Mercury Decontamination of Dental Clinic Wastewater Using Electrochemical Alloy Formation

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Mercury Decontamination of Dental Clinic Wastewater Using Electrochemical Alloy Formation

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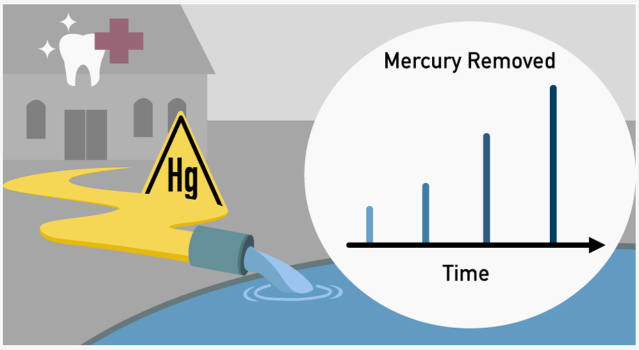

ABSTRACT: This study explores electrochemical alloy formation for mercury removal from dental clinic wastewater. Laboratory-scale experiments using wastewater from a Swedish dental clinic found a total mercury concentration of 0.68 mg/L, with 0.45 mg/L in dissolved or small particulate (<0.45 μm) form and the remainder as larger particulate mercury. Particulate mercury refers to mercury bound to solid-phase materials, including fine particles and fragments of dental amalgam. Electrochemical removal successfully captured 87% of mercury within 150 h by reducing dissolved Hg^{2+} ions at a platinum cathode, forming a stable Pt−Hg alloy. To investigate the effectiveness of this technique in practical applications, a flow reactor system based on the same electrochemical alloy removal method was installed in four dental clinics across Sweden. The reactors were installed downstream of the existing amalgam separators. While amounts vary, the reactors consistently achieved substantial mercury removal, with an estimated 340 mg to 7.5 g of mercury captured from the wastewater during 1 year of operation at each site. In total, approximately 19 g of mercury was removed, and 125,000 L of wastewater was treated. Thus, this electrochemical method effectively removes mercury not caught by amalgam separators, preventing environmental contamination.

KEYWORDS: *mercury decontamination, water treatment, dental clinic, wastewater, electrochemistry*

INTRODUCTION

Pollution of water is a critical and increasing global concern. Toxic chemicals are continuously released into rivers, lakes, and oceans, posing a significant threat to the essential role of water in supporting all forms of life.[1,2] Among the various pollutants, mercury (Hg) stands out as one of the most severe global environmental threats. This heavy metal is known for its acute toxicity, bioaccumulation, and mobility, making its pollution a critical issue recognized worldwide.[2−5] As a result, strict limits for Hg in drinking water have been set as 1 μg/L by the European Union (EU) and 6 μg/L by the World Health Organization (WHO).[4,6] In water, Hg can travel vast distances, resulting in widespread contamination and devastation. It is unique in its ability to continuously cycle within the environment, with water bodies contributing to over 60% of the global Hg cycle.[7] Hg pollution is exacerbated by human activities such as mining, fertilizer production, waste incineration, and disposal.[7] Another potentially less apparent source of Hg pollution is wastewater generated by dental clinics. For over 150 years, Hg has been used in dental practices as a filling material.[8] Today, the use of dental amalgams has been banned or is being phased out in some regions. However, it remains prevalent in many others, contributing to the risks of amalgam waste management.[9,10] In 2013, the United Nations Environment Program (UNEP) estimated that over 300 tonnes of Hg are used each year for dental amalgams, with approximately 100 tonnes entering the wastewater annually.[11]

Current Hg decontamination methods for dental clinic wastewater rely mainly on amalgam separators, which separate large fragments and particles of dental amalgams through sedimentation, filtration, or centrifugation.[12,13] However, amalgam separators are not without limitations, as studies show that significant amounts of Hg can still remain in the wastewater, allowing Hg to enter the environment, both bound in amalgam and particulates as well as in the form of dissolved Hg.[12,14−16] Amalgam separators capture solid amalgam pieces and small particles. Separators using sedimentation can capture amalgam particles down to approximately 0.2 mm in diameter (see the Supporting Information for further discussion) but do not capture smaller particles or dissolved Hg. Alternative

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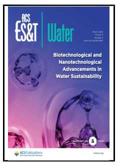

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removal strategies include adsorption-based methods, such as functionalized zeolite tuff with green rust and sulfhydryl groups, which have shown high Hg^{2+} removal capacities.[17] These materials effectively bind Hg^{2+} but rely on micropores, which can clog over time in environments with a lot of particles and organic material, such as dental clinic wastewater. This limits their lifetime and therefore makes them unsuitable for dental clinics where it is expected that installations function, without servicing or other upkeep, for about 1 year. Other options are ion-exchange media and activated carbon, which also can achieve very high levels of Hg capture, but similarly suffers from the risk of clogging due to the small effective pore sizes. Thus, there is a need for more efficient and effective Hg removal technologies specifically designed to handle wastewater generated by dental clinics.

We have recently developed a novel method for Hg removal from various aqueous solutions based on electrochemical alloy formation in which Hg ions in solution are reduced on the surface of a metal cathode electrode, such as Platinum (Pt), and then forming a stable alloy ($PtHg_4$).[18−22] This method can utilize electrodes with a large pore size, thus reducing the risk of clogging significantly. Our previous studies have shown that this removal method enables efficient Hg removal across a wide range of solution types, including industrial effluents and acidic environments.[20,21] The method has been shown to be efficient in the presence of numerous other cations and anions, as well as at different pH levels.[18,20] This versatility and relatively simple operation make the electrochemical removal method an interesting candidate for treating dental clinic wastewater.

In this study, we investigate the potential of the electrochemical removal method for Hg removal from dental clinic wastewater. Laboratory-scale (lab-scale) experiments were conducted to evaluate the removal of Hg from wastewater collected at a dental clinic in Sweden. In addition, the Hg species in the wastewater was investigated by filtration, allowing the determination of the ratio between particulate and dissolved Hg. To investigate practical implementation, a flow reactor system utilizing the electrochemical alloy formation was installed and operated for 1 year in four different dental clinics across Sweden.

■ EXPERIMENTAL SECTION

Laboratory Scale: 60 mL Batch Cell. The wastewater used in the lab-scale experiments was collected from a dental clinic in Köping, Sweden, after passing through the clinic's amalgam separator. It can be noted that the time between the wastewater collection from the dental clinic and the start of lab-scale experiments was around 1 week. The clinic's specifications are shown in Table 1.

Table 1. Locations of the Dental Clinics Included in the Field Studies, along with the Average Daily Water Flow and the Number of Dental Chairs at Each Clinic[a]

clinic no.	location	no. of chairs	avg. daily water volume [L]
1	Gothenburg	2	40
2	Degerfors	5	100
3a	Köping	4.5	90
3b	Köping	4.5	90
4	Kalmar	9	180

[a] In Köping, two flow reactors were installed in parallel.

To estimate the ratio of dissolved Hg and solid Hg in the wastewater, a small sample, approximately 0.5 mL, was extracted and filtered through a 0.45 μm filter (nylon syringe filter, article number 15131499, Thermo Fisher). This provides an approximate upper bound for the dissolved Hg concentration, as some amalgam particles may be smaller than 0.45 μm. However, the vast majority of amalgam particles are larger than 0.45 μm.[23,24]

Electrochemical Hg removal was performed using a three-electrode system with 60 mL of wastewater as the electrolyte solution. Pt wire (1 cm^2) was used for both the cathode and the anode, and a Ag/AgCl electrode (B2820+, SI Analytics) as the reference electrode (RE). A potentiostat (Reference 600, Gamry) was used to apply a constant potential of 0.1 V vs the reversible hydrogen electrode (RHE) at the cathode. The use of Pt cathodes for efficient Hg removal have been showcased in earlier publications and this specific potential was chosen in order to provide a large enough overpotential for electrochemical alloy formation, while avoiding the hydrogen evolution reaction.[18,22]

Field Studies: Flow Reactor. Five identical prototype electrochemical flow reactors, built by Atium, were installed at four different dental clinics in Sweden; see Table 1. The reactors were equipped with 17 cathode and anode electrodes enabling electrochemical alloy formation for Hg removal. At each clinic, the flow reactors were installed downstream of the amalgam separator, and upstream of the sewer outlet. The reactors were designed to treat varying volumes of dental wastewater, targeting a processing capacity of 10 L/h. To simplify operations, the reactors were powered by a constant potential, direct current (DC) power supply, where the supplied power was around 5 W and the cathode potential was in the range of electrochemical alloy formation.[22]

Hg Quantification. In all experiments, the concentration of Hg in solution was analyzed using a direct mercury analyzer (DMA, DMA-80 Hg analyzer, Milestone Srl). It can be noted that DMA-80 is an analyzer for the total Hg determination of solid and liquid samples. It is a certified method for Hg quantification, certified under US EPA Method 7473, ASTM methods D-6722-01, and D-7623-10. For lab-scale experiments, samples were taken from the electrolyte before, during, and after electrochemical treatment. Filtered samples were taken prior to and during the electrochemical Hg removal process. In the field studies, one cathode was extracted from the reactor at each measurement point during the experiment. The reactor continued to operate with the remaining electrodes. The removed cathode was completely dissolved in strong acid in order to release all captured Hg into solution. The Hg concentration in the solution was then measured, and the total amount of Hg captured by the reactor was estimated by assuming a homogeneous Hg uptake across all cathodes within the reactor.

■ RESULTS AND DISCUSSION

Laboratory Scale: 60 mL Cell. Dental clinic wastewater generally refers to all water generated during day-to-day activities within dental clinic facilities. While this wastewater is often legally classified as domestic waste, it typically contains a range of contaminants specific to dental clinic operations.[25] These contaminants can include Hg from amalgam fillings along with various other metals, disinfectants, and dental materials. Despite the wide range of chemicals present in the wastewater, its exact composition remains largely unknown, as most research is primarily focused on dental amalgams.[26]

A dental amalgam is a mixture of Hg and at least one other metal, with the Hg ratio determined by the manufacturer.[27] In Sweden, dental amalgams typically contain about 50% Hg, with the remainder composed of metals, such as copper, silver, gold, tin, and zinc.[15] In 2009, Sweden implemented a general ban on Hg, including its use in dental amalgams.[28] Despite this ban, a significant number of patients still have amalgam fillings that were placed before the regulation came into effect. Consequently, amalgam waste remains a concern in Sweden, as it continues to be generated during dental procedures such as the removal or replacement of old fillings.[15] In 2018, Hg emissions from Swedish dental clinics into outgoing wastewater were estimated to range from 0.1−56 g of Hg per dental chair annually, despite the implementation of amalgam separation processes. This corresponds to total emissions of between 1−500 kg of Hg annually that is released into the environment through the wastewater from dental clinics in Sweden.[15]

Hg contamination in dental wastewater can occur in various forms and is typically a combination of dissolved Hg species such as Hg ions (Hg^{2+}), methylmercury ($[CH_3Hg]^+$), dissolved elemental Hg (Hg^0) and undissolved Hg bound in amalgam particles.[14] While dental amalgam is generally stable in water,[29] the drain cleaners commonly used in dental clinics to prevent biological growth often contain chlorides and/or have low pH, which can significantly accelerate the dissolution rate of amalgams and stabilize Hg^{2+}.[30]

To assess the possibility of using electrochemical alloy formation to remove Hg from dental wastewater, real wastewater from a dental clinic in Köping, Sweden, was collected and treated. To investigate the dissolved and solid Hg contents in the wastewater, samples were filtered prior to any electrochemical treatment and at specific intervals during the Hg removal process. Figure 1 shows the results from the electrochemical treatment, and Table 2 presents the data along with the results from two filtration measurements taken at the start and after 76 h of treatment.

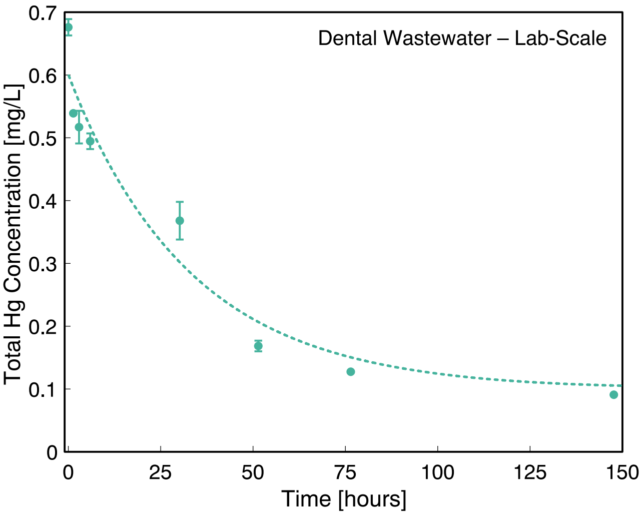

Figure 1. Hg removal at the lab scale (60 mL) from wastewater from clinic 3 in Köping. The data are fitted with an exponential fit with an offset: $y = 0.1 + 0.5e^{-0.03x}$.

Table 2. Hg Concentrations in Wastewater from Clinic 3 in Köping at the Lab Scale (60 mL) before and after Filtration, Including Dissolved Ionic Hg Forms

time [h]	total Hg [mg/L]	Hg after filtration [mg/L]	dissolved Hg [mg/L]
0	0.68	0.45	0.25
30	0.37	n/a	n/a
76	0.13	0.03	0.10
148	0.10	n/a	n/a

Dental wastewater was passed through a 0.45 μm filter that effectively captures the vast majority of larger amalgam particles, and the concentration in the filtered water provides an upper bound for the concentration of dissolved Hg. At the start of the experiment, the concentration of dissolved Hg was determined to be equal to or less than 0.45 mg/L. It is assumed that the electrochemical alloy formation process requires dissolved Hg or Hg ions to be captured at the cathode, which would mean that the lowest total concentration that would be reached would be about 0.22 mg/L in this experiment. However, the results in Figure 1 show that the removal process is more efficient than would be expected from removing only dissolved Hg. Approximately 87% of the Hg content was removed over a period of 148 h, reducing the initial Hg concentration from approximately 0.68 mg/L to a final concentration of 0.09 mg/L. Specifically, around 20% of the Hg was removed within just over 1 h, 45% within the first 30 h, 75% in 50 h, and over 80% in 76 h. The rate of Hg removal during the 1st hour of electrochemical treatment was calculated to be approximately 0.10 mg/L cm^2 h, normalized to the projected cathode area of 1 cm^2.

Several different mechanisms could explain the observation that not just dissolved and ionic Hg is removed but also a large fraction of Hg that appeared to be bound in/to larger particles. (i) Larger amalgam particles dissolve, releasing Hg that was initially inaccessible to the electrochemical removal method. However, this explanation does not explain why the total Hg concentration ultimately does not approach zero. It is, e.g., difficult to motivate why some of the amalgam particles would fail to dissolve completely. (ii) A more likely explanation is that some Hg is adsorbed onto particles suspended in the solution, likely in ionic form. Specifically, an equilibrium may exist between the Hg ions adsorbed to these particles and those dissolved in the solution (see eq 1). This mechanism has been extensively documented in the context of Hg pollution in oceans and other natural bodies of water.[31]

$$Hg^{2+}_{\mathrm{adsorbed}} \rightleftharpoons Hg^{2+}_{\mathrm{dissolved}} \qquad (1)$$

As the dissolved Hg ions are captured on the cathode, the concentration of the dissolved Hg decreases. This reduction prompts some of the adsorbed ions to be released into the solution to maintain equilibrium. This process is driven by the relative potential energy of Hg ions in solution compared with their energy when adsorbed to a particle. During the course of electrochemical treatment and Hg removal experiments, the first adsorbed Hg ions to detach and enter the solution are those most loosely bound to their respective particles. Consequently, as the experiment proceeds, the remaining Hg ions would require an increasingly lower concentration of dissolved Hg ions to detach and enter the solution. Reaching these lower concentrations would require exponentially more and more time and, at some point, could become practically unfeasible. This behavior could thus explain both the observation that more Hg than what was estimated to be dissolved could be removed and why some Hg can ultimately remain inaccessible for electrochemical removal.

The results in Figure 1 demonstrate successful removal of Hg from dental wastewater using electrochemical alloy formation at a lab scale. The error bars in Figure 1 represent the spread of repeated concentration measurements, at least two for each point. However, to transition from lab-scale experiments to real-world applications, particularly in dental clinics, where waste streams can be significantly larger, the electrochemical method needs to be scaled up. For this practical application in dental clinics, it is essential to adapt the process to a flow reactor system that can handle larger volumes of dental wastewater. This involves designing a commercial flow reactor capable of treating multiple liters per hour, ensuring continuous operation and integration into existing waste management systems in dental clinics.

Field Studies: Flow Reactor. Field studies were conducted in dental clinics in southern Sweden to investigate the feasibility of decontaminating wastewater from Hg using a flow reactor system based on electrochemical alloy formation. Five flow reactors were installed across four different dental clinics, as listed in Table 1.

In clinic 1 in Gothenburg, five samples were collected over a period of 384 days and the Hg captured by the flow reactor was quantified, as shown in Figure 2. After 23 days, approximately 5 mg of Hg was captured by one cathode (out of the 17 cathodes in total in the reactor) from the wastewater. After 101 and 174 days, around 38 and 82 mg were captured, respectively. By 298 days, over 330 mg had been captured, and by 384 days, approximately 348 mg of Hg had been captured from the wastewater. The total removal of Hg in clinic 1, assuming homogeneous uptake across all 17 cathodes, was 5.9 g. For clinic 2 in Degerfors (Figure 3), two samples were taken in total, with around 93 mg of Hg captured in 35 days and 186 mg in 183 days on one cathode. The reason for only two measurement points from this clinic was that an electrical connection failure occurred, causing the reactor to stop functioning properly. The measurement at this clinic was aborted after this, and thus, there are no samples representing 1 year or longer for this clinic. In Clinic 3 in Köping (Figure 4), two reactors were installed, for reactor a, three samples were collected after 38, 186, and 376 days with 9, 64, and 84.3

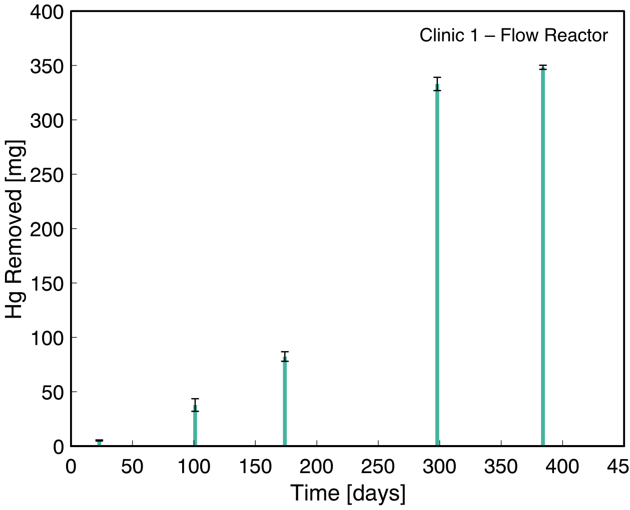

Figure 2. Amount of Hg captured by one cathode (out of 17) at clinic 1 in Gothenburg.

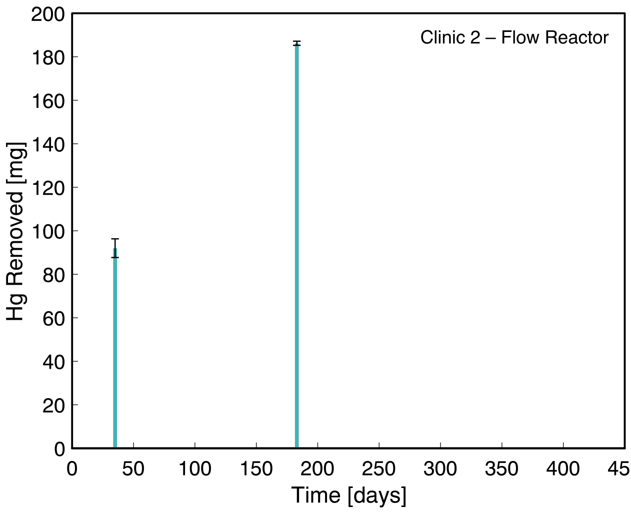

Figure 3. Amount of Hg captured by one cathode (out of 17) at clinic 2, in Degerfors.

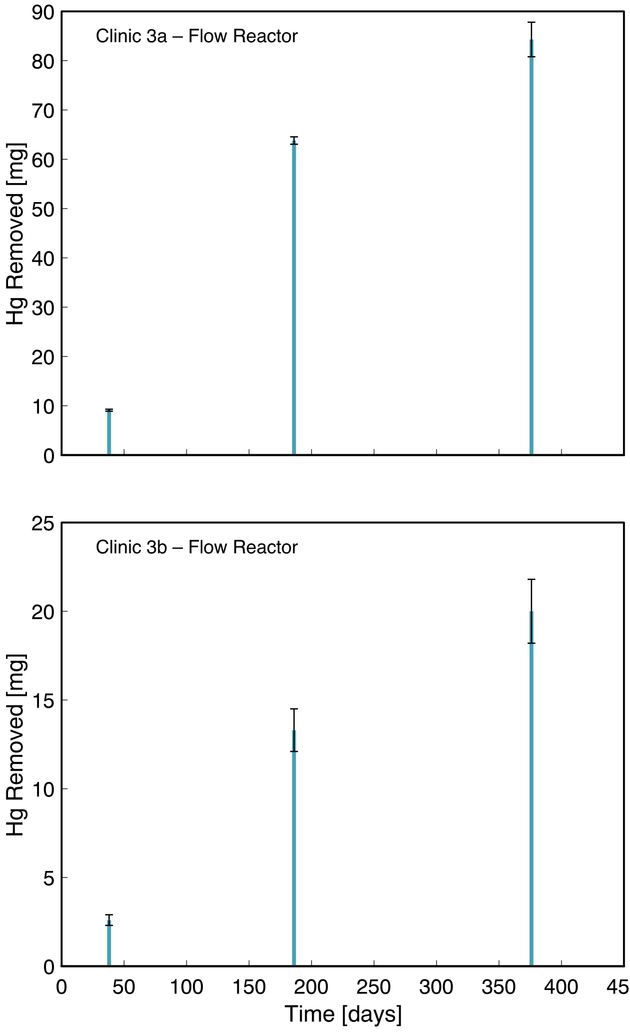

Figure 4. Amount of Hg captured by one cathode (out of 17) at dental clinic 3, in Köping (reactors 3a and 3b).

mg of Hg captured in one cathode, respectively. For reactor b, samples were collected at the same time points, resulting in the capture of 0.3, 1.2, and 20 mg of Hg, respectively for one cathode. For Clinic 4 in Kalmar (Figure 5), 22 mg of Hg was captured after 116 days on one cathode. After 1 year, or 439 days, a total of 439 mg of Hg was captured, leading to an estimated 7.5 g of Hg removed in total. An overview of estimated total Hg removal across all clinics over the course of around 1 year is provided in Figure 6. The error bars in Figures 2–6 represent the standard deviation of repeated measurements. Tables with experimental results from the field studies can be found in the Supporting Information.

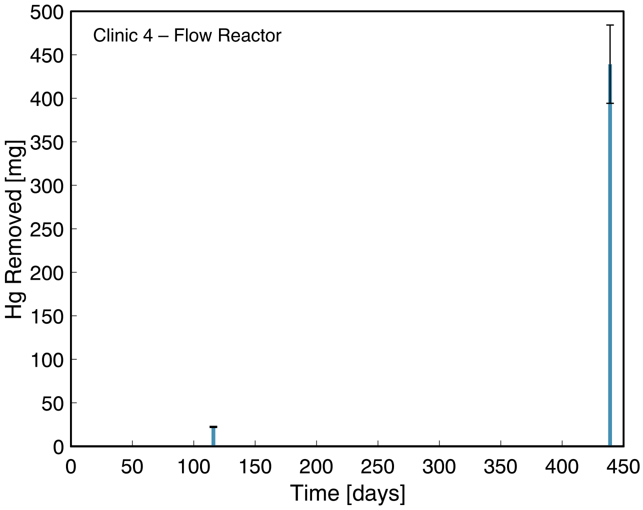

Figure 5. Amount of Hg captured by one cathode (out of 17) at clinic 4 in Kalmar.

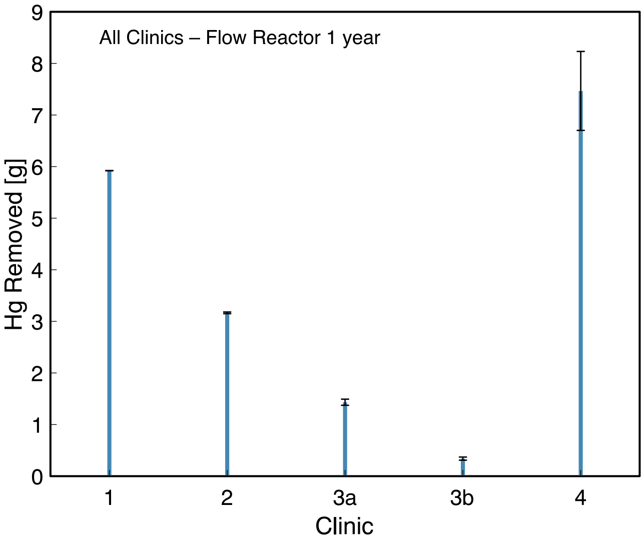

Figure 6. Amount of Hg removed in over a year of testing in all four dental clinics: (1) Gothenburg, (2) Degerfors* (data for 183 days), (3a, 3b) Köping, and (4) Kalmar, assuming homogeneous Hg uptake across all 17 cathodes.

The sample frequency varied at each clinic based on its location, as the clinics are spread across different parts of Sweden. Clinic 1 is, e.g., within walking distance of our lab at Chalmers University of Technology, which allowed for more frequent sampling, whereas, e.g., Clinic 4 is located 5 h away by car, limiting the frequency of sample collection. The difference in the amount of Hg removed over approximately the same time period between clinics can be attributed to several factors: (i) The four dental clinics differ in size, measured by the number of dental chairs, which correlates with the total amount of water passing through the reactor per day. (ii) The design of the suction system can result in a pulsed rather than continuous water flow. Water can accumulate in the suction system until its tank is full, after which the entire volume is released. The size of the tank depends on the model but is generally around 10 L. When the water is released, it flows due to gravity rather than being pumped; thus, the flow rate is a function of the pressure of the water, the pressure drop caused by the piping, the amalgam separator, and the flow reactor. The time it takes for the tank to empty is typically on the scale of a few minutes, while the interval between pulses can range from 30–60 min, depending on water usage at the dental chairs. As a result, the water in the reactor is mostly stagnant on average, leading to the majority of Hg uptake occurring during these stagnant phases. (iii) The configuration of the water pipes can lead to accumulation of dental amalgam particles, particularly in U-shaped sections, which then can gradually leach into the water. (iv) The demographics of patients, such as age distribution, vary across clinics, influencing the amount of Hg present in the wastewater due to different dental treatment needs and the prevalence of amalgam fillings. (v) The specific types of disinfectants and cleaning agents used in each clinic may also affect Hg^{2+} levels in the wastewater. Some disinfectants contain chlorides or other oxidizing agents, which can enhance the dissolution of Hg from the amalgam particles. This effect is particularly relevant in relation to amalgam accumulation in plumbing systems (as discussed in point (iii), where residual amalgam may gradually leach Hg into the wastewater over time). The chemical composition of cleaning solutions can therefore influence both the rate and the extent of Hg dissolution, contributing to variations in Hg levels between clinics. These factors (i–v) are likely to generate significant variations in the levels of Hg in the wastewater at the four different dental clinics, leading to expected differences in Hg uptake by the flow reactors.

These field studies confirm that the amalgam separators installed at dental clinics are insufficient for fully capturing Hg, leaving significant amounts in the wastewater with a large portion in dissolved form. This highlights the need for improved Hg removal processes to prevent Hg release into local water bodies. Even small residual amounts pose environmental risks, as dissolved Hg can undergo microbial methylation, forming methylmercury, which is a highly toxic and bioaccumulative compound that readily enters food webs and ultimately leads to human exposure through, e.g., seafood consumption.[31] By incorporating additional treatment steps, such as the electrochemical removal investigated here, dental clinics can more effectively capture dissolved and ionic Hg, reducing its environmental and human health impacts.

■ CONCLUSIONS

Hg was efficiently removed from wastewater generated by dental clinics in Sweden using the electrochemical alloy formation method. Lab-scale experiments on wastewater collected from one of the clinics, after passing through the clinic's amalgam separator, revealed substantial amounts of both dissolved and particulate Hg still present in the wastewater. To further decontaminate the wastewater, electrochemical treatment was used, and approximately 87% of Hg was successfully removed, reducing the initial Hg level from around 0.68 to 0.09 mg/L in 148 h. When scaled up to a flow reactor system for practical applications, the reactors demonstrated consistent performance across four dental clinics in Sweden, removing up to an estimated 7.5 g of Hg in one clinic over approximately 1 year of continuous operation, assuming homogeneous uptake across all cathodes in the flow reactor. Without such treatment, significant amounts of Hg can enter the public sewer system, thereby polluting local water sources and posing public health risks. This technology, based on electrochemical alloy formation, effectively targets and captures Hg directly from dental clinic wastewater, preventing its release into the environment and safeguarding water quality.

This study demonstrates the effectiveness of electrochemical removal for Hg decontamination in dental clinic wastewater; however, several key areas warrant further research. One critical aspect is the dissolution behavior of dental amalgam in wastewater, particularly how different disinfectant solutions used in dental clinics influence both the amount of Hg released and the rate of dissolution. Understanding these interactions could help refine wastewater treatment strategies and minimize Hg emissions at the source. Future work should also focus on expanding the data set by including more sampling points and test sites to better capture variations in wastewater composition across different clinics. This would provide a broader understanding of how factors, such as disinfectant use, patient demographics, and plumbing configurations influence Hg levels in the wastewater. Additionally, there is a need for more accessible and cost-effective Hg monitoring technologies tailored to dental clinics. Current analytical methods, such as thermal decomposition atomic absorption spectrometry, provide high accuracy but require specialized equipment and expertise (which leads to additional costs), making routine monitoring challenging for dental clinics. Developing reliable, real-time, and affordable Hg sensors could improve regulatory compliance and allow for better control of the discharge of Hg into wastewater systems. Addressing these challenges will be essential for enhancing Hg management in dental care settings and ensuring more effective environmental protection. However, large-scale implementation of the flow reactor system depends not only on technological feasibility but also on regulatory enforcement. In many cases, businesses and institutions are unlikely to adopt new treatment technologies unless stricter discharge regulations make compliance mandatory. Existing wastewater treatment practices, such as the use of amalgam separators, are often seen as sufficient under current regulations despite their limitations in removing dissolved Hg. Stricter enforcement of Hg discharge limits, along with clearer regulatory requirements for dissolved Hg removal, could drive broader adoption of advanced treatment technologies.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsestwater.4c01255.

Additional details on dental amalgam separators and experimental data from field studies. (PDF)

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Author Contributions

CRediT: **Vera Roth** conceptualization, data curation, formal analysis, investigation, methodology, visualization, writing - original draft, writing - review & editing; **Henric Ernbrink** conceptualization, data curation, formal analysis, investigation, methodology, resources, writing - original draft, writing - review & editing; **Björn Wickman** conceptualization, formal analysis, funding acquisition, methodology, project administration, resources, supervision, writing - original draft, writing - review & editing.

Notes

The authors declare the following competing financial interest(s): The authors declare the following competing interests: A patent application (EP 17199244.9) has been filed, and B.W. is listed as one of the inventors. B.W. is one of the minority owners of a start-up company Atium that is currently developing a commercial water treatment system based on the patent. In addition, H.E. is the CTO at Atium.

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