Modern Environmental Science and Engineering (ISSN 2333-2581)
March 2026, Volume 12, No. 1-3, pp. 1-12
Doi: 10.15341/mese(2333-2581)/01-03.12.2026/001
Academic Star Publishing Company, 2026
www.academicstar.us

Bench-Scale Testing for Sustainable Flowback Water Reclamation

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Abstract: Freshwater scarcity and increased cost put pressure on the need to find innovative ways to reuse water in the oil and gas sector. Flowback water represents the crucial period following fracking activity where the injected frack fluids in the formation returns to the surface and collected for proper management. Flowback has undesirable characteristics which makes it unusable for practical reuse, unless adequately treated. The purpose of this study is to evaluate viable and practical methods to reclaim flowback water for beneficial industrial and societal reuse, aiming to enhance environmental protection. Batch treatments of flowback water included ozone, electrocoagulation, and combined charcoal-ozone. Results showed that electrocoagulation was more effective, yielding a 90% reduction in COD compared to 52% with the ozone treatment while TSS and turbidity were reduced to 99% and 90% by the electrocoagulation and the ozone treatment, respectively. Reduction in priority heavy metals ranged from 21% to 62% while destruction of heterotrophic bacteria was 90% and 100%, respectively. The charcoal-O_3 technique proved largely ineffective as a treatment modality.

Key words: flowback, treatment, COD, turbidity, TSS, reuse

1. Introduction

Deep shale and other unconventional formations can provide access to very large quantities of oil and natural gas [1]. Fracking also referred to as hydraulic fracturing, is a well stimulation technique commonly used to extract oil and natural gas from deep, tight rock formations or other unconventional formations. Fracking fluids represent complex fluid mixtures composed of proppant, chemicals, iron controlling agents, clay stabilizer, gelling agents, quartz, surfactant [2]. These chemical additives are injected at a high-pressure and volumes into the deep well to create and prop open fractures, improve the flow of fluids, kill bacteria, and allowing hydrocarbons to flow to the surface [3]. After fracturing is completed, the pressure of the formation declines, and the zone settles down on the proppant, but leaving the fracture open. The thixotropic characteristics of the fluid mixture dissipates and the fluid flows back to the surface [4]. Fracking is often combined with horizontal drilling, unlocked vast unconventional reserves, boosting energy production but raising environmental concerns regarding water usage, potential contamination, and induced seismicity, making it a significant, yet debated, part of the modern oil and gas industry.

Two distinct types of wastewater streams known as flowback and produced water, are generated during the fracking operations timeline. Due to time and operation dependency, each waste stream has distinct chemical compositions that varies with the formation. Flowback consists of a mixture of chemical additives and natural occurring substances that comeback to surface following the hydraulic fracturing operations. Produced water is the combination of the fluid mixtures that return to the ground surface following gas well

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production and generated throughout the lifespan of the well. Flowback fluid comes out of the well within the first two weeks post fracking and the volume is contingent on the geologic formation, and operating procedures. Current estimation states that approximately 10-40% of the fracturing fluid flows back to the surface as flowback and produced water [5].

The management of flowback fluids is of great concern to regulators and the public at large. Water requirements for typical fracking operation on the average requires several millions of gallons of water per well. Furthermore, the fracking process itself contaminates millions of gallons of water that are drilled at each site with an array of chemicals that may carcinogenic [6]. Securing the water necessary for use in shale or unconventional gas development is progressively challenging operators to adopt innovative, affordable, and sustainable sourcing strategies. In the oil and gas sector hydraulic fracturing currently has the lowest rate of water recycling; and this is partially due to the limited use of flowback fluid and the smaller amount of waste water created [6]. Beyond the sourcing challenge, highly saline flowback and produced water cannot be discharged to its original ecosystem because the sourced water once used does not meet the water quality criteria as it relates to the Canadian Council of Ministers of the Environment [7]. Because most of that water is hauled away to be injected in deep disposal wells, there is concern that this highly contaminated water could migrate into existing aquifers actively used for domestic purposes or may discharge in surface water systems. Given the excessive costs associated with water procurement and management throughout the shale gas development cycle, this research evaluated potential treatment solutions. Opportunities to reduce freshwater consumption and optimizing flowback management as a resource in the quest for achieving operational sustainability and increasing reuse, may be created and adopted. The primary objective of this study is to evaluate various treatment methods for treating flowback and its reuse in society for beneficial purposes.

2. Material and Methods

2.1 Flowback Sample

The flowback sample used in the study originated from the Montney Formation, NE, BC, and was collected during the final stage of the fracking operations. The Montney Shale Formation lithology is mainly composed of silt, siltstone, limestone with varying degrees of dolomization and rich in natural gas and liquids. This brittle composition of the lithology contributes to successful hydraulic fracturing operations. The surface to the top depth of Montney Formation ranges from 500-4500 m that further increases from northeast to southwest [8]. A total of 30 L of contaminated hydraulic fracturing fluid was collected in 3×10 L HDPE, labelled and capped with a six cm ID plastic lid. Field screening with the GR-110E does not reveal the presence of naturally occurring radioactive constituents in the flowback samples. The samples were stored in a cooler filled with ice, transported to the laboratory where they were consolidated in a 30-gal HDPE container, labelled and refrigerated at 6°C±1. Before use, the sample was gently homogenized to allow collection of representative subsamples. The screening test found no NORM constituents in the flowback fluid.

2.2 Sample Analysis

Parameters of interest were based on strategic environmental concerns and reported in Table 1 along with the methods and the quality assurance and control (QA/QC) of the testing program. Additionally, QA/QC program included duplicate and method blank. The parameters of concern for the study consisted of COD, turbidity, TDS, TSS, heterotrophic bacteria, heavy metals, and pH, respectively. Of particular note, COD is a parameter of particular interest in oil and gas development; high COD creates an environment where bacteria could rapidly degrade the efficiency of additives in frac water [9].

2.3 Experimental Design

Several treatment methods were investigated involving ozonation, electrocoagulation, and charcoal bed sorption. Furthermore, a 400 mL portion of the original frack water sample underwent physical treatment, which involved placing the raw sample in a blender and mixing it for 0.50 minute at high speed. The homogenized sample was subsequently labelled, capped and stored in the fridge prior to be used in various treatment processes. The relative efficiency of a treatment (RTE) was calculated according to Eq. (1):

$$\%RTE = \frac{C_o - C_f}{C_o} \times 100 \tag{1}$$

Where C_0 and C_f is the initial and the final concentration, respectively.

Table 1 Summary of parameters QA/QC

[1] **Parameters**	[2] **Method**	[3] **QA/QC**
[4] pH	[5] Orion Ag/AgC1 glass probe	[6] As specified by manufacturer
[7] COD	[8] Method 10212	[9] As per procedures manual for Hach DR/2010
[10] TSS	[11] Method 8006	[12] As per procedures manual for Hach DR/2010
[13] TDS	[14] Dissolved Solid Meter	[15] As specified by manufacturer
[16] EC	[17] Orion Star A212	[18] As specified by manufacturer
[19] Cr^{+6}	[20] Method 8023	[21] As per procedures manual for Hach DR/2010
[22] Cu	[23] Method 8506	[24] As per procedures manual for Hach DR/2010
[25] Mn	[26] Method 8034	[27] As per procedures manual for Hach DR/2010
[28] Turbidity	[29] Nephelometric	[30] As per procedures manual for Hach Turbidimeter Mode1 2100 A
[31] Radioactivity (NORMs)	[32] GR-110E field system	[33] As specified by manufacturer
[34] DO	[35] HQ40d Multi-Parameter	[36] As specified by manufacturer
[37] ORP	[38] Orion5Star^{TM} Meter	[39] As specified by manufacturer
[40] *HPC-Total	[41] Millipore Sampler	[42] As specified by manufacturer

*Heterotrophic Plate Count

2.3.1 Ozone Treatment

As an oxidizer, ozone (O_3) was evaluated because of it is an eco-friendly, strong and water-soluble oxidant (2.07 V potential). Additionally, this method has high effectiveness and rapid reaction time (5-30 minutes). It provides strong disinfection capabilities and oxidizes organic substances and heavy metals while generating no harmful byproducts. A further benefit, ozone increases dissolved oxygen levels and reduces the overall formation of sludge. These combined attributes make ozonation an attractive and versatile technology for the treatment of complex wastewater matrix such as flowback fluids. A multifunction compact and portable with corona discharge, Model MP-3000, ozone generator was used. The unit has an output capacity of 800 mg of O_3/h. Ozone treatment consisted of quantitatively transferring 200 mL of flowback samples in a 250 ml partially capped Erlenmeyer flask. The batch treatment systems were ozonated at a constant ozone concentration of 2100 mg/hr and subjected to varying settling times. The combination of the experimental pairings for ozonation and settling times were as follows: treatment 1: (2 min ozonation and 30 min settling); treatment 2: (2 min ozonation and

60 min settling); treatment 3: (2 min ozonation and 180 min settling); treatment 4: (45 min ozonation and 0 min settling). Following the treatment, the samples were transferred into labelled centrifuges tube and centrifuge for three minutes at 6500 RPM and the supernatants collected for analysis. All supernatants were capped and refrigerated prior to be analyzed. The analytical program involved pH, COD, TDS, EC, DO, Cr^{+6}, Mn, Cu, turbidity, HPC-Total, and TSS. The analyses were generally performed in duplicate within a few hours of treatment and reported as the average value.

2.3.2 Electrocoagulation Treatment

The 110 V laboratory electrocoagulation prototype features a 0.50 L empty treatment cell capacity that accommodates up to nine metal electrodes. Sacrificial anode and cathode were made of the same aluminum elemental metal. Electrode dimensions are 3 cm wide × 36 cm long × 0.3 cm thick and fits in a 0.5 × 30 cm slot having 0.40 cm groove carved in the treatment cell. Electrode material strongly influences the nature of electrochemical reactions occurring in an electrocoagulation system. It is generally found that aluminum yields a higher level of treatment effectiveness [10]. All treatments in the reactor chamber were conducted with two aluminum electrodes configured in parallel. The gap between the two electrodes was 2 cm. Such gap allows to minimize significant ohmic change and the electrode overpotentials which may result in the fluid and the electrodes being over heated. The treatment process involved batch-treating 450 ml of flowback sample in the treatment cell by passing an electric current across the metal electrodes. The flowback having a pH of 6.5, consequently favors the formation of aluminum hydroxide, i.e., $Al(OH)_3$ as the coagulant. Electrocoagulation tests were conducted at 10 V, with treatment 1: 10 V for 60 s and treatment 2: 10 V for 120 seconds. The experimental design allowed for comparing the effect of voltage duration on the frac fluid characteristics. Following the electrolysis time in the electrocoagulation reactor, the treated sample was transferred to a 1 L glass beaker, sealed, and left to settle overnight to allow the coagulation process to proceed. The clear liquid was then carefully transferred to a 1 L Erlenmeyer flask using a 60 m plastic syringe, parafilmed, and kept refrigerated prior to analysis. The analytical program was similar as in ozonation.

2.3.3 Charcoal Treatment

This treatment method was applied to both original flowback sample and samples previously treated with ozone within a treatment train concept. Raw samples were contacted with a commercial grade, medium size bamboo charcoal at a 2:1 liquid-to-bamboo ratio for a 5-minute equilibrium time. After a 5-minute centrifugation at 6500 RPM, the supernatant was collected and analyzed for EC, pH, TDS, Cu, Cr^{+6}, and Mn. The separated charcoal was then retrieved and reused on fresh treated samples. This reuse cycle was repeated three times to assess the charcoal's effectiveness over multiple uses. An additional test was conducted on a blended sample at a ratio of 2:1; 10:1; 5:1, 3.33:1, and 2.5:1 liquid-to-bamboo ratio and followed the same protocol as above.

3. Results and Discussion

3.1 Ozone Treatment

The impact of ozone treatment on EC evolution is depicted in Fig. 1. Change in ionic strength in the flowback fluid ranged from 83 to 174 mS/cm. Compared to the original frac sample, the O_3 treated samples showed a marginal increase in EC across varying settling times, with the highest value (174 mS/cm) denoted in the O_3 treated sample analyzed without prior settling.

In this context, the marginal yet noticeable increase in EC values ranging from 83 to 174 mS/cm is attributed to formation of organic compounds into ionic intermediates, mineralization of dissolved substances and formation of inorganic byproducts in the complex fluid matrix created by the strong oxidant reactions. Additionally, not allowing the O_3 treated sample to settle, kept these ionic species suspended and dissolved, whereas allowing O_3 treated samples to settle, removed them from the supernatant via various precipitate forms. Unlike EC, Fig. 2 presents a graph that shows the changes in pH values when settling of the O_3 treated samples is allowed.

As the settling time decreases, we observe a sharp decrease in pH from 7.45, near neutral, to 4.36, acidic. This indicates a significant formation of organic acids as ionic intermediates in the complex, with carboxylic acids being major oxidation products. Other types of organic acids may include aldehydes, acetic acid, oxalic acid, and glycolic acid. They all directly contribute to the high acidity, thereby an increase in hydrogen ion (H^+) concentration. By not allowing the O_3 treated sample to settle (i.e., Treatment 4), these organic species remain in the supernatant, which might otherwise be removed as precipitates, in the solution.

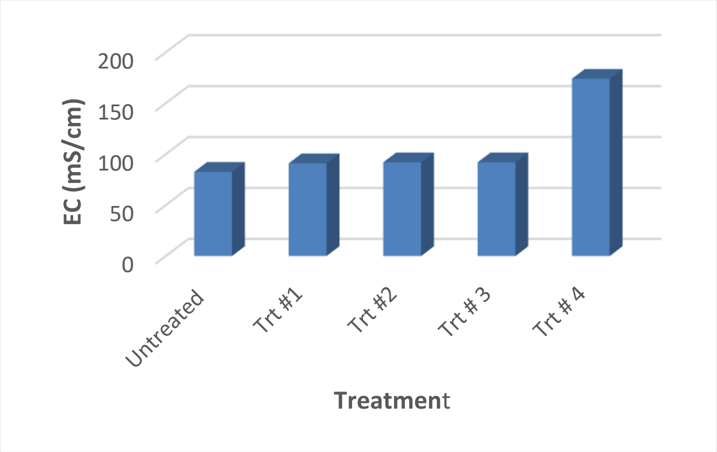


Fig. 1 Effect of O_3 treatment on EC levels.

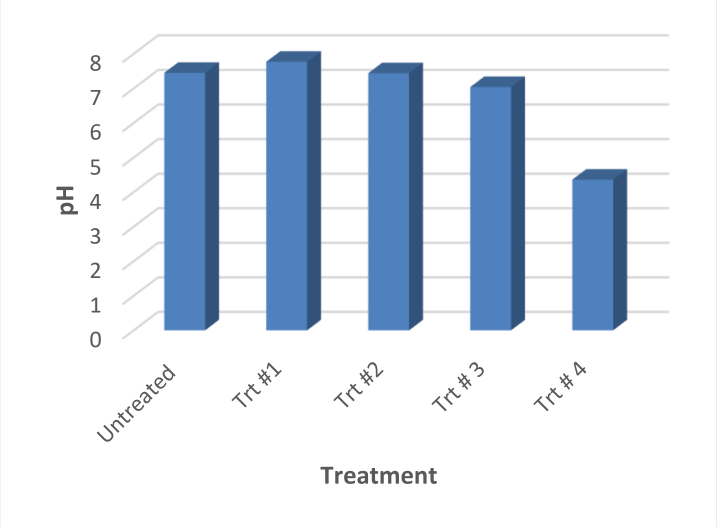


Fig. 2 Effect of O_3 treatment on pH levels.

Turbidity of the frac water decreased in all the treated samples, with final levels ranging from 35 to 60 NTU (Fig. 3), corresponding to 84% to 90%, respectively. A combination of mechanisms is acting to induce change in the fluid turbidity, including adequate reaction time for fine particles to aggregate into larger micro-flocs, the reduction of color causing molecules, and oxidative degradation of large molecules organic suspended solids into smaller compounds. The low turbidity in Treatment 4 is likely due to low light scattering from micro particles and macro coagulants.

Fig. 4 shows an equal and marked reduction in TSS for all O_3 treated samples, with final values ranging from 32 to 41 mg/L, which represents a reduction of 93% and 91%. The reduction of TSS is also directly related to the reduction in turbidity. Higher reductions in turbidity result in lower TSS. Although they are not directly interchangeable as the relationship may vary with coagulants size and treated frac water color. Fig. 5 shows the linear regression using turbidity as the independent variable and TSS as the dependent variable. The linear model is TSS = 0.3814×Turbidity +18.799; R^2 = 0.9911 and the log-linear model is Ln(TSS) = 0.0105×Turbidity+3.1019; R^2 = 0.9892 or in exponential form: TSS = $22.24e^{0.0105\times\text{Turbidity}}$. Both models can be used in some instances to calculate the predicted TSS for a specific turbidity value.

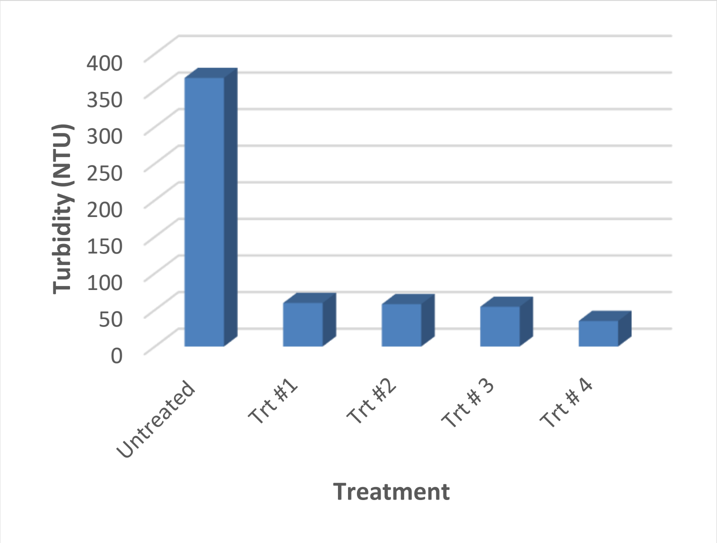


Fig. 3 Effect of O_3 treatment on turbidity.

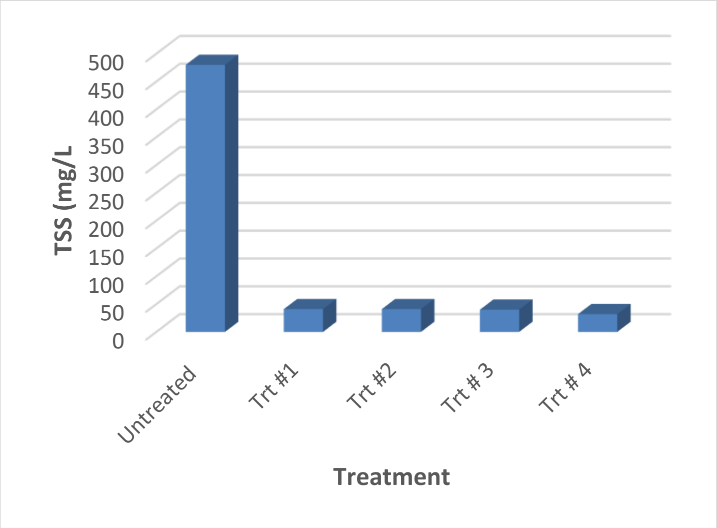
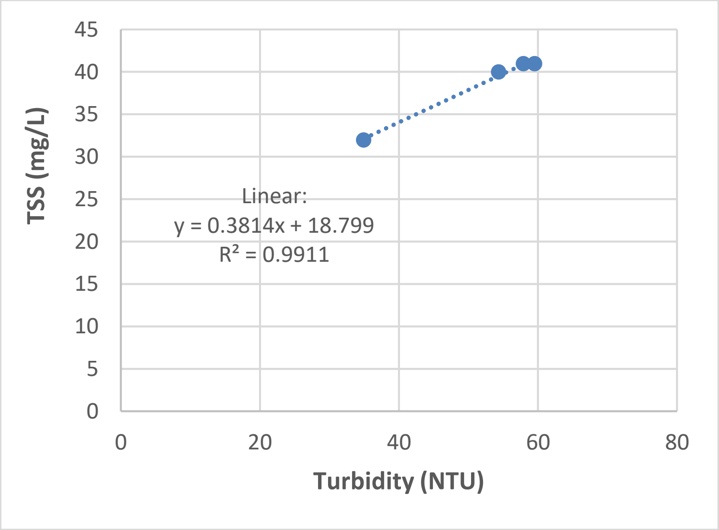
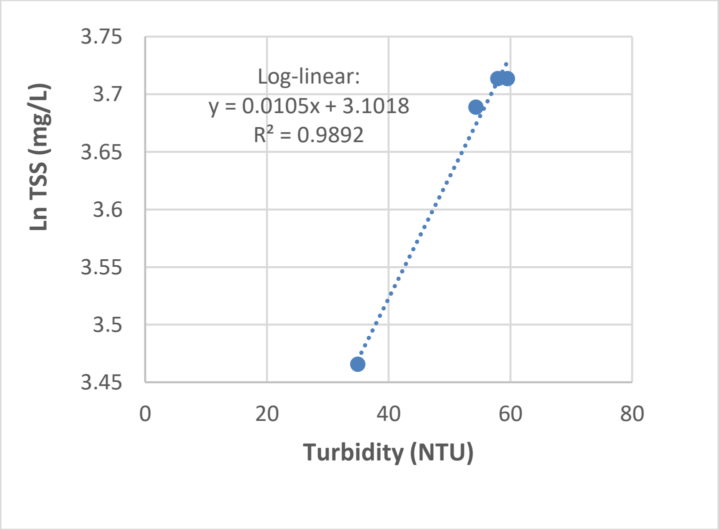


Fig. 4 Effect of O_3 treatment on TSS.



(a)



(b)

Fig. 5 Relationship between turbidity and TSS, a) and b).

As shown in Fig. 6, the TDS of the frac water was reduced from 155,000 to 130,000 mg/L. Since TDS is a measure of all particles lesser than 2 µm, the O_3 treatment resulted in the degradation of small particles of organic matter and compounds, and the formation of larger inorganic complexes. This shift in particle size effectively resulted in a decrease in TDS concentration across all the O_3 treated samples by moving matter out of the dissolved range. Reduction in TDS levels ranged from 13% to 19%.

Fig. 7 shows the linear regression using total dissolved solids as the independent variable and TSS as the dependent variable. The linear model is $TDS = 48.047\times \text{TSS}+132008$; $R^2 = 0.9547$ and the log-linear model is $Ln(TDS) = 0.0003\times \text{TSS}+11.794$; $R^2 = 0.9383$ or in exponential form: $TDS = 132{,}455e^{0.0003\times \text{TSS}}$, which can be used to predict TDS for a specific TSS value.

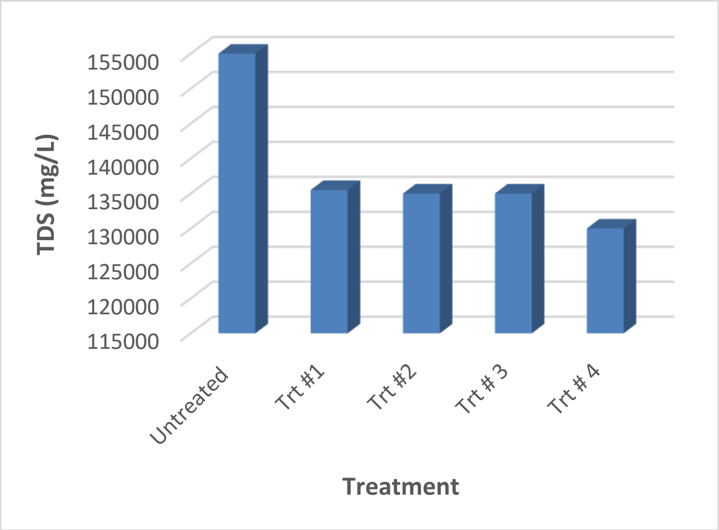
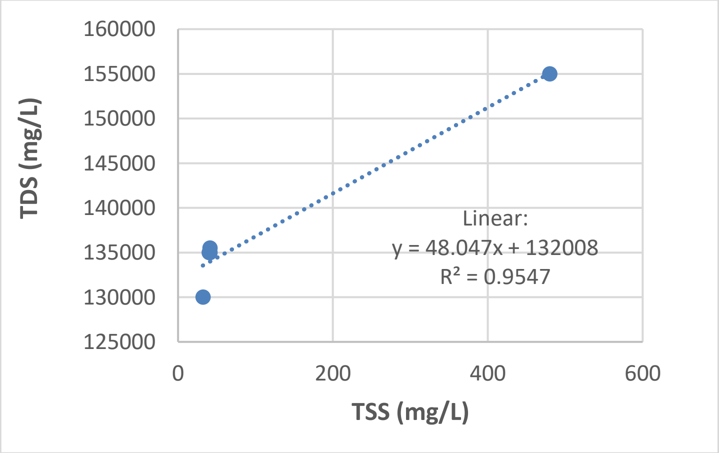
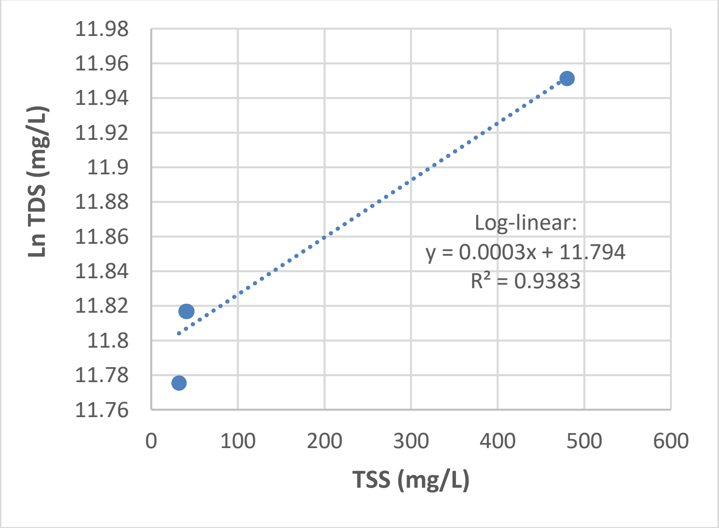


Fig. 6 Effect of O_3 treatment on TDS.



(a)



(b)

Fig. 7 Relationship between TSS and TDS, a) and b).

Ozone treatment usually increases oxidation-reduction potential (ORP) and commonly associated with higher dissolved oxygen (DO) meaning that the treated wastewater is in now a more oxidizing state (higher positive mV). Fig. 8 indicates a temporary decrease in ORP value during ozone treatment of the frac fluid accompanied with an increase in DO (Fig. 9). High, immediate demand from reducing agents in the complex fluid, rapid radical consumption (i.e., HO_2^*, O_{2*}^-), initial pH change, or interfering chemical reactions such as H_2O_2 reacting hydroxyl radicals, may cause this phenomenon [11]. Furthermore, the significant rise in ORP and DO result from a higher degree of ozone-derived radicals fully oxidizing reducing agents in the fluid matrix that previously lowered ORP while more residual O_3 decomposed into oxygen.

O_3 treatment equally reduced COD by 52% in all treatments, decreasing levels from 2500 to 1200 mg/L (Fig. 10). This efficiency results from direct oxidation of organic chemicals and compounds by attacking electron-rich sites, such as aromatic rings and aromatics rings combined and indirect oxidation via highly reactive, non-selective free radicals and non-radical species, which aggressively mineralize them into CO_2 and H_2O. There has also been an across-the-board reduction in priority heavy metals, with concentrations dropping by 62% for Cu, 56% in the case of Mn, and 21% for Cr^{+6}.

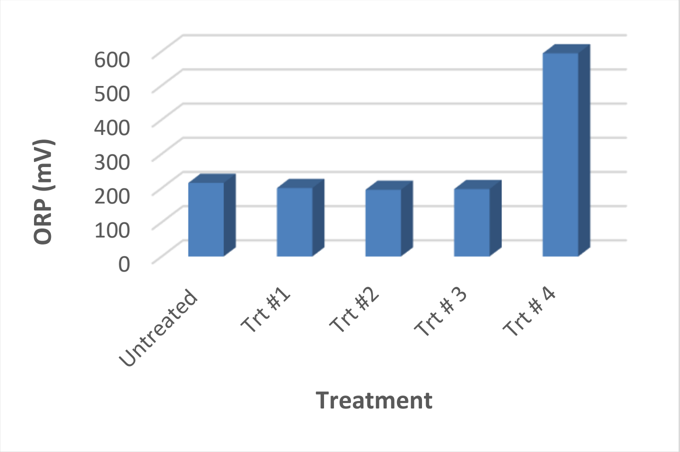


Fig. 8 Effect of O_3 treatment on ORP.

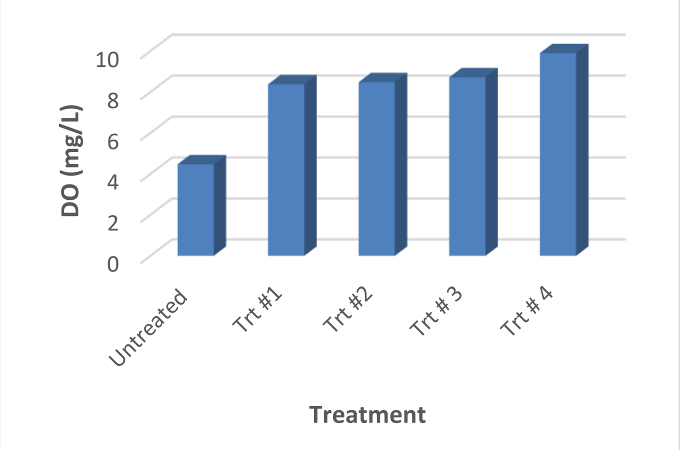


Fig. 9 Effect of O_3 treatment on DO.

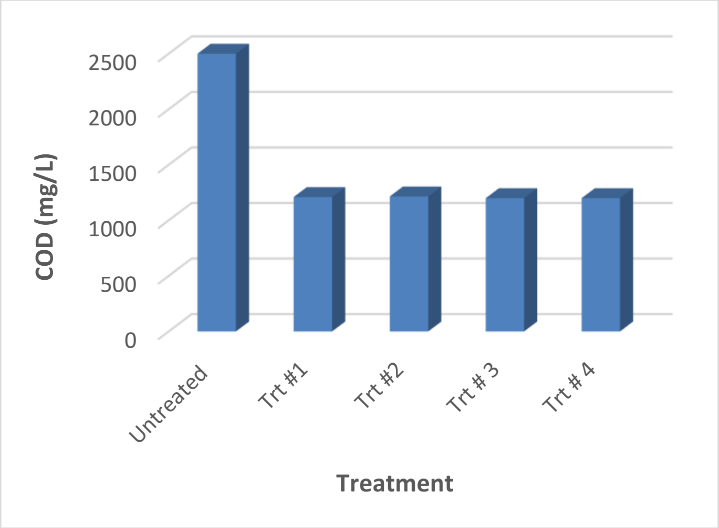
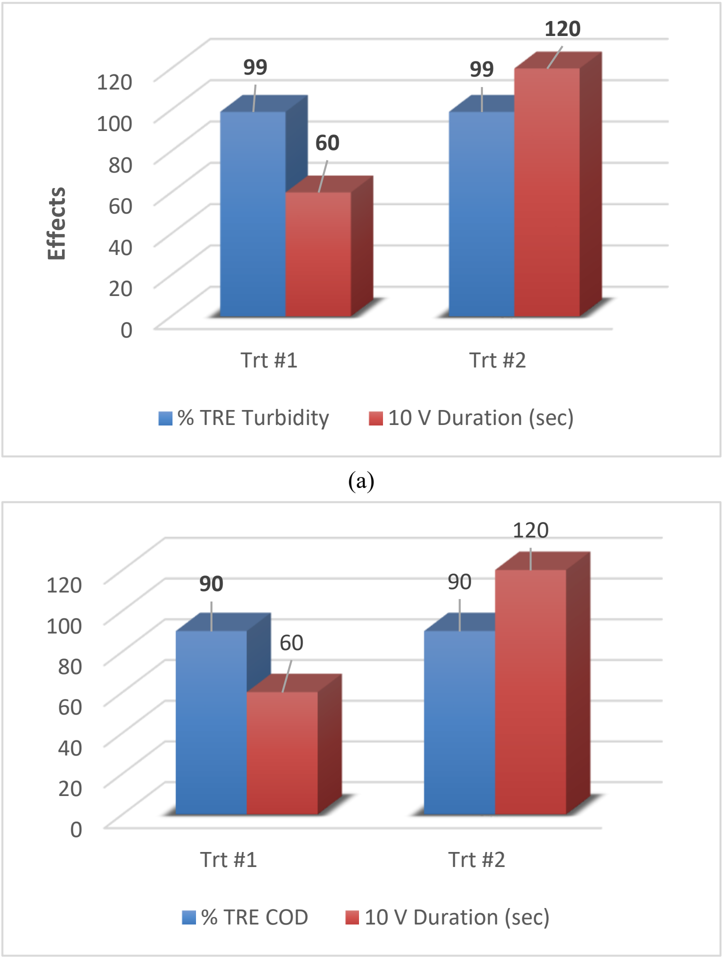


Fig. 10 Effect of O_3 treatment on COD.

3.2 Electrocoagulation

The results show that electrocoagulation was effective at significantly decreasing turbidity and COD with marginal drop in pH while an increase in EC. The reactions [1] occurring at the anode: $2Al \rightarrow 2Al^{+3} + 6e^-$ while at the cathode: $6H_2O + 6e^- \rightarrow 3H_2 + 6OH^-$ resulting in $2Al^{+3} + 6OH^- \rightarrow 2Al(OH)_3$. When expressed according to Faraday's law: $Al + 3H_2O \rightarrow Al(OH)_3 + 3/2H_2$. However, excessive current can lead to electrode passivation and higher energy costs. The parameters turbidity and COD decreased as current duration increased while EC increased. Change in the tested parameters is expressed in % TRE in relation to the treatments are illustrated in Fig. 11. Both treatments reduced turbidity by 99% and COD by 90%, respectively, with a drop in initial pH 7.45 by 1.53 in treatment 1 and 1.23 in treatment 2. The drop in pH indicates a greater presence of H^+ and formation of organic acid substances in the fluid matrix relative to an increasing removal of OH^-. Specifically, electrocoagulation has been proven to reduce COD and Turbidity, which improves wastewater quality for reuse [12, 13]. Through multiple simultaneous pathways, in-situ metallic coagulants are produced from the Al sacrificial anode through electrolytic dissolution. At the same time, the cathode produces hydrogen gas and hydroxyl ions. This process triggers a series of key chemical and physical mechanisms of constituent's removal in the frack. The generated electro-generated coagulants neutralize the negative charge of finely dispersed and colloidal particles via destabilization, allowing them to aggregate into larger flocs. These flocs entrap suspended solids and adsorb organic substances that are removed by settling out or flotation by gas bubbles contributing to reduce turbidity of the frac. Organic molecules and refractory compounds that contribute to COD are adsorbed onto the surface of the Al hydroxide flocs through electrostatic attraction or complexation, and through this mechanism, COD in the fluid is reduced. Optimal treatment efficiency was observed at 10 V and 60 sec.



(a)

(b)

Fig. 11 Effects of treatments on turbidity and COD (a) and (b).

3.3 Charcoal-Ozone Treatment

After the frack sample was O_3 treated for 45 minutes, it was contacted with charcoal and the later was then reused in treatments 2 and 3, respectively. EC was reduced in all the three treatments at a similar efficiency of 50%, TDS dropped marginally by 8% for the first two washes and remained unchanged for the third wash while pH increased by an average value of 1.52 despite the original drop in pH created by the O_3 treatment. After a 58% drop following the second wash, Cu levels stabilized during the third wash. Conversely, Mn levels showed a 19% increase during this period, indicates residual presence in the charcoal. These findings suggest distinct behavioral patterns for Cu and Mn during sequential washing. Additionally, applying a 2:1 liquid-to-solid ratio to the treatment 4 supernatant (O3-treated flowback) dropped EC, Cu, and Cr^{+6} levels by 16%, 27%, and 21%, respectively. In contrast, Mn concentration rose by 28%, likely due to its desorption from charcoal binding sites and a distinct behavioral pattern.

3.4 Evaluation of Treatments Efficacy

Both ozone and electrocoagulation treatments proved highly effective for the flowback sample sanitation, eliminating heterotrophic bacteria counts to non-detectable levels (0 CFU) for electrocoagulation and 90% for ozone. By eliminating heterotrophic bacteria in a flowback water for reuse in oil and gas operations could prevent microbially-influenced corrosion (MIC), mitigate biofilm buildup and infrastructure clogging, and reduce the risk of souring. Conversely, charcoal showed no significant effectiveness.

4. Conclusions

Ozone, electrocoagulation, and charcoal-ozone treatments were investigated as possible techniques for reducing turbidity, COD, TSS, TDS, heavy metals, and EC in a flowback sample. Ozone provided an overall reduction of 90% in turbidity, 52% in COD, 93% in TSS, 19% in TDS, and a reduction in priority heavy metals, with concentrations dropping by 62 % for Cu, 56% in the case of Mn, and 21% for Cr^{+6}. Electrocoagulation achieved an overall reduction of 99% in turbidity which parallels TSS reduction, 90% in COD, and a100% destruction in heterotrophic bacteria. Charcoal was generally ineffective as a treatment technique when combined with ozone.

Acknowledgement

The authors would like to thank Ovintiv Inc for supporting this project.

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