND |
| 10.00 | ND | ND | 188.2 (3.35) | 96.65 | ND | ND |

ND, not determined.
 *Percent Hg remaining in the supernatant after treatment and precipitation is shown in parentheses.
 †pH 2.2 sample, when pH adjusted to 7.4, Hg content was 0.200 µg/l, an 18.3% decrease.

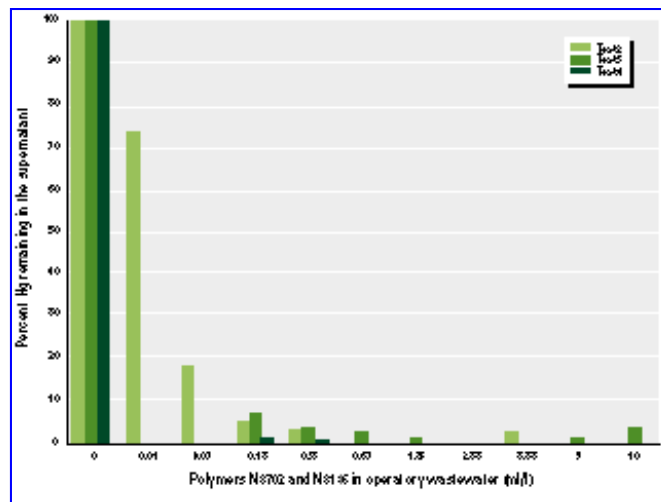


Figure 2. Tests 2a, 3, and 4: percent Hg remaining with polymers used together in equal volumes in three separate tests.

The effects of pH on polymer treatment of wastewater are shown in Table 4 and Figure 3. About 20% of the Hg precipitated when the pH was adjusted to near neutral (spiked and nonspiked controls). In every case, the use of 3.33 ml/l of both test polymers removed the largest percentage of Hg. The effect of pH was most apparent between the Hg-spiked clinical samples in which the pH 7.5 aliquots consistently yielded much larger Hg removal percentages at all treatment levels over the pH nonadjusted aliquots. The recovery of the field spike was determined to be 100.22%, showing an analysis error of 0.22%. A relative percent difference of 0.76% was found when 5,000 µg/l was subtracted from the spiked sample.

| Both polymers concentration (ml/l) | Clinical samples | | | | Mercury spiked clinical samples | | | |
|---------------------------------------|------------------------|-----------------------|------------------------|-----------------------|---------------------------------|-----------------------|------------------------|-----------------------|
| | pH Nonadjusted | | pH Adjusted | | pH Nonadjusted | | pH Adjusted | |
| | Hg remaining (µg/l) | Percent Hg removed | Hg remaining (µg/l) | Percent Hg removed | Hg remaining (µg/l) | Percent Hg removed | Hg remaining (µg/l) | Percent Hg removed |
| 0.01 (Control) | 210 (0.00) | 0 | 178 (0.15) | 11.6 | 210 (0.00) | 0 | 576 (0.10) | 21.8 |
| 0.03 | 128 (0.02) | 64.6 | 20 (0.18) | 11.6 | 101 (0.02) | 11.6 | 128 (0.11) | 11.6 |
| 0.15 | 101 (0.01) | 91.6 | 41 (0.34) | 91.6 | 52.8 (0.00) | 25.0 | 140 (0.1) | 91.6 |
| 0.57 | 52 (0.17) | 91.7 | 24 (0.1) | 91.7 | 2.08 (0.02) | 91.6 | 47 (0.00) | 91.6 |
| 0.63 | 27 (0.27) | 91.7 | 20 (0.22) | 91.7 | 4.02 (0.18) | 41.6 | 21 (0.22) | 91.6 |

Percent Hg remaining in the supernatant and the amount of Hg precipitated is shown in parentheses.
Percentages are based on 210 µg/l.

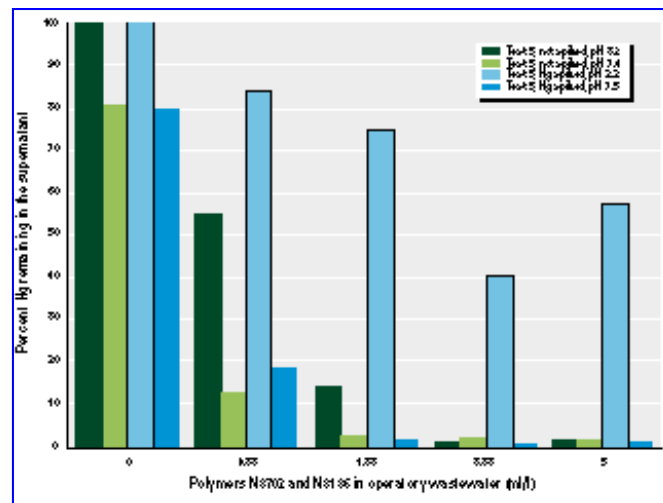


Figure 3. Test 5: effect of pH on polymer treatment efficiency shown as percent Hg remaining.

Figure 4 shows the mean levels of Hg remaining in the five separate dental wastewater samples from the holding tanks of two different dental treatment facilities. All received concomitant use of equal volumes of both polymers at various concentrations. Hg content consistently decreased as the polymer concentrations increased, until the optimal concentrations were exceeded; beyond that, the effectiveness of Hg removal by the polymers decreased. The results of these analyses indicated the optimal concentration was near 2.33 ml/l of each polymer.

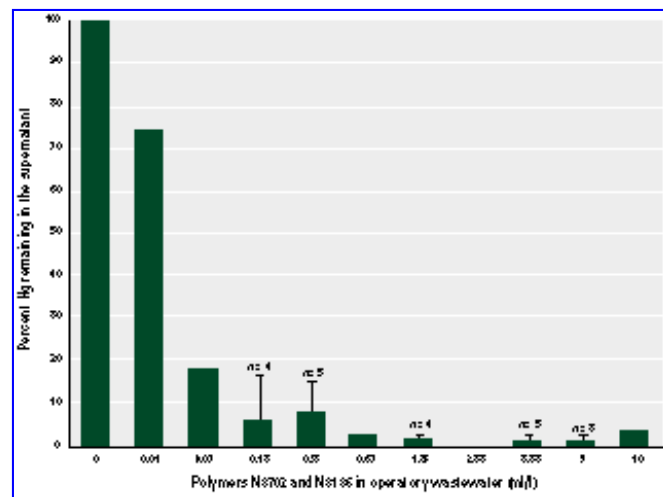


Figure 4. Tests 2, 3, 4, and 5: mean percent Hg remaining after treatment with

both polymers in equal volumes. Error bars demonstrate standard deviations where sample size (n) was between 3 and 5. Where number is not given, $n=1$.

Differences in settling rates and precipitate volumes between the various polymer concentrations used in tests 2 through 4 were observed. Figures 5 through 7 demonstrate these differences with the test 4 samples where paired wastewater samples at pH 3.3 and pH 7.5 were treated with 0, 0.33, 1.33, and 3.33 ml/l of both polymers. The photograph in Figure 5, taken immediately after mixing, shows the formation of flock in all treated samples. Differences in flock characteristics are readily apparent. The photograph in Figure 6, taken 15 min after mixing ceased, shows increased supernatant clarification with increased polymer concentrations, especially with the pH 7.5 aliquots. When higher levels of the polymers were used, the precipitants tended to settle more slowly. The differences in supernatant clarity and in the volume of precipitate formed are more pronounced in Figure 7 taken 7.5 hr after mixing ceased. Generally, the supernatants of pH-adjusted samples were more completely clarified than low pH samples, and these supernatants always contained less total Hg upon analysis.

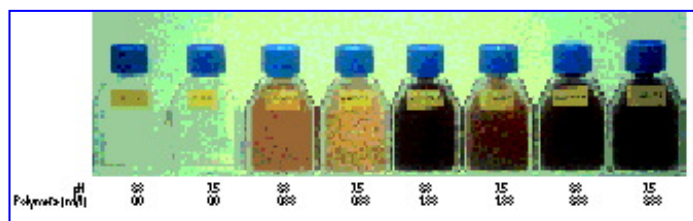


Figure 5. Polymer-treated dental-operator wastewater immediately after mixing.

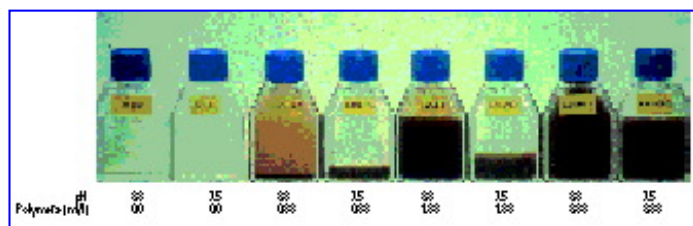


Figure 6. Polymer-treated dental-operator wastewater 15 min after mixing.

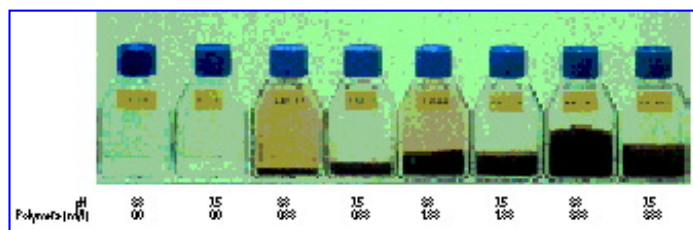


Figure 7. Polymer-treated dental-operator wastewater 7.5 hr after mixing.

Discussion

A combination of the two polymers used in this study removed more Hg than each polymer used individually. The addition of polymers N8186 and N8702 at 1.333-3.333 ml/l were found to remove Hg to levels that permit discharge of the supernatants from treated waste directly to the publicly owned sewage treatment facilities serving the dental clinics. The polymers react quickly and the formation of flocculent material is often observed while mixing is taking place. The precipitation of the "sludge" often begins immediately upon cessation of mixing, provided that the pH is within the 6.0-9.0 range as recommended by the polymer manufacturer.

Even with the aid of the polymers, some floc remains in suspension. It is possible that excess individual polymers at higher concentrations compete for the heavy metals so that many of the polymer fragments do not bind sufficient material to make them dense enough to precipitate to the bottom of the treatment container. This potential problem is not as apparent when both polymers are used. In combination, the polymers may bind common mercury molecules, thus achieving sufficient density to cause precipitation.

The pH-adjusted samples containing both polymers tend to settle as a uniform mass with a distinct boundary between the clarified zone and the settled particle mass. This phenomenon is characterized as a "blanket clear-water interface" (23) and should be maximized by identifying the optimal concentration of the polymers. Overtreatment can result in decreased Hg removal efficiencies.

Wastewater samples containing the acidic evacuation system cleanser developed a suspension gradient with respect to the height of the container. This closely resembled "hindered settling" (23) in which the fractional volume occupied by the floc is too high. Therefore, the need to monitor the pH is readily apparent. The process of pH adjustment apparently results in the formation of hydroxide compounds, allowing additional precipitation due to the increased density, thus reducing the Hg content of the supernatant by 18-20%.

Conclusion

While each polymer used separately removed up to 75-88% of the Hg from dental-operator wastewater samples, polymers N8186 and N8702 were much more effective when used in combination at the appropriate pH. The use of both polymers at 2.33-3.33 ml/l wastewater may be considered optimal and capable of removing almost 100% of the Hg from dental-operator wastewater. These preliminary results are promising and point to the need for further polymer studies. This laboratory has initiated additional studies using polymers and other treatment agents, together with waste de-watering and system automation methods, to remove Hg and other heavy metals from dental wastewater at several large dental treatment facilities. These results may lead to the development of automated treatment methods better suited for use in smaller private dental offices.

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