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Experimental Study of Produced Water Treatment Using Activated Carbon with Aluminum Oxide Nanoparticles, Nanofiltration and Reverse Osmosis Membranes

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ABSTRACT
This work inspected the produced water discharged from the Amara oil field in (Misan-Iraq) to improve the quality of water before reuse and reinjection or disposal. The process of treatment included a pretreatment step using activated carbon and post-treatment using flat polymeric nanofiltration membrane (NF) (1.0 nm) and reverse osmosis membrane (RO) (0.3 nm), respectively. Therefore, activated carbon without aluminum oxide (Al_2O_3) nanoparticles and with (Al_2O_3) nanoparticles (20 nm) was used to examine the removal efficiency of the total organic compound (TOC). The height of the fixed bed of activated carbon and its diameter were 35 cm and 2.5 cm, respectively. The volumetric flow rates of the produced water flowing through the activated carbon column were taken as (25, 20, 15, 10 and 5)×10^{-4} m^3/h respectively, at transmembrane pressure (TMP) of 1.0 bar, pH equals 6, and the temperature of 25 °C. The TOC removal efficiencies attained using activated carbon without Al_2O_3 nanoparticles were (52, 64, 77, 83 and 87%), respectively, and (65, 72.7, 83.4, 92.5 and 95.2%) with the use of Al_2O_3 nanoparticles, respectively. Produced water effluent from the activated carbon column was treated by flat NF and RO membranes to reduce the total dissolved solids (TDS). The cross-flow rates through NF and RO membranes were 0.1 and 0.25 m^3/h, TMP (1–12 bar) and 60 bar, respectively. The removal efficiency of TDS was enhanced up to 40% and 99.67%, respectively. In addition, the TOC removal efficiency was 100% in the effluent of the RO membrane.

Keywords: produced water, wastewater treatment, nanofiltration membrane, reverse osmosis membrane, TOC and TDS removal, reuse.

INTRODUCTION

The production of petroleum is the most significant activity; it has been the main source of income and energy for several countries (AlJaberi et al. 2020a; Oliveira et al. 2005). However, the petroleum and petrochemical activities discharge huge amounts of produced water which is increased long the lifetime of the reservoir (AlJaberi et al. 2020b). The source of freshwater begins to minimize; therefore, alternative sources of water, such as the produced water, have been considered that could be regained for purposes of industries and instantly minimize freshwater withdrawals (AlJaberi et al. 2020c). This aim will minimize the amount of wastewater discharged to the environment, thus, reducing the risk of aquatic systems and soil. The produced water contains numerous contaminants such as suspended solids, dissolved, total dissolved solids, volatile organic compounds, dispersed oil, heavy metals, and bacteria (Zsirai et al. 2016; Jepsen et al. 2019). Both characteristics and volume of produced water highly depend on the reservoir's lifetime, hydrocarbon amount, and geological characteristics (Ebrahimi et al. 2009; Igunnu and Chen 2012). The large volume and its characteristics are challenging for handling the produced water to reuse for different beneficial purposes (Horner et al. 2011). Thereby, considerable fractions of contaminants should be rejected to meet the quality standards of freshwater (Li and Lee 2009).

A considerable volume of oily water has been reused for enhancing oil recovery by injecting water into formation wells (Graham et al. 2015). Moreover, the reinjection process is essential for disposal purposes. The main targets of both near-zero discharging and saving freshwater usage are required along with minimizing waste disposal. Thereby, different treatment techniques like precipitation, reverse osmosis, adsorption, ion exchange, membrane filtration, oxidation and biosorption process, electrochemical treatments, evaporation, and filtration are extensively used (AlJaberi 2020d; Feini et al. 2008; AlJaberi et al. 2019). Several materials have been used to remove contaminants from oily wastewater, such as bentonite, activated carbon, and deposited carbon (Okiel et al. 2011). These materials suffer from some disadvantages which impact their performance in wastewater treatment; therefore, they should be enhanced using other materials such as nanoparticles to be used efficiently in wastewater treatment. The particles are diagnosed at the nano-scale level, which participated extensively in the modification, production, and shaping of structures to use in several environmental applications (Bhattacharya et al. 2013). There are several kinds of nano-scale metal oxides, such as aluminum oxide, titanium oxide, ferrous oxide, and iron oxides. They are effective for eliminating contaminants from wastewater (Lu et al. 2006; Deliyanni et al. 2003; Alardhi et al. 2020).

The conventional methods used in wastewater treatment suffer from some drawbacks where providing the requirements that are complicated to be met with the traditional technologies (Gardner 1972; Huang et al. 2009; Lee 2000). The membrane separation process is an effective and flexible treatment technique possessing several advantages, such as the wide application, energy saving, minimal influence of the quality of feed water on the outlet permeate quality, and no chemicals required (Gardner 1972; Pan et al. 2012; Cui et al. 2008). Reverse osmosis (RO) is one of the membrane technologies that is employed to eliminate numerous contaminants from saline and wastewater such as COD, BOD, salinity, and TOC (Malaeb and Ayoub 2011). The diffusion mechanism is the main parameter that caused the mass transfer in the RO process. Other parameters affecting this process are the size and charge exclusion, the physical-chemical interactions between solvent, solute, and the RO membrane (Malaeb and Ayoub 2011). The efficiency of the RO process is controlled by several conditions involving the operating variables, membrane, and influent water specifications. Flat sheet and spiral-wound are the common available RO membrane designs with the integrally asymmetric structure of thin-film composite to cellulose acetate, composite membranes (polyamide-based) to other composite membranes (sulfonated polysulfone) (Bilstad 1997). The strength and valence of the membrane charge are controlled by the functional groups of the polymer structure. In turn the roughness, charging, and membrane hydrophobicity control the capacity of adsorption of dissolved species (Malaeb and Ayoub 2011; Lee et al. 2011; Bulut et al. 2008; Crini et al. 2007).

The purification of polluted water and desalination have been performed worldwide to provide humanity with usable water. For four decades, systems of filtration equipped with nano-porous membranes have been widely employed in several industrial activities (Yamjala et al. 2016; Hansen et al. 2018; Lively and Sholl 2017). Purification of wastewater using membrane processes proceeds depending on the particle size of the retained species. Types of membrane processes are categorized depending on the size of the pollutants particles into (Moslehyani et al. 2019; Zhao and Yu 2015):

- microfiltration (MIF) – retaining bacteria, and suspended species,
- ultrafiltration (UF) – retaining viruses, bacteria, and suspended species,
- nano-filtration (NF) – retaining multivalent ions, viruses, bacteria, and suspended species,
- reverse osmosis membrane (RO) – retaining monovalent ions, multivalent ions, viruses, bacteria, and suspended species.

This work aimed to investigate the ability of the use of a hybrid system including activated carbon as pretreatment, with and without nanoparticles, as well as the post-treatment process involving a flat polymeric (NF) membrane and (RO) membrane, respectively, to enhance the quality of produced water discharged from Amara oil field located in (Misan-South of Iraq) then to reuse.

EXPERIMENTAL WORK

Chemicals

The produced water was collected from the Amara oil field located in Misan, Iraq. The

Table 1. The chemical specification of the produced water released from Amara oil field

Parameters	TOC (mg/L)	Conductivity (ms/cm)	TDS (g/L)	pH
Value	280	220	137.5	6

chemical specification of this water is shown in Table 1.

Other chemicals used in the present work are explained as follows:

- activated carbon – characteristics of activated carbon are (particle density 0.85 g/ml, bulk density 0.54 g/ml, pore volume 0.8 ml/g and surface area 1200 m^2/g) which is purchased from a local market,
- aluminum oxide – alumina, gamma-Al_2O_3, 99.9%, 20 nm, Skyspring Nanomaterials, inc. 2935 west hollow Dr. Houston, TX77082,
- NF membrane – the filtration rig contains a flat NF membrane type (GE Osmonics TF (Thin Film) DK membrane (190×140 mm), PN: YMDK5P19O5, is supplied by GE Osmonics, USA (dimension of 190×140 mm),
- RO membrane – the RO membrane-type GE, Pore size 0.3 mm, Composite Polyamide (Polymer) – TFC, pH 1–11, is supplied by GE Osmonics, USA (dimension of 190×140 mm).

Instruments

The following details contain the instruments used in the present treatment of produced water:

- column – glass column contains an activated carbon bed (diameter of 2.5 cm and length of 50 cm),
- pump – centrifuge type (Model: MO35ASGSN-SCA, Sterliltech Co., USA); operating pressure range (0–69 bar),
- feed container – QVF glass vessels with a volume of 5 liters,
- flow meter – two rotameters type (Gemu Gebe Muler, Germany). The range of flow rate (0–18) L/min,
- flow meter – FKB model (Dropnweg, Deursen-Dennenburg, Netherlads). The range of flow rate (6.9–69) mL/min,
- pressure gauges – three pressure gauges, type (WIKA, range 0–25 bar, Germany),
- piping, fittings, and valves – pining and different fittings of reinforced (PVC), and four Ball valves (St.st), were installed at the inlet and the outlet of the activated carbon column, the outlet of the NF filtration cell, and at the recycle line of the process,
- measuring TOC – total organic compounds analyzer type (LPG408-99.00012, USA),
- conductivity and total dissolved solids (TDS) – content of TDS and conductivity in produced water samples were measured using a bench meter (Inolab Cond 7110, WTW, Germany),
- digital balance – type (AZ214: Sartorius weighing technology, Germany),
- a cell of NF and RO membranes (see Figures 1 and 2) – the specification of NF and RO membranes cells are shown in Table 2.

Apparatus

Figure 3 shows the schematic of the experimental setup for contaminants removal from produced water by adsorption technology using fixed bed activated carbon column (ACC) in produced water effluent. Then, the membrane treatment of TOC and TDS pollutants was conducted using the NF membrane. Furthermore, the bench-scale of flat plate RO membrane (Figure 4) was used for more reduction of TOC and TDS concentrations.

Granular activated carbon was used as an adsorbent alone or with five grams of nanomaterial (Al_2O_3) that were mixed by shaking well inside a

Table 2. Specifications of NF and RO system

Specification	Value
Number of cells	1
Cell type	Sepa CF, 316 stainless steel, 1000 psi rated
Effective membrane area	(140 cm^2) 24 in^2
Membrane sample size	(19 × 14 cm) 7.5 × 5.5 in
Operating pressure range	(0–69 Bar) 0 – 1000 PSI
Feed flow rate	(1.8 LPM) 1.8 GPM max

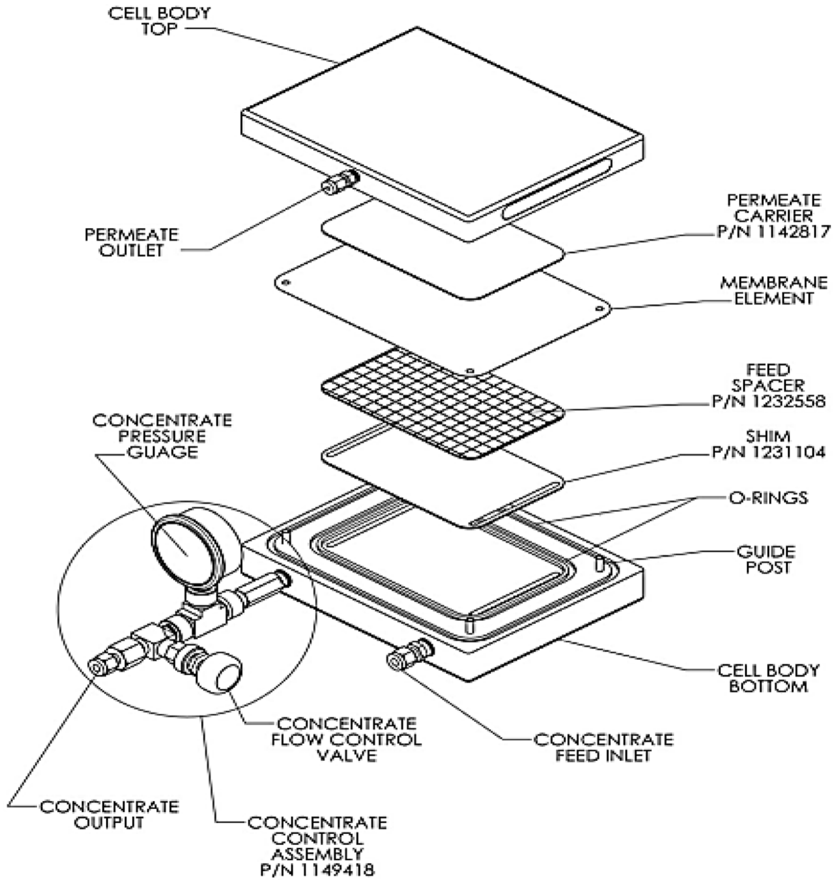

Figure 1. Typical cell body assembly (adopted from Sepa CF cell-manual-sterliech corporation)

Figure 2. NF and RO membranes cell

sealed glass container to allow the nanoparticles to penetrate well into the pores of the activated carbon. They are employed as the adsorbent in the (ACC) for the adsorption process and it was supported in the column using fiberglass. The aluminum oxide nanoparticles have a high potential for treating water pollutants due to their unique properties, low concentration, and large surface areas. Therefore it has the potential to improve decontamination efficiency and water purification.

The filtration rig worked at different flow rates of produced wastewater (25×10^{-4}, 20×10^{-4}, 15×10^{-4}, 10×10^{-4} and 5×10^{-4}) m^3/h, respectively, and applied TMP of 1.0 bar for organic compounds adsorption experiments using a fixed bed of activated carbon. It must ensure that the valves of input, output, and recycling of the tubular module adsorption column are opened, and the valve of the flat module feed line is closed. Before running the experiment, the activated carbon was wetted by passing distilled water through the ACC to improve the activated carbon wetting characteristics, which are essential for the high interfacial area issue. The distilled water was allowed to be drained, and the feed of produced water was allowed to flow through the fixed bed of the tubular column. Different feeding flow rates were conducted by regulating the input, the output, and the recycle valves.

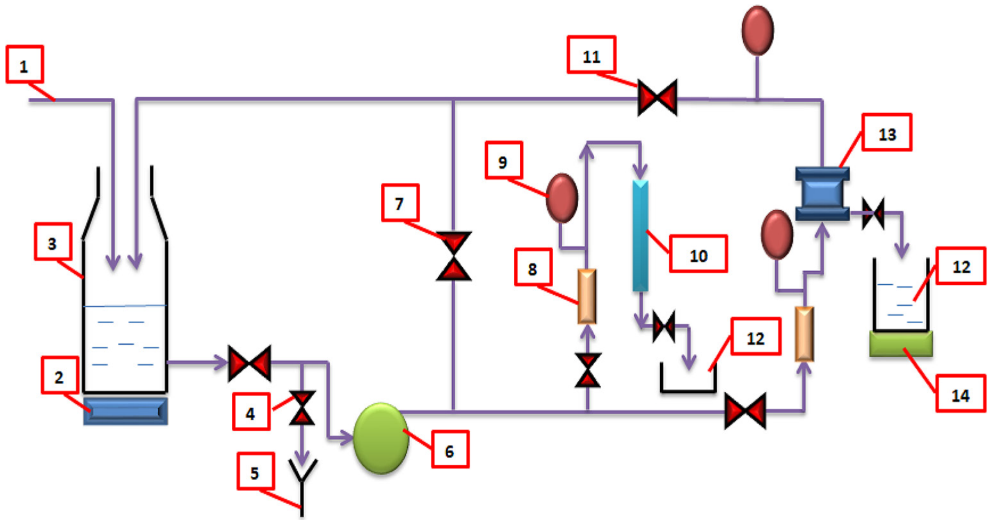

Figure 3. Schematic of the integrated treatment system for the removal of 280 mg TOC/L and 137.5 g TDS/L from oilfield produced water treatment: (1) produced water influent, (2) magnetic stirrer, (3) reservoir, (4) sampling port, (5) drain, (6) centrifugal pump, (7) by-pass valve, (8) flow meter, (9) pressure gauge, (10) adsorption column (acc), (11) back pressure valve, (12) permeate storage, (13) nf membrane module, (14) electronic balance

Figure 4. Bench scale of flat plate RO membrane filtration system

After that, the input valve of the tubular module was closed, while the input and the output valves of the NF membrane were kept open. The effluent of the produced water from the ACC was collected and placed in the reservoir and allowed to flow through the flat module of the NF feed line with a feed volumetric flow rate of 0.1 m^3/h and TMP (1–12 bar). The permeate of the activated carbon column and NF membrane were collected as well as the conductivity and TDS measurements were carried out. Then, the permeate of NF membrane was treated by RO membrane at flow rate 0.25 m^3/h and TMP 60 bar. The permeate of the RO membrane was collected, and the estimation of TDS and TOC concentrations was measured. The removal ratio of contaminants is determined using Eq. 1 (Labban et al. 2017).

$$R\% = \left(1 - \frac{C_{ip}}{C_{if}}\right) \times 100 \quad (1)$$

where: C_{ip} and C_{if} – the concentration of the contaminant (i) in the permeate and in the feed (g/m^3), respectively.

Moreover, the transmembrane pressure (TMP) is evaluated using Eq. 2 (Avula et al. 2009; Sarkar et al. 2009).

$$TMP = \left(\frac{P_{inlet}+P_{outlet}}{2}\right) \quad (2)$$

where: TMP – the transmembrane pressure, $P_{inlet.}$ – inlet pressure (bar), P_{outlet} – outlet pressure (bar).

RESULTS AND DISCUSSION

Effect of flow rate on TOC removal

The oily wastewater was treated using a tubular column of fixed bed-activated carbon. Figure 5 shows a comparison of the TOC removal efficiency using activated carbon alone and with nanoparticles Al_2O_3, respectively. As observed in Figure 5, the increase of TOC removal (52%, 64%, 77%, 83%, and 87%) was obtained with decreasing flow rate (25×10^{-4}, 20×10^{-4}, 15×10^{-4}, 10×10^{-4} and 5×10^{-4}) m^3/h, respectively, by using fixed bed activated carbon without nanoparticles (Al_2O_3). In turn, the TOC removal is improved attaining (65%, 72.7%, 83.4%, 92.5%, and 95.2%) respectively when a fixed bed of activated carbon-containing nanoparticles (Al_2O_3) is performed. This behavior can be explained by the increase of the residence time in the fixed bed column due to the use of nanoparticles with activated carbon, which increases the surface area and improves the physical as well as chemical properties of activated carbon and, consequently, the TOC removal efficiencies have enhanced.

The mathematical relations between the response of TOC removal efficiency and flow rate (FR) of produced water for both cases of with and without the use of nanoparticles Al_2O_3 are shown in Eq. 3 and Eq. 4, respectively, as follows:

$$\text{TOC removal (\%)} = -16040\ (FR) + 105.82 \quad (R^2 = 0.972) \quad (3)$$

$$\text{TOC removal (\%)} = -17800\ (FR) + 99.30 \quad (R^2 = 0.951) \quad (4)$$

Effect of TMP on conductivity and TDS in the permeate

Table 3 as well as Figures 6 and 7 show the decreasing of the conductivity and TDS with the increase of TMP that may occur due to the increases of the pure water permeate. The removal efficiencies of TDS and TOC after using RO membrane achieved 99.67% and 100%, respectively, at a flow rate of 0.25 m^3/h, pH 6, and a temperature of 25 °C. The conductivity response related to the applied transmembrane pressure (TMP) as shown in Eq. 5:

$$\text{Conductivity (ms/cm)} = -6.955\ (\text{Applied TMP}) + 199.31 \quad (R^2 = 0.906) \quad (5)$$

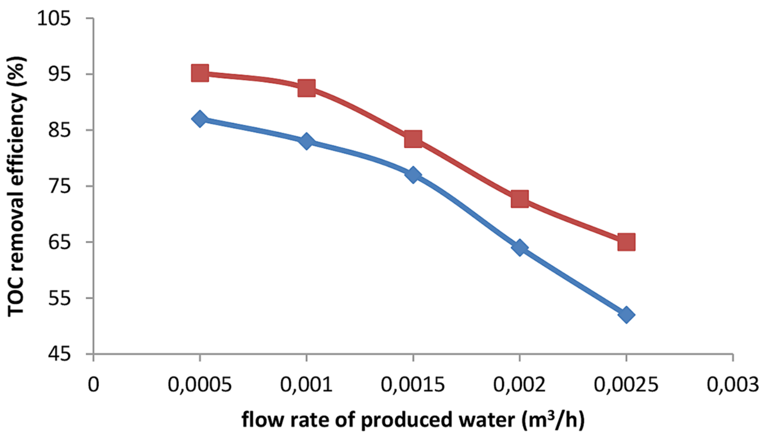

Figure 5. Effect of flow rate on TOC removal

Table 3. Conductivity and TDS results at flow rate 0.1 m^3/h., transmembrane pressure 1–12 bar and temperature of 25 °C

TMP bar	1	2	4	6	8	10	12
Conductivity (ms/cm)	203.7	190	160	146	141	130.4	125
(TDS g/l)	127.3	118.66	98.83	90.75	87.5	85.3	82.5
Removal %	7.42	13.7	28.2	34	35.8	38	40

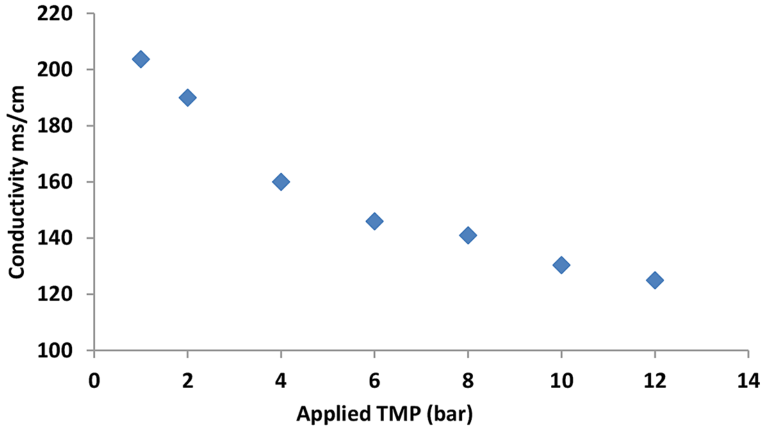

Figure 6. Conductivity of NF membrane permeate versus TMP (1–12) bar at cross flow rate 0.1 m^3/h, pH (6) and temperature 25 °C

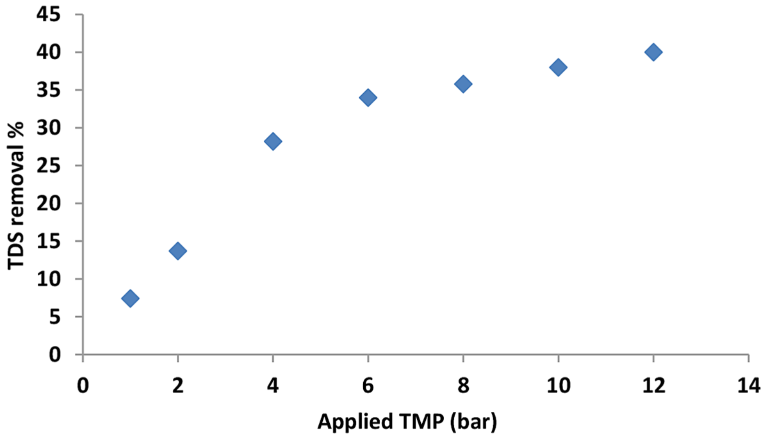

Figure 7. TDS of NF membrane permeate versus TMP (1-12) bar at cross flow rate 0.1 m^3/h., pH (6) and temperature 25 °C

Eq. 6 relates the response of TDS removal to the applied transmembrane pressure (TMP) with significant regression coefficient as follows:

$$\text{TDS removal (\%)} = 2.847\ (\text{Applied TMP}) + 10.671 \quad (R^2 = 0.844) \qquad (6)$$

Effect of pH on TOC removal

The capacity of adsorption for activated carbon depends not only on its properties, such as distribution of pore size, surface area, and other physical properties but also on its surface chemical nature. The change of pH value in the adsorption system could lead to the chemical properties transformation of the activated carbon surface and the adsorbate form; therefore, pH impacts the performance of the adsorption process. The experiments were conducted by changing pH from 3 to 10 with a fixed TOC concentration of 280 mg/L, the volumetric flow rate of (5×10^{-4} m^3/h). The pH value affects the adsorptive uptake of the adsorbate molecule due to its impact on the adsorbent molecule (ionization/ dissociation).

Figure 8 shows the variation of TOC removal at different values of solution pH where the highest removal efficiency of TOC was achieved at pH equal to 3. This behavior may be occur due to the activated surfaces of carbon obtained (Ghouma et al. 2015) and it is hydrophobic. At a low pH value, the growth of the (H^+) ions in the solution will lead the activated carbon system to be a positive charge by absorbing. When the surface of activated carbon is positively charged, a notably strong attraction appears between anionic TOC molecule and activated carbon surface of positively charged leading to highest adsorption of TOC. Thus, the number of negatively charged sites increases along with the pH value, and the number of the positively charged sites decreases.

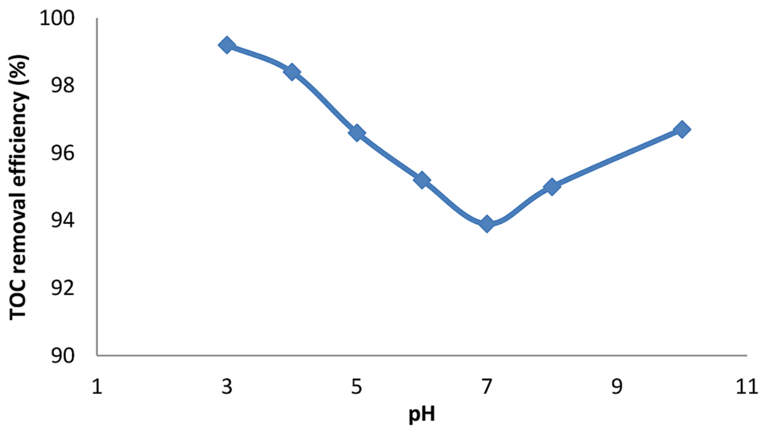

Figure 8. Effect of pH value on the TOC removal using activated carbon with nanoparticles (Al_2O_3) at flow rate 5×10^{-4} m^3/h

Due to electrostatic repulsion, the surface of negatively charged activated carbon does not favor the adsorption of anionic TOC molecules'. Moreover, minimizing adsorption of the TOC in alkaline medium is also because the contest the anionic TOC molecule with excess OH^- ions for the adsorption sites (Hassan et al. 2020; Maghrabi et al. 2020).

The range of TOC removal was increased in the alkaline medium as the pH increased more than 8. The removal behavior could be explained as the pH value is greater than or equal to 8, the formation of small amounts of coagulants of TOC was observed. This participation occurred in the range of TOC removal at pH 8. It also observed that the TOC of produced water was precipitated when the pH value increased larger than 10.

CONCLUSIONS

In this study, the removal behavior of (fixed bed activated carbon) and the integrated system of (NF and RO) membranes have been investigated using conditions of fixed bed activated carbon with and without Al2O3 nanoparticles, nanofiltration membrane NF of 1.0 nm, and RO membrane of 0.3 nm. The study proved the efficient performance of the present systems for the removal of TOC and TDS from produced water. Fixed bed column contains activated carbon with nanoparticles Al_2O_3 as one of the first efficient steps for TOC removal. The use of the combination of (NF and RO) membranes leads to the effective removal of TDS and TOC contaminants. The TOC removal attained 87% and 95.2% when activated carbon treatment without and with Al_2O_3 nanoparticles are used, respectively. For NF and RO membrane treatment, TOC removal achieved 99.3% and 100%, respectively, at pH equal to 6, while TDS removal was maximized to 40% and 99.67%, respectively, for one cycle.

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