ISSN (Online) 2456-1290

International Journal of Engineering Research in Mechanical and Civil Engineering (IJERMCE)
Vol 3, Issue 3, March 2018

Analysis and Treatment of Oil Field Produced Waters of Upper Assam Basin

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Abstract: **-- The largest volume in oil industries comprise of Produced Water (PW) from oil fields. The water besides containing hydrocarbons in soluble, emulsified and free form, also is high in suspended and dissolved solids. Hydrocarbons are considered among the most harmful pollutants as they are lethal to human, animal and plant on direct consumption. Therefore the PW samples have to be treated before discharging it into the environment as it contains harmful constituents. Results showed that the PW samples had water content above 80% along with Oil and Grease (O&G), salinity, turbidity, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Ca, Fe, Na, K, Li, Mg, Mn, Pb and Zn. However the presence of Cr, Cu, Mo, and Ni has not been observed in the samples. These samples were treated with microfiltration and ultrafiltration in the continuous cross flow cell. Most of the results after the treatment processes were found to be within range as per Central Pollution Control Board (CPCB) of India. However, the result of dissolved oxygen while treating with microfiltration was not found to be within range. Also, the treatment of oil and grease by both the treatment processes did not meet the specified range set by the pollution control board of India. Therefore further filtration has to be done to bring all the parameters within range.**

Keywords: **- Filtration, Inorganic, Pollutants and Produced water.**

I. INTRODUCTION

Water is generally present with oil in the reservoir. As the oil production continues its saturation slowly decreases and the water saturation increases which in later stage is produced with oil sometimes in the form of emulsion. This water besides containing hydrocarbons in soluble, emulsified and free form, also usually is high in suspended and dissolved solids. Hydrocarbons are considered among the most harmful pollutants as they are lethal to human, animal and plant on direct consumption. It is thus, obvious that the effluent cannot be let off as such in the environment. Realizing the threat posed by industrialization and rapid development, government all over the world have imposed strict legislations to the industries. Various agencies such as US environmental protection agency, World Health Organisation (WHO), CPCB, Indian Council of Medical Research and MINAS have formulated standards for quality of water used for different purpose and for safe disposal by various means. From natural gas production operations, PW account for 80% of the wastes and residuals produced. Besides oil wells are producing with 98% PW and 2% fossil fuel in some oil fields of Upper Assam Basin [1]. For each barrel of oil, 7 to 10 barrels of PW is produced in US [2]. After treatment the PW can be discharged into the environment or can be used in water injection wells for reservoir pressure maintenance [2, 3]. The PW quality vary considerably depending on the location of the reservoir oil field, the characteristic and type of the producing well, the geochemistry of the producing formation and the type of hydrocarbon product being produced [2, 3, 4, 5]. The membrane separation is a technology which separates two fluids via pores or gaps in the molecular arrangement of a continuous structure. This technology is being used for treatment of contaminated water so as to bring the level of contamination within the limits set by the pollution control board of both the centre and state.

II. EXPERIMENTAL

(A) Materials

Five samples of formation water and crude oil were collected from oil fields of Upper Assam Basin.

Table 1 – Materials Used

Sl. No.	Materials	Specification	Firm
1	Formation Water 1		
2	Formation Water 2	Barail formation	Oil Industry
3	Formation Water 3		
4	Formation Water 4		

5	Formation Water 5		
6	Potassium hydroxide (KOH)	M.W. – 56.11 g/mol	Merck specialities private limited
7	Ethanol (C_2H_5OH)	M.W. – 46.07	Biochem life sciences
8	Petroleum Ether	Wt. Per ml at 20^0C, 0.630 – 0.645 g	Fisher Scientific
9	Ethylene di-amine tetra acetic acid (EDTA) Disodium Salt Dihydrate	0.01 M	RFCL Limited
10	Erichrome Black T	0.5 g in 100 ml	RFCL Limited
11	Potassium Chloride (KCl)	M.W. – 74.56 g/mol	Avantor Performance Materials India Limited
12	Sodium Chloride (NaCl)	M.W. – 58.44 g/mol	RFCL Limited
13	Calcium Carbonate ($CaCO_3$)	M.W. – 100.09 g/mol	Merck specialities private limited
14	Lithium Carbonate (Li_2CO_3)	M.W. – 73.89 g/mol	Spectrochem Pt. Ltd. Mumbai (India)
15	Hydrochloric Acid (HCl)	M.W. 36.46 g/mol, Sp. Gr. at 25^0C is about 1.18	RFCL Limited
16	Phenolphthalein	pH range 8.2 – 10 (colourless to pink)	RFCL Limited
17	Filter Paper	Pore size 0.45 µm, Diameter 47 mm	Whatman
18	Toluene (C_7H_8)	M. W. – 92.14 g/mol	Merck Specialities private Limited

(B) METHODS

The following instruments were used for the experimental work

Table 2 – Parameters determined

Sl. No.	Parameters	Instruments	Chemicals Used
1	pH	***Make***- Systronics ***Model***- Water Analyser 371	-
2	Salinity		
3	Conductivity		
4	Total Dissolved Solids (TDS)		
5	Turbidity		
6	Dissolved Oxygen (DO)		
7	Total Suspended Solids (TSS)	***Make***- Vacuubrand ***Model***- D 97877	-
8	Water Content	***Make***- Electra ***Model***- Electra Lab Centrifuge	Toluene (C_7H_8)
9	Biochemical Oxygen Demand	***Make***- VELP Scientifica ***Model***- FOC	Potassium Hydroxide (KOH)
10	Sodium (Na)	***Make***- Systronics ***Model***- Flame Photometer128 Compressor 126	Sodium Chloride (NaCl)
11	Potassium (K)		Potassium Chloride (KCl)
12	Calcium (Ca)		Calcium Carbonate ($CaCO_3$)
13	Lithium (Li)		Lithium Carbonate (Li_2CO_3)
14	Manganese (Mn)	Atomic Absorption Spectrometer ***Make***- Perkin Elmer ***Model***- Analyst 200	-
15	Iron (Fe)		
16	Chromium (Cr)		
17	Copper (Cu)		
18	Magnesium (Mg)	Inductively Coupled Plasma Optical Emission Spectrophotometer	-
19	Molybdenum (Mo)		
20	Nickel (Ni)		
21	Lead (Pb)		
22	Strontium (Sr)		
23	Zinc (Zn)		

24	Membrane separation	***Make-*** Perkin Elmer, USA ***Model-*** Optima 2100 DV Continuous Cross Flow Cell with reciprocating pump ***Make-*** Pumps India Pvt. Ltd. ***Model-*** CI

III. EXPERIMENTS

The following experiments will be performed both before and after treatment.

(A) Water analyzer
The pH, conductivity, salinity, TDS, Dissolved Oxygen (DO) and turbidity by using Systronics water analyser 371.

(B) Water content
For evaluating water content, the oldest and most widely used method is the centrifuge test, where equal volumes of oil and solvent are placed in a graduated centrifuge tube. After centrifugation, the volume of the higher gravity water and sediment layer at the bottom of the tube is read. Therefore, 5 ml of CO + 5 ml of toluene is completely mixed. The new sample is run in the centrifuge for 5 minutes. And then two layers is easily visible. It can be calculated by using the formula

$$\text{Water content} = \frac{Amount\ of\ water\ sample\ visible}{Amount\ of\ \ sample\ initially\ taken} * 100\ \%$$

(C) Oil & grease (O&G)
20 ml sample water + 40 ml petroleum ether in separating funnel was mixed and shaken for a half an hour. Appearance of two layers was observed where the lower layer was separated out in the beaker then dried in the oven and weighed = y, x= weight of the empty beaker.

$$\textbf{O\&G (ppm)} = \frac{(y-x)\times 10^6}{sample}$$

(D) Total suspended solid (TSS)
The TSS can be calculated by using Millipore assembly. It can be calculated by using the formula below

$$\textbf{TSS (ppm)} = \frac{Wt\ of\ filter\ paper\ with\ cake - Wt\ of\ filter\ paper}{Volume\ of\ water\ filtered\ in\ 30\ mins}$$

(E) Biochemical Oxygen Demand
The BOD can be calculated by measuring the dissolved oxygen (DO) before putting the sample in the BOD incubator for 5 days. After 5 days DO is calculated again. The difference gives the value of BOD.

(F) Titration methods
Alkalinity*:* 50 ml sample water + methyl orange titrated with 0.05 N HCl = T

$$HCO_3 = \frac{T\times 0.05\times 50\times 1000}{sample}$$

Total Alkalinity (ppm) = HCO_3 × 0.82
Total hardness*:* 50 ml sample water + buffer solution (3-4 drops) + Erichrome black indicator, then titrated with EDTA 0.01 M or 0.02N. A change to ink colour was observed after titration.

$$\text{Total hardness (ppm)} = \frac{EDTA\times 1000}{sample}$$

(G) Flame Photometer
Flame Photometer instrument is used to determine Na, K, Li and Ca present in the FW.
Preparation of stock standard solution

a. Na: A standard solution of 1000 ppm is prepared by dissolving 2.5416g NaCl in one litre of distilled water.
b. K: A standard solution of 1000 ppm is prepared by dissolving 1.9070 g KCl or 2.5869 g KNO_3 in one litre of distilled water.
c. Ca: A standard solution of 1000 ppm is prepared by dissolving 2.497 g $CaCO_3$ in approx 300 ml glass distilled water and adding 10 ml conc. HCl in one litre of distilled water.
d. Li: A standard solution of 2000 ppm is prepared by dissolving 4.945 g Li_2CO_3 in approx 300 ml glass distilled water and adding 15 ml conc. HCl in one litre of distilled water.

(H) Atomic Absorption Spectrometer (AAS)
Determination of Fe, Cu, Mn and Cr of PW was conducted in the instrument Atomic Absorption Spectrophotometer (AAS). Prior to the determination of the amount present, the AAS equipment was calibrated and analyzed using blank solution of 2% Nitric acid (HNO_3) solution and standard solutions of 1ppm, 2ppm, 3ppm and 4ppm of Fe solution for

ISSN (Online) 2456-1290

International Journal of Engineering Research in Mechanical and Civil Engineering (IJERMCE)
Vol 3, Issue 2, February 2018

Fe test. Similarly for Cr, Mn and Cu 1ppm, 2ppm, 3ppm and 4ppm of Cr, Mn and Cu standards samples were made respectively. After calibration samples were aspirated, the results were obtained.

(I) Inductively coupled plasma optical emission spectrometry (ICP-OES)

In ICP-OES when plasma energy is given to an analysis sample from outside, the component elements are excited. When the excited atoms return to low energy position, emission rays are released and the emission rays that correspond to the photon wavelength are measured. The element type is determined based on the position of the photon rays, and the content of each element is determined based on the rays intensity. The elements such as Mg, Mo, Ni, Pb, Sr and Zn of PW were determined using this instrument.

(J) Continuous Cross Flow Cell

The Continuous Cross Flow Cell consist of a heavy duty dual piston reciprocating pump which was used for driving the solution of PW from the feed tank to the membrane test rig. The membrane of 14.5 × 5.5 cm dimension was fitted in the membrane test rig. Two bypass lines were provided in suction. The cross flow rate was regulated by valves, attached to the bypass line and measured using rotameter. The treated water comes from the permeate line.

IV. RESULTS

(A) Results of water content

Table 3 – Values of Water content

Sl. No.	Sample ID	Geologic formation/ depth (m)	Reservoir pressure (kg/cm^2)	Reservoir temperature (^0C)	Water cut i.e. water in crude oil (%)
1	Sample 1	Barail (3200)	240	87	90
2	Sample 2	Barail (3100)	200	88	82
3	Sample 3	Barail (3200)	240	87	94
4	Sample 4	Barail (3250)	190	85	92
5	Sample 5	Barail (3100)	200	88	84

The oil fields of Upper Assam Basin produces crude oil with high water cuts of more than 80% [6]. From the above results we can easily evaluate that the oil fields of Upper Assam Basin are producing a huge amount of water along with crude oil. This shows that the oil fields of this basin are aged. Therefore we should find some effective Enhanced Oil Recovery (EOR) method so that we could extract the residual oil left in the wells. As the water produced with oil cannot be directly disposed in the environment, so these water has to be treated properly to meet the standards imposed by the pollution control boards of both the centre and state. The physical, chemical and biological parameters were evaluated and gradually the PW has been treated in the Continuous cross flow cell for both Microfiltration (MF) and Ultrafiltration (UF).

MF and UF utilize high pressure across the membranes to accomplish filtration of contaminants from the PW. These technologies are the most common techniques of water purification. MF and UF are based on the principle of rejection of species higher than the pore size of the membrane under pressure.

(B) Results of Physical Analysis

(i) Results of Water Analyser

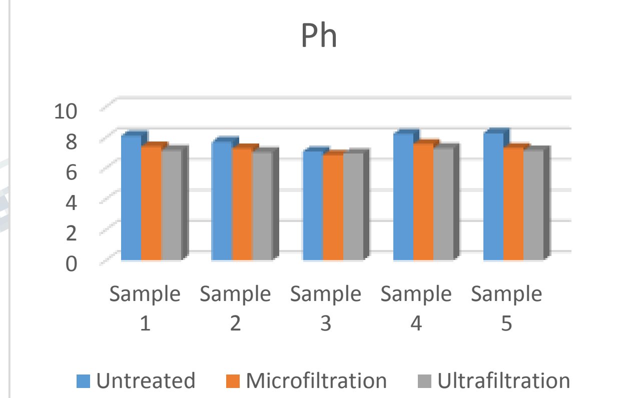

Fig 1: Comparison of pH after stages of membrane treatment

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ISSN (Online) 2456-1290

International Journal of Engineering Research in Mechanical and Civil Engineering (IJERMCE)

Vol 3, Issue 2, February 2018

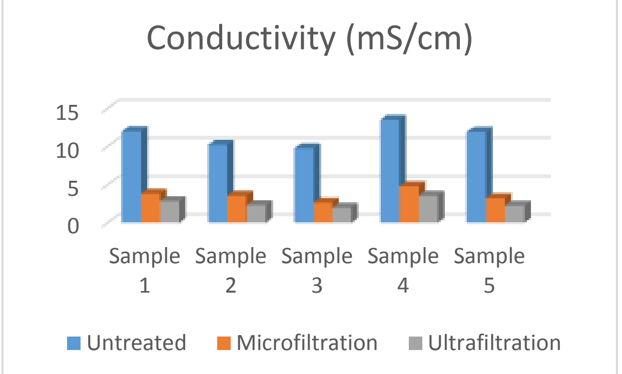

Fig 2: Comparison of Conductivity after stages of membrane treatment

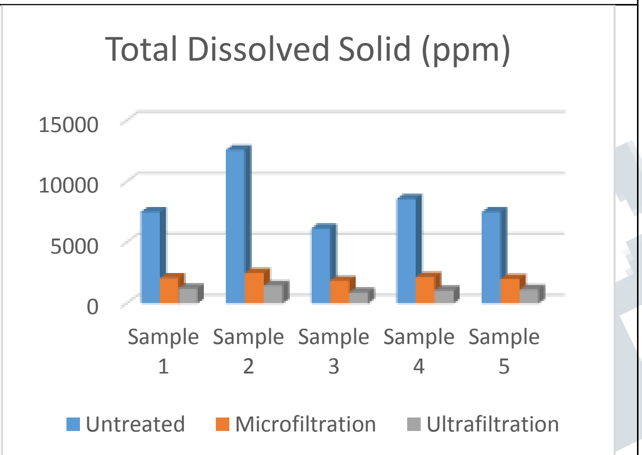

Fig 3: Comparison of Total Dissolved Solid after stages of membrane treatment

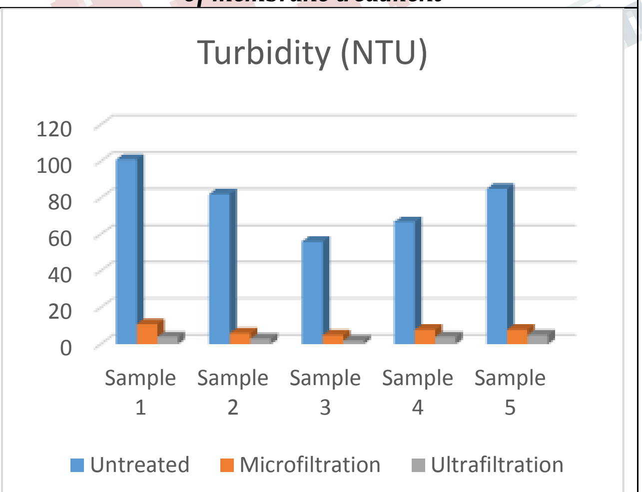

Fig 4: Comparison of Turbidity after stages of membrane treatment

The chemical and biological properties of PW are affected due to a high value of pH where we can see the alkaline nature in three cases [6]. But after treatment the values proceeds towards normal. The carbon dioxide/bio-carbonate/carbonate governs the pH of PW [6]. The conductivity of PW is well above that of pure water whose conductivity is 0.55 mS/cm due to the presence of ions in it [6]. One of the most important part of water treatment system is the removal of dissolved solid, salts or impurities. TDS gives the sum of all ion particles which are smaller than 2 micron and ranges from <2000 ppm to >150,000 ppm [8]. But after treatment all the values are less than 2100 ppm which is below the range set by the pollution control board of India. The TDS accounts for organic solutes such as hydrocarbons and sodium, calcium, chloride and bicarbonates [8]. The partial removal of TDS can be done by ion exchange and precipitation methods but for complete removal of all dissolved solids from water can be done through filtration processes like reverse osmosis. This process is not intended to be used for wastewater treatment, but to provide drinking water from seawater in desalinization plants as of its high costs. Conductivity is a process to measure the capacity of water to pass electrical flow which is related to the concentration of ions in the water. The conductive ions basically come from dissolved salts and inorganic materials such as alkalis, chlorides, sulphides and carbonate compounds. With the higher presence of ions the conductivity of ions becomes higher which is being measured at 25^{0}C. The turbidity measurements are useful to determine the optimum amount of coagulants to treat industrial wastewaters. Also it is used to evaluate the performance of water treatment plants.

(ii) Results of Millipore Assembly

ISSN (Online) 2456-1290

International Journal of Engineering Research in Mechanical and Civil Engineering (IJERMCE)
Vol 3, Issue 2, February 2018

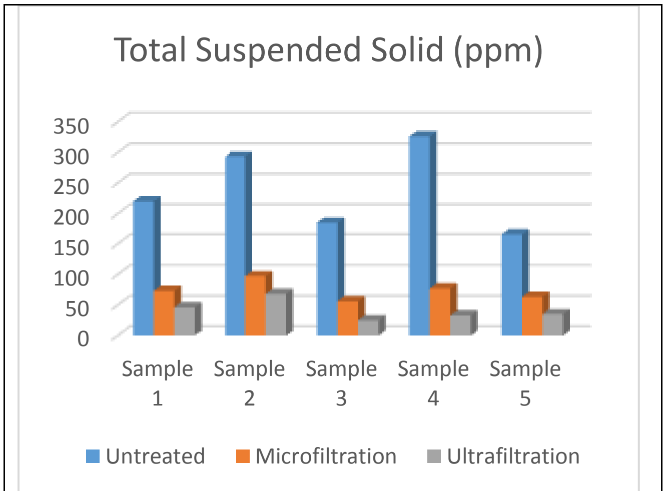

Fig 5: Comparison of Total Suspended Solids after stages of membrane treatment

The concentration and composition of suspended solids are fundamental indicators of water quality. In a water flood process possibility exists that these suspended solids will cause the injection wells to become impaired. Therefore, selection of specific process or combined processes for removal of suspended solids from water depends on the character of the solids, their concentration and the required filtrate clarity. The main physical processes of formation damage in water injection wells can result from plugging by solids present in injection water.

(C) Results of Chemical Analysis

(i) Results of Water Analyser

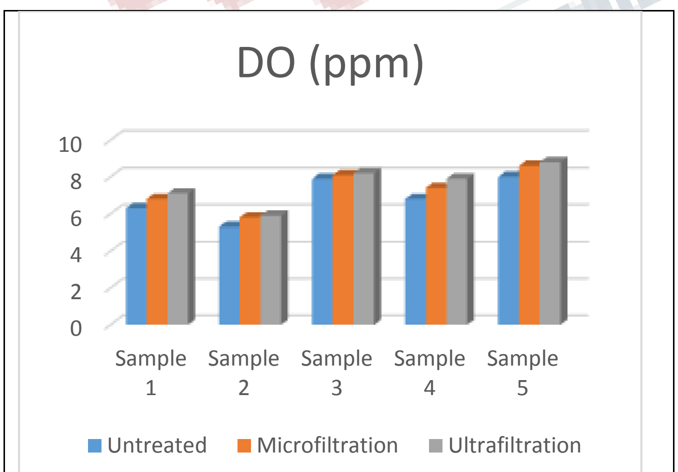

Fig 6: Comparison of Dissolved Oxygen after stages of membrane treatment

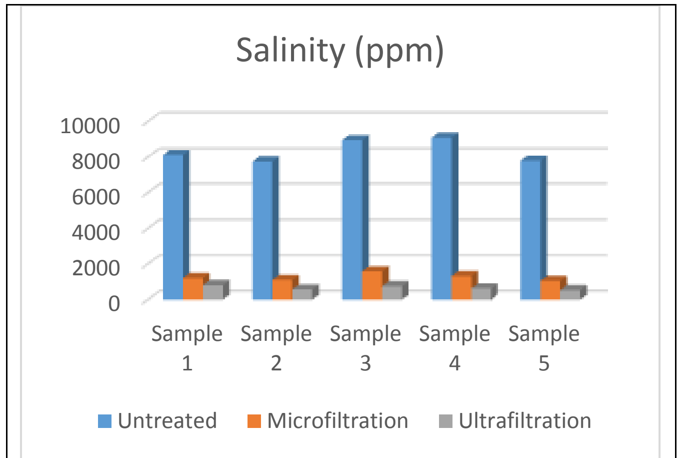

Fig 7: Comparison of Salinity after stages of membrane treatment

The PW is highly saline which is very rich in dissolved minerals [6]. Salinity is the total concentration of all dissolved salts in water whose range is from 7000 – 9000 ppm. But after treatment the values comes well below 1000 ppm. The major contributing ions for salinity are sodium, magnesium, calcium, potassium, chloride, sulphate, bicarbonate and bromine. DO analysis measures the amount of gaseous oxygen dissolved in an aqueous solution. The diffusion process leads to entry of oxygen into the water from the surrounding air. Higher level of total dissolved solid gas concentration can be harmful to aquatic life [10]. But, adequate DO is necessary for good water quality.

(ii) Results of Titration Methods

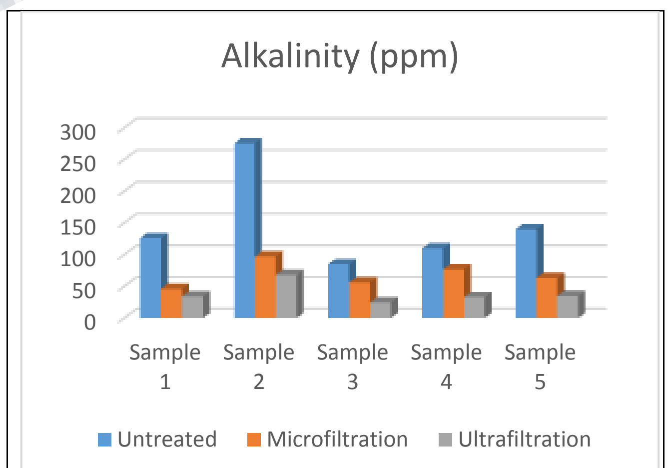

Fig 8: Comparison of Alkalinity after stages of membrane treatment

ISSN (Online) 2456-1290

International Journal of Engineering Research in Mechanical and Civil Engineering (IJERMCE)
Vol 3, Issue 2, February 2018

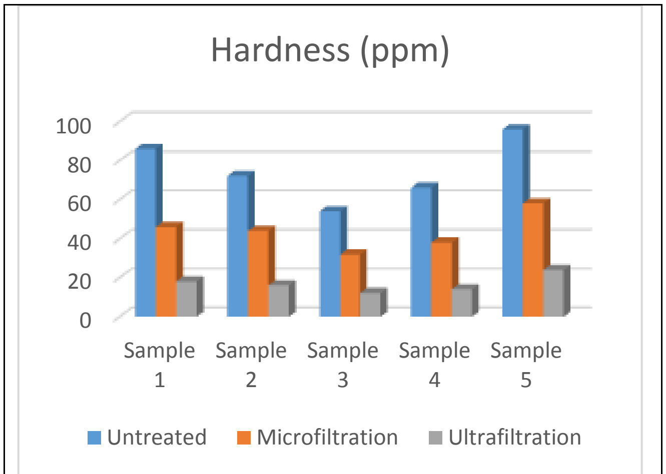

Fig 9: Comparison of Hardness after stages of membrane treatment

The sum of total cation present in water give us the hardness which is perhaps due to minerals coming from underground reservoir and drilling activities [6]. It is predominantly caused by divalent cation such as Calcium, Magnesium and others (e.g. aluminium, barium, iron, manganese, strontium and zinc). The dissolved polyvalent metallic ions from sedimentary rocks, seepage and runoff from soils are the principal sources of hardness in water [9]. Alkalinity is primarily due to hydroxide, carbonate and bicarbonate contents.

(iii) Results of Oil and Grease (O&G)

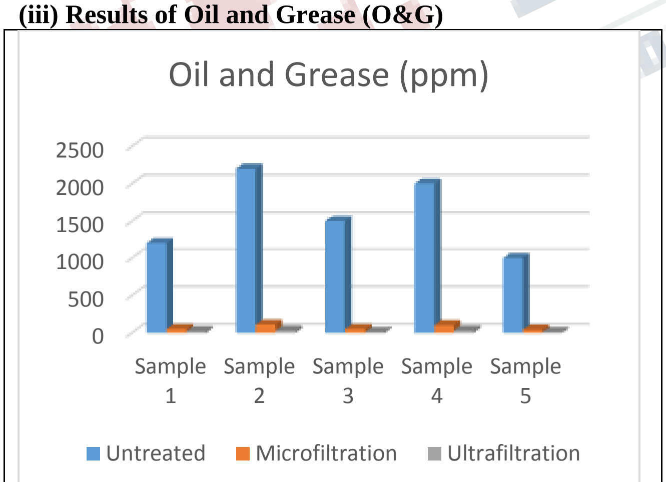

Fig 10: Comparison of Oil & Grease after stages of membrane treatment

The hydrocarbons as oil and grease is present in PW in the form of free oil, dispersed oil and emulsified oil. The disposal limit of O&G is 10 ppm but the values of raw PW is very high, even after treatment by microfiltration and ultrafiltration all the values are not within range. Therefore further filtration has to be done to bring it within range. The separation of two immiscible liquids is governed by Stokes law which gives the rate of fall of a small sphere through a viscous fluid. Stokes law states that when this sphere is under the influence of gravity it attains a constant velocity, which is given by the stokes equation

$$Vt = \frac{\{(\rho - \rho o)gdo2\}}{18\mu}$$

where,
V_t – Terminal velocity of the drop
ρ – Water density
ρ_o – Oil density
g – Gravitational acceleration
d_o – oil drop diameter
μ - water viscosity

(D) Results of Biological Analysis
(i) Results from BOD Incubator

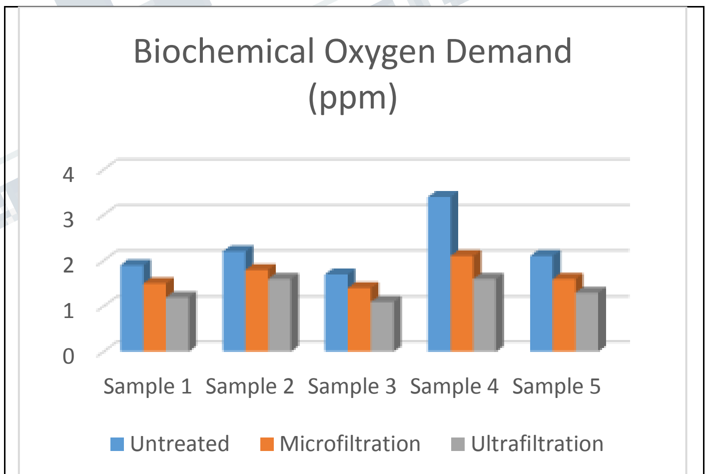

Fig 11: Comparison of Biochemical Oxygen Demand after stages of membrane treatment

It is a measure of the quantity of oxygen used by microorganisms in the oxidation of organic matter. When there are high levels of contaminants in PW then the microorganisms consume more oxygen to decompose the organic matter, and hence it decreases the amount of oxygen required by other aquatic organisms to live.

International Journal of Engineering Research in Mechanical and Civil Engineering (IJERMCE)
Vol 3, Issue 2, February 2018

(E) Results of Inorganics

(i) Results of Flame Photometer (FP)

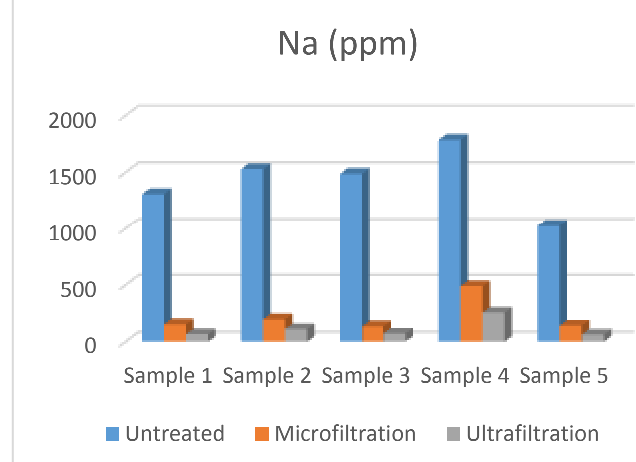

Fig 11: Comparison of Sodium (Na) after stages of membrane treatment

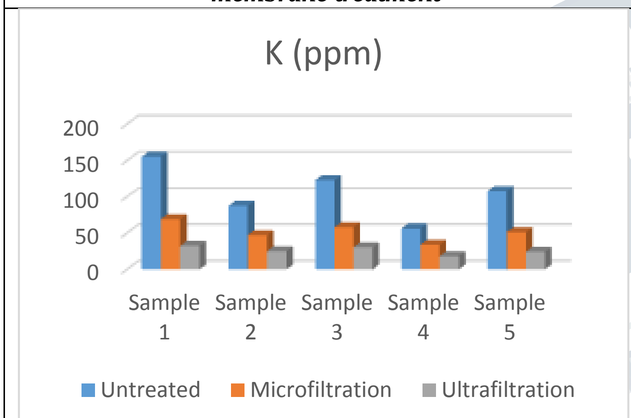

Fig 12: Comparison of Potassium (K) after stages of membrane treatment

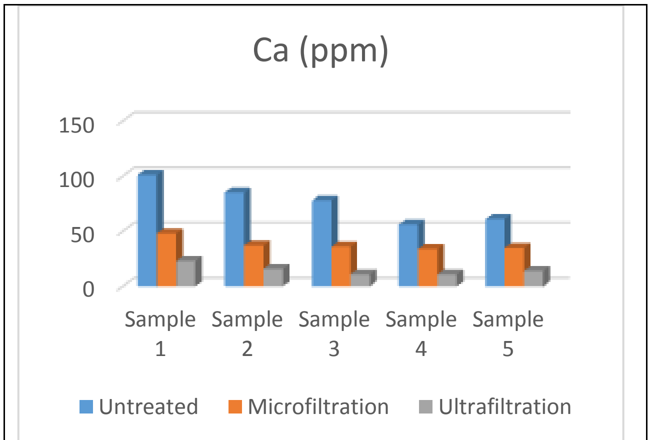

Fig 13: Comparison of Calcium (Ca) after stages of membrane treatment

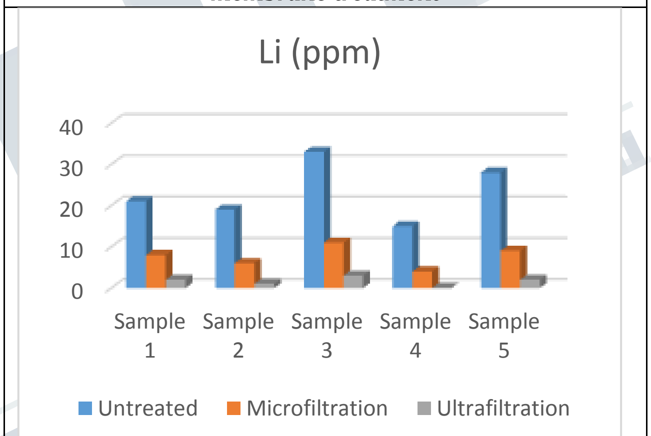

Fig 14: Comparison of Lithium (Li) after stages of membrane treatment

High levels of salts present in PW adversely affect organic removal efficiency and sludge settle ability during the treatment process. Also it cannot be disposed in the environment, which will increase the salinity of the water where it is being disposed [11]. The elements Na, K and Ca are naturally occurring elements which is present in natural water from the weathering of various rocks. But these quantities increase in PW due to contamination of hydrocarbons. Therefore it has to be treated to bring down the parameters within its permissible limits.

(ii) Results of Atomic Absorption Spectrometer (AAS)

ISSN (Online) 2456-1290

International Journal of Engineering Research in Mechanical and Civil Engineering (IJERMCE)
Vol 3, Issue 2, February 2018

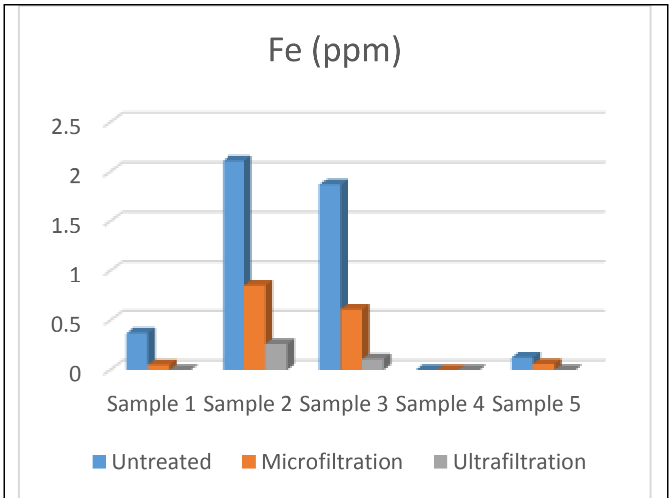

Fig 15: Comparison of Iron (Fe) after stages of membrane treatment

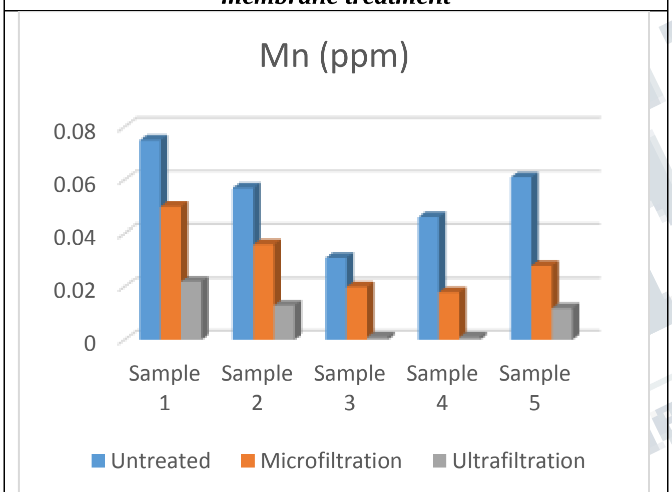

Fig 16: Comparison of Manganese (Mn) after stages of membrane treatment

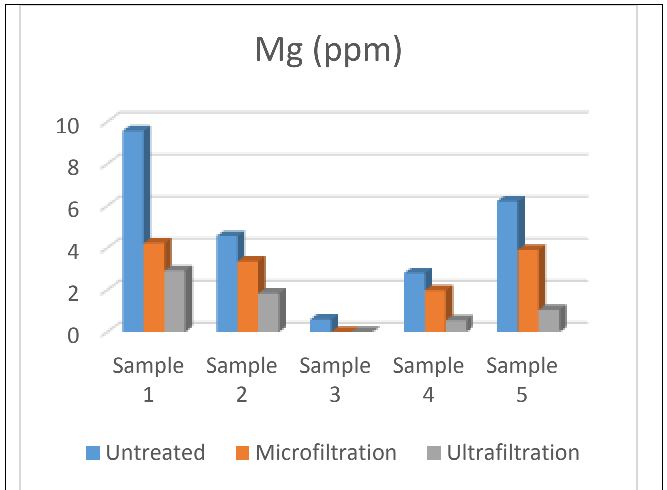

Fig 17: Comparison of Magnesium (Mg) after stages of membrane treatment

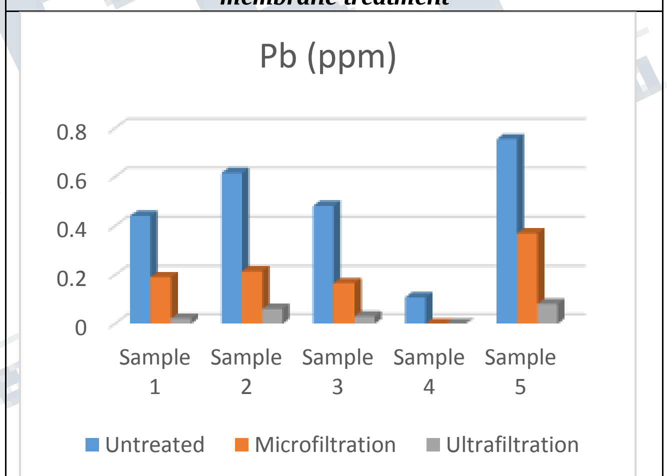

Fig 18: Comparison of Lead (Pb) after stages of membrane treatment

Natural water contains variable amounts of iron but in ground water it is normally present in the ferrous or bivalent from (Fe^{++}). The quality varies depending upon the geological area and it is required both by plants and animals [10]. High levels of Fe and Mn in PW can promote the growth of bacteria which obtains its energy from the chemical reaction that occurs when Fe and Mn mixed with DO. It form thick slime growths on the walls of the pipe which finally mixes with the PW. The reduced levels of Fe after treatment determines that the Fe is fully oxidized.

(iii) Results of Inductively Coupled Plasma Optical Emission Spectrophotometer (ICP - OES)

ISSN (Online) 2456-1290

International Journal of Engineering Research in Mechanical and Civil Engineering (IJERMCE)
Vol 3, Issue 2, February 2018

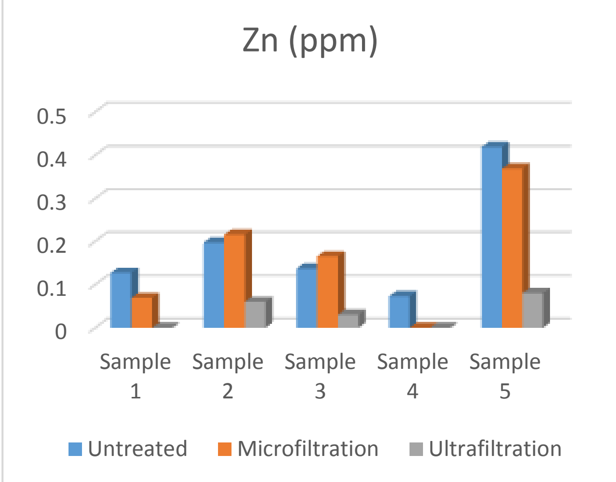

Fig 19: Comparison of Zinc (Zn) after stages of membrane treatment

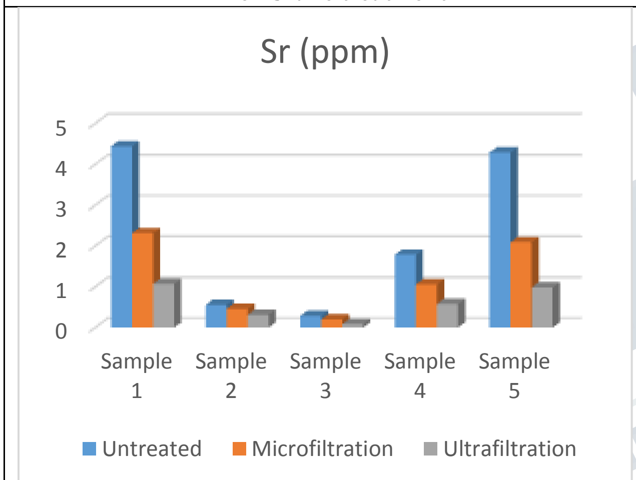

Fig 20: Comparison of Strontium (Sr) after stages of membrane treatment

The heavy metals are directly related with environmental pollution and biological toxicity problems [12]. The metallic elements are human carcinogens which may induced multiple organ damage even at lower levels of exposure [13].

V. CONCLUSION

In any case if the effluent is discharged into environment without treatment, this will cause pollution, affecting the salinity, BOD, COD and many more of the water sources into which they may be discharged on contaminated land. The effluents reduce DO concentration and formation of sludge deposits leading to environmental disturbances that may damage aquatic biota. Also injection of effluent into formation may decrease its injectivity due to plugging and choking of pores if suspended solids and oil are not removed before injection. Also improved EOR techniques are being used extensively involving polymers, surfactants and various gases that will adversely affect the interfacial properties of the produced waste waters making the resolution of emulsions more difficult in future. In some cases, significant treatment of produced water is required to meet the quality required for beneficial uses such as irrigation, farmland restoration, cattle and animal consumption, and drinking water for private use or in public water systems [7]. To prevent scaling and water contamination from PW removal of bacteria, viruses, microorganisms, algae, etc. is very necessary. Microorganisms occur naturally in the PW or may be added during de-oiling treatments and to remove it advanced filtration techniques are one of the effective technologies. UV light treatment, chorine or iodine reaction, ozone treatment and pH reduction are other treatments available to disinfect produced water [8].

Table 4 – Produced water treatment performance

Sl. No.	Constituents	Range	CPCB specification	Overall removal (%)
1	pH	6.85 – 8.27	6.5 – 8.5	17.17
2	Conductivity (ppm)	1.3 – 8.65	200	84.97
3	TDS (ppm)	880 – 12600	2100	93.02
4	DO (ppm)	5.3 – 8.8	10	39.77
5	Salinity (ppm)	530 – 9100	600	94.18
6	Turbidity (NTU)	2 – 101	10	98.02
7	O&G (ppm)	10 – 2200	10	99.54
8	TSS (ppm)	25 – 326	100	92.33
9	Alkanity (ppm)	25 – 275	600	90.91
10	Hardness (ppm)	12 – 96	600	87.5
11	BOD (ppm)	1.1 – 3.4	30	67.65
12	Na (ppm)	63 – 1785	100	96.47
13	K (ppm)	18 – 155	20	88.38
14	Ca (ppm)	11 – 101	200	89.1
15	Li (ppm)	1 – 33	2	96.96
16	Fe (ppm)	0 – 2.11	1	100
17	Mn (ppm)	0.001 –	0.05	98.66

		0.075		
18	Mg (ppm)	0 – 9.57	100	100
19	Pb (ppm)	0 – 0.755	0.1	100
20	Zn (ppm)	0 – 0.419	5	100
21	Sr (ppm)	0.081 – 4.441	0.1	98.17

VI. ACKNOWLEDGEMENT

We would like to acknowledge

1. Indo-Tunisia Project no. DST/INT/TUNISIA/P-02/2017 entitled **"Characterisation of oil field water to mitigate a major environmental hazard with recovery of usable water".**
2. Indo-US Project F. No. 194-1/2009(IC) dated February, 2015 entitled **"Foam-assisted CO_2 Flooding for the depleted Reservoirs of Upper Assam Basin and in Candidate Reservoirs in Louisiana"** of 21st century knowledge initiative program under University Grants Commission.
3. DBT Twinning Project No. BT/485/NE/TBP/2013 entitled "**Remediation of produced water from NE oil field by microbial and membrane intervention with modeling of an effluent unit for the recovery of usable water".**
4. Dibrugarh University for the laboratory facilities

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International Journal of Engineering Research in Mechanical and Civil Engineering (IJERMCE)
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