



## Evaluation of phenol removal from aqueous solutions by UV, RO, and UV/RO hybrid systems

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### ABSTRACT

In this paper, an efficient method for phenol removal from wastewaters by a combination of UV degradation and rejection by a low-pressure reverse osmosis membrane system has been presented. The results showed that phenol removal by the UV/RO hybrid system is more efficient than by individual UV degradation or rejection by RO system. In order to compare the efficiencies of the individual and hybrid systems, a multipurpose experimental setup was assembled, and phenol removal by UV degradation and rejection by RO and UV/RO hybrid systems was examined. In degradation of phenol by UV system, phenol solutions with constant concentration and various pH values (3, 5, 7, and 9) were examined. The results showed that the highest degradation efficiency was obtained at pH=5 using a single 6W UV system. The removal of phenol by the RO membrane at various pH (3, 5, and 7) at two different feed pressures (50, and 70 psi) and at different feed concentrations (35, 50, and 100 ppm) was examined. The removal efficiencies after 60 min for UV, RO, and UV/RO hybrid systems were obtained as 17, 20, and 58%, respectively. In addition, the results showed that an increase in feed concentration increases the removal efficiency of phenol in the UV/RO hybrid system, while the effect of pressure showed the presence of a critical pressure after which the removal is efficient. The critical pressure in the present study was obtained as 50 psi.

*Keywords:* Phenol; UV/RO; Membrane processes; Wastewater treatment; Hybrid system

### 1. Introduction

Phenol is one of the most hazardous organic pollutants in wastewaters due to its toxicity, structural stability, and resistance to degradation even at low concentrations [1]. The presence of phenol in natural waters can also lead to the formation of substituted compounds (such as chlorophenols) during disinfection and oxidation processes which are difficult to remove by conventional treatment methods such as

activated sludge digestion, and other biological methods. These compounds inhibit the growth of microorganisms in biological treatment processes because of their biotoxic and recalcitrant properties [1].

Nevertheless, phenol is a basic raw material for various products such as herbicides, drugs, paints, creams and shaving soaps, and lubricants. Phenol and its derivatives are found in the effluents and wastewaters of coal conversion processes, coke ovens, petroleum refineries, fiberglass production, textile industry, and petrochemicals [2]. Phenolic compounds can

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directly affect the health of humans through contamination of surface and ground waters, soil, and sedimentations. Due to safety and environmental issues, it is essential to remove the phenolic compounds from the wastes of various production units.

Phenol is also pertinent in the field of environmental research, because it has been chosen frequently as a representative of the pollutants and much data on its removal or destruction in wastewater treatments are available [1–4].

In this regard, several processes have been suggested such as distillation, extraction, adsorption, total chemical oxidations, biodegradation, photo-oxidation processes, and membrane separation methods [5,6].

Use of UV irradiations has been proven as one of the safest and most reliable technologies for wastewater treatment [7–10]. The basic principle for this method is the photo-degradation reaction that takes place between energy emitted by low-pressure ultraviolet lamps' light quantum, the photons, and the molecules [10]. Photo-degradation based on the free radical mechanism is initiated by the interaction between the energized photons and the organic molecules with or without the presence of catalyst. If the photocatalytic mechanism occurs on the surface of semiconductors, free radicals are generated, and hence the rate of degradation increases considerably [7]. The most common disadvantage of the photocatalytic oxidation method is the low efficiency of the current photocatalysts due to the adsorption of contaminants on the surface and blocking of the UV-activated sites. The combination of ultraviolet (UV) and the ultrasonic (US) irradiations, and utilization of UV irradiation and electrolysis with Fenton's reagent have also been investigated for degradation of phenol [11,12].

Membrane separation technologies are very useful separation systems that are used in many applications in various industries including petrochemical, water and wastewater treatment, pharmaceutical, etc. [13]. Water and wastewater treatment industry is the most important market for membranes. Currently, different membranes are used for the removal of dissolved solids, anions, cations, organic matters and pollutants, and suspended solids from various water and wastewater streams. Lower energy consumption and easy scale-up are the main advantages of the membrane technology, whereas attention must be paid to membrane fouling due to accumulation of particles and colloids from the feed stream on the surface of the membrane and limit the lifetime of the membranes [14–19]. For dilute aqueous mixtures (water and a solute), the selectivity of a membrane towards the mixture is usually expressed in terms of the solute rejection coefficient. The parameter that is shown by  $R$

is the ability of the membrane to separate the solute from the solvent. The permeation rate of flux,  $J$ , is defined as the volumetric flux rate of the product (permeated flow). Based on the solution–diffusion model, it can be assumed that both of the solvent and solute are able to be dissolved at the nonporous and homogeneous surface layers of the membrane and diffuse because of the concentration and pressure gradient across the membrane [20–24]. These membranes have excellent salt rejection but often have lower ability for rejection of many small organic molecules; therefore, their application in combination with other processes is being developed to a great extent [23].

Reverse osmosis (RO) is a pressure-driven membrane-based technique that is used to separate dissolved solids, such as ions, mostly from water-based solutions. RO membranes generally act as perm-selective barriers that allow some species such as water to permeate selectively, while other dissolved species such as ions, organic components, and dissolved solids are retained [25]. Removal of organic and toxic contaminants such as phenol by selective transport through membranes, especially RO membranes, has been found to have widespread applications [12,25–27]. Bodalo et al. studied phenol removal from aqueous solutions by using different membranes and obtained low rejections in all cases even at high pressures [28,29].

Khokhawala and Gogate [30] and Gogate et al. [31], used sono-chemical reactors for the degradation of phenol in the presence of different additives to enhance the rate of phenol degradation. Ultrasonic irradiation to organic matters in wastewaters leads to the generation of hydroxyl free radicals. They are responsible for the removal of pollutants [32]. A combination of ultraviolet UV and US irradiations for degradation of phenolic compounds has been reported [10]. Different modes of operation such as UV, US, UV/US, UV/TiO<sub>2</sub>, UV/H<sub>2</sub>O<sub>2</sub>, UV/NaCl, and UV/US/TiO<sub>2</sub> for phenol degradation were examined. Based on a new research by simultaneous utilization of UV and electrolysis by the Fenton reagent, the efficiency of the photo-electro-Fenton system for phenol removal degradation was increased considerably. Photo-electro-Fenton and sono-electro-Fenton processes showed complete degradation of 200 ppm phenol after 40 min [33].

Unlike the sonochemistry in which ultrasound waves do not directly interact with the organic molecules, the photochemistry occurs when there is an interaction between a light quantum, a photon, and a molecule. Two basic conditions must exist for the reaction to occur between photons and the organic molecule. First, the photons should have enough energy,

which is determined from its wavelength, typically lower than 700 nm. Second, as the first law of photochemistry states: “only the light which is absorbed by a molecule can be effective in producing photochemical change in the molecule.” Therefore, for a reaction to proceed, the molecule must be able to absorb the wavelength of light irradiated [10].

Fig. 1 shows the absorption of a photon by a molecule which results in the formation of an excited state. This excitation can be dissipated by four mechanisms: irradiative processes such as luminescence, radiationless processes, bimolecular deactivation, and dissociation of the molecule. Dissociation typically results in free radical formation. Utilizing light for degrading the compounds relies on dissociation and free radical formation.

In this work, phenol elimination from wastewaters by UV, RO, and UV/RO hybrid systems was investigated. The main purpose of this study was to retain the phenol molecules in the RO-rejected stream until they were degraded by UV irradiation. For this purpose, the degradation of phenol by UV irradiation was primarily studied. Then phenol rejection by a low-pressure RO system was investigated. The main purpose of these experiments was the determination of the individual systems for degradation and rejection of phenol molecules and determination of the pertinent parameters for each system. At the third step, phenol removal using a hybrid UV/RO system was performed. The effects of various parameters such as feed pressure, feed concentration, and pH, and the time on stream on phenol removal for the three systems were studied and the optimum conditions were reported.

In fact, selection of the hybrid system was based on the phenol present in the solution to be retained until it is degraded by UV irradiation. Based on our

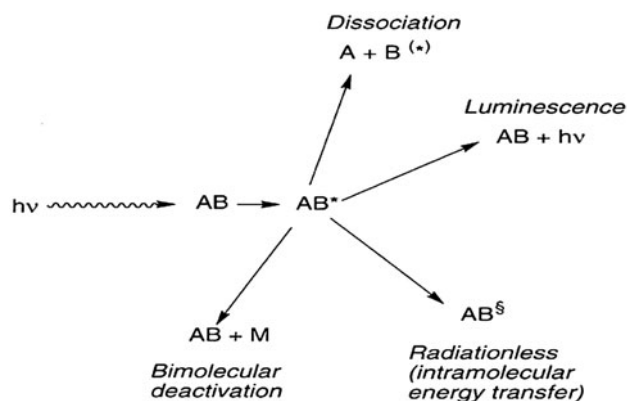


Fig. 1. The effect of photon irradiation on a molecule [10].

previous experiences, as the phenol concentration in the feed solution is increased, the concentration in the permeate stream is also increased, but the rejection percentage is initially increased and then decreased as the phenol concentration is increased. Thus, we concluded that the phenol concentration for attaining the maximum rejection should be kept at an optimized value. On the other hand, the phenol degradation rate by UV irradiation depends on phenol concentration; the more the phenol concentration, the greater the degradation rate. Then, it seems that there is an optimum point at which the maximum efficiency could be observed. By using the UV/RO hybrid system, the concentration of phenol could be kept at high levels in the rejected stream, which is a favorable condition for photolysis of phenol by UV, while in simple photolysis of phenol the concentration of phenol is reduced during the photolysis period. Thus, the average degradation rate would be lower than that at high concentrations. In addition, the RO membrane retains most of the unreacted phenols, while UV degradation prevents the development of concentration polarization on the membrane surface. Thus, it is predicted that the hybrid system gives better results than the sum of the individual systems.

## 2. Materials and methods

### 2.1. Materials

Phenol crystals (with purity of 99.5%), sodium hydroxide, hydrochloric acid, potassium ferricyanide, and ammonia were of reagent grade and purchased from Merck Co. (Germany). Chloroform (99% purity) and 4-aminoantipyrine were purchased from Dr Mojallali Laboratory Chemicals Co. and Alfa Aesar, respectively, and were used without further purification. The other chemicals that were used in this research were of analytical grade. Distilled water was used throughout.

### 2.2. Experimental setup and procedure

The experimental setup is shown in Fig. 2. A UV-C Philips lamp (CE UV101/UV1011, 6 W) was used for phenol degradation experiments. A low-pressure polyamide thin film composite RO membrane (TW30-1812-100) manufactured by Dow Filmtec Company was used for phenol rejection from the wastewater solutions. Table 1 shows the main specification of the applied RO membrane [34]. A diaphragm pump (HEADON model HF-8367) with maximum pressure of 125 psi and 1.2 L/min flow rate, pressure gages (Marsh, 0–100 psig), stainless steel diaphragm valves (Nupro,

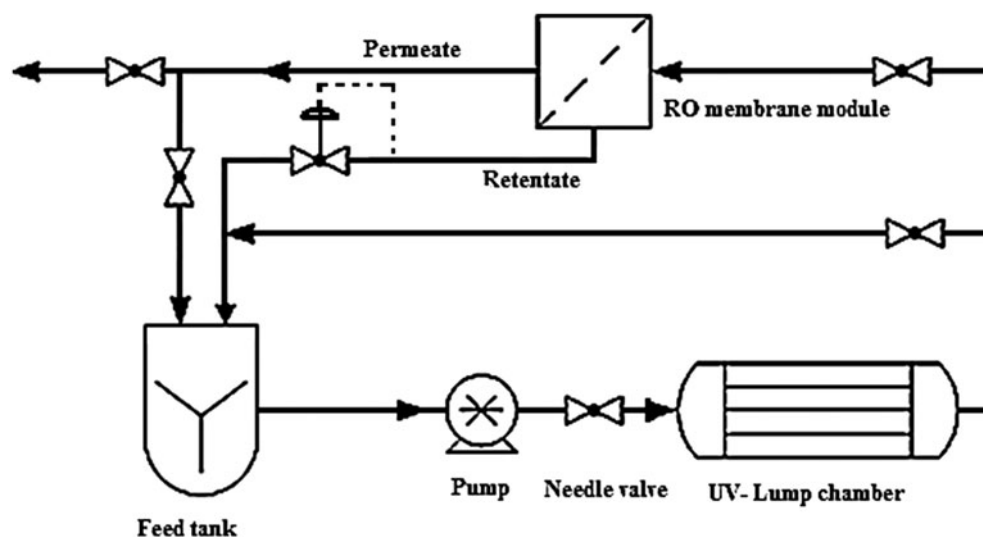


Fig. 2. A schematic view of the experimental setup for phenol degradation.

SS-4DAL) for samplings, stainless steel needle valves for flow adjustment, and back pressure regulator were the other components of the experimental setup.

pH measurements were carried out by a precision pH meter (Metrohm 780 Herisau, Switzerland). A borosilicate beaker with effective capacity of 2 dm<sup>3</sup> (2L) was used as the feed tank. The feed tank was filled with 2Ls of the feed solution with adjusted pH and concentration and the UV and RO experiments were performed in batch mode, where the products were recirculated into the feed tank. The phenol concentration in the aqueous solutions was determined by the known “sensitive 4-aminoantipyrine method” [35] spectrophotometric method at 460 nm using a Cecil CE-1010 spectrophotometer. The sensitivity of this method has been reported to be as low as 0.2 ppb [35].

The first series of the experiments were conducted for evaluation of UV irradiation for phenol degradation. The second series of experiments include the rejection of phenol by the RO system and finally phenol removal by the UV/RO hybrid system was performed in the third series of experiments. Next, the effect of initial phenol concentration, pump pressure, and time for phenol removal was analyzed by the use of the RO rejection system. Some of the experimental runs were replicated and the results showed good repeatability. The average deviation was found within  $\pm 5\%$ , and all of the experiments were done at room temperature of  $25 \pm 2^\circ\text{C}$ . The feed flow rate in all experiments was kept unchanged at 1.172 L/min to minimize the concentration polarization in the RO membrane. The design of the setup was done so as all of the experiments could be conducted in a single

Table 1

The specification of the applied RO membrane used in the experiments [34]

Specification	Type/value	Unit
Product name	TW30-1812-100	–
Manufacturer	Dow/Filmtec	–
Membrane Type	Polyamide thin-film composite	–
Diameter	1.75	in
Length	10	in
Effective membrane surface area	0.446	m <sup>2</sup>
Maximum operating temperature	113 (45)	°F (°C)
Maximum operating pressure	300 (21)	psig (bar)
Maximum flow rate	7.6	L/min
pH range, continuous operation	2–11	–
pH range, short-term cleaning (30 min)	1–13	–

unit. The change between the three modes was done by the needle valves. Phenol removal efficiency was calculated from the difference between the initial concentration and the final phenol concentration in the treated stream. The removal percentage of phenol was calculated by using Eq. (1):

$$\text{Removal (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

where  $C_t$  (ppm) is the phenol concentration at time  $t$ , and  $C_0$  (ppm) is the initial phenol concentration.

### 3. Results and discussion

#### 3.1. UV irradiation system

In the first series of experiments, direct photolysis of phenol using a 6 watt UV-C lamp was investigated at a constant feed concentration (100 ppm). Then, the effect of feed pH on phenol decomposition was evaluated after 60 min at pH = 3, 5, 7, and 9.

Fig. 3 shows the effect of feed pH on the photolysis efficiency after 60 min UV irradiation. The results showed that the maximum phenol degradation was 17% and attained at pH = 5. Based on these results, the phenol molecules in highly acidic (pH = 3) and alkaline solutions (pH = 9) exhibit resistance to degradation by UV irradiation, whereas the best result was obtained at pH = 5. In addition, the photolysis efficiency at alkaline solutions is considerably lower than that for acidic solutions.

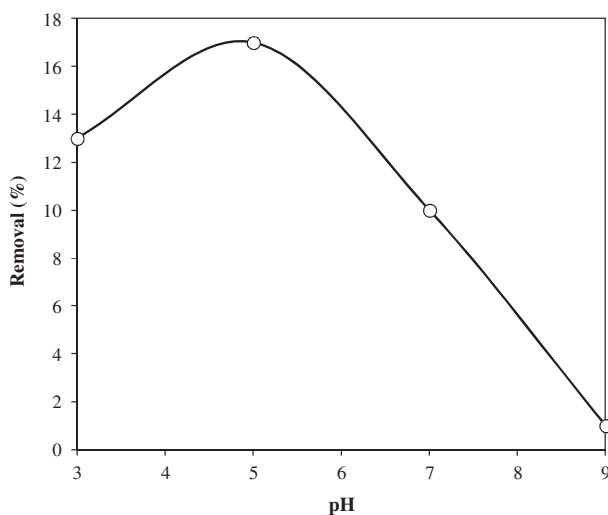


Fig. 3. Effect of pH on the phenol removal by UV system, after 60 min radiation (feed = 100 ppm).

The UV energy should degrade the phenol molecules. For this purpose, it is important that the maximum amount of available energy be consumed for dissociation of C–H or O–H bonds in the phenol molecule. In highly acidic solutions (i.e. pH = 3), there is a strong hydrogen bonding between phenol molecules and the molecules form clusters [2]. Although this hydrogen bond is not as strong as covalent C–H or O–H bonds, a part of available UV energy is consumed for dissociation of these hydrogen bonds. Then, in highly acidic solutions, the degradation efficiency is low. In neutral and alkaline solutions, the quantum yield diminished because of secondary reactions [36]. Thus, the photolysis efficiency decreases more considerably than acidic solutions.

#### 3.2. RO membrane system

In the next step, phenol removal using the RO system was examined. The feed solutions (with adjusted pH, and concentration) were pumped into the membrane module while the feed pressure was set at the desired value (50 and 70 psi) using a back pressure regulator. In order to establish a quasi-steady-state condition, the rejected and permeated streams were recycled into the feed tank. Sampling from the permeate and the rejected streams was done at different times until the equilibrium condition was monitored. The results showed that, at least 30 min is necessary to reach the steady-state condition. This time is named as “equilibration time,” and after that the system was at steady-state condition and the streams could be analyzed for phenol concentration. As this equilibration time may change by changes in the experimental conditions such as concentration and pressure, then 60 min is considered for the equilibration time.

At the steady-state condition, the membrane retention  $R$  (%) remains unchanged and could be expressed as:

$$R (\%) = \frac{C_R - C_P}{C_R} \times 100 \quad (2)$$

where  $C_P$  and  $C_R$  are the permeate and retentate stream concentrations, respectively. In RO processes, it should be noted that the permeation flux depends on the net hydraulic pressure applied across the membrane (apparent hydraulic pressure minus the difference in the osmotic pressures of the solutions at the two sides of the membrane). In addition, it depends on the membrane structure, solute concentration, while the solute permeability is a function of the solute physical and chemical natures, and composition.

The effect of feed pH on phenol rejection for feed concentration of 100 ppm is shown in Fig. 4. The results showed that for both pressures, the removal efficiency is time dependent. In addition, after 30 min of operation at pH=5, the steady-state condition did not establish. The phenol removal at feed pressure of 70 psi was higher than that for 50 psi for all pH values and the maximum phenol removal was achieved after 60 min and at pH = 5.

The reason could be expressed as the applied pressure affects the rejection coefficient. As the feed pressure is increased, the permeate flow rate and consequently the rejection coefficient is increased. It is reasonable that the time needed for establishment of the steady state at higher pressures is lower than the lower pressure because of higher permeation flux. Then, the feed pressure and pH were considered as the most effective parameters on phenol rejection by the RO system. It was also concluded that the time on stream is important and the system required a long time to reach a steady-state condition. Fig. 4 illustrates that for short times on stream (i.e. 30 min), the rejection of phenol at low pH values (pH=5) was higher than that for longer times on stream. This is mainly due to lower physical adsorption of phenol molecules at the surface of the incompletely swollen RO membrane. As the swelling of the membrane is increased by time on stream, the water flux is also increased and consequently the rejection of large solutes such as phenol molecules is increased. On the other hand, as

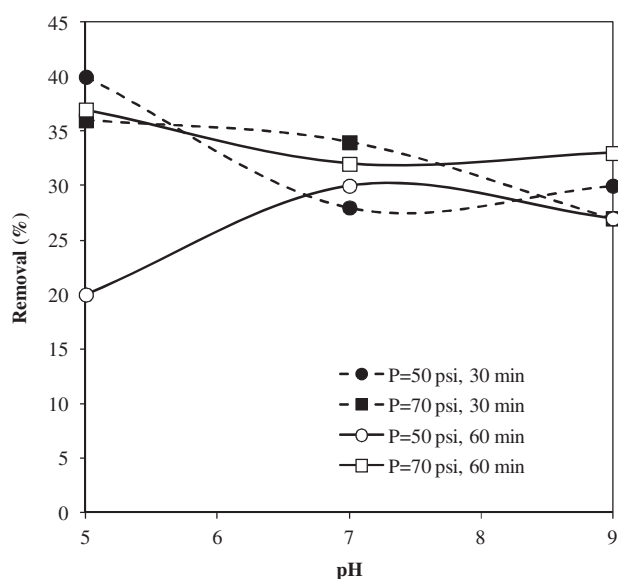


Fig. 4. Effect of feed solution pH on phenol removal by RO system for two feed pressures after 30 and 60 min (feed = 100 ppm).

time goes on, the surface of the membrane becomes more saturated by phenol molecules and also concentration polarization at the surface of the membrane reduces the phenol removal percentage and concentration of phenol in the permeate stream increases. Nevertheless, as it was reported by other researchers, by increasing the time on stream, the system reaches a better condition and the difference between the rejection percentage at acidic and alkaline feeds (in the range of pH=5–9) is reduced [37].

However, in some researches, no significant differences in phenol rejection at different feed concentrations and pH have been reported for high-pressure RO membranes, which may be related to the more compact structure of the applied membranes [38]. The hydrogen bonding ability of phenol due to the strong interaction between the phenol and membrane causes adsorption of phenol on the membrane. It seems that the degree of mobility on the sorption layer increases by increasing the operating pressure, and so consequently, rejection decreases. Thus, the retention was slightly increased with an increase in the applied pressure. On the other hand, membrane retention is not simply a filtration process, and to explain the process, the molecular shape alone is not sufficient. A net preferential sorption occurs at the membrane solute interface as well as preferential transport occurring inside the membrane that is the overall result of the interaction between the membrane material and the solute molecules [37,38].

On the other hand, the importance of the solution pH on the performance of the membrane retention is especially related to possible changes on the surface charge of the membrane. Some researchers found that the charge of the membrane surface was shifted from negative to positive with a pH less than 5.0 [36,38]. Akbari et al. reported that the differences in electrical charge between the membrane and solute might cause a concentration polarization phenomenon [23]. However, it seems that the acidic feeds have a minor effect on phenol retention.

### 3.3. UV/RO hybrid system

For increasing the phenol removal, a combination of UV and RO systems was used. Based on the individual UV and RO systems results, the hybrid system was considered to improve phenol elimination by the addition of the UV degradation system to the RO membrane system as the UV/RO system. The experiments were conducted at the same conditions that were used for individual UV and RO systems. Fig. 2 shows the setup that was used for this purpose. This system was consisted of a feed tank, a diaphragm

pump, UV-lamp chamber, RO membrane module, pressure gauge, needle valves, and back pressure regulator. In this system, the phenol containing aqueous feed was passed through a UV photolysis system and was then introduced into a RO system. The permeate stream was collected and the rejected stream (which is phenol rich) was recirculated to the feed tank. The effect of influencing parameters such as initial feed concentration, pump pressure, and processing time for phenol removal by the UV/RO system was evaluated.

The influence of initial feed concentration on the phenol removal was tested at various feed concentrations (35, 50, and 100 ppm). Fig. 5 shows the results of the experiments. Based on the results of individual RO and UV experiments, the feed pH was set at pH=5 and the efficiency was calculated after 60 min. The results showed that higher phenol removal could be obtained from higher phenol containing feeds. The phenol removal efficiency was increased from 43 to 58% as the feed concentration was increased from 35 to 100 ppm, respectively.

The effect of feed pressure on phenol removal by the UV/RO hybrid system was also examined. As the best results were obtained from the feed containing 100 ppm phenol in the previous experiment, the effect of pressure was studied for only 100 ppm feed concentration at three feed pressure levels (30, 50, and 70 psi). Fig. 6 shows the effect of feed pressure on the phenol removal efficiency by the UV/RO hybrid system. By an increase in the feed pressure from 30 to 50 psi, the phenol removal was increased, but it was

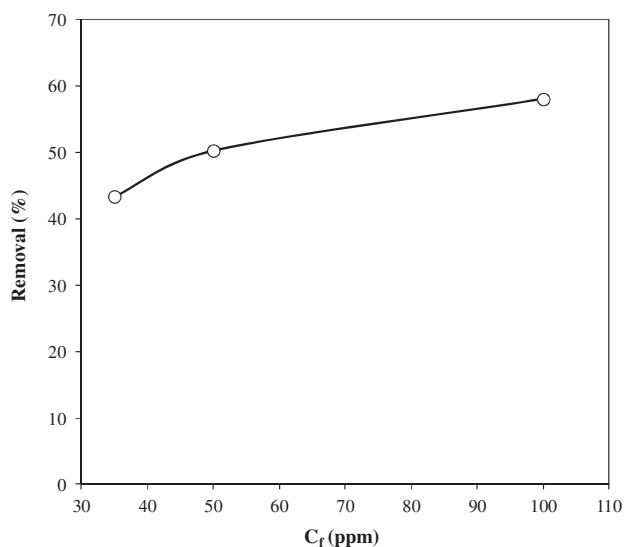


Fig. 5. Effect of feed concentration on the phenol removal from the UV/RO system after 60 min ( $\Delta p = 50$  psi and feed pH = 5).

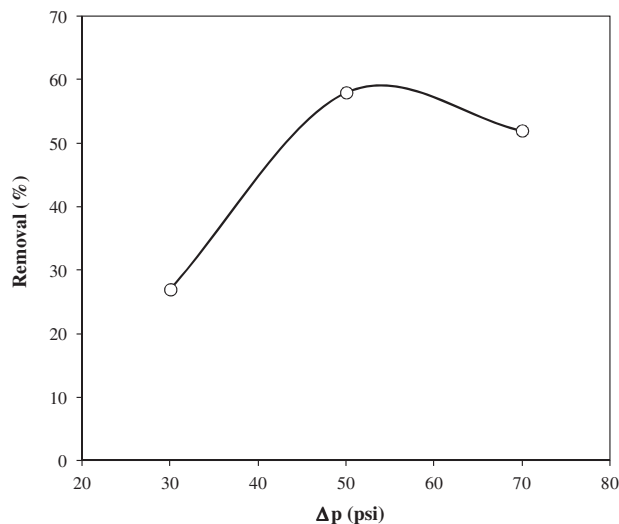


Fig. 6. Effect of feed pressure on the phenol removal from the UV / RO system after 60 min (feed = 100 ppm and pH = 5).

decreased when the feed pressure was increased to 70 psi. The reason is the competition between increasing the permeation flux by an increase in feed pressure and increase of concentration polarization by an increase in permeation flux. As the UV lamp in this study was a low-power lamp (6 W), the effect of the RO process on the phenol removal efficiency is more considerable than the effect of UV degradation of phenol. The figure also shows a critical pressure beyond it, and the effect of concentration polarization decreased the phenol removal efficiency. The existence of a critical pressure for the removal of phenolic compounds by the low-pressure RO system has been reported by other researchers [38].

As the recirculation rate in the UV/RO hybrid system is high, then the order of RO and UV systems in the process line is not important. Thus, it is expected that the RO/UV hybrid system would result in UV/RO system efficiency.

### 3.4. Comparison of UV, RO, and UV/RO hybrid systems

The result of phenol removal in UV, RO, and UV/RO hybrid systems is shown in Fig. 7. The results show that a combination of UV irradiation with the RO membrane separation system (UV/RO hybrid system) is able to remove phenol up to 58% after 60 min, which is about 40% more than that for the sum of individual UV and RO systems. In the UV/RO hybrid system, phenol is removed under both degradation and rejection mechanisms, so it seems that using this

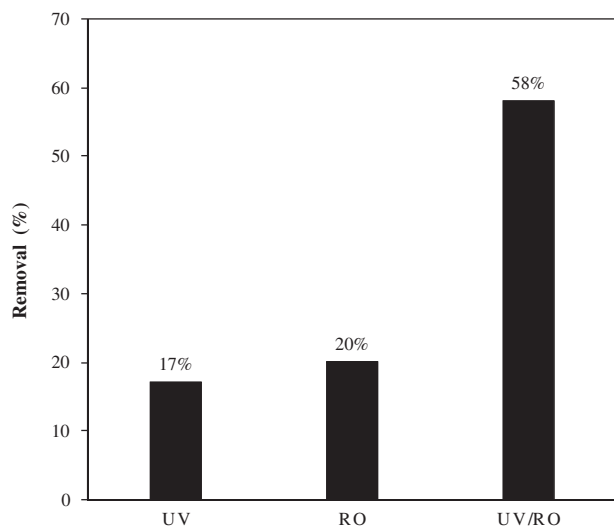


Fig. 7. The comparison between RO, UV, and UV/RO systems for phenol removal efficiency after 60 min (feed = 100 ppm and pH = 5,  $\Delta p$  = 50 psi).

hybrid system under reasonable operating conditions (based on the source of wastewater and the post treatment strategies), could be considered as an effective method for treatment of phenolic wastewaters.

#### 4. Conclusions

The use of UV irradiation, rejection by the RO system, and UV/RO hybrid systems are three methods for phenol removal from wastewaters. The following conclusions could be inferred from the current study:

- Highly alkaline and acidic phenolic feeds are not suitable for degradation by UV. The best results were obtained at pH = 5.
- Different behaviors were observed during phenol removal by the RO system. A critical pressure was observed below which the removal efficiency is decreased. In addition, higher feed pressures give better results than lower pressures and rejection efficiency is time dependent. In addition, pH of the feed solution in the range of 5–9 showed little effect on the rejection efficiency, while the rejections for acidic solutions were slightly higher than those for alkaline solutions in this range of pH.
- The hybrid UV/RO system for phenol removal showed the best results. The removal efficiency of the hybrid system was obtained three times more than that for individual UV and RO systems. In addition, the overall efficiency of the hybrid system was obtained nearly two times more than the sum of the individual systems efficiencies.

- The results showed the dependency of the removal efficiency to the phenol concentration in the UV section, and thus we recommend RO/UV combination for large-scale systems.
- It is proposed that the use of a hybrid UV/RO system with a higher rejection coefficient membrane would probably give better results for the phenol removal process because the RO system had a key role in the UV/RO hybrid system in the range of the studied parameters.

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