Removal of Perfluorooctanoic Acid and Microcystins from Drinking Water by Electrocoagulation

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Perfluorooctanoic acid (PFOA) and microcystins are some of the well-known chemical contaminants in drinking water in the USA. Despite the availability of filtration technologies like ion-exchange resins, activated-carbon, and high-pressure membrane filters, these contaminants still remain widespread in the environment. In the present study, two innovative aspects of electrocoagulation techniques were tested, (a) cheap and easy-to-operate field-unit instead of hi-tech electrocoagulation and (b) reverse-polarity instead of conventional polarity, and applied to remove PFOA and microcystins from drinking water sources. The method presented here outperformed commercial activated-carbon filtration by nearly 40%. When the efficiency of electrocoagulation was examined in terms of voltage discharge, pH, and reverse-polarity, the results averaged 80% decontamination for individual treatment, while their combined effects produced 100% detoxification in 10–40 minutes, exceeding recently published results. The method shows great economic promise for water and wastewater treatment and chemical recycling.

1. Introduction

Perfluorooctanoic acid (PFOA) and microcystins (Figure 1) are some of the well-known drinking water contaminants in the USA [1]. Unfortunately, consumption of PFOA and microcystins is associated with very serious health problems such as elevated blood cholesterol, gastrointestinal infection, depressed immune system, kidney and liver disease, diabetes, and cancer. Also, growth deficiency among little children has been reported [2–9].

The US EPA health regulatory limit for PFOA in drinking water is < 0.07 μg/L and the WHO guidelines limit for microcystins lifetime exposure is 1.0 μg/L [11]. Despite current regulations, high concentrations of PFOA (1.0–2.4 μg/L) and microcystins (12.5–225.6 μg/L) have been detected in drinking water in multiple US communities [12–14].

The environmental distribution of PFOA and microcystins is closely related to their chemistry. The persistence of PFOA, for example, is attributable to the strength and low polarizability of the carbon-fluorine (C-F) covalent bond. Also, its thermal stability, surfactant behavior, and stain-resistant properties are the reason for its wide use in the production of industrial items like fluoropolymer products, firefighting foams, stain-resistant coatings, and electroplating [15]. PFOA is not lipophilic, which means that it can bind to serum albumin making it hard to excrete from the human body [4–7, 16]. On the other hand, microcystins belong to a family of stable monocyclic heptapeptide compounds, of which nearly 80 variants are now known. The two most widely known moieties are microcystin-LR and microcystin-RR and are considered to be the most potently infectious toxins against human internal organs [16, 17].

A number of methods have been developed to remove PFOA and microcystins from drinking water, including activated-carbon filtration [18, 19], ion-exchange resins [19], high pressure membranes [20], peat absorbance [21], iron-based absorbance [22], and biosorbert material (crustacean-shell) filtration [23]. The biggest problem is that many of these methods are sophisticated, expensive, and hard to
operable. Other laboratory-based techniques do not easily lend themselves to industrial application. Still, some lack the required efficiency to meet EPA decontamination standards. Consider, for example, eleven groundwater water wells in Vienna (W.Va.) (latitude: 39° 19′ 36″ W and longitude: 81° 32′ 55″ N) which are all fitted with granulated activated-carbon filters. Yet, a statewide EPA survey in 2016 revealed that PFOA levels in drinking water were in excess of 0.40 μg/L compared to the health regulatory limit of <0.07 μg/L (personal communication with Vienna City Council–May 16, 2016). The Grand St. Mary’s Lake drinking water source in Mercer County (Ohio) has similar problems. Water algal data monitored between 2015 and 2019 by the Ohio EPA showed that microcystin load in the lake ranged from 0.0 to 79.7 μg/L, compared with the tolerable limit of 1.0 μg/L [24]. One of the most effective ways of removing PFOA and microcystins is electrocoagulation, mainly because of its deployment of bond-splitting electrical energy [25, 26]. Nevertheless, only a few studies have examined cheaper, easy-to-operate electrocoagulation techniques or studied system optimization based on electrical reverse polarity. In view of this, we hypothesize that reverse-polarity electrocoagulation factors like radiative energy, hydroxyl radicals (OH·), and solvated electrons will effectively degrade PFOA and intermediate fragments (e.g., PFBA, PFHxA, and PFPeA) as well as microcystins from contaminated drinking water.

The purpose of this study is to (1) examine the efficiency of a cheap, easy-to-operate electrocoagulation field-unit at removing PFOA and microcystins from drinking water sources, (2) investigate decontamination efficiency on the basis of the electrocoagulation reverse polarity (ECRP) method, and (3) validate results against activated-carbon filtration and published data. The study will raise questions about electrocoagulation residue recycling.

### 2. Materials and Methods

#### 2.1. Equipment and Raw Materials. An electrocoagulation unit (Figure 2(a)) was deployed in this study following a design by Rutberg et al. [26]. The basic raw material is groundwater samples from Vienna (W.Va.) and untreated water from the Grand St. Mary’s Lake in Celina (Ohio). Potassium aluminum sulfate (KAl₂(SO₄)₂·12H₂O) (potash alum) solution was used as both electrolyte and coagulant. Potash alum exhibits strong coagulation properties when dissolved in impure water.

The electrocoagulation unit consists of a 400 gallon steel tank powered by a high amperage and low voltage generator designed to provide energy via switching polarity from direct current electric discharge. A characteristic component is the approach by Bao et al. [35]. Nevertheless, only a few studies have examined cheaper, easy-to-operate electrocoagulation techniques or studied system optimization based on electrical reverse polarity. In view of this, we hypothesize that reverse-polarity electrocoagulation factors like radiative energy, hydroxyl radicals (OH·), and solvated electrons will effectively degrade PFOA and intermediate fragments (e.g., PFBA, PFHxA, and PFPeA) as well as microcystins from contaminated drinking water.

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#### 2.2. Experimental Methods. Treated water was sampled every 10 minutes for 90 minutes based on ALS Field Sampling Handbook instructions. Filtrates were analyzed by the ASL Environmental Laboratory (ASL) in Middletown (PA) and Industrial Laboratories (ICL) in Denver (CO), while ASL is specialized in general and PFOA testing, and ICL is specialized in microcystin and bacteriological testing.

The ASL procedure for analyzing aqueous samples of PFOA is summarized below. The analysis was done using the Agilent 1100 tandem High Performance Liquid Chromatography-Mass Spectrometer (HPLC-MS). First, Teflon materials were avoided, and all glassware was thoroughly washed and rinsed with methanol and distilled water to prevent contamination. Next, aliquots of 25 μL were injected onto the system’s column (2.1 mm x 100 mm) with 10 mM ammonium acetate and acetonitrile as mobile phases, initializing with 40% acetonitrile at a flow rate of 250 μL/min and column temperature of 40°C. The gradient was increased to 90% acetonitrile at 9 minutes and then held for 2 minutes. A 5-minute reequilibration interval was run before each subsequent sample. The gas temperature and ion spray voltage were maintained at 350°C and 4000 V. Finally, ions were monitored via the multiple reaction monitoring mode, and PFOA transitions matched against standards following the approach by Bao et al. [35].
The ICL microcystin analysis procedure is also described here. The analysis was implemented using the HPLC Agilent 1100 instrument coupled with a diode array detector. Because of the saturated water from the Grand St. Mary’s Lake, water samples were first filtered with Whatman filter paper (1.2 μm) and chilled overnight at −20°C. The filtrate was dissolved in 400 μL methanol and treated with a 2mg/L sodium thiosulphate and acidified with trifluoroacetic acid (TFA, 0.1%, v/v), concentrated via solid phase cartridges (SuperClean LC-18, 3mL Tube), and eluted with 15mL of 0.1% TFA in methanol. Like before, aliquots of 20 μL were injected into system’s column (150 × 4.60mm) at a flow rate of 1mL/min at 30°C column temperature. The mobile phase consisted of H2O plus 0.05% TFA and acetonitrile plus 0.05% TFA, with a linear increase from 30 to 70% of the latter between 0 and 30min. Finally, chromatograms were recorded at 238nm based on literature. UV spectra and all chromatographic peaks were examined and compared to spectra standards of microcystin moieties. Peaks possessing the UV spectrum characteristic for microcystins were quantified using a calibration curve. Unidentified peaks possessing the UV spectrum characteristic for microcystins but not matching the retention time of the standards were quantified as microcystins-LR equivalents with a detection limit of 0.01 μg/L [28]. To determine PFOA concentrations for 2016 and microcystins for 2017, each contaminant was measured seven consecutive days in spring, summer, and fall, and a regional mean was calculated.

The electrocoagulation reverse polarity (ECRP) reaction follows a complex electrolytic procedure [27]. Here, the primary reactions at the anode and cathode are described by the following equations:

$$2H_2O(l) \rightarrow O_2(g) + 4H^+(aq) + 4e^- \rightarrow \text{(anode)}$$

$$4H_2O(l) + 4e^- \rightarrow 2H_2(g) + 4OH^- (aq) \rightarrow \text{(cathode)}$$

$$6H_2O(l) \rightarrow 2H_2(g) + O_2(g) + 4H^+(aq)$$

$$+ 4OH^- (aq) \rightarrow \text{(overall)}$$

while reductants (free electrons) are released from the anode, oxidants, and flocculation aggregates (e.g., H2O2, Al(OH)3, and Al2O3) are generated at the cathode [3].

The pollutant removal efficiency (%) was calculated using the following:

$$\% r = \left(\frac{C_0 - C_t}{C_0}\right) \times 100\%,$$

The diagram shows the electrocoagulator setup with untreated and treated water flow paths, and the key components labeled.
where \( C_0 \) is initial concentration of pollutant and \( C_t \) is concentration of pollutant at time \( t \).

The filtrate results were compared with data derived from a commercial gravity block ionic adsorption unit fitted with granular activated-carbon filters, which are coated by silver-impregnated ceramic outer shells [27].

### 3. Results and Discussion

#### 3.1. PFOA Removal Efficiency

The regional mean concentration of PFOA in drinking water was first determined. This is important for two reasons: (1) to measure electrocoagulation efficiency against EPA’s health advisory limit and (2) to determine the effect of electrocoagulation parameters on PFOA removal. The PFOA concentration in the Vienna district (\( \sim 0.126 \mu g/L \)) was calculated as the average of seasonal means is shown in Table 1.

The data shows that PFOA toxicity is nearly 44% above the EPA health regulatory limit of 0.070 \( \mu g/L \), indicating that the Vienna (West Virginia) community has PFOA levels similar to or greater than many communities in the USA and other parts of the world [1, 3, 28, 29]. The data in Figure 3 is a comparative analysis between PFOA results (Table 1) and a commercial source activated-carbon filtration.

The results in Figure 3(a) show that the coagulator degraded PFOA by nearly 60% below the EPA health advisory limit of 0.070 \( \mu g/L \) in 40–50 minutes, following a negative exponential model (Figure 3(b)). This compares well with results published by Bao et al. [25], Rutberg et al., [26] and Gadja et al. [30]. When the results were evaluated against granular activated-carbon filtration, the coagulator outperformed its counterpart by more than 40% (Figure 3(a)).

The question is what factors make electrocoagulation effective at PFOA removal? Electrocoagulation condenses electrical energy to split C-F bonds when voltage discharge, pH, and electron polarity are optimized in electrolytic cells [25, 26]. Gadja et al. [30] demonstrated the potential of fuel-cell pulse discharges at splitting C-N bonds in wastewater. Also, Choi et al. [31] used an iridium catalytic approach to clamp dialkylphosphino molecules and used the intermediate radicals to cleave C-F bonds. In the present study, experiments were performed to determine the effects of voltage discharges, pH, and polarity on PFOA removal; the results are displayed in Figure 4.

Voltage discharge is a key factor at removing dissolved contaminants in the electrocoagulation process [25, 31, 32]. Voltage degradation of PFOA molecules starts with dissolution of sacrificial electrodes driven by high oxidation potential. Consequently, hydroxyl radicals (OH\(^-\)) are generated ((1) and (3)) leading to the attack and dissociation of C-F bonds [25, 33, 34].

The data in Figure 4(a) shows 60% removal of PFOA in the first 10 minutes of voltage treatment and confirms previous findings that voltage discharge is directly proportional to chemical decontamination (Bao et al. [35]). In this study, 24 V was considered the optimum pulse discharge, degrading nearly 85% of PFOA in 30 minutes. Bao et al. [25] recently reported 70–90% removal of per- and polyfluoroalkyl substances (PFAS) using a 12 V laboratory-scale electrocoagulation device.

But what is the physics behind voltage decontamination? The answer lies with Faraday’s law which explains the relationship between voltage (electromotive force) and magnetic media [25]. Here, cations were dissolved from the sacrifice anode, while electrolytic reaction at the cathode increased the concentration of solvated OH\(^-\) ions to accelerate the formation of metal hydroxide flocculants needed to adsorb and eliminate PFOA.

The role of pH in electrocoagulation processes is emphasized in many freshwater and wastewater detoxification studies (e.g., Luz-Pedro et al. [36] and García-García et al. [37]). The reason is that pH is closely related to solution equilibria and sensitive to cation adsorption, flocculation, and decontamination (Bao et al. [25]). In the present study, the effect of pH values at 4.0, 6.0, 8.0, and 10.0 on PFOA removal was examined. As shown in Figure 4(b), slightly alkaline condition (pH = 8.0) was most favorable for PFOA degradation contrary to previous studies by Bao et al. [25] and Kim et al. [38]. The difference was attributed to OH\(^-\)-rich flocculants in this experiment.

Electrical polarity defines the exchange of pulse discharge between electrodes, which directly affects electrocoagulation determinants such as stirring or agitation speed, collision contact with pollutants, and flocculation. The electrocoagulation reverse polarity (ECRP) technique was employed, with the chief benefit being doubled adsorption at the electrodes by reverse turbulence. As expected, increasing polarity is directly proportional to PFOA removal (Figure 4(c)), with optimal polarity measured at 5 seconds (~86% removal). The decline in decontamination at higher polarity (10 seconds) may be related to a stirring threshold, which when exceeded is likely to destroy flocculation. It is worth noting that none of the individual coagulation treatment exceeded 85% decontamination efficiency. However, their combined effect readily produced 100% removal as evidenced in Figure 4(d). This result exceeded findings from similar studies such as Bao et al. [25] and de la Luz-Pedro et al. [36].

#### 3.2. Microcysts Removal Efficiency

The electrocoagulation method was further validated based on microcystin detoxification using untreated lake water (Grand St. Mary’s Lake in Celina, Ohio). The data in Table 2 represents seasonal microcystin loads of the lake. Following the previous approach, the pollutant removal efficiency (Figure 5) was measured starting from the regional mean microcystin load of 147.0 \( \mu g/L \) (Table 2). Figure 5 shows that microcystins removal efficiency was better than PFOA cognizant that the coagulator nearly completely removed 99.2% of the toxins in less than 10 minutes, compared to 60% PFOA in 40 minutes. Detoxification at this rate was attributed to easier dissociation of \( \alpha \)-peptide bonds (C-N) in contrast with the more stable C-F bonds in PFOA. Still, the results closely matched findings by authors like Miao et al. [39] who previously examined decontamination of microcystin-LR and microcystin-RR moieties using ozone bond dissociation.
Table 1: Measured PFOA concentration (μg/L) from drinking water in Vienna (West Virginia) in 2016.

| Measurement day | Spring   | Summer  | Fall    |
|-----------------|----------|---------|---------|
| 1               | 0.128    | 0.126   | 0.126   |
| 2               | 0.138    | 0.136   | 0.099   |
| 3               | 0.098    | 0.128   | 0.126   |
| 4               | 0.122    | 0.142   | 0.129   |
| 5               | 0.126    | 0.128   | 0.132   |
| 6               | 0.125    | 0.102   | 0.126   |
| 7               | 0.129    | 0.132   | 0.136   |
| **Mean**        | **0.124**| **0.128**| **0.125**|
| **STDEV**       | 0.12     | 0.13    | 0.12    |

Figure 3: (a) Rate of PFOA removal by electrocoagulation compared with activated carbon and (b) negative exponential model of PFOA degradation.

Figure 4: Continued.
Aside from flocculation and adsorption, electrocoagulation further utilizes ozone and hydroxyl radical attack on polar bonds [35]. The present study followed mechanisms previously elucidated by Miao et al. [39]. We agreed with the above authors that the conjugated diene structures of the microcystin-LR moiety were attacked by hydroxyl radicals to produce dihydroxylated products. These were then cleaved into aldehyde or ketone peptide residues, which were

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### Table 2: Measured concentration (μg/L) of microcystins in Grand St. Mary’s source of drinking water (Celina, Ohio) in 2017.

| Measurement day | Spring | Summer | Fall |
|-----------------|--------|--------|------|
| 1               | 147.8  | 155.4  | 144.8|
| 2               | 146.3  | 144.8  | 145.3|
| 3               | 149.9  | 163.1  | 142.6|
| 4               | 131.4  | 146.4  | 145.2|
| 5               | 144.6  | 140.6  | 146.6|
| 6               | 155.5  | 142.8  | 144.5|
| 7               | 148.9  | 145.3  | 154.2|
| **Mean**        | **146.3** | **148.3** | **146.1** |
| **STDEV**       | 7.433  | 7.998  | 3.773 |

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![Figure 4: PFOA removal efficiency based on (a) voltage capacity, (b) pH, (c) polarity, and (d) combined factors.](image)

![Figure 5: Rate of electrocoagulation removal of microcystin compared with WHO health advisory limit.](image)
subsequently oxidized into carboxylic acids and neutralized by alkaline media. A similar attack was anticipated on the benzene ring of the microcystin-RR side chain [39].

The role of voltage discharge, pH, and reverse polarity in electrocoagulation processes has already been emphasized. By comparison, the optimal effect of individual factors on microcystins removal (Figure 6) was found to be very similar to PFOA (∼80%) as shown in Figure 3. The main difference was the ease of microcystins peptide bond dissociation explained in the preceding section. As expected, effect of the combined electrocoagulation factors resulted in complete decontamination of microcystins (100%) within 30 minutes, quite comparable to PFOA.

3.3. End Products of PFOA and Microcystin Decomposition. Figure 7 shows sample residues from PFOA and microcystins degradation. Subsequent studies will focus attention on the chemistry of mineralized end-products; but for now, a crude analysis is pursued to just highlight some expected fragments. A study by Zhang et al. [40] on PFOA mineralization lends itself to comparison. They investigated PFOA decomposition using $^{60}$Co γ-ray irradiation and observed initial elevation of key intermediate products like PFHpA (C$_6$F$_{13}$COOH) and PFHxA (C$_5$F$_{11}$COOH). They concluded, however, that organic intermediates were transient and completely degraded after 20 minutes of irradiation. The current study is similar to the above in two ways. First, both employed radiative energy, hydroxyl (OH$^-$) radicals, and solvated electrons under alkaline conditions to decompose PFOA molecules. Secondly, both studies achieved highly comparable results at complete (100%) PFOA removal within 20–40 minutes. We therefore assume that degradation of PFOA and intermediate compounds including PFBA (C$_3$F$_7$COO$^-$) and PFPeA (C$_4$F$_9$COO$^-$) is quite comparable to previous research. This will be subjected to further investigation in future studies.

A second study of interest is by Triantis et al. [41]. In that study, aqueous samples of microcystin were irradiated using nitrogen doped TiO$_2$ (N-TiO$_2$) photocatalyst, under UV-A, solar and visible light. The researchers observed 99% degradation of microcystin molecules after 20 minutes of illumination and determined total organic carbon (TOC) and inorganic ions including NO$_2^-$, NO$_3^-$, and NH$_4^+$ as end-products. Similar to PFOA, there is substantial agreement between the current and previous studies, in terms of methodology and 100% efficiency of microcystins removal. We therefore conclude that the end-products of the present case are very similar, being harmless carbon and nitrogen-based fragments. Still, this will be a topic for further investigation.

3.4. Comparison of Technical and Economic Benefits of ECRP with Other Methods. Table 3 compares the technical and economic performance of the electrocoagulation reverse-polarity technique (ECRP) with other published methods. First, the ECRP compares well with similar radiative...
methods at complete removal of PFOA and microcystin molecules and outperforms traditional (single polarity) electrocoagulation and granular activated-carbon filtration by more than 20%. Additional benefits of this experiment include its applicability to drinking water and wastewater decontamination, low cost, zero health risk, and easy operability (Figure 2). Let us consider the cost aspect (which is the easiest), for example. In the USA, the average cost of drinking water per household is about $2.40/400 gal/day (capacity of prototype in Figure 2). At the same time, the cost of industrial water filtration by activated carbon is nearly $0.06 per household consumption. With the estimated cost of electrocoagulation at $0.04 at the same rate, the current method is not only efficient, but also cheaper by more than 30% [27]. The conclusion here is that the technical and economic benefits from this approach are better or highly comparable to similar published methods (Table 3).

3.5. Challenges and Research Questions. The greatest challenge to this study is further validation and elucidation of chemical end-products with related mechanisms. This makes the following research questions imperative: (1) what is the chemical nature of flocculent residues and mechanisms describing the same? (2) How toxic are the residues? (3) How can residues be recycled or disposed of? Future studies will target infrared and mass spectroscopy, HPLC, and cation/anion exchange analytical methods to help address some of these questions.

4. Conclusions

This study has confirmed previous studies, where perfluorooctanoic acid (PFOA) and microcystins are a major source of hazardous contaminants in drinking water in the USA. Two major conclusions are that (1) PFOA contamination of drinking in the Vienna community (West Virginia) can be as high as 0.126 μg/L compared with the EPA health regulatory limit of 0.07 μg/L and (2) microcystins toxicity at the Grand Lake St. Mary’s in Celina (Ohio) can reach 147.0 μg/L, compared to the limit of 1.0 μg/L. Two innovative aspects of decontamination techniques were investigated and applied to PFOA and microcystins removal: (a) field-unit instead of laboratory-based electrocoagulation and (b) reverse polarity instead of conventional one-way...
Data Availability

The raw PFOA and microcystins measurement data have been discussed in the text. The remaining analytical spreadsheet data are being restricted while the research findings are being further validated for commercial application. Requests for the entire research data may however be considered by the corresponding author 12 months after the publication of this article.

Conflicts of Interest

The authors declare no conflicts of interest.

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