



Suitability of water treatment chemicals in the remediation of produced water: a data-driven approach

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Abstract

There exist numerous counts of research works on produced water. We got to know about them because they made it to publishing probably by indicating a positive or promising result. Contrarily, there exist a hundred times unpublished, unreported works on produced water; works rejected based on not yielding desirable results or not being innovative enough. We might have encountered undesirable results but to what depths and time have we committed to mining out intricate details. The world is thinking and demanding sustainability. Is it sustainable for the future of water treatment, the ease and pace at which we transition to the next chemical or treatment option? In this data-centred approach, three common chemicals, aluminium sulphate, ferrous ammonium sulphate and calcium chloride, were used to treat produced water. The collected data (both initial and final analysis) were inferentially analysed. The first statistical analysis was the testing of 2 hypotheses using the Analysis of Variance test. This was done to reveal to compare the dependence of produced water properties on two categorical variables (sample type and treatment chemicals). The second was the test for relevance: correlation and regression analyses. The laboratory experimental analysis revealed that aluminium sulphate was most suitable for the alteration of physical effluent characteristics; ferrous ammonium sulphate for salinity concerns and calcium chloride for a particular heavy metal's stability. The overall effluent characteristics indicated a greater dependency on 'sample type' than 'treatment chemicals'. Certain produced water properties relationships were highlighted and quantified for instance iron(II) and chloride ion concentrations were dependent on total solids and indicated a significance F of 0.01.

Keywords Produced water · Data-driven approach · Statistical hypothesis testing · Cost-effective treatment

Introduction

With the beginning of a new decade, one would expect new challenges, but some continuously exist. Presently, one of such for the oil and gas industry is produced water. An inevitable and toxic form of our most essential resource—water. Inevitable because one cannot exploit our underground resource (oil and gas) without it coming along eventually. Toxic? This water contains variable levels of pollutants such as dissolved solids, heavy metals, unstable anions, oils and grease, organic compounds. Currently, the world is waking up to the effects of climate change and how pollution is an undeniable hinder to sustainability. Produced water is wastewater generated alongside oil and gas (Clark and Veil 2009).

Produced water is often referred to as brine or formation water. It represents the largest waste generated from the production process. Clark and Veil (2009) predicted produced water volumes to increase by 32% by 2025.

Why should we treat produced water 'better'?

On the 3rd of March, 2021, at the United Nations Human Rights Council, UN HRC in Geneva, David Boyd, the United Nations' Special Rapporteur on human rights and environment stated "The world faces a water crisis and it is getting worse...3/4 of all the natural disasters in the last 20 years were water-related...water pollution, water scarcity, water-related disasters and damage to healthy freshwater ecosystems have major impacts on a wide range of human rights...". He recommended five steps for addressing the global water crisis—one of which includes a state-of-the-art water assessment (UN OHCHR 2021). The toxicity and complexity of the constituents of produced water make it a factor

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in the mitigation of the global water crisis. Surface interactions between hydrocarbons and their enclosed geologic formations encourage chemical reactions which yield organic and inorganic products with high toxicity levels (Benko and Drewes 2008). Products such as radionuclides (a radioactive atom/element which has an unstable nucleus due to its excess nuclear energy) and oil droplets are very difficult to treat and they pose direct harm to both the environment and human life (Liangxiong et al. 2004). Also, environmental protection agencies have been established worldwide on different government tiers with strict rules and regulations to control and monitor the discharge of produced water due to its different complex chemical nature and large production volumes. Produced water contains heavy metals that biomagnify and other organic contaminants such as asphaltenes, naphthenic acids and resins (Pimentel et al. 2008; Li et al. 2006). Produced water could ruin earth's terrain, pollute water bodies thereby endangering our aquatic ecosystem, our land, crops, the often-forgotten microbial ecosystem and raise stable elements above trophic levels when not treated and disposed of properly. The conventional treatment method which involves subsequent discharge is the gravity-based separation (Fakhru'l-Razi et al. 2009). Furthermore, disposal as surface waters requires optimum treatment of all suspended and dissolved components. The dissolved components contribute to the chemical oxygen demand which reduced the dissolved oxygen levels creating an anaerobic aquatic ecosystem (Liangxiong et al. 2004). For instance, alkylphenols which do not require bioaccumulation due to their already high concentration (present in produced water and is also incorporated in detergents) causes feminization of fish in polluted rivers and upsets the reproductive make-up of rodents (Markey et al. 2001).

Cost of treatment

Colorado School of Mines/ Advanced Water Technology Center (n.d.) summarized the overall treatment cost into 5: construction cost treatment and disposal structures; operating cost of these structures; management cost of by-products generated during treatment; transportation cost; permits, reports and monitoring costs. The website also reports that total cost ranges from less than 1 cent/bbl to more than \$5/bbl. For agricultural standards, (Burnett and Siddiqui 2006) report treatment costs to range from \$0.5 to \$1.5/bbl. For instance, thermal treatment technologies such as hybrid multi-effect distillation-vapour compression (MED-VCD) have a capital cost ranging from \$250–\$360 per bpd, operating cost and total unit costs of ~\$0.12/bbl and \$0.19/bbl, respectively (Igunnu and Chen 2014). Igwe et al. (2013) examined the factors and methods for handling wastes stating; “from practical experience, the feasibility of choosing a particular disposal system is usually

dependent on cost contributing factors (such as transportation, treatment and development of disposal site) as well as environmental regulations. Some of the techniques being currently used are disposal to surface water; disposal to sewer; re-injection into the reservoir (through injection well); discharge to evaporation pond; spray evaporation and application of zero liquid discharge”.

The chemical treatment of produced water

Various classes of chemicals are widely used in water treatment. Some of these include oxidants such as ozone; alkalinity control agents for example lime; coagulants such as aluminium sulphate and organic polymers; corrosion inhibitors for instance silicates and morpholine. They can either be used as standalone treatments or incorporated with other treatment technologies (usually in the pre-treatment phase or as cleaning agents) such as ceramic MF/UF membrane, reverse osmosis, vapour compression distillation, macro-porous polymer extraction technology, gas floatation, media filtration.

These chemicals have been experimented with and utilized in produced water treatment. Hosny and Ramzi (2017) compared the treatment efficacy between two natural polymers (chitin and chitosan) on produced water for the reduction of formation damage. Despite using a simultaneous mixture of local materials in their treatment design, Udeagbara et al. (2020) had to initially wash the ground materials with 0.4 mol/L HNO₃. Zakwan et al. (2018) designed a treatment solution using an advanced oxidation process from H₂O₂ and UV radiation to degrade toxic components in chemically enhanced oil recovery (CEOR) produced water. Carus Group Inc. (2019) stated permanganate oxidizes soluble iron, manganese, hydrogen sulphide and mercaptans in produced water. Inorganic coagulants such as aluminium sulphate and ferric chloride consist of some of the most widely used coagulants for the removal of suspended and colloidal particles. Rodriguez et al. (2020) stated a downside to their usage includes large masses of residual sludge and their discouraging compound to element ratio—1 ton of ferric chloride FeCl₃·6(H₂O) yields 210 kg of Fe(III).

When treating produced water, one of the recurring processes is chemical dosing. Manual chemical dosing isn't usually recommended for large-scale applications because it is error-prone. Mechanical chemical dosing is achieved using dosing pumps and meters such as peristaltic pumps and diaphragm pumps. Intelligent chemical dosing incorporates AI, machine learning and the Internet of Things (IoT) to achieve continuous automated dosing optimization. Examples of this dosing system include Emagin and OpWorks technologies.

A data-driven approach

When an experimental treatment analysis is carried out on produced water, comparisons are made between final and initial results and oftentimes when undesired change is achieved, the results, chemicals used and treatment design are often discarded and the next treatment option is sought-after. The end result isn't all there is to a mixture as complex as produced water.

Inferential statistics covers an array of decision-making tools that utilize inductive reasoning to yield a probability for validity instead of the traditional right or wrong. Statistical hypothesis testing (or confirmatory data analysis) is one of those tools. It is used to verify an experimental aim against a conventional belief—this is often referred to as statistical significance. It is utilized in the medical field to test drugs and procedures (Dubois n.d.). It is also widely utilized in philosophical science. Repeated testing is an alternative to statistical hypothesis testing but it isn't lean. Repeated testing requires several repeated experimental runs and an increase in sample size. (Nickerson 2000). Inferential statistics might be criticized as time-consuming but presently there are several software applications with user-friendly experiences and interfaces (UX and UI) capable of automating calculations within seconds such as R, Python, Microsoft Excel, Minitab and IBM SPSS.

Another sustainable application of produced water experimental data is for correlation and regression analysis. These two have continuously formed the basis for foundational theories in science and engineering for instance the relationship between density and volume, coagulant dosage and settling time. Hypothesis testing might validate this, but it cannot quantify and mathematically express these theories. Results derived from correlation and regression analysis are indispensable; and since produced water still presents a threat to global water pollution control, it is paramount that every data on its composition, properties, treatment be collected. We can't say when we might need it but we can still keep it.

Aim and selection criteria

The option of using easily sourced, relatively inexpensive methods has been overlooked due to the nagging existence of this toxic water. In this work, chemicals—'essential toxins', were used to treat, alter, limit alarming ones (contained in produced water). A widely used coagulant (aluminium sulphate), a double salt of two treatment salts (ferrous ammonium sulphate) and a generally used laboratory chemical (calcium chloride) were each utilized. According to C. N. Harmony (personal communication, July 1, 2019), the selection criteria included simplicity in treatment design and affordability of these chemicals to encourage treatment and manage waste. Before the actual designed treatment process, produced water often goes through a dosing phase which involves the addition of flocculants and scale inhibitors (Nwosi-Anele and Illedare 2016).

The aim is to extensively investigate the tri-fold suitability which includes dosing suitability, statistical suitability and output models suitability. These will combine to create an archetypical template that could be widely utilized for wastewater treatment considerations and options selection.

Methodology

Two (2) Produced water samples collected from the different reservoirs in the Niger Delta region of Nigeria were analysed. The parameters analysed include pH, capillary viscosity, temperature, apparent colour, total dissolved solids, total suspended solids, total solids, turbidity, oils and grease, sulphate ion, chloride ion, nitrate ion, calcium carbonate, calcium ion, sodium ion, barium ion, iron(II) ion, magnesium ion concentrations. See Table 1 for method of analysis.

To determine the suitable concentration of treatment chemicals needed, 200 mg/l, 500 mg/l, and 1000 mg/l of the 3 treatment chemicals concentration were dissolved in the produced water samples. The 18 treated samples were

Table 1 Method of analysis

Effluent characteristics	Method of analysis
pH and Temperature (°C)	Electronic Method
Apparent Colour	Human Eye
Total dissolved solids (mg/l)	Gravimetric Analysis
Total suspended solids (mg/l)	
Total solids(mg/l)	
Turbidity (NTU)	Nephelometer
Oils and Grease (mg/l)	Extraction, Acidification and Gravimetric Analysis
SO ₄ ²⁻ (mg/l)	Ba ²⁺ (mg/l) Acidification, BaCl Addition and Gravimetric Analysis
Cl ⁻ (mg/l)	Na ⁺ (mg/l) AgNO ₃ Titration
CaCO ₃ (mg/l)	Ca ²⁺ (mg/l) EDTA Titration
NO ₃ ⁻ (mg/l)	Mg ²⁺ (mg/l) Atomic Absorption Spectroscopy, AAS
Fe ²⁺ (mg/l)	

stirred. The flocs and other suspended matter were decanted. The capillary viscosity and pH of each trial sample were measured and compared. The dosage of 200 mg/l was chosen since it did not alter the initial capillary viscosities of the produced water samples and posed the least significant change in pH.

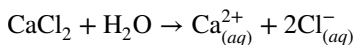
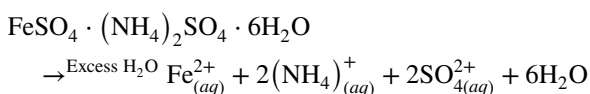
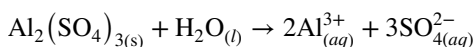
200 mg/l concentrations of each treatment chemicals in the produced water samples were created. The samples were rapidly agitated for 60 s for the production of a micro-floc. The 6 samples (2 by 3) were agitated slowly for 25 min to form a floc capable of settling. The samples were then left to settle for 3 h. A sieve (with mesh size capable of not altering total solids values) was used to clear off flocs.

Final physiochemical laboratory analyses of the 6 samples (2 by 3) were carried out.

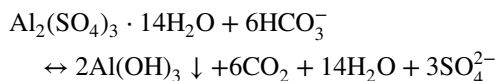
Statistical analyses were carried out on the experimental results. The first was using a suitable hypothesis testing tool (Analysis of Variance). The second, correlation and regression analyses were done in other to reveal useful insights.

The treatment chemicals utilized are inorganic coagulants which are known for forming metallic precipitates capable of absorbing impurities in water (Jones 2020). Dissolving aluminium sulphate in water causes a fraction of the aluminium to dissociate into the highly charged Al^{3+} , $\text{Al}(\text{OH})^{2+}$, $\text{Al}(\text{OH})_2^+$ which neutralize the negatively charged impurities suppressing their zeta potential. However, calcium chloride and ferrous ammonium sulphate produce divalent cations (Ca^{2+} and Fe^{2+}) resulting in a lower neutralization potential on impurities (Bennett 2006).

The dissociation of the treatments chemicals is expressed below:



For bicarbonate, carbonate and hydroxide impurities, a significant concentration of these treatment chemicals is needed to precipitate $\text{Al}(\text{OH})_3$, $\text{Fe}(\text{OH})_2$ and $\text{Ca}(\text{OH})_2$ (Bhanderi and Ranade 2014). An example of the reaction is shown below:



According to Bhanderi and Ranade (2014), the precipitating ability of the treatment chemicals to produce $\text{Al}(\text{OH})_3$, $\text{Fe}(\text{OH})_2$, and $\text{Ca}(\text{OH})_2$ is a function of the produced water's pH. Conventionally, most inorganic coagulants require quick mixing mechanisms for effective distribution of the intermediate products stated above because these products are the destabilizing agents and they are short-lived.

Observation

Table 2 contains the physical observations recorded when the treatments chemicals were added to the produced water samples.

Results

Table 3 contains the results of the analyses of the two produced water samples both before and after treatment with respect to the three treatment chemicals. The last two columns contain Nigeria's Department of Petroleum Resources (DPR) limits for both inland and nearshore disposal.

Table 4 contains two split heat maps illustrating the percentage changes before and after treatment for the 2 produced water samples. Each map was delimited to add up the cumulative change in physical and chemical properties. For samples "A" and "B", aluminium sulphate yielded the most positive change in physical properties, while the samples treated with calcium chloride yielded the least negative change in chemical properties.

Since three treatment chemicals were used, we can graphically depict their normalized concentrations (to sum 100)

Table 2 Observations upon addition of chemicals to samples before and after settling hours

Treatment chemicals	Immediate observations	Observations after settling time
Aluminium Sulphate	Lighter Contrast. The appearance of suspended matter. No apparent heat loss or gain	Settled bottom suspended matter. Collection of lesser matter on the top layer. The increased mass of suspended matter
Ferrous Ammonium Sulphate	Deeper Contrast. The appearance of suspended matter. No apparent heat loss or gain	Settled bottom suspended matter. The increased mass of suspended matter
Calcium Chloride	No noticeable colour changes. Collection of oil films on beaker's walls. No apparent heat loss or gain	The increased mass of oil films

Table 3 Initial and final analysis with DPR limits

Effluent characteristics	SAM PLE 'A'	Sampl e "A" (aluminium sulphate)	Sampl e "A" (ferrous ammonium sulphate)	Sam ple "A" (calcium chloride)	Sam ple "B" (initial)	Sampl e "B" (aluminium sulphate)	Sampl e "B" (ferrous ammonium sulphate)	Sam ple "B" (calcium chloride)	Depart ment of petroleum resources (DPR) limit for inland disposal	Depart ment of petroleum resources (DPR) limit for nearshore disposal
pH	8.31	7.99	8.26	7.98	7.68	7.35	7.19	7.43	8.5	8.5
Temperature (°C)	26.1	25.9	26	25.9	25.9	25.9	25.9	26	25	30
Apparent Colour	Custard yellow	Light yellow	Orange	Custard yellow	Custard yellow	Light yellow	Orange	Custard yellow	–	–
Total dissolved solids (mg/l)	45	20	20	25	55	35	35	45	2000	5000
Total suspended solids (mg/l)	10	25	35	30	15	30	35	25	30	50
Total solids (mg/l)	55	45	55	55	70	65	70	70	–	–
Turbidity (NTU)	2.086	0.763	1.539	1.192	1.982	1.438	2.286	1.621	10	10
Oils and Grease (mg/l)	13	7.8	12.5	13.4	20.6	19.4	19.4	23	10	20
SO ₄ ²⁻ (mg/l)	0.5143	3.0863	3.0863	0.5143	0.009	1.5431	1.5431	0.009	200	200
Cl ⁻ (mg/l)	60	68	64	68	90	104	100	104	–	–
NO ₃ ⁻ (mg/l)	7.63	2.102	5.973	4.286	3.463	1.82	2.049	1.038	–	–
CaCO ₃ (mg/l)	40	85	50	120	15	20	22	56	–	–
Ca ²⁺ (mg/l)	16	34	20	48	6	8	8.8	22.4	–	–
Na ⁺ (mg/l)	38.9	44.1	41.5	44.1	58.4	67.5	64.9	67.5	–	–
Ba ²⁺ (mg/l)	2.0572	4.4148	4.4148	0.7357	0.009	2.2074	2.2074	0.009	–	–
Fe ²⁺ (mg/l)	3.8125	1.8526	3.1271	2.56398	4.92375	3.61105	5.1812	4.18029	1	–
Mg ²⁺ (mg/l)	1.00386	0.8139	0.53928	0.41823	1.11316	0.89281	0.52392	0.32965	–	–

in equilateral triangles as shown in Figs. 1, 2, 3 and 4 above. The ternary diagrams were designed separately for the physical and chemical properties to avoid clustering of points due to the number of parameters analysed. Figures 1 and 3 illustrate the quantifiable physical properties while Figs. 2 and 4 are for the chemical properties. The figures might also reveal a hypothesized tri-fold compositional effect should the one-chemical-per-sample design be replaced by three-chemicals-cocktail-per-sample given that ternary plots are widely incorporated in phase diagrams. Addinsoft (2021) was used in designing the ternary graphs.

Table 5 contains the frequency distribution, measures of variability and the central tendency for all quantitative variables in Table 3. Skewness and Kurtosis reveal the disparity between an observed distribution and the ideal normal distribution. SO₄²⁻ concentrations for both samples yielded skewness values of 0 which is typical of a normal distribution. Also, SO₄²⁻ concentrations for both samples were bimodal. Sample B's Ba²⁺ values are also bimodal. For both samples, CaCO₃ had the maximum ranges. CaCO₃ also had the largest difference between mean and median values. A zero variance indicates a high similarity within recorded observations. This is so for Sample B's temperature values and to an extent that of sample A.

Discussion

Table 6 explains each property's experimental result.

Comparison of the dependence of produced water properties on sample type and treatment chemicals

Table 3 can be classically summarized as one containing 2 categorical independent groups namely sample types with 2 levels (A and B) and treatment options with 4 levels (initial, aluminium sulphate, ferrous ammonium sulphate and calcium chloride) and one dependent continuous variable (each numerical effluent characteristics). Due to the relatively large number of dependent variables compared to observations, the Multivariate Analysis of Variance (MANOVA) would not be suitable. The Two-Way Analysis of Variance (ANOVA) without replication is, therefore, the suitable hypothesis-testing method for the following hypotheses:

H01 For a particular water property, the final analysis results were not significantly influenced by the sample types.

Ha1 For a particular water property, the final analysis results were significantly influenced by the sample types.

Table 4 Percentage change between initial and final analysis

Effluent characteristics	Sample "A" (aluminium sulphate)	Sample "A" (Ferrous Ammonium Sulphate)	Sample "A" (calcium chloride)	Sample "B" (aluminium sulphate)	Sample "B" (ferrous ammonium sulphate)	Sample "B" (calcium chloride)
pH	3.85%	0.60%	3.97%	4.30%	6.38%	3.26%
Total dissolved solids (mg/l)	55.56%	55.56%	44.44%	36.36%	36.36%	18.18%
Total suspended solids (mg/l)	-150.00%	-250.00%	-200.00%	-100.00%	-133.33%	-66.67%
Total solids (mg/l)	18.18%	0.00%	0.00%	7.14%	0.00%	0.00%
Turbidity (NTU)	63.42%	26.22%	42.86%	27.45%	-15.34%	18.21%
Oils and Grease (mg/l)	40.00%	3.85%	-3.08%	5.83%	5.83%	-11.65%
Cumulative change in physical properties	31.01%	-163.77%	-111.80%	-18.92%	-100.10%	-38.67%
SO ₄ ²⁻ (mg/l)	-500.10%	-500.10%	0.00%	-1701.11%	-1701.11%	0.00%
Cl ⁻ (mg/l)	-13.33%	-6.67%	-13.33%	-15.56%	-11.11%	-15.56%
NO ₃ ⁻ (mg/l)	72.45%	21.72%	43.83%	47.44%	40.83%	70.03%
CaCO ₃ (mg/l)	-112.50%	-25.00%	-200.00%	-33.33%	-46.67%	-273.33%
Ca ²⁺ (mg/l)	-112.50%	-25.00%	-200.00%	-33%	-47%	-273%
Na ⁺ (mg/l)	-13.37%	-6.68%	-13.37%	-15.58%	-11.13%	-15.58%
Ba ²⁺ (mg/l)	-114.60%	-114.60%	64.24%	-24426.67%	-24426.67%	0.00%
Fe ²⁺ (mg/l)	51.40%	17.98%	32.75%	26.66%	-5.23%	15.10%
Mg ²⁺ (mg/l)	18.92%	46.28%	58.34%	19.79%	52.93%	70.39%
Cumulative change in chemical properties	-723.62%	-592.08%	-227.55%	-26131.68%	-26154.82%	-422.29%

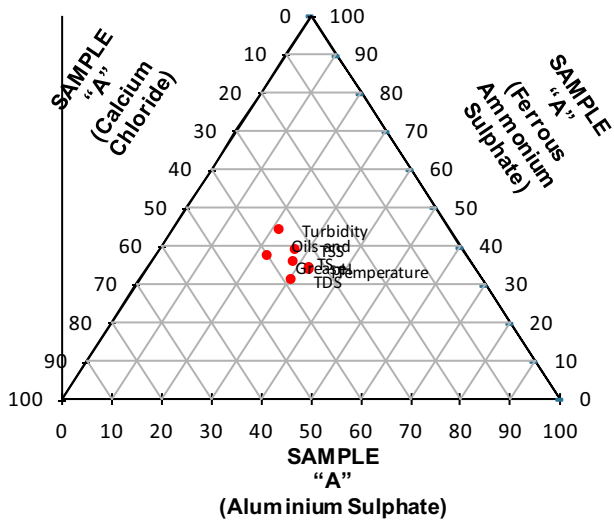


Fig. 1 Ternary diagram of sample A's continuous final physical properties

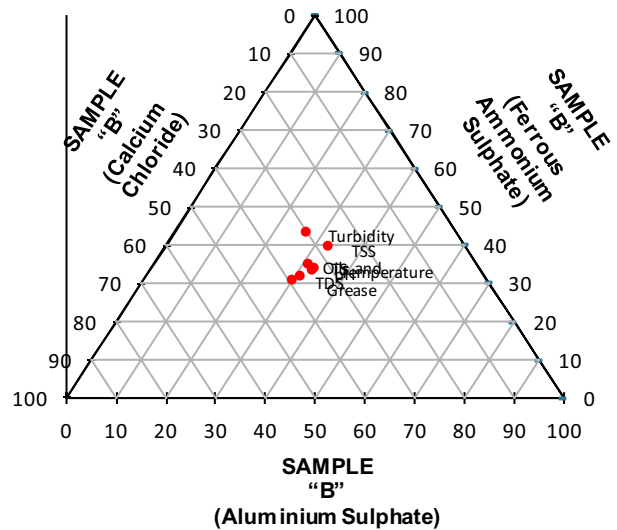


Fig. 3 Ternary diagram for sample B's continuous final physical properties

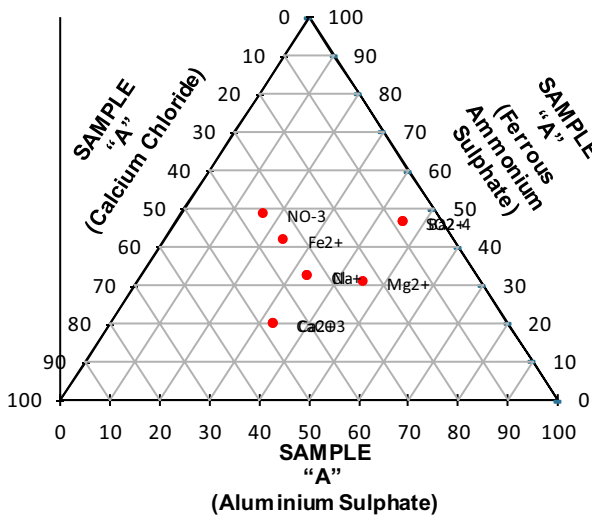


Fig. 2 Ternary diagram for sample A's continuous final chemical properties

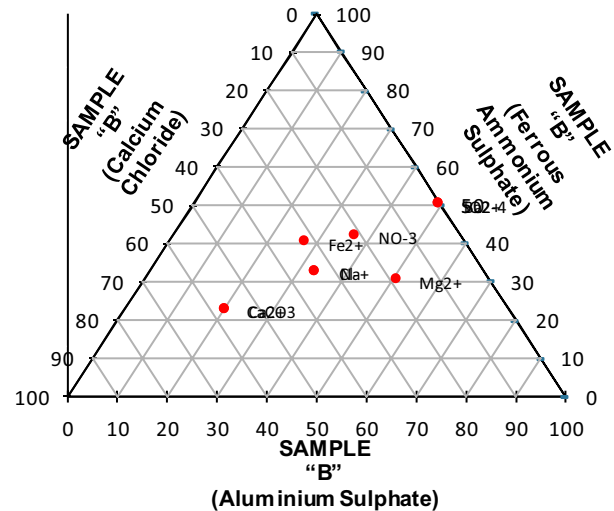


Fig. 4 Ternary diagram for sample B's continuous chemical properties

H02 For a particular water property, the final analysis results were not significantly influenced by the treatment chemicals.

Ha2 For a particular water property, the final analysis results were significantly influenced by the treatment chemicals.

The significance level $\alpha=0.05$. The block/row headers were the treatment options/chemicals while the column headers were the sample types for Table 7. Statistical Significance is achieved when P-value is less than alpha and the critical *F*-ratio is less than *F*-ratio. For total dissolved solids, total solids, pH, oils and grease, SO_4^{2-} , Cl^- , NO_3^- , $CaCO_3$, Ca^{2+} , Na^+ , Ba^{2+} and Fe^{2+} , the 1st null hypothesis

was rejected (these properties were significantly influenced by the sample types) while for temperature, total suspended solids, turbidity and Mg^{2+} , the 1st null hypothesis was accepted (these properties were not significantly influenced by the sample types). For total suspended solids, total dissolved solids, total solids, SO_4^{2-} , Cl^- , Na^+ , Ba^{2+} , Fe^{2+} , Mg^{2+} the 2nd null hypothesis was rejected (these properties were significantly influenced by the treatment chemicals). On the other hand, pH, temperature, turbidity, oils and grease, NO_3^- , $CaCO_3$ and Ca^{2+} accepted the 2nd null hypothesis (these properties were not significantly influenced by the treatment chemicals). Total solids was on the

Table 5 Descriptive statistics for all continuous variables

	pH	Temperature	TDS	TSS	TS	Turbidity	Oils and Grease	SO ²⁻ ₄	Cl ⁻	NO ⁻ ₃	CaCO ₃	Ca ²⁺	Na ⁺	Ba ²⁺	Fe ²⁺	Mg ²⁺	
SAMPLE "A"																	
Mean	8.14	25.98	27.50	25.00	52.50	1.40	11.68	1.80	65.00	5.00	73.75	29.50	42.15	2.91	2.84	0.69	
Standard Error	0.09	0.05	5.95	5.40	2.50	0.28	1.30	0.74	1.91	1.18	18.19	7.27	1.24	0.91	0.42	0.13	
Median	8.125	25.95	22.5	27.5	55	1.3655	12.75	1.8003	66	5.1295	67.5	27	42.8	3.2360	2.8456	0.6766	
Mode	-	25.9	20	-	55	-	-	0.5143	3.0863	68	-	-	44.1	4.41	-	-	
Standard Deviation	0.17	0.10	11.90	10.80	5.00	0.56	2.61	1.48	3.83	2.36	36.37	14.55	2.49	1.82	0.83	0.26	
Sample Variance	0.03	0.01	141.67	116.67	25.00	0.31	6.81	2.21	14.67	5.59	1322.92	211.67	6.20	3.33	0.69	0.07	
Kurtosis	-5.58	-1.29	3.13	1.50	4.00	-0.24	3.60	-6.00	-1.29	-0.76	-1.68	-1.68	-1.29	-3.55	-0.61	-2.80	
Skewness	0.07	0.85	1.78	-1.19	-2.00	0.27	-1.88	0.00	-0.85	-0.28	0.67	0.67	-0.85	-0.43	-0.04	0.25	
Range	0.33	0.20	25	25	10.00	1.32	5.60	2.57	8	5.53	80	32	5.2	3.68	1.96	0.59	
Minimum	7.98	25.9	20	10	45	0.763	7.8	0.5143	60	2.102	40	16	38.9	0.7357	1.85269	0.41823	
Maximum	8.31	26.1	45	35	55	2.086	13.4	3.0863	68	7.63	120	48	44.1	4.4148	3.8125	1.00386	
Sum	32.54	103.90	110.00	100.00	210.00	5.58	46.70	7.20	260.00	19.99	295.00	118.00	168.60	11.62	11.36	2.78	
Count	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	
SAMPLE "B"																	
Mean	7.41	25.93	42.50	26.25	68.75	1.83	20.60	0.78	99.50	2.09	28.25	11.30	64.58	1.11	4.47	0.71	
Standard Error	0.10	0.03	4.79	4.27	1.25	0.19	0.85	0.44	3.30	0.51	9.37	3.75	2.15	0.64	0.36	0.18	
Median	7.39	25.9	40	27.5	70	1.8015	20	0.7761	102	1.9345	21	8.4	66.2	1.1082	4.55202	0.70837	
Mode	-	25.9	35	-	70	-	19.4	0.009	1.54313	104	-	-	67.5	0.009	2.2074	-	
Standard Deviation	0.20	0.05	9.57	8.54	2.50	0.38	1.70	0.89	6.61	1.01	18.73	7.49	4.30	1.27	0.71	0.35	
Sample Variance	0.04	0.00	91.67	72.92	6.25	0.14	2.88	0.78	43.67	1.02	350.92	56.15	18.45	1.62	0.51	0.13	
Kurtosis	1.00	4.00	-1.29	0.34	4.00	-2.20	1.50	-6.00	2.17	1.72	3.55	3.55	2.17	-6.00	-2.61	-2.94	
Skewness	0.62	2.00	0.85	-0.75	-2.00	0.33	1.41	0.00	-1.56	0.89	1.85	1.85	-1.56	0.00	-0.41	0.07	
Range	0.49	0.1	20	20	5	0.848	3.6	1.5341	14	2.425	41	16.4	9.1	2.2064	1.57015	0.78351	
Minimum	7.19	25.9	35	15	65	1.438	19.4	0.009	90	1.038	15	6	58.4	0.001	3.61105	0.32965	
Maximum	7.68	26	55	35	70	2.286	23	1.5431	104	3.463	56	22.4	67.5	2.2074	5.1812	1.11316	
Sum	29.65	103.70	170.00	105.00	275.00	7.33	82.40	3.10	398.00	8.37	113.00	45.20	258.30	4.42	17.90	2.86	
Count	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	

Table 6 Discussion of analysis data

pH	Since the chemicals used were salts, the slight shifts in pH reveal the nature of these salts. According to Brandt et al. (2017), when Aluminium sulphate is dosed in water, alkalinity is usually reduced due to the acidic nature of the coagulant. A solution of ferrous ammonium sulphate in water turns blue litmus red hence, the reduction in both samples A and B. CaCl is a salt of a strong acid and strong acid thus forming a neutral solution in water
TEMPERATURE	None of the chemicals initiated a thermal reaction. Hence, there is no significant change in temperature
TDS and TSS	The flocculation phase yields a decrease in TDS. The use of a sieve with a fairly large mesh size and the possibility of the flocculation process been ongoing even after 3 h could explain the irregularities in TSS. The formation of chemical precipitates during treatment also supports this argument since chemical precipitates are considered a form of suspended solids (Fondriest Environmental Inc. 2014)
TURBIDITY	Coagulants have long been demonstrated to encourage aggregation and settling of suspended particles by increasing salinity for instance the visibly clear oceanic salt waters. the discouraging result from the ferrous ammonium sulphate perfectly corresponds to its high TSS values
OILS and GREASE	It's worthy to note the reduction for this parameter in the aluminium sulphate-treated water. The calcium chloride treated water increase suggests an ongoing deoiling process since oil films were initially observed in Table 2
SULPHATE ION	Aluminium sulphate and ferrous ammonium sulphate both partly dissociate into sulphate ions when dissolved in the water. This combined with those recorded in the initial analysis led to an increase in its ionic concentration
CHLORIDE ION	Here all treatment chemicals increased the chloride concentration and this raises serious concerns
NITRATE ION	All 3 treatment chemicals yielded reductions in all samples
CALCIUM CARBONATE AND CALCIUM ION	The calcium chloride treated water produced the highest increase, hence the least suitable here
SODIUM ION	Since high salinity prevents nitrogen intake in soils, ferrous ammonium sulphate had the safest result here
IRON(II) ION	Aluminium sulphate is the best option. Calcium is more reactive than iron. Hence, it is capable of displacing it in any reaction
BARIUM ION	High barium levels trigger a high concentration of chloride, manganese, iron, strontium etc. Heavy metals bio-magnify but despite being a heavy metal, barium does not tend to bio-magnify. However, it forms insoluble complexes with complex organic compounds (Oram n.d.). Therefore, its reduction and stability are crucial. Calcium chloride is the best option here
MAGNESIUM ION	In the reactivity series, calcium is above magnesium thereby it has the greater tendency to lose electrons and form cations- this explains its highest reduction in magnesium ions but highest increase in calcium ions concentration (oxidation)

edge of significance. The 1st and 2nd hypotheses were both rejected for total dissolved solids, SO_4^{2-} , Cl^- , Na^+ , Ba^{2+} , and Fe^{2+} .

Investigation of the inter-dependence between produced water properties

How can the experimental result be useful to the design and understanding of past and future chemical treatment data? By investigating the degree of interdependence and predictability of certain water properties, we reveal applicable knowledge which could serve as determining factors for treatment chemical selection and further insights into the complexity of coagulation chemistry. This is where the output model suitability comes in. The previous ANOVA test only covered the singular significance of each parameter. Produced water is a complex mixture and a mixture that complex requires an investigation into the relationship and interdependence between its measured parameters. A combination of correlation and regression analyses is utilized here to reveal the inter-dependence between several effluents' characteristics.

Correlation tests quantify the relationship strength between two continuous variables. It offers two main insights: the correlation coefficients and the p values. Correlation coefficients range from -1 to 1 . Inverse proportionality between two variables indicates a negative correlation coefficient while direct proportionality is positive. Table 8 presents a halved correlation matrix for all analysis data. Light Red highlights indicate coefficients less than 0 (negative). Light yellow highlights cover coefficients greater than 0 but less than 1. The 'ideal' correlation coefficients of 1 observed between Na^+ and Cl^- ; CaCO_3 and Ca^{2+} were because each pair had the same method of analysis (see Table 1) backed by long proven and widely used tests.

Assuming a significance level of $\alpha = 0.05$, Table 9 displays the P values for Pearson's correlation test. The light red highlights shade P values greater than 0.05; the statistically non-significant correlations. The light green highlights indicate P values less than 0.05 which are commonly deemed statistically significant. Correlations involving Fe^{2+} had the maximum number of statistically significant tests.

The coefficients of determination (R-squared) shown in Table 10 interpret the fitness of the regression models with

Table 7 Contingency table for two-way ANOVA test without replication

Effluent Characteristics	Source of Variation	Sum of Squares SS	Degree of Freedom df	Mean Square MS	F-ratio	P-value	Critical F-ratio
pH	Block/Row	0.134	3	0.045	1.611	0.352	9.277
	Column	1.044	1	1.044	37.764	0.009	10.128
	Error	0.083	3	0.028			
Temperature (°C)	Block/Row	0.010	3	0.003	0.400	0.764	9.277
	Column	0.005	1	0.005	0.600	0.495	10.128
	Error	0.025	3	0.008			
Total dissolved solids (mg/l)	Block/Row	675.000	3	225.000	27.000	0.011	9.277
	Column	450.000	1	450.000	54.000	0.005	10.128
	Error	25.000	3	8.333			
Total suspended solids (mg/l)	Block/Row	534.375	3	178.125	15.545	0.025	9.277
	Column	3.125	1	3.125	0.273	0.638	10.128
	Error	34.375	3	11.458			
Total solids(mg/l)	Block/Row	84.375	3	28.125	9.000	0.052	9.277
	Column	528.125	1	528.125	169.000	0.001	10.128
	Error	9.375	3	3.125			
Turbidity (NTU)	Block/Row	1.144	3	0.381	5.138	0.106	9.277
	Column	0.382	1	0.382	5.138	0.108	10.128
	Error	0.223	3	0.074			
Oils and Grease (mg/l)	Block/Row	22.334	3	7.445	3.317	0.176	9.277
	Column	159.311	1	159.311	70.976	0.004	10.128
	Error	6.734	3	2.245			
SO ₄ ²⁻ (mg/l)	Block/Row	8.430	3	2.810	15.652	0.025	9.277
	Column	2.098	1	2.098	11.687	0.042	10.128
	Error	0.539	3	0.180			
Cl ⁻ (mg/l)	Block/Row	161.500	3	53.833	11.963	0.036	9.277
	Column	2380.500	1	2380.500	529.000	0.000	10.128
	Error	13.500	3	4.500			
NO ₃ ⁻ (mg/l)	Block/Row	15.024	3	5.008	3.121	0.187	9.277
	Column	16.881	1	16.881	10.519	0.048	10.128
	Error	4.814	3	1.605			
CaCO ₃ (mg/l)	Block/Row	4297.000	3	1432.333	5.931	0.089	9.277
	Column	4140.500	1	4140.500	17.145	0.026	10.128
	Error	724.500	3	241.500			
Ca ²⁺ (mg/l)	Block/Row	687.520	3	229.173	5.931	0.089	9.277
	Column	662.480	1	662.480	17.145	0.026	10.128
	Error	115.920	3	38.640			
Na ⁺ (mg/l)	Block/Row	68.234	3	22.745	11.963	0.036	9.277
	Column	1005.761	1	1005.761	529.000	0.000	10.128
	Error	5.704	3	1.901			
Ba ²⁺ (mg/l)	Block/Row	14.070	3	4.690	18.411	0.020	9.277
	Column	6.476	1	6.476	25.422	0.015	10.128
	Error	0.764	3	0.255			
Fe ²⁺ (mg/l)	Block/Row	3.380	3	1.127	14.522	0.027	9.277
	Column	5.346	1	5.346	68.914	0.004	10.128
	Error	0.233	3	0.078			
Mg ²⁺ (mg/l)	Block/Row	0.573	3	0.191	46.837	0.005	9.277
	Column	0.001	1	0.001	0.218	0.673	10.128
	Error	0.012	3	0.004			

Table 8 Correlation coefficients (Pearson) for all continuous variables

	pH	Temperature	TDS	TSS	TS	Turbidity	Oils and Grease	SO ₄ ²⁻	Cl ⁻	NO ₃ ⁻	CaCO ₃	Ca ²⁺	Na ⁺	Ba ²⁺	Fe ²⁺	Mg ²⁺	
pH	1.000																
Temperature	0.541	1.000															
TDS	-0.330	0.221	1.000														
TSS	-0.341	-0.470	-0.678	1.000													
TS	-0.776	-0.150	0.709	0.037	1.000												
Turbidity	-0.275	0.332	0.675	-0.246	0.682	1.000											
Oils and Grease	-0.771	-0.111	0.717	0.004	0.978	0.570	1.000										
SO ₄ ²⁻	0.265	-0.155	-0.818	0.532	-0.603	-0.468	-0.653	1.000									
Cl ⁻	-0.962	-0.406	0.455	0.239	0.848	0.301	0.874	-0.344	1.000								
NO ₃ ⁻	0.836	0.668	-0.039	-0.387	-0.424	0.239	-0.478	0.026	-0.798	1.000							
CaCO ₃	0.437	-0.094	-0.605	0.181	-0.649	-0.714	-0.576	0.122	-0.530	0.072	1.000						
Ca ²⁺	0.437	-0.094	-0.605	0.181	-0.649	-0.714	-0.576	0.122	-0.530	0.072	1.000	1.000					
Na ⁺	-0.962	-0.406	0.455	0.239	0.848	0.301	0.874	-0.344	1.000	-0.798	-0.530	-0.530	1.000				
Ba ²⁺	0.411	0.055	-0.760	0.362	-0.687	-0.380	-0.740	0.966	-0.482	0.221	0.093	0.093	-0.482	1.000			
Fe ²⁺	-0.583	0.051	0.769	-0.150	0.902	0.913	0.816	-0.541	0.626	-0.136	-0.781	-0.781	0.626	-0.543	1.000		
Mg ²⁺	0.187	0.039	0.428	-0.684	-0.074	0.172	-0.113	-0.060	-0.135	0.281	-0.496	-0.496	-0.135	0.051	0.125	1.000	

the observed data. It ranges between 0 and 1. The more the coefficients progress towards 1, the better the linear fit between the correlated variables. Analysis variables with strong correlation coefficients and near-1 coefficients of determinations were further examined in Table 11 to provide further insights regarding their inter-relationship.

In Table 11, Turbidity and Fe²⁺; Total Solids and Oils and Grease relationships were further examined using simple linear regression and ANOVA techniques. Each pair had direct proportionality with very impressive coefficients of determination. The dependability of total solids on Fe²⁺ and Cl⁻ concentrations and the dependability of pH on NO₃⁻ and Cl⁻ concentrations were interpreted using multiple linear regression and ANOVA. All but Cl⁻ had positive independent variable coefficients. Its negative coefficient confirms that for every unit increase in Cl⁻ concentration, pH will decrease by the value of its coefficient. Relative to all variables' coefficients, their respective standard errors are small except for turbidity. The standard error tells how sufficiently precise the regression model is by calculating the average distance of the data points from the regression line (Frost 2017). Significance F represents the probability of not rejecting the regression model. It applies to the entire model while the *P* value applies to each respective coefficient (Mathews n.d.). All significance *F* values are below the 0.01, 0.05 and 0.1 significance levels making the models highly statistically significant. The 95% confidence intervals give estimated ranges of real coefficient values (Mathews n.d.). For

instance, the coefficient for total solids in its first model is 31.943, there is a 95% probability that it could be as low as 25.594 and as high as 38.293. Figures 5 and 6 displays the plot of turbidity (NTU) against Fe²⁺ (mg/l) and total solids (mg/l) against oils and grease (mg/l). Figures 7, 8, 9 and 10 are for the multiple linear regression outputs. They depict observed and predicted data for dependent variables against their associated independent variables. Microsoft Excel (2016) was used in creating Figs. 5, 6, 7, 8, 9 and 10.

Conclusion

The suitability of the 3 chemicals in the treatment of produced water can be summed up as follows:

Dosing suitability

The incorporation of more ionic parameters other than the conventional ones showed the reducing effects aluminium sulphate, ferrous ammonium sulphate and calcium chloride had on NO₃⁻ and Fe²⁺ concentrations. Aluminium sulphate represents the best treatment option for physical parameters correction. For highly saline waters, ferrous aluminium sulphate is the best option. Calcium chloride is best considered when sulphate and barium levels are required to be maintained. With these groups of chemicals, the possibility of them being used as stand-alone treatment chemical isn't yet

Table 9 P values (Pearson) from the correlation analysis

	pH	Temperature	TDS	TSS	TS	Turbidity	Oils and Grease	SO ₄ ²⁻	Cl ⁻	NO ₃ ⁻	CaCO ₃	Ca ²⁺	Na ⁺	Ba ²⁺	Fe ²⁺	Mg ²⁺	
pH	0.000																
Temperature	0.166	0.000															
TDS	0.425	0.599	0.000														
TSS	0.408	0.239	0.064	0.000													
TS	0.024	0.722	0.049	0.931	0.000												
Turbidity	0.509	0.421	0.066	0.557	0.063	0.000											
Oils and Grease	0.025	0.793	0.046	0.993	0.000	0.140	0.000										
SO ₄ ²⁻	0.525	0.715	0.013	0.175	0.114	0.243	0.079	0.000									
Cl ⁻	0.000	0.319	0.257	0.569	0.008	0.469	0.004	0.404	0.000								
NO ₃ ⁻	0.010	0.070	0.927	0.344	0.295	0.569	0.231	0.951	0.018	0.000							
CaCO ₃	0.279	0.825	0.112	0.667	0.081	0.047	0.135	0.774	0.177	0.865	0.000						
Ca ²⁺	0.279	0.825	0.112	0.667	0.081	0.047	0.135	0.774	0.177	0.865	0.000	0.000					
Na ⁺	0.000	0.319	0.257	0.569	0.008	0.469	0.004	0.404	0.000	0.018	0.177	0.177	0.000				
Ba ²⁺	0.312	0.897	0.029	0.378	0.060	0.353	0.036	0.000	0.227	0.599	0.827	0.827	0.227	0.000			
Fe ²⁺	0.129	0.904	0.026	0.723	0.002	0.002	0.013	0.166	0.097	0.747	0.022	0.022	0.097	0.164	0.000		
Mg ²⁺	0.657	0.928	0.290	0.061	0.862	0.683	0.789	0.889	0.750	0.500	0.211	0.211	0.750	0.904	0.767	0.000	

Table 10 Coefficients of determination (Pearson)

	pH	Temperature	TDS	TSS	TS	Turbidity	Oils and Grease	SO ₄ ²⁻	Cl ⁻	NO ₃ ⁻	CaCO ₃	Ca ²⁺	Na ⁺	Ba ²⁺	Fe ²⁺	Mg ²⁺	
pH	1.000																
Temperature	0.293	1.000															
TDS	0.109	0.049	1.000														
TSS	0.117	0.221	0.460	1.000													
TS	0.602	0.023	0.503	0.001	1.000												
Turbidity	0.076	0.110	0.455	0.060	0.465	1.000											
Oils and Grease	0.595	0.012	0.513	0.000	0.957	0.325	1.000										
SO ₄ ²⁻	0.070	0.024	0.670	0.283	0.363	0.219	0.426	1.000									
Cl ⁻	0.926	0.164	0.207	0.057	0.719	0.091	0.765	0.118	1.000								
NO ₃ ⁻	0.698	0.446	0.002	0.150	0.180	0.057	0.228	0.001	0.637	1.000							
CaCO ₃	0.191	0.009	0.366	0.033	0.422	0.510	0.331	0.015	0.281	0.005	1.000						
Ca ²⁺	0.191	0.009	0.366	0.033	0.422	0.510	0.331	0.015	0.281	0.005	1.000	1.000					
Na ⁺	0.926	0.164	0.207	0.057	0.719	0.091	0.765	0.118	1.000	0.637	0.281	0.281	1.000				
Ba ²⁺	0.169	0.003	0.578	0.131	0.472	0.144	0.548	0.932	0.232	0.049	0.009	0.009	0.232	1.000			
Fe ²⁺	0.340	0.003	0.592	0.023	0.813	0.833	0.667	0.292	0.392	0.019	0.610	0.610	0.392	0.295	1.000		
Mg ²⁺	0.035	0.001	0.183	0.468	0.005	0.030	0.013	0.004	0.018	0.079	0.246	0.246	0.018	0.003	0.016	1.000	

likely given the chloride, barium, and sodium ion concentration increase. The work also confirms the highly complex nature of produced water given the sometimes-non-corresponding trend between 2 different samples treated with the same chemical.

Statistical suitability

50% of the continuous effluent characteristics (total suspended solids, total dissolved solids, total solids, SO₄²⁻, Cl⁻, Na⁺, Ba²⁺, Fe²⁺, Mg²⁺) were statistically significant for the

treatment chemicals hypothesis while 75% of the continuous effluent characteristics (total dissolved solids, total solids, pH, oils and grease, SO₄²⁻, Cl⁻, NO₃⁻, CaCO₃, Ca²⁺, Na⁺, Ba²⁺ and Fe²⁺) were statistically significant for the sample type hypothesis. This indicates that effluent characteristics were more influenced by the nature/composition of the produced water than the treatment chemicals used. Only 37.5% of the continuous effluent characteristics were statistically significant for both hypotheses.

Table 11 Regression and ANOVA summary output for some correlated variables

		Coefficients	Multiple R	R Squared	Adjusted R Squared	Significance F	Standard error	P value	Lower 95%	Upper 95%	Regression equation
Simple linear regression	Intercept on turbidity axis	0.139	0.913	0.833	0.805	0.002	0.281	0.638	-0.548	0.826	Predicted Turbidity = $0.403Fe^{2+} + 0.139$
	Fe ²⁺	0.403					0.074	0.002	0.223	0.584	
	Intercept on total solids axis	31.943	0.978	0.957	0.950	0.000	2.595	0.000	25.594	38.293	Predicted Total Solids = $1.777(\text{Oils and Grease}) + 31.943$
	Oils and Grease	1.777					0.154	0.000	1.401	2.154	
Multiple linear regression	Intercept on total solids axis	23.141	0.972	0.945	0.923	0.001	4.402	0.003	11.825	34.456	Predicted Total Solids = $0.230Fe^{2+} + 5.085Cl^{-} + 23.141$
	Fe ²⁺	0.230					0.066	0.018	0.059	0.400	
	Cl ⁻	5.085					1.119	0.006	2.207	7.962	
	Intercept on pH axis	9.136	0.969	0.938	0.914	0.001	0.441	0.000	8.001	10.270	Predicted pH = $0.035NO_3^{-} - 0.018Cl^{-} + 9.136$
	NO ₃ ⁻	0.035					0.034	0.356	-0.053	0.122	
	Cl ⁻	-0.018					0.004	0.007	-0.029	-0.008	

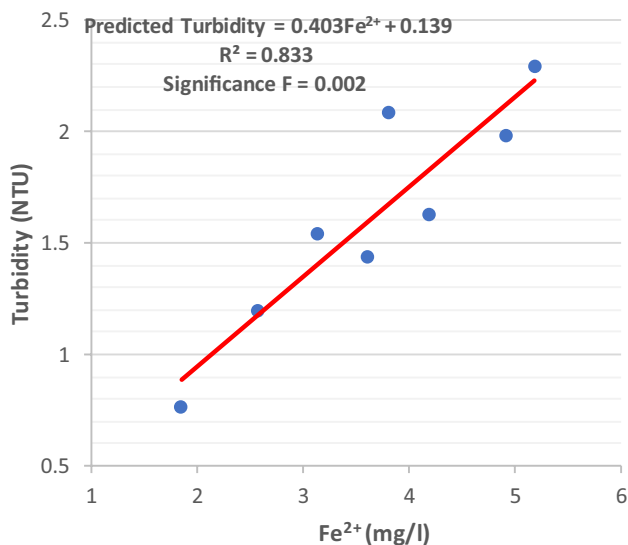


Fig. 5 Scatter plot of turbidity (NTU) against Fe²⁺ (mg/l)

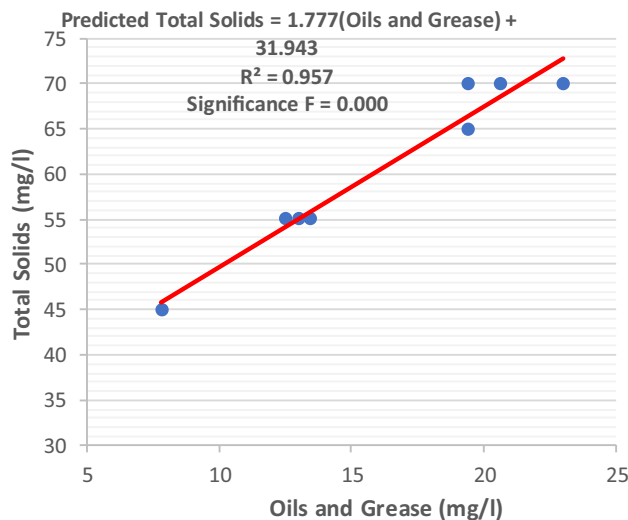


Fig. 6 Scatter plot of total solids (mg/l) against oils and grease (mg/l)

Output model suitability

Significant strength and variable dependence were recorded and examined for 4 relationships namely turbidity and Fe²⁺; total solids with oils and grease; total solids, Fe²⁺ and Cl⁻; pH, NO₃⁻ and Cl⁻. All models were statistically significant using the three most widely used significance levels.

Recommendation

Increase in settling time to yield better total suspended solids and oils and grease results. The incorporation of more treatment chemicals especially those with double salt natures to create a wider spectrum of treatment options. The Department of Petroleum Resources (DPR) should set limits for newly discovered parameters with potential harm to the environment. Regulatory bodies (especially the DPR) should implement measure standards such as WQI (Water

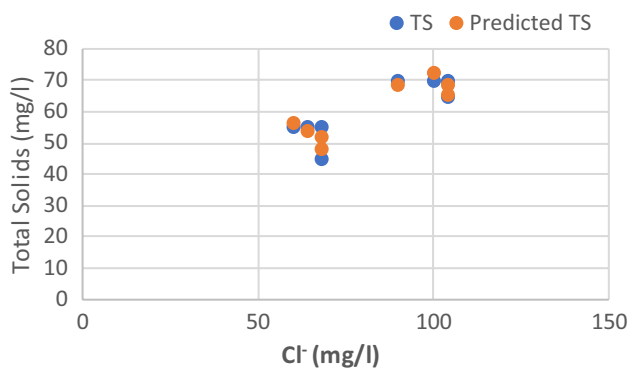


Fig. 7 Line fit plot of observed and predicted total solids (mg/l) against Cl^- (mg/l)

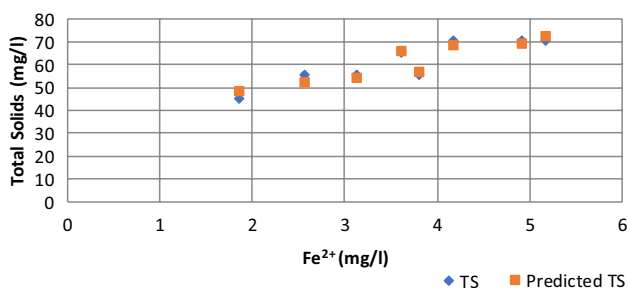


Fig. 8 Line fit plot of observed and predicted total solids (mg/l) against Fe^{2+} (mg/l)

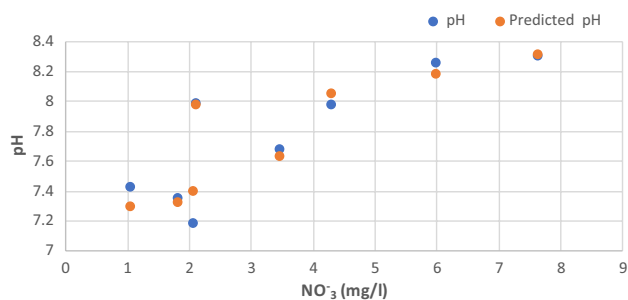


Fig. 9 line fit plot of observed and predicted pH values against NO_3^- (mg/l)

Quality Index), THQ (Target Hazard Quotient) for produced water. Better liaison between the petroleum and chemistry fields in order to investigate the reaction chemistry in these options.

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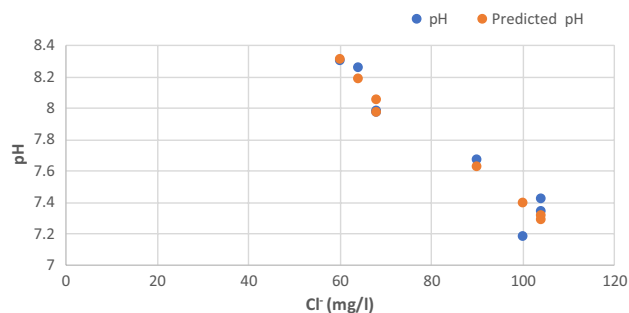


Fig. 10 Line fit plot of observed and predicted pH values against Cl^- (mg/l)

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Declaration

Conflict of interest I, Clifford Okwudili Aniakor, the sole author of this research hereby declare that I have no conflicting interests.

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