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Effectiveness of coconut shell activated carbon for the treatment of produced water to meet DPR standards for disposal

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Abstract

Produced water is a complex inorganic and organic mixture of compounds[1]. It is also the main waste produced in oil and gas recovery operations[2]. This investigation will address the issue of pollution caused by produced water disposal into the environment, by treating produced water with Coconut shell activated carbon using the adsorption principle, to ensure produced water meet Department of Petroleum Resources (DPR) standards for disposal in the Niger Delta. Physio-chemical test were carried out on a produced water sample. Results obtained showed that the produced water sample is toxic, and can pose serious human health hazards and contaminate the environment if disposed off. Remedial treatment was offered to the produced water sample using Coconut shell activated carbon, and significant improvements were noticed as compared with the standards of Department of Petroleum Resources (DPR) for disposal of produced water in the Niger Delta. The produced water temperature was reduced from 29.7^{o}C to 28^{o}C, the pH of the sample was acidic with pH of 6.61 prior to treatment improved to 7.90. The amount of Oil and Grease diminished from 64mg/L to 12mg/L for 5ml of untreated and treated produced water respectively, Turbidity also experienced significant improvement, it was reduced from 103.5 NTU to 06.0 NTU. Total Dissolved Solids (TDS) was reduced from 0514ppm to 0483ppm, while Total Suspended Solids (TSS) was reduced from 0.03g to 0.02g, appreciable reduction was also noticed for the Chemical Oxygen Demand (COD), as it was reduced from 327.9mg/L to 156.6mg/L, as well as the Biochemical Oxygen Demand (BOD) which dropped from 80mg/L to 68.47 mg/L. Improvements were observed from the results obtained for the heavy metals.

Keywords: Produced Water, Pollution, Toxic, Hazard, Treatment, Niger Delta.

Introduction

Produced water is a complex inorganic and organic mixture of compounds[1], is also the main waste produced in oil and gas recovery operations[2]. Essentially it contains water from formation as well as water injected with trace amount of dissolved organics, heavy metals, suspended oil, dissolved minerals, solids (silt and sand) and production chemicals in the separation/production line[3]. If produce water is not properly managed it can cause serious harm to the environment and give bad publicity to oil and gas industry. Proper produced water management comes with challenges and one of them is its significant cost[4].

This research aim is to treat produced water obtained from an Oil and Gas Company in Sapele Delta State to the standard of Department of Petroleum Resources (DPR) for disposal in the Niger Delta. Produced water which was, was analyzed and treated using Coconut shell activated carbon. The physio-chemical properties tested and analyzed were Temperature, pH, Chemical Oxygen Demand (COD), Electrical conductivity, Total Suspended Solids (TSS), Oil/Grease, Salinity, Turbidity, Lead, Iron, Total Dissolved Solids (TDS), Copper, Zinc, Chromium and Biochemical Oxygen Demand (BOD).

Produced water management system performance and reliability is very crucial for a seamless oil and gas production especially in fields that are mature where production of water immensely affects oil production. Currently the gas and oil industry manages more produced water than even oil[5]. It is estimated globally that about 250 million barrels of produced water from oil and gas fields are produced daily and more than 40% of this is discharged into the environment. How to effectively manage the large amount of unwanted produced water is one challenge oil and gas companies have to contend with[6]. Cost of disposal which encompass capital, transportation, maintenance and infrastructure cost, sums up to about $4.00/bbl. The treatment and disposal of produced water has now become a matter of extreme importance in gas and oil recovery operations for the following reasons[7]. i. Large volumes of produced water are being produced as more fields gets old. ii. Stringent disposal standards are being introduced in many operating areas of the world. iii. The need to reduce operating cost as well as capital[7].

Petroleum is produced along with huge amount of waste. Wastewater accounts for about 80% of liquid waste and can get up to 95% in matures fields, Oil and gas companies manage produced water through variety of options that include; i. Injection of produced water into formations. ii. Using polymer gel or down hole water separators to block fractures and passage of water to avoid water production[4]. iii. If produced water meets offshore and onshore discharge standards/regulations it may be disposed into the environment. iv. Treated produced water can be tailored for use for work over operations and even drilling operations[4]. v. Produced water can be used for industrial purposes, irrigation and even drinking water, but this will involve intense treatment[8].

Regulation of Produced Water Discharge in Niger Delta: Produced water discharge makes up the larger part of wastes that result from gas and oil exploration and production offshore activities.

One of the Department of Petroleum Resources statutory function is to make sure oil and gas industry operators do not degrade the environment in the course of their operations. The Department of Petroleum Resources has continuously developed standards and guidelines for the environment and since 1981 which cut across the control of petroleum, exploration and production and pollutants in Nigeria[9].

Effluent limitations, procedures and standards for monitoring and evaluating discharge of Production Exploration and wastes into the environment are contained in Environmental Guidelines and Standard for the Petroleum Industry (EGASPIN)[10].

For the disposal of produced water a permit is required from EGASPIN, and point sources must be registered with the Director of Petroleum Resources. The Director must give a written approval before changes to process or operation that can change or cause a material decrease or increase in terms of quantity and quality of the discharged produced water can be made.

Produced water discharge in Nigeria without a permit is not allowed and even with a permit produced water must meet allowable standards for disposal as stipulated by DPR before it can be discharged into the environment. If produced water is discharged in conditions not within the allowable limit as stipulated by DPR for the Niger Delta, it is registered as an offence and it's punishable by imprisonment, fine and/or license revocation.

Materials and methods

Materials: Major materials used for this research include; Produced water sample, Coconut shell, Zinc Chloride, n-hexane, Calcium Chloride, Magnesium Sulphate, Iron chloride, Phosphate buffer, Pi-chloric acid and Nitric acid.

Equipment: Some of the major equipment used for this research include; SX-5-12 Electric Muffle Furnace, Eletric hot plate, EcoSense ODO200, Hy – 4A Cycling Vibrator, Turbidity Meter, Desktop pH Meter, Electro-heating standing Oven, Thermometer, Salinity Meter, Tong, Crucible, Electrical Conductivity Meter, Separating funnel, COD Meter, Atomic Absorption Spectrometer (AAS), Whatman Filter paper, Beakers, Conical flask and Corked bottles.

Preparation of Coconut shell activated carbon: Coconut shell carbon preparation: 500g of Coconut shell was washed with water to remove dirt and then dried under the sun for three days. It was then placed on a crucible, and a tong was used to hold the crucible containing the Coconut shell. The crucible was placed into the SX-5-12 Electric Muffle Furnace with a tong and the furnace was set at 600^oC, it was left for 2hrs to carbonize. After allowing cooling the carbonized Coconut shell was pounded and sieved[12].

Activation of Coconut shell carbon: 34.075g of $ZnCl_2$ was measured with a digital weighing scale and then dissolved in 250ml of water. The sieved Coconut shell carbon was added into the Zinc Chloride solution in a 500ml beaker[11]. It was stirred and left to thoroughly soak for 24hrs. The solution was boiled using electrical hot plate for 30min, it was then allowed to cool and rinsed severally with water until salt content reduced to a minimum value as confirmed with a salinity meter. The activated carbon was turned into a dish and placed into an Electro heating standing Oven till it became dried and ready for use as an adsorbent to treat the produced water sample.

Produced water treatment: Treatment of produced water with the prepared Coconut shell activated Carbon: 400ml of produced water was placed in a 500ml conical flask and 4.0g of Coconut shell activated carbon initially prepared was added into the 400ml of the untreated produced water sample. It was then covered with a cork and then taken to the Hy – 4A Cycling Vibrator, and placed firmly into the top holder of the shaker. The shaker was set at 200rpm and allowed to mix for 90min, after 90min of thorough mixing the conical flask was then removed and the entire content filtered using Whatman filter paper into another clean 500ml conical flask, a treated produced water was obtained[13].

Effluent Characteristics Test: pH: The Desktop pH meter calibration was done with a buffer solution of known pH, 100ml of treated produced water sample was poured into a 250ml beaker, and the Desktop pH meter probe cup was filled with some quantity and tested, the value was read off from the digital screen. Similar test was conducted for the un-treated produced water sample.

Salinity: The Salinity meter was calibrated and then hand-deeped into the produced water sample, it was held until a stable value was gotten and the value was read off from the screen. Test was conducted for both un-treated and treated produced water sample.

Turbidity: Turbidity meter was used for this measurement, the turbidity measuring cup was removed and pure turbidity calibration fluid was poured into the cup to calibrate the meter, the pure fluid gave a turbidity value of 0.00 NTU, the fluid was poured out, and the cup was filled with produced water sample and nub and covered, value was read off from the digital screen. Test for Turbidity was conducted for both un-treated and treated produced water sample.

Chemical Oxygen Demand (COD): The Digital COD meter was used for this measurement, water was poured into the COD cup inside the COD meter to fill level, and the COD of the water was checked and value read off from the screen, 2.5ml of produced water sample was poured into a 250ml beaker, and toped to 100ml with the same water initially tested, appreciable quantity was poured into the COD cup inside the COD meter and the COD was checked and the value was read off from the screen, necessary calculations were made to arrive at the final COD value of the produced water sample. COD Test was conducted for both un-treated and treated produced water sample, and result were computed using equation (1).

$$\mathrm{COD} = (\mathrm{D\%} \times \mathrm{W_{COD}}) - \mathrm{Tf_{COD}} \quad (1)$$

D% = Dilution in percentage, $\mathrm{W_{COD}}$ = Measured COD of water (mg/l) and $\mathrm{Tf_{COD}}$= Total fluid COD (mg/l).

Total Suspended Solids (TSS): Whatman filter paper was thoroughly dried in the Electro heating standing Oven at 50°C for 15mins, the filter paper was removed and measured in grams using the digital weighing scale. 100ml of produced water sample was poured into a 100ml beaker, the Whatman filter paper initially dried was used to filter the 100ml of the produced water sample into a 500ml beaker, the filter paper was then placed into the Electro heating standing Oven at 50°C and dried thoroughly, it was then removed and weighed. TSS value in grams was gotten from initial weight and final weight difference of the Whatman filter paper[2].

TSS test was conducted for both un-treated and treated produced water sample and the values were computed using equation (2).

$$TSS = W_f - W_i \quad (2)$$

W_f= Final filter paper weight (g), and W_i = Initial filter paper weight (g).

Total Dissolved Solids (TDS): The TDS and EC meter was switched to TDS mode, distilled water was used to thoroughly rinse the TDS and EC meter, and then hand-deeped into the sample of the produced water, hand-held until a stable value was gotten, and the value was read off from the digital screen of the TDS and EC meter. TDS test was conducted for both un-treated and treated produced water sample.

Temperature: TM-902C Digital temperature Meter was used to carry out this test, the metal probe of the meter was placed into some quantity of produced water sample in 100ml beaker, and the value was read off from the screen.

Electrical Conductivity (EC): The TDS and EC meter was placed on EC mode with the mode button, its sensitive sensor was rinsed thoroughly with water, and then hand-deeped into the produced water sample, it was held until a stable value was gotten, and the value was read off from the screen of the TDS&EC meter.

Oil/Grease: The un-treated produced water sample was thoroughly mixed, and 100ml of the sample was poured into a 250ml beaker, 5ml was removed using the glass syringe, and poured into a clean conical flask. It was then measured in grams using the digital weighing scale, another 200ml of un-treated produced water was prepared and 50ml of n-hexane was added. It was then turned into a separating funnel and allowed to settle, bottom fluid in the separating funnel was drained and allowed to fill a 250ml beaker to 100ml point. The glass syringe was again used to collect a 5ml sample from it and then measured in grams using the digital weighing scale. The initial value in grams was subtracted from the final value, and divided by the initial volume of the produced water sample to arrive at the Oil and Grease content value in mg/L. The Oil/Grease content test was also conducted on the produced water treated sample and results were computed with equation (3) in mg/l.

$$Oil\,\&\,Grease = \frac{(Wpw - Wn) \times 1000}{Vpw \times 0.001} \quad (3)$$

Wpw = Weight of 5ml of PW sample (g), Wn = Weight of 5ml of PW sample with n-hexane (g), and Vpw = Volume of PW sample (ml).

Dissolved Oxygen: The EcoSense ODO200 digital meter probe was placed in a 250ml beaker containing 250ml of the produced water sample to be tested, and the Dissolved Oxygen value was read off from the equipment screen. Dissolved Oxygen (DO) test was conducted for both un-treated and treated produced water sample.

Biochemical Oxygen Demand (BOD): A 2000ml beaker was obtained and filled with distilled water to the 2000ml point. 2ml each of $CaCl_2$, $MgSO_4$, $FeCl_2$ and Phosphate buffer was put in the 2000ml of distilled water to prepare the dilution water a 267ml corked bottle was obtained and 10ml of the un-treated produced water sample was put in it with the help of a glass syringe, the bottle was then topped with the dilution water to fill it to the brim and then corked. Dissolved Oxygen value of the dilution water and the Dissolved Oxygen value of the corked fluid were recorded using the EcoSense ODO200 digital meter. The cocked fluid was then left for 5 days[14]. The Dissolved Oxygen was again recorded after 5 days, same procedure was carried out for the treated produced water sample. BOD values were calculated for both un-treated and treated produced water sample from the initial and final values of Dissolved Oxygen recorded for day zero and day five, and the total volume of the fluid in the corked bottles using equation (4).

$$BOD_5 \text{ PW Sample} = \frac{D_1 - D_2}{P} \quad (4)$$

P = Cork Volume of untreated PW (ml), D_1= Day Zero Dissolved Oxygen (mg/l) and D_2= Day Five Dissolved Oxygen (mg/l).

Heavy Metal: A Pi-chloric-Nitric acid mixture was prepared in the ratio of 1:3, and was used to digest the un-treated produced water sample, 10ml of the solution was placed in 10ml of the un-treated produced water sample[15].

It was then heated for about 5 minutes till it became clear, and then taken to the Atomic Absorption Spectrometer (AAS) machine for analysis[16].

The treated produced water sample was analyzed directly without digesting. The Heavy metals analyzed for the un-treated and treated produced water sample were Lead, Iron, Copper, Chromium and Zinc.

Results and discussion

Presentation of Results and Discussion: Table-1 shows the Effluent Produced Water Discharge Limit in the Niger Delta[17]. Table-2 shows various effluent test parameters of the Produced Water sample obtained from an Oil and Gas Company in Sapele Delta State. It also shows results from each test parameter for both un-treated and treated Produced Water (PW) sample.

The pH of the un-treated Produced water sample was 6.61, after treatment it improved to 7.90 as shown in Figure-1. This shows that the level of produced water pH falls within DPR statutory limit of 6.5 to 8.5[17]. The untreated produced water temperature was 29.7°C and after treatment fell to 28°C. Which indicated that the temperature of the Produced water dropped as shown in Figure-2 after treatment, which is lower than the limit stipulated by DPR of 30°C[17].

The concentration results for oil and grease in the un-treated produced water was 64 mg/L for 5ml of the un-treated produced water, and dropped to 12 mg/L for 5mls for the treated produced water as shown in Figure-3.

Table-1: The Effluent Produced Water Discharge Limit in the Niger Delta[17].

Effluent characteristics	Inland	Near shore	Offshore
PH	6.5-8.5	6.5-8.5	No limit
Temperature °C	25	30	-
Oil/Grease Content	10	20	40
Salinity	600	2,000	-
Total Dissolved Solids	2,000	5,000	-
Total Suspended Solids	>30	>50	-
Chemical Oxygen Demand	10	125	-
Biochemical Oxygen Demand	10	125	-
Lead	0	No limit	-
Iron	1	No limit	-
Copper	2	No limit	-
Chromium	0	0	-
Zinc	1	5	-
Mercury	0	-	-
Turbidity	10 NTU	10 NTU	10 NTU

Table-2: The effluent characteristics test results.

Effluent characteristics	**Un-treated Produced Water**	**Treated Produced Water**
pH	6.61	7.90
Temperature	29.7^{o}C	28^{o}C
Oil/Grease Content	64 mg/L	12 mg/L
Salinity	0.54ppt	1.72 ppt
Turbidity	103.5 NTU	06.0 NTU
Total dissolved Solids	0514 ppm	0483 ppm
Total suspended solids	0.03 g	0.02 g
Chemical Oxygen Demand	327.9 mg/L	156.6 mg/L
Electrical Conductivity	1016 µm/cm	1008 µm/cm
Dissolved oxygen	1.76 mg/L	1.37 mg/L
Biochemical Oxygen Demand	80 mg/L	68.47 mg/L
Lead	0.02 mg/L	0.01 mg/L
Iron	0.7 mg/L	0.2 mg/L
Copper	0.06 mg/L	0.1 mg/L
Chromium	0.05 mg/L	0.01 mg/L
Zinc	1.19 mg/L	0.33 mg/L

The drop in oil and grease concentration in the treated produced water shows it is below DPR statutory limit of 20mg/l for near shore disposal[17]. The measured Total Suspended Solids (TSS) and Total Dissolved Solids (TDS) in the produced water before and after the treatment are shown in Figure-4 and Figure-5 respectively.

The Total Dissolved Solids (TDS) concentrations was reduced from 0514ppm to 0483ppm and Total Suspended Solids (TSS) was reduced from 0.03g to 0.02g for 100ml of produced water sample filtered for both un-treated and treated produced water sample respectively. The concentrations of TSS and TDS of the produced water were not only reduced but were below DPR limit of 2000mg/l and 50mg/l respectively[17].The improvement in the produced water sample quality can be attributed to the efficiency of Coconut shell activated carbon used for the treatment of the produced water sample.

Turbidity of the untreated produced water when measured registered a spike as shown in Figure-6 with a value of 103.5 NTU, from visual inspection, the un-treated produced water sample was un-clear, with visible presence of suspended solids, after treatment with Coconut shell activated carbon, the treated produced water sample when tested with the Turbidity meter gave a value of 06.0 NTU, registering a tremendous improvement as it is now below DPR limit of 10 NTU for Inland, Near shore and Offshore disposal.

Figure-7 shows the values of the measured Chemical Oxygen Demand (COD), which gave values of 327mg/L and 156.6mg/L respectively, which registered a very significant reduction in the Chemical Oxygen Demand (COD) even though it was not reduced below the limit stipulated by DPR.

The Biochemical Oxygen Demand values measured for the treated and un-treated Produced water gave values of 80mg/L and 68.47mg/L respectively as shown in Figure-8, there was a considerable reduction in the BOD of the produced water sample. This suggest that the produced water treatment with Coconut shell activated carbon is effective, because BOD is one of the several indicators used to know the effectiveness of produced water treatment[18].

As seen in Figure-9, Salinity of the produced water experienced an increase from 540mg/L to 1720mg/L, this is because of the Zinc chloride ($ZnCl_2$) used in the activation of the Coconut shell carbon, which was used for the produced water sample treatment.

Heavy Metals in the sample of the produced were present in small elemental quantity, which were all within the DPR limit[17]. They experienced minimal reduction except for Copper that increased from 0.06mg/L to 0.1mg/L, Lead went from 0.02 mg/L to 0.01mg/L, Iron went from 0.7mg/L to 0.02mg/L, also Chromium reduced from 0.05mg/L to 0.01mg/L, and zinc reduced from 1.19mg/L to 0.33mg/L. Electrical conductivity also reduced from 1016µm/cm to 1008µm/cm.

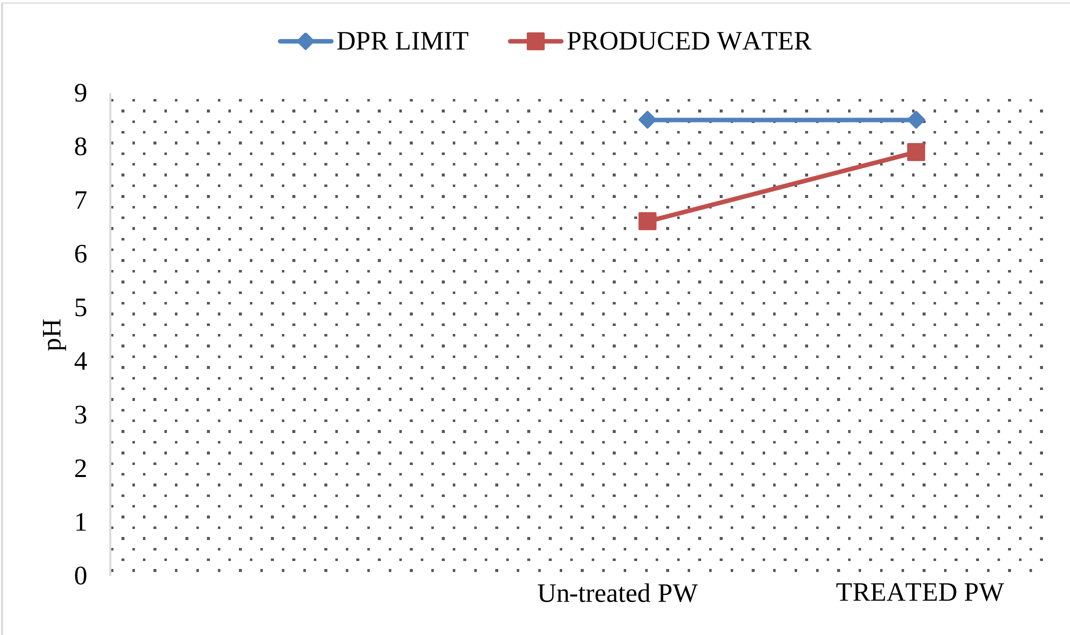

Figure-1: Graph showing pH values for un-treated and treated produced water with DPR limit for Ph.

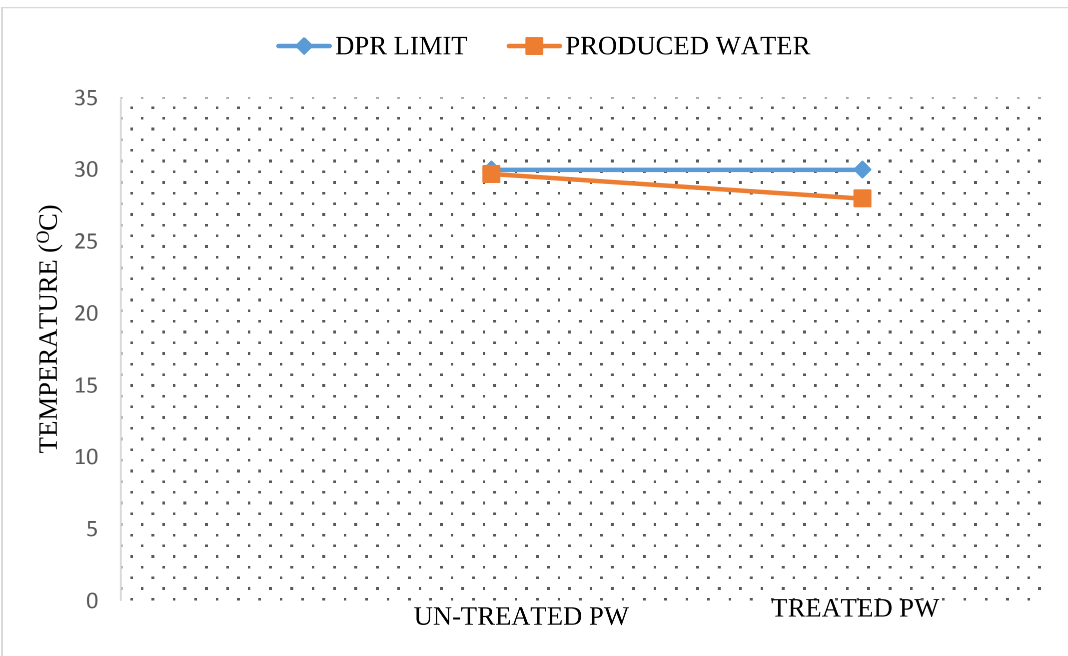

Figure-2: Graph showing Temperature values for un-treated and treated produced water with DPR limit for Temperature.

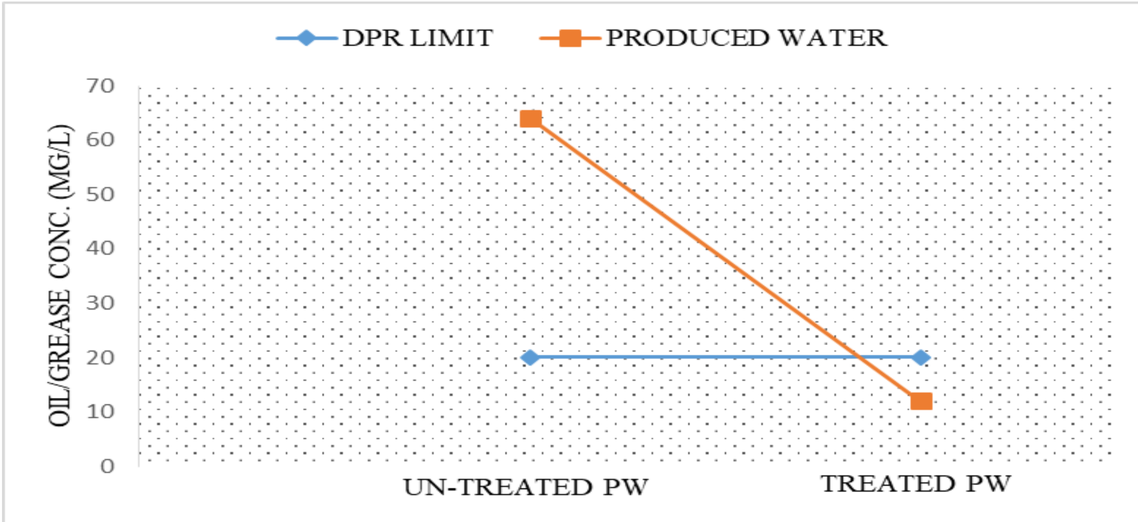

Figure-3: Graph showing Oil/Grease content values for un-treated and treated produced water with DPR limit for Oil/Grease.

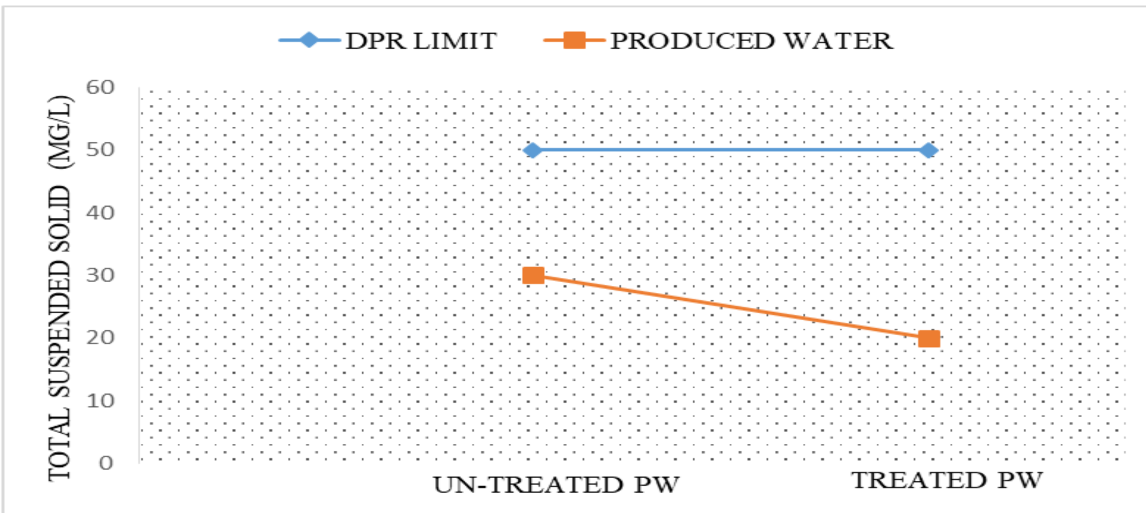

Figure-4: Graph showing TSS values for un-treated and treated produced water with DPR limit for TSS.

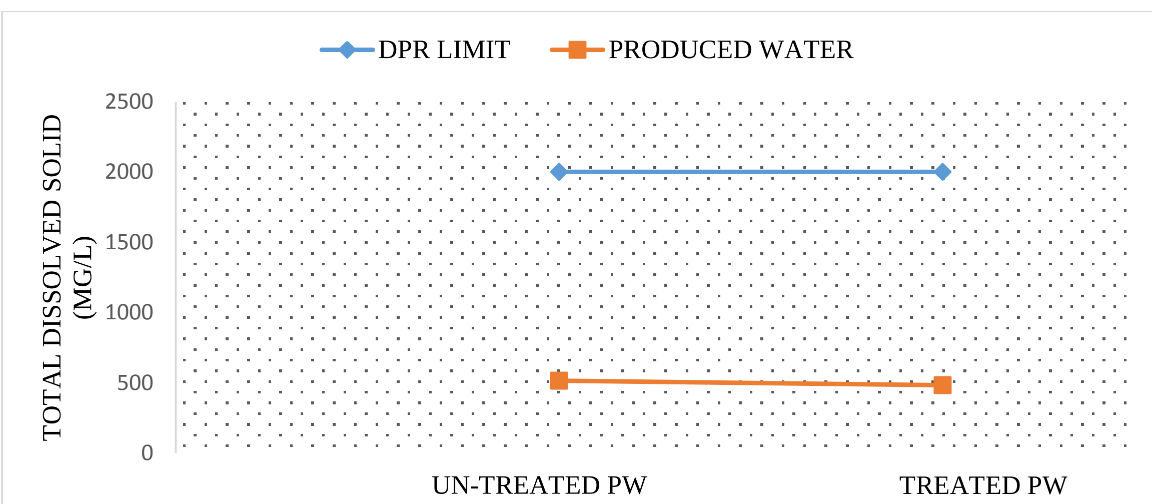

Figure-5: Graph showing TDS values for un-treated and treated produced water with DPR limit for TDS.

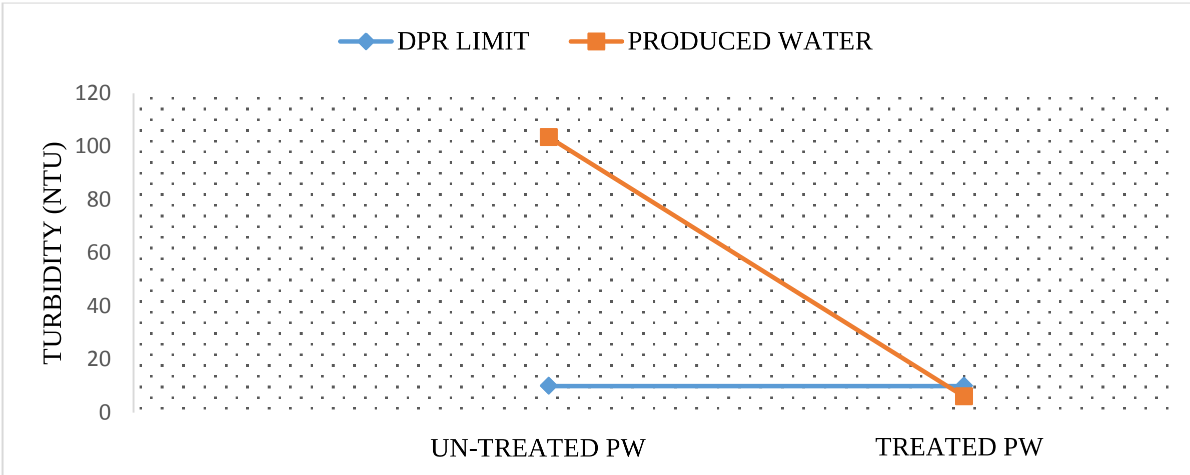

Figure-6: Graph showing Turbidity values for un-treated and treated produced water with DPR limit for Turbidity.

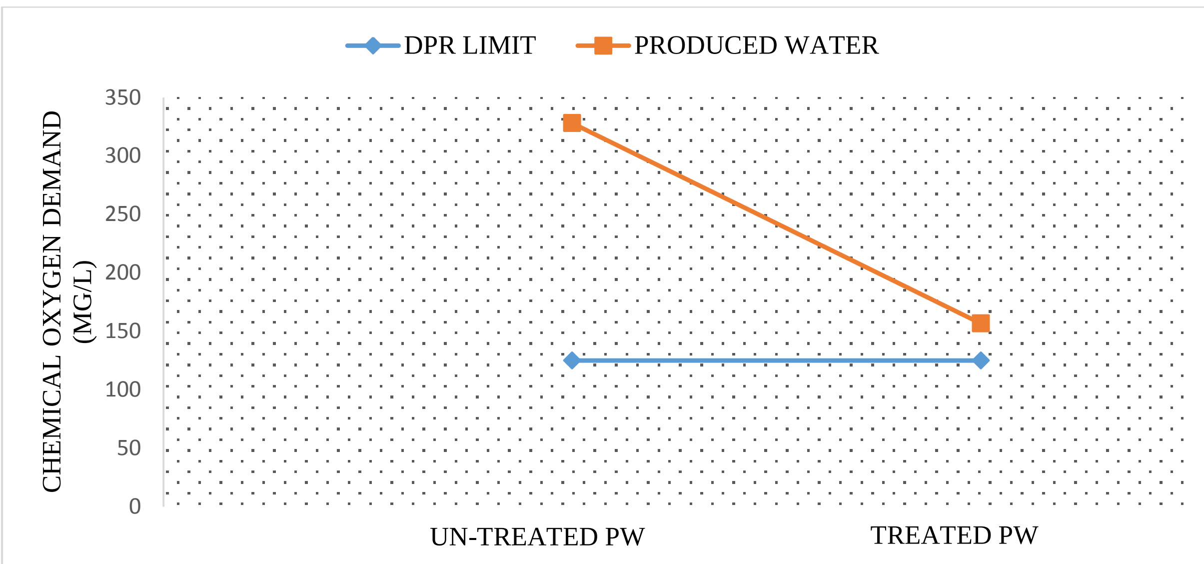

Figure-7: Graph showing COD values for un-treated and treated produced water with DPR limit for COD.

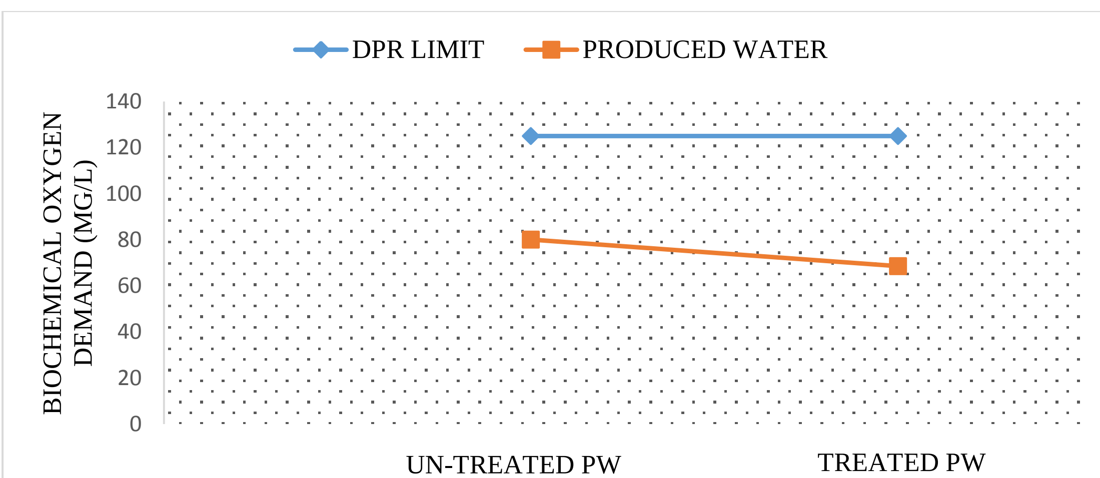

Figure-8: Graph showing BOD values for un-treated and treated produced water with DPR limit for BOD.

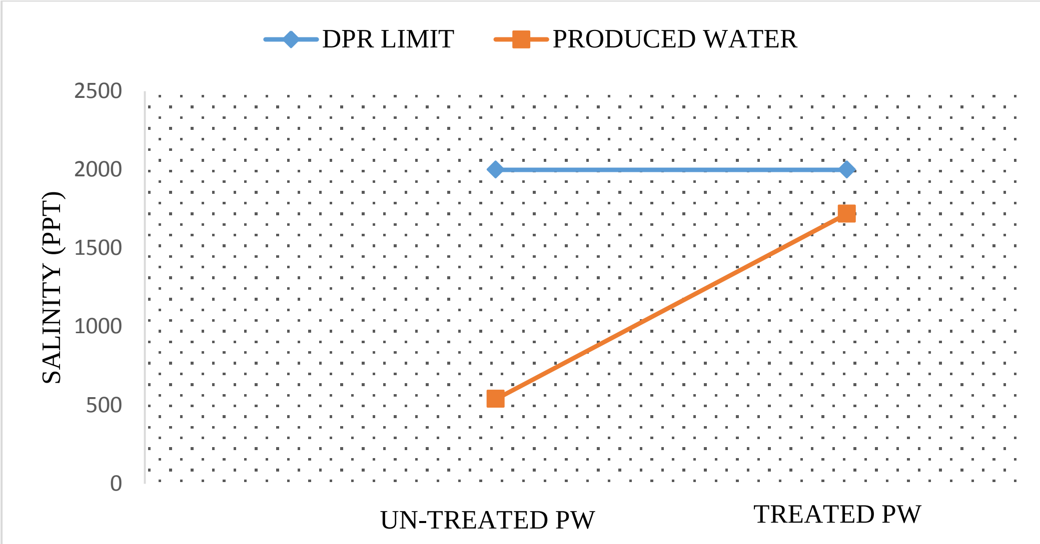

Figure-9: Graph showing Salinity values for un-treated and treated produced water with DPR limit for Salinity.

Conclusion

This research showed that the obtained produced water sample is below statutory limits of DPR, and must be treated properly to meet allowable disposal standards. This research also confirmed that the use of Coconut shell activated carbon to treat produced water applying the adsorption technique is an effective means to treat and bring produced water to allowable limit for disposal.

This also showed that the produced water treatment technology is effective in treating and preparing produced water for discharge to the environmental haven met allowable standards. Although further refining techniques may be used to support the adsorption technique for produced water treatment if available, this could further cause a drop in dispersed oil contents, as well as the COD and in some cases a drop in the level of organic pollutants and soluble aromatics, especially persistent organic pollutants.

Water production is on the high especially as Oil and gas wells mature, this increase in production volumes of produced water has resulted in an enormous pollution threat. Techniques that are effective for removing organic compounds that are toxic in produced water have now drawn huge interest, and from this research it was proven to a large extent that Coconut shell activated carbon is a locally sourced effective adsorbent for produced water treatment to meet allowable standards for disposal.

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