

Treatment of wastewater by a combined technique of adsorption, electrocoagulation followed by membrane separation

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ABSTRACT

In this study, a combined lab-scale purification system was set up to treat wastewater from the National Iranian Oil Company. The combined system was composed of three main sections: pre-filtration using activated carbon filter (ACF), electrocoagulation (EC) system, and a filtration section (MF followed by RO). The performance of the treatment system was evaluated by measuring heavy metals, BOD, COD, TDS, TSS, and O&G. The results showed that prefiltration using ACF could lead to the removal of the BOD, COD, TDS, TSS, and O&G by 24.6%, 21.12%, 31.07%, 36.9% and 8.49%, respectively; the heavy metals were removed significantly. In the EC section, heavy metals were rejected by more than 98% using both the Al and Fe electrode, except for the Cr ions that were mostly removed with Fe electrodes. The removal of BOD, COD, TDS, TSS, and O&G using the AI and Fe electrode was 95.6%, 96%, 91%, 76.6%, and 98.6% and 93.2%, 92.1%, 76%, 83% and 99%, respectively. EC followed by MF/RO filtration led to a remarkable purification performance, and the rejection rate of all pollutants was obtained over 99% after this section. The experimental results indicated that the optimum time for ACF and EC processes were 20 and 50 minutes.

1. Introduction

In oil and gas reservoirs, oil and water are usually found together [1]. Also, the reservoir pressure decreases after a while; hence, enhanced oil recovery (EOR) methods are used by injecting a large amount of water. These two reasons lead to the production of large amounts of water along with the oil during oil production, which is called produced water (PW) [2]. PW is a combination of several organic and inorganic compounds whose amounts can be different in various reservoirs [1]. Generally, PW can contain different amounts of oil residues, sand or mud, naturally occurring radioactive materials, chemicals from fluids used in EOR, bacteria, and dissolved organic compounds [3]. It is estimated that in 2009 more than 70 million barrels of PW were produced per year [4]. Discharging a large volume of untreated PW into the environment can lead to dire environmental issues such as soil and freshwater contamination [5]. Therefore, the treatment of PW is necessary before discharging it into the environment. Based

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on the kind of pollutants and their concentrations in PW, several treatment methods are available. Using a single method usually does not achieve a significant effect on water purification [6]. So a combination of physical, chemical, and biological treatment processes can lead to achieving proper treatment targets [5]. Generally, the process of PW purification has three stages: pre-treatment, main treatment, and final treatment [7]. To enhance the efficiency of PW treatment, a combination of various purification systems can be significantly effective [6]. Several purification methods can be used in a suitable topology to increase the treated water quality in combined treatment systems. Membrane filtration is one of the most common methods used in PW treatment due to its low cost, simplicity, and high efficiency [8]. Membrane treatment is classified into three main categories based on membrane pore size, i.e., microfiltration, ultrafiltration, and nano-filtration. Reverse osmosis is also one of the most prevalent membrane filtration methods used for removing salts and water salinity, as well as total hardness [9]. Besides filtration methods, electrochemical technologies can be used to enhance treatment efficiency. Electrocoagulation (EC) is an electrochemistry method that has a successful history in wastewater treatment [10]. However, the use of this method in PW treatment is rare [7]. EC can be effective in PW purification due to the reduction of chemicals requirements; also, it has a high-efficiency removal of oil, heavy metal ions, and petroleum contaminants [11,12]. The goal of this study is to investigate a combined purification system for the treatment of PW from the Iranian Oil Company. Four heavy metal ions (Cu²⁺, Cr²⁺, Ni²⁺, and Zn²⁺) were added to the PW to evaluate the performance of the proposed treatment system. The purification system consisted of a pretreatment section using activated carbon filtration (ACF), EC as the main treatment stage, a filtration section including microfiltration (MF), and reverse osmosis (RO) as the final treatment method. Each section plays a specific role in this system, and samples are taken after each step to evaluate the effectiveness of each purification method. Although the mentioned methods have been well studied individually, a few studies have focused on the simultaneous use of pre-treatment and

advanced treatment. Hence, this study focuses on the development of a novel three-step method for the treatment of PW on a lab-scale. Also, the simultaneous removal of heavy metals, TDS, TSS, BOD, COD, and O&G is studied in this paper, which has rarely been studied before.

2. Material and methods

2.1. The characteristics of PW

In this study, Iranian oil company wastewater was used as PW. To evaluate the performance of the proposed treatment system, four heavy metal ions $(Cu^{2+}, Cr^{2+}, Ni^{2+}, and Zn^{2+})$ were added to the PW by the dissolution of Zinc nitrate hexahydrate (\geq 98.0 %), nickel(II) chloride (\geq 98.0 %), potassium dichromate (\geq 99.0 %), and copper(II) nitrate (\geq 99.0 %), which were purchased from Sigma-Aldrich. Table 1 shows the characteristics of the PW samples.

Table 1. The characteristics of the PW sample.

Factors	Initial value (mg/l)
O&G	15.2
TSS	275.8
TDS	8526.4
COD	231
BOD ₅	1200.25
рН	7.47
Cu ²⁺	30.86
Cr ²⁺	0.35
Ni ²⁺	2.76
Zn ²⁺	18.9

2.2. Pre-treatment

In combined treatment systems, pre-treatment can improve the performance of subsequent processes. Pre-treatments should have high performance, no need for special equipment, and low cost [13]. Before EC, the pre-treatment methods were considered to reduce pollutants, which could lead to higher purification efficiency in the main treatment stage [14]. In this research, ACF was used before the EC process to reduce the concentration of pollutants before entering EC. The characteristics of activated carbon used in this study are presented in Table 2.

 Table 2. Characteristics of activated carbon.

Туре	Granular Activated Carbon
Brand	Jacobi
Average size	1.5 mm
Surface area	900 m²/g
Bed porosity	0.35
Flow rate	2.84 l/min
Operating temperature	38 °C
Operating pressure	8.6 bar
Module dimension	Diameter=15 cm,
	length=30 cm

2.3. Electrocoagulation process

EC is a well-known method for removing total suspended solids (TSS), heavy metals, emulsified oils, and other contaminants [10]. The EC process performance can be affected by some factors such as the chemistry of the aqueous medium, pH, chemical particle size, and constituent concentrations [15]. The choice of electrode materials is also one of the main factors that can affect performance and treatment efficiency [16]. Iron and aluminum are the most widely used materials for the electrode in EC due to their low price and higher efficiency in pollutant removal [17]. The dimensions of the electrodes used in this study were 12×4×0.2 cm, and the distance between the electrodes was 3 cm. The mechanism of EC is based on the electrode types listed below.

For the aluminum electrode [18]:

 $AI \rightarrow AI3+ (aq) + 3e$ (At the anode) (1)

 $3H2O + 3e \rightarrow 3/2 H2$ (g) + 3OH - (At the cathode) (2)

Al3+ (aq) + 3H2O (aq) \rightarrow Al (OH) 3 + 3H + (aq) (In the solution :) (3)

For Iron electrode [19]:

Fe (s) \longrightarrow Fe²⁺ (aq)+2e⁻ (At the anode) (4)

$$Fe^{2+}(aq)+2OH^{-}(aq) \longrightarrow Fe(OH)_{2}(s)$$
 (5)

(At the anode)

 $2H_2O(I)+2e^{-} \rightarrow H_2(g)+2OH^{-}(aq)$ (6)

(At the cathode)

 $4Fe^{2+} (aq)+10H_2O(L)+Q_2(g) \longrightarrow$ Fe(OH)₃+8H⁺ (aq) (Overall) (7)

2.4. Filtration section

In this study, the filtration section consists of two parts: MF and RO. MF is a kind of membrane filtration uses a driving force such as pressure and concentration gradients. MF is capable of removing colloids, proteins, bacteria, pyrogens, and other organic molecules larger than 0.1 in size [20]. MF has a successful background in many areas such as chemical and pharmaceutical processes, wastewater treatment, and food industries [21, 22]. Table 3 shows the properties of MF filters.

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Brand	C.C.K
Model number	SC-10-1 and SC-10-5
Material	Polypropylene fiber
Pore size	1µ and 5µ
Maximum temperature	52°C
Service life	2500 GAL

RO is a membrane purification process for water and wastewater used to remove a variety of organic and inorganic pollutants such as biochemical oxygen demand (BOD), chemical oxygen demand (COD), total organic carbon (TOC), total suspended solids (TSS), oil and grease (O&G), etc. [23]. RO is used in different applications, including semiconductors, food processing, power generation, pharmaceuticals, desalination, biotechnology, coproduced water from oil and gas production, textile, pulp and paper, mine and diary wastewater, process and boiler water, tanneries, and beverage industry [24]. The main limitation of RO systems is membrane fouling due to the pore-clogging or adsorption of solutes on the membrane surface [25]. Investigation shows that using filtration followed by RO can prevent membrane fouling, leading to increased water treatment performance [26]. Table 4 illustrates the properties of the RO filter.

Table 4. The properties of F	२० filter
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Brand	DOW Filmtec
Model number	SW30-2521
Duration	300 GAL/day
Maximum pressure drop	15 psi
Туре	Polyamide Thin-Film
	Composite

2.5. Experimental setup

An experimental combined purification system was set up based on Figure 1. This system was composed of three main sections: pre-filtration using activated carbon, EC system, and filtration section (MF followed by RO).



Fig. 1. A schematic of the proposed hybrid system: a) ACF b) EC c) Filtration sections.

PUMP

The pre-filtration section of the experimental setup consisted of ACF, which was located before the EC section. Two modules with a diameter of 15 cm and a length of 30 cm were filled with granular activated carbon and placed in series. The EC container was constructed from thick plastic with a dimension of $40 \times 25 \times 25$ cm. The tank consisted of two parts, namely the upper part and the lower part. These two parts were connected by a pipe with a radius of 4.5 cm. Figure 2 shows the combined treatment system. The PW entered the lower part, and the treated water went to the upper part by the pressure of the pump (Pentax, PM45, 220V) through the connector pipe. A mesh plate was installed at the inlet of the connector pipe to prevent the sludge from entering the upper

part through the pipe. Four electrodes (two anodes and two cathodes) with a size of $12 \times 4 \times 0.2$ cm were located 2 cm apart at the lower part of the tank [16]. Iron (Carbon steel, ST37, Aria Industrial Group, Iran) and aluminum (5754 aluminum alloy, AlumMetal, Iran) were used as electrodes in this section. The electrodes were connected to a 12V power supply, and the current intensity varied from 15mA to 30mA. The membrane filtration section was placed after EC and included two membrane filters with a pore size of 1µm and 5µm, which were used for MF followed by a RO membrane filter.



Fig. 2. The designed hybrid treatment system.

2.6. Process description

In this study, the treatment of PW was carried out in three different batch processes. First, PW entered the ACFs using a pump (DOW RO booster pump, 100GPD, 24VDC) from the storage tank and circulated in this cycle for 60 minutes. The ACFs have an input and an output. In this step, one sample was taken every 10 minutes from the output stream of the second ACF. Pre-treated wastewater was stored and used as the EC feed flow. The EC feed entered into the lower part of the EC container. The EC reactions were carried out based on the Eqs. 1 to 6 in this step. The hydrostatic pressure created by the pump (Pentax, PM45, 220V) caused the wastewater to go the upper part through the connector pipe. EC has an input and an output. The output stream was recycled to the tank to increase the EC performance. This cycle took 60 minutes, and one sample was taken every 10 minutes from the output stream of the EC container. The experiments were performed using Al and Fe electrodes. One sample was taken every 10 minutes in the EC process. After the EC tests

were completed, the treated wastewater was collected in an open tank with a size of $50 \times 50 \times 100$ cm and left for 24 hours to let the contaminants settle. The temperature was kept below 10° C to prevent the evaporation of the pollutants. After that, the treated wastewater was directed to the filtration section using a pump (DOW RO booster pump, 100GPD, 24VDC). The MF filters had one input, one output, and a flow rate of 1 lit/min. The RO filter had one input and two outputs: permeated and concentrated. The permeated stream with a flow rate of 0.2 lit/min was stored for analysis. In the filtration, section samples were taken after MF and RO.

2.7. Measurements

The BOD and COD measurements were performed using a standard incubation method over five days and an oxidation method using potassium dichromate. O&G was measured using a partition gravimetric method. In this method, hexane was used as the extracting liquid. TDS measurements were carried out using a TDS meter (JENWAY 4510 bench conductivity meter, UK). TSS was measured by filtering samples, drying the filter and captured solids, and then weighing the filter to determine the weight of the captured suspended solids in the sample. The concentrations of metal ions were determined using atomic adsorption (Varian Spectra A 250 Plus). The metal ions concentrations were measured by a Perkin Elmer Inductively Coupled Plasma-Optical Emission Spectrometer (Optima 200 DV).

3. Results and discussion

3.1. Performance of pre-filtration

Figure 3 shows the heavy metal removal efficiency using ACF. As can be seen from Figure 3, heavy metals are eliminated significantly using ACF. The removal of Cu^{2+} , Cr^{2+} , Ni^{2+} , and Zn^{2+} are 74.3%, 78.85%, 83%, and 44%. The results are in good agreement with the literature. Baby et al. reported that depending on the adsorbent and contact time, the removal rate of chromium and zinc using the activated carbon of the kernel shell of palm oil could be around 85% and 70%, respectively [27]. Aboli et al. showed that nickel biosorption from aqueous solution onto activated carbon prepared from citrus limetta leaves was strongly dependent on the pH; the amount of adsorbent and its removal percentage could reach 90% [28]. Poultry litter-based activated carbon for removing Cu²⁺ ions in water was investigated by Guo et al.; they revealed that the Cu²⁺ removal on activated carbon from an aqueous phase could reach 73.2 [29]. The highest amount of heavy metal removal efficiency occurred during the first two minutes. Although the removal of heavy metals increased over time, the reduction was small after 20 minutes. Since the removal of contaminants had not reached a steady-state condition after 60 minutes, it could be concluded that the ACFs did not reached the saturated state. And if the contact time increased, it was possible to increase the removal of contaminants. Because EC was considered after pre-filtration, it was not necessary to entirely remove the contamination in this section. Hence, it can be concluded that the suitable time for the prefiltration step is 20 minutes. Although the main purpose of ACF is to reduce heavy metals to increase EC performance, pre-filtration can also be effective in rejecting other contaminants. Figure 4 illustrates the pollutant removal of wastewater using ACF. It is clear that the ACF effectively eliminates the contamination so that the BOD, COD, TDS, TSS, and O&G removal is 24.6%, 21.2%, 31.07%, 36.9%, and 8.94%, respectively. Adsorption capacities of activated carbon for the pollutant were calculated based on Eq. 8, where C_i is the initial concentration, Ce is the final concentration, V is the volumetric flow rate (=1 lit/min), m is the mass of activated carbon (=500g), and g is the amount of each pollutant adsorbed onto the adsorbent. The results of the adsorption capacity of activated carbon are presented in Table 5. The results are consistent with other publications. Devi et al. reported that using activated carbon, BOD, and COD could remove more than 99% [30]. Mortula et al. showed that the TDS could be eliminated by about 50% using activated carbon [31]. Sia et al. used activated carbon to remove TSS from palm oil mill effluent; their results showed that this method removed 39% of TSS [32]. Okiel et al. also showed that activated carbon had great potential to remove oil from wastewater [33].

$$q = \frac{(C_i - C_e) \times V}{m} \tag{8}$$



Fig. 3. Heavy metal removal efficiency using AC.





3.2. Performance of EC

3.2.1. The effect of current density

The contamination removal in EC can pertain to the destabilization mechanism that includes three main steps: compression of the double layer, charge neutralization, and floc formation [34]. The current density is one of the most important parameters in the EC reactor that can affect treatment performance. The pre-filtered

wastewater was used as EC feed flow. The effect of the different current densities of 15, 20, 25, and 30 mA/cm² at a fixed voltage of 12 V on heavy metal removal using AI and Fe electrodes are shown in Figures 5 and 6, respectively. As observed in Figures 5 and 6, a high removal rate was obtained using both electrodes by increasing the current density. And the final concentration value of the heavy metals was notably decreased. However, it could be said that the removal of heavy metals using aluminum electrodes was more affected by changes in current density; therefore, changing the current density from 15 to 30 mA/cm2, the rejection of Cu^{2+} , Cr^{2+} , Ni^{2+} , and Zn^{2+} increased by 17.48%, 38.76%, 33.18% and 5.63% using Al electrodes. And by applying the iron electrodes, the changes of these materials increased by 2.4%, 10.89%, 1.21%, and 0.03%, respectively. So, it can be said that compared to Fe electrodes, Al electrodes require more energy to remove heavy metals effectively.

Table 5. The capacity of activated carbon used in thisstudy.



Fig. 5. The effect of the different current densities on the removal of heavy metal using Al electrodes.



Fig. 6. The effect of the different current densities on the removal of heavy metal using Fe electrodes.

Changing the current density also affects the removal of other pollutants. Figures 7 and 8 depict the effect of current density on the pollutant elimination of the wastewater using Al and Fe electrodes, respectively. The comparison of Figures 7 and 8 reveals that at a low current density using the Al electrode, the rejection of contamination is higher than using the Fe electrode. At a high current density, the Al electrode was more effective in removing BOD, COD, and TDS, while applying the Fe electrode led to the better rejection of TSS and O&G. The electrode weight loss was calculated by determining the weight of the electrodes before and after the experiments. Figure 9 shows the weight loss percentages of the electrodes by changing the current density. As can be seen, the consumption of Fe is more than Al in the same current density. Because Fe has a higher atomic weight and the number of electrons exchanged during the EC process, it is lower than Al [35].



Fig. 7. The effect of current density on the pollutant elimination of the wastewater using Al electrodes.



Fig. 8. The effect of current density on the pollutant elimination of the wastewater using Al electrodes.



Fig. 9. The weight loss of electrons in different current densities.

3.2.2. The effect of electrodes material

In the EC process, pollutants can be removed by various mechanisms such as adsorption, coprecipitation, oxidation, and reduction [36]. During the EC process, metal ions can be chemically adsorbed to iron hydroxide species and form mixed bimetallic hydroxides. During coprecipitation reactions, metal-OH and metal-Ometal bonds can form [37-39]. Iron (III) hydroxide (Fe (OH)₃(s)) or aluminum (III) hydroxide (AI(OH)₃(s)) precipitates should be formed to effectively eliminate metals by co-precipitation, adsorption, and sedimentation mechanisms [40]. Figure 10 depicts the reduction of the heavy metals using different electrodes during the EC process. From Figure 10, it is obvious that Cu^{2+} , Ni^{2+} , and Zn^{2+} decreased remarkably using both Al and Fe electrodes. However using the Al electrode, Cr²⁺ was not eliminated effectively. Cr⁶⁺ was formed during the EC process based on Eqs. 9 and 10 [41]. The reduction of Cr^{6+} to Cr^{3+} is a decisive step in the removal of chromium species [42] due to the formation of a solid hydroxide (Cr(OH)₃), which can be eliminated from wastewater easily [43]. Using Fe as the electrode, Fe^{2+} is generated at the anode, which can help to reduce Cr^{6+} to Cr^{3+} (Eqs. 10 and 11) [44]. So, the Fe electrode is more effective in removing Cr ions as well as Cu, Ni, and Zn ions from wastewater. Similar results were reported by Kim et al. They investigated the removal mechanism of heavy metal (Cu, Ni, Zn, and Cr) by EC using Al and Fe electrodes. The results of their study showed that the Fe electrode was more effective than the Al electrode for removing

Cr ions. They attributed this result to the reduction of Cr^{6+} by Fe^{2+} .

$$HCrO_{4}^{-} + 7H^{+} + 3e^{-} \to Cr^{3+} + 30H^{-}$$
 (9)

$$CrO_4^{2-} + 4H_2O + 3e^- \to Cr(OH)_3 + 5OH^-$$
 (10)

$$CrO_4^{2-} + 8H^+ + 3Fe^{2+} \tag{11}$$

$$\xrightarrow{} Cr^{3+} + 3Fe^{3+} + 4H_2O$$

$$Cr^{6+} + 3Fe^{2+} \rightarrow Cr^{3+} + 3Fe^{3+}$$
(12)

The EC process is also effective in rejecting other contamination in the wastewater. Figure 11 shows the removal of BOD, COD, TDS, TSS, and O&G. From Figure 11, it can be said that BOD and COD elimination have similar behavior using both Al and Fe electrodes. The higher BOD and COD removal is achieved using the Al electrode. The removal of COD and BOD by EC can be due to the removal of TDS and the precipitation of dissolved organic molecules as organometallic compounds [46]. Higher COD removal using an Al electrode arises from the dissolution of (AI (OH) 3) in water and the formation of a hydroxo complex ($[AI(OH)_n]^{(n-3)-}$) [46]. TDS and TSS were also removed significantly using both AI and Fe electrodes. The results showed that TDS removal was achieved using Al electrodes, while TSS was eliminated more effectively using Fe electrodes. O&G removal by both electrodes was observed almost identically. It could be said that the EC could break oil/water emulsions and separate the oil effectively [47]. The outcomes of Figures 10 and 11 illustrate that the proper time for the EC process is 50 minutes. Similar results were obtained by Uğurlu et al. [48]. They showed that the BOD and COD were respectively removed from mill effluents by 70% and 75% using Al electrodes and 80% and 55% using Fe electrodes in the EC process. Rusdianasari et al. also reported that the TDS and TSS of integrated wastewater could be removed by 88.96% and 50% using the Al electrode and 80.27% and 57.55% using the Fe electrode, respectively [49]. The removal of oil from an oilwater emulsion using EC was also investigated by Fouad [50]. Findings showed that EC could remove more than 98% of the oil in the effluent. The higher rejection performance of EC in this study could be due to the pre-filtration system placed before EC, which reduced the initial concentration of pollutants.





Fig. 10. Heavy metal reduction using different electrodes during EC.



Fig. 11. The pollutants removal using EC applying Al and Fe electrodes.



Fig. 11. Continued

3.3. The performance of the filtration section

As earlier stated, the filtration section is composed of MF and RO filters. The treated wastewater using the EC process was used as filtration feed flow. Samples were taken after MF and RO at a pressure of four bar. Figures 12 and 13 show the effect of MF and RO in removing pollutants, respectively. By using MF, most pollutants such as BOD, COD, TSS, and O&G are removed by 95.63%, 92.3%, 98.3%, and 99.9%, respectively, while TDS is not eliminated effectively (68.3%). On the other hand, the RO is able to reject all pollutants significantly (> 98). This is due to the smaller membrane pore size of the RO than the MF, as well as the low concentration of pollutants as a result of treatment using ACF, EC, and MF.





rising pressure leads to an increase in the driving force, and as a result, more water can pass through the membrane with a high rate of contamination rejection [51].





Fig. 14. The effect of varying pressure on the performance of MF.



Fig. 15. The effect of varying pressure on the performance of RO.

4. Conclusion

In this study, the purification of Iranian oil company wastewater was investigated. A hybrid system composed of a pre-treatment section using an activated carbon filter, EC, and filtration section including MF and RO processes was proposed. Results depict that the performance of EC was increased by applying a pre-filtration step. In the EC process, it was revealed that the Al electrode leads to rejection of Ni²⁺, BOD, COD, and TDS by 98.71%, 95.6%, 96%, and 91%, respectively. Also using Fe electrode Cr²⁺, TSS and O&G can remove by 99.86%, 83%, and 98.05%, respectively. The removal of Cu^{2+} and Zn^{2+} was the same using both electrodes and obtained as 99.8% and 99.76%. EC followed by MF/RO filtration had a great impact on the treatment performance of the hybrid system. MF and RO caused remove any contamination in the wastewater so that the rejection rate of all pollutants obtain is over 99%.

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