

## Combined SBR and RO pilot scale treatment for pharmaceutical wastewater

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### ABSTRACT

Complexity, presence of active pharmaceutical ingredients (API) and need to meet stringent rules have made treating of pharmaceutical wastewater highly challenging, which lead to an increase in attention towards these wastewaters seeking alternate and new methods. Some of the most commonly and widely accepted such treatment methods are activated carbon, membrane filtration and reverse osmosis. The present study focusses on treating wastewater from pharmaceutical industry using a combination of sequential batch reactor (SBR) and reverse osmosis (RO). After a series of four SBR treatments (SBR – I, II, III and IV) 53.7% of COD reduction was obtained by the end of SBR – IV. 99% removal in TDS, 99% reduction in COD and 83% reduction in Ammonia were obtained after treating the effluent from SBR by reverse osmosis. From the result it is concluded that series of SBR treatment proved to be suitable pretreatment for Reverse Osmosis which has shown promising results in reduction of the tested pollution parameters.

*Keywords:* Sequencing batch reactor; Reverse osmosis; Chemical oxygen demand; Total dissolved solids; Ammonia

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### 1. Introduction

Production of pharmaceutical compounds is a batch process that leads to presence of wide variety of active pharmaceutical ingredients (API) compounds in the wastewaters generated from different operations. These varied operations include requirement of copious amounts of water which eventually is generated as wastewater. Pharmaceutical compounds presence in drinking water can be traced to two different sources i.e., from production processes and from use of pharmaceutical compounds ending in urban and farm wastewaters. The fact of water resource scarcity makes it necessary to understand and develop methodologies for treatment of pharmaceutical wastewater as part of water management. Conventional methods are not successful in meeting required standards owing to the same reason [1].

Search for new technologies which would prove efficient in treating pharmaceutical effluents has identified the technologies of membrane separation processes. These have become much popular for effective treatment of different industrial wastewater owing to the reason that they have combine process stability resulting in remarkable quality of effluent [2–5].

Reverse osmosis (RO), which is one among many membrane process that is extensively adopted round the world has reported to be promising [6–11]. The key problem using RO is membrane fouling which is predominant in water reclamation applications. This membrane fouling is also the main reason for permeate flux decline and also loss in product quality of the in reverse osmosis Systems [6,12,13]. Fouling sources are divided into four main classes: biofouling, particular fouling, scaling and organic fouling. Reverse osmosis systems are required to be coupled along with an affective pretreatment in order to avoid common problems which might can result in system failure.

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Sequencing batch reactor (SBR) system has demonstrated to offer considerable benefits over alternative conventional systems for the biological treatment of both domestic and industrial wastewaters. SBR is tremendously flexible in its capability to meet varied diverse treatment objectives, while physically it is very simple. Sequencing batch (fill-and-draw) biological reactors might be operated in order to provide equalization, treatment, and sedimentation in the same set of reactors. The wastewaters are to be held in the treatment systems till the desired effluent quality is achieved [14]. SBR has been extensively studied and operated on full scale for wastewater treatment and it is believed to be a new pretreatment for reverse osmosis in water recovery and reuse among many diverse pretreatment schemes for RO.

Membrane bioreactor (MBR) processes, use membrane filtration units to replace the secondary settler. These offer numerous well-known benefits over conventional activated sludge processes, like brilliant quality of effluent, virtuous disinfection capability, enhanced volumetric loading, reduced footprint and sludge production, process flexibility toward influent changes, and upgraded nitrification [15]. This technology has demonstrated effectiveness in removing organic and inorganic contaminants including microorganisms from wastewaters [16] and thus gained superior popularity. MBR processes prove to be an attractive option for treatment of industrial waste streams which are complex with high-strength, having problems like settling and clarification [17]. A study conducted by Emmanuel and Evan, 2009 [18] have showed that a combination of MBR and RO provided excellent quality effluent which was devoid of heavy metals along with very low concentration of organic matter (DOC level below 4 mg/L) but incomplete removal of total nitrogen (20 mg/L).

The current work is carried out focusing on the appraisal of the pilot scale operation of combined treatment of SBR and RO for pharmaceutical wastewaters. The removal of certain pollution parameters such as chemical oxygen demand (COD), total dissolved solids (TDS) and ammonia ( $\text{NH}_3$ ) were monitored.

## 2. Material and methods

### 2.1. Flow chart of effluent treatment

**Multi effect evaporator:** Wastewaters arising from manufacturing processes are separated based on the total dissolved solid (TDS) content into two streams. One stream which contains high TDS (HTDS) which needs to be pre-treated before biological treatment and the other stream containing low TDS (LTDS).

The pretreatment of HTDS is done by first neutralization the effluent in a neutralization tank and then passing these effluents to Multi Effect Evaporator (MEE) where in reduction in TDS is obtained.

**Anoxic tank:** Condensate from MEE and LTDS are then fed to Anoxic Tank - I for retention time (RT) of 24 h with addition of food source (return sludge from SBR) and a mixer for the constant agitation of the mixed effluent.

**Sequential batch reactor:** The wastewater from anoxic tank is fed to a series of SBR i.e. SBR - I, II, III and IV. Retention time of individual SBR is 20 h (which includes feeding and settlement) after which the effluent is decanted. The decanting of SBRs is done only if all the parameters are within the specifications, particularly with reference to COD which should be less than 3000 ppm. From SBR the decanted effluent is fed to Anoxic tank - II for removal of ammonical nitrogen. After this pretreatment the treated water from anoxic tank - II is fed to reverse osmosis for further treatment.

**Reverse osmosis:** The decanted effluent from anoxic tank - II pumped to the RO feed tank through Cartridge filter 20 micron. After treatment permeate from reverse osmosis is reused for various utilities whereas the reject is pumped to the MEE feed tank for further processing (Table 1).

### 2.2. Sample collection points

Samples are collected from

1. Inlet to SBR, outlets from SBR - I, II, III and IV (5)
2. Feed, Permeate and Reject of Reverse Osmosis (3)

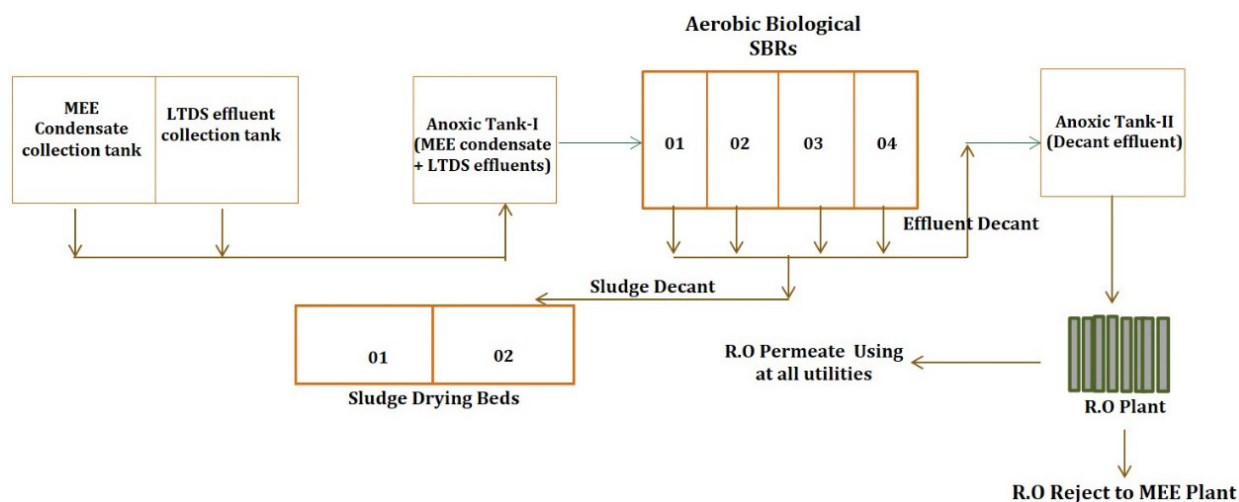


Fig. 1. Line diagram of process sequence.

Table 1  
Characteristics of the RO membrane used in the study

Details	MHP-90 XXL
Membrane type	Polyamide thin film composite
Application	Reverse osmosis
Membrane area module, m <sup>2</sup>	11.40
Outer dia of module, mm	216 ± 2
Inner dia of module, mm	202 ± 1
Length of module, mm	1430
Length of tie rod, mm	1630
Operating pressure max, bar	90
pH operating range	3–11
Rejection as per testing conditions, %	>98.0
Max. operating temperature, °C	40

Permeate flux operated in the present study is 273 L/h.

### 2.3. Analytical methods

Characterization of pharmaceutical wastewater is done as per the standard methods given by APHA, 2012 [19]. The parameters considered in the present study for evaluation of the treatment processes are pH, TDS, COD and NH<sub>4</sub><sup>+</sup>, additionally Sludge Volume is also determine for samples from SBR. Data presented was averaged by at least 2 experiment results at each process.

### 3. Results and discussion

Table 2 presents the initial parameters of the pharmaceutical wastewater, which includes characteristics of both high TDS and low TDS. It is observed that the characteristics of the high TDS wastewater is far beyond the limits and would require extensive and advanced treatment, in order to comply with the effluent discharge standards.

Studies carried out by others have reported similar results. Mayabhate et al. [20] observed that the characteristics of wastewater originating from manufacturing of pharmaceuticals varied greatly, which was depended on raw materials and equipments used along with the manufacturing, compounding and formulation process employed. Characterization of pharmaceutical wastewater by Madukasi et al. [21] showed the following characteristics of parameters in mg/l total suspended solids 425 ± 2.3, total dissolved solids 1,600 ± 1.1, total nitrogen 533.7, BOD 146.7 ± 0.3, Zn 0.056, Iron 2.1, Mn 0.605, Cu 0.022, acetic acid 422.7, butyric acid 304.5 and propionic acid 201.3. In their study regarding the pharmaceutical industries in and around Hyderabad, India, Raj and Anjaneyulu [22] mentioned that these industries produce a variety of products as they use both organic and inorganic substances as raw materials, resulting in generation of large quantity of complex toxic organic liquids waste containing high concentrations of inorganic TDS, BOD and COD.

The key intention of conventional methods is to improve the life of RO and to reduce the cost of the treatment. Performance of pilot scale treatment using SBR and

Table 2  
Physico-chemical parameters of raw effluents from manufacturing process

Parameters	High TDS	Low TDS
pH	6.81	7.20
Temperature °C	38	33
Total dissolved solids	52,573	2690
Total suspended solids	2,038	300
Chemical oxygen demand	86,078	3500
Biochemical oxygen demand (3 d at 27°C)	29,290	1192
Nitrates as NO <sub>3</sub> <sup>-</sup>	654	39
Chlorides as Cl <sup>-</sup>	6,686	510
Sulphates as SO <sub>4</sub>	2,135	110
Oil & Grease	19.60	11.70
Phenolic compounds as C <sub>6</sub> H <sub>5</sub> OH	0.62	0.28
Ammonical nitrogen as N	2,125	98
Cyanide as CN	2.80	BDL
Chromium hexavalent Cr <sup>+6</sup>	BDL	BDL
Chromium (total) as Cr	0.50	0.23
Copper as Cu	1.05	0.18
Lead as Pb	0.82	0.56
Nickel as Ni	0.70	0.32
Zinc as Zn	0.60	0.98
Arsenic as As	BDL	BDL
Mercury as Hg	BDL	BDL
Cadmium as Cd	0.20	0.09
Selenium as Se	BDL	BDL
Fluoride as F	6.35	2.06
Boron as B	0.70	0.84

Note:

1. All values except pH are expressed in mg/L.
2. BDL- Below detectable limit.

RO for pharmaceutical wastewater has been studied. Effluent from MEE condensate is equally pumped into SBRs I, II, III and IV. Decant from these SBRs is either fed to anoxic tank or sent to reverse osmosis. Figs. 2–5 present efficiency of SBRs, expressed in terms of parameters like reduction in COD, increase in DO, sludge volume and trends of pH. Figs. 6–9 predict the efficiency of reverse osmosis expressed in terms of reduction in total dissolved solids, chemical oxygen demand and ammonia, along with trends of pH. These parameters are selected as indicators of organic pollution.

COD reduction in SBRs – I, II, III and IV are presented in Fig. 2. SBR – III had technical difficulty during from June to November and hence values are not presented. Reduction of COD was observed to be maximum of 68% during January in SBR – I and minimum of 34% during May. Average COD reduction during the nine months duration was between 53 and 54% in all the SBRs (Fig. 2). The result shows that SBR system was efficient in removal of organic constituents achieving good removal of COD, reducing organic load effectively. Similar study by Elmolla et al. [23] reported 94% BOD<sub>5</sub> removal and 83% COD removal at 24 h HRT and 4000

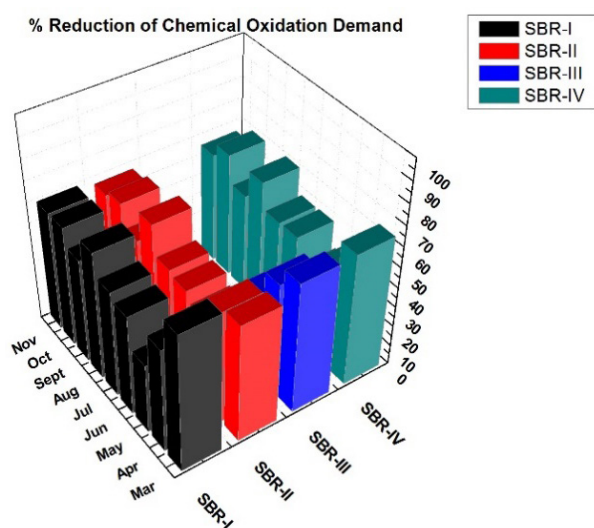


Fig. 2. Percentage reduction of chemical oxygen demand in SBRs I, II, III and IV.

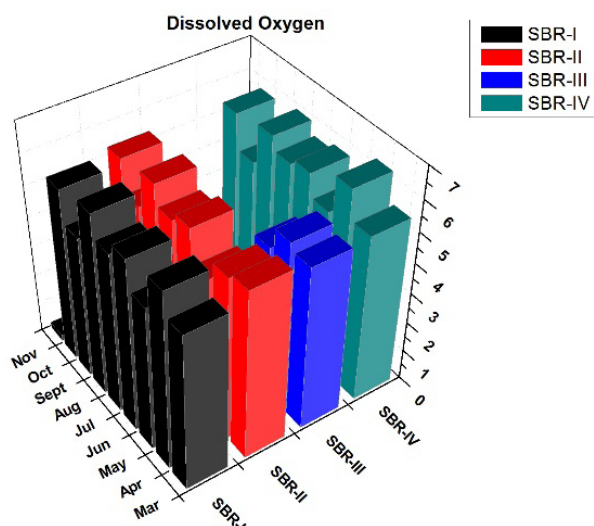


Fig. 3. Replenishment of dissolved oxygen in SBRs – I, II, III and IV.

mg/L of MLSS. Another study reported 98% of reduction in COD with combined solar photo-Fenton process and aerobic sequential batch reactor treatment applied for pharmaceutical wastewater by varying pH, concentration of ferrous ion, dosage of  $H_2O_2$  and treatment time [24].

Pharmaceutical industry generates wastewater containing toxic organic chemicals and the composition of the wastewater is highly variable resulting in drastic reduction of dissolved oxygen levels. Highest replenishment (6.4 ppm) of DO was recorded in SBR - IV during April. The lowest (0.2 ppm) was detected in all SBRs during the month of November (Fig. 3). Studies conducted by other researchers have reported similar results [25,26].

Low dissolved oxygen concentrations apart from indicating pollution also mobilize trace metals. Also low DO

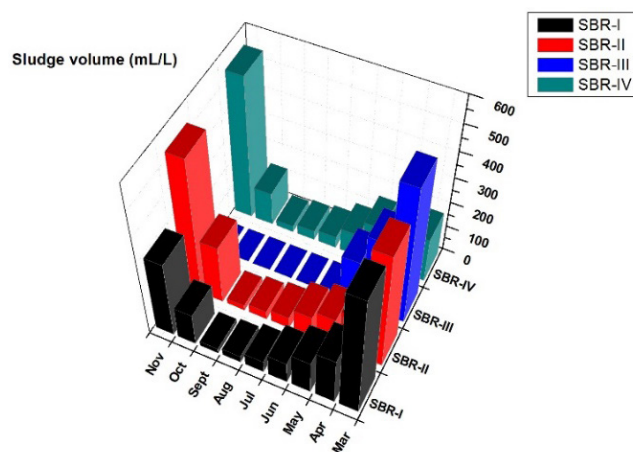


Fig. 4. Sludge volume SBRs – I, II, III and IV.

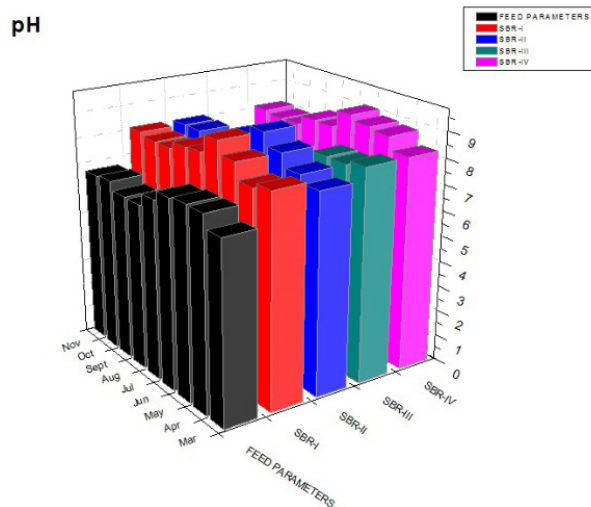


Fig. 5. Trends of pH in SBRs – I, II, III and IV.

values indicate the production of organic substances which result in high oxygen demanding wastes [27]. Effluents with low DO released into natural waters effect the aquatic life. In the present study SBR treatment has resulted in enhanced DO, which will also enable aerobic conditions in the system.

For initial three months sludge volume was between 540 ml/L and 118 ml/L, which later on reduced to as low as 18 ml/L during September. Higher values of sludge volume requires attention on treatment and disposal of sludge which would be an additional load on the treatment process. Reduction in sludge volume with effective removal of pollutant parameters will offer a feasible and promising treatment option (Fig. 4). Favourable conditions for formation of good settling characteristic sludge is a high food to microorganism (F:M) ratio which results in floc forming organisms versus filamentous organisms (Wastewater Technology Fact Sheet, 1999) [28]. On the other hand structure and settleability of sludge resulting in increased sludge volume index (SVI) is due to the problem of bulking [29]. According to

many studies conducted previously stated that an important role is played by exopolymeric substances (EPS) like proteins and carbohydrates in flocculation and settleability of sludge [30,31]. The present study reported good settling quality sludge (18 ml/L) which is in lines with the studies conducted by Texier and Gomez, 2004 [32], where in they obtained a well settled sludge in correlation with the microbial protein concentration of  $1.04 \pm 0.05 \text{ g}\cdot\text{L}^{-1}$ .

pH of the inlet effluent streams into all SBRs was between 6.5–7.5. The resultant decant from all SBRs is observed to be in the range of 7.8–9.1, which is towards alkaline. pH plays an important role in utilization rate and metabolic activity of microbial metabolic substrates and also influences sludge yield and pollutant removal [33]. Presence of ammonia might be a reason for the basic conditions (Fig. 5). As reported by Li et al. [34], their investigation of alkalinity as a reliable indicator of effluent nitrogen concentration has presented a better indication over other method adopted (ORP). This was especially clear when there was a progressive decrease in nitrification/denitrification efficiency of the SBR, where alkalinity exhibited a linear reverse correlation with effluent nitrogen concentration.

### 3.1. Performance of reverse osmosis

Performance of reverse osmosis has been evaluated in terms of change in pH, reduction in parameters like chemical oxygen demand, ammonia and total dissolved solids. The quality of the permeate (filtrate), in terms of ammonia - nitrogen and COD, is highly dependent on the actual feed concentration.

### 3.2. pH

During the test period the pH of feed was in the range of 5.4–7.0, whereas the permeate pH has increased during the entire study the range of permeate pH was observed to be 6.0–7.4 (Fig. 6). As pH increases it can bring out changes in water chemistry through affecting size, charge or solubility of specific constituents in the feed. pH increase can

also effect charge of RO membrane opening highly cross-linked molecules which form the polyamide structure. Such changes in both membrane characteristics and water chemistry can be used to the advantage of the designer of RO system through improving rejection, increasing recovery and reducing fouling [35].

Franks et al. [35] studied the effect of passage through three different nanofiltration membranes at different pH corresponding to variation in membrane charge. Of the three membranes they studied membrane C was characteristic of a typical polyamide RO membrane used for treating the high pH produced waters. They reported that at lower pH the hydrogen ion attaches to the membrane's negative carboxyl groups and neutralizes the charge of the membrane. The lack of a negative charge at lower pH reduces the membrane's ion rejection. At neutral and high pH, the lack of the hydrogen ion attached to the membrane's carboxyl groups, results in a negatively charged membrane. The presence of a negative charge improves the rejection at neutral pH. At high pH, the membrane retains its negative charge. However, as pH increases and the hydroxyl ion concentration increases, the carboxyl groups on membranes surface are repelled or opened and the membrane "swells". This "swelling" effect increases the channel of certain ions. hydroxyl ions ( $\text{OH}^-$ ), for example, transfer through the membrane more readily than chloride ions ( $\text{Cl}^-$ ).

### 3.3. Total dissolved solids

Dissolved solid content of the feed was in the range of 1196–2490 mg/L, in permeate the range was 6–424 mg/L. Maximum of 99% and minimum of 77% removal of total dissolved solids was seen (Fig. 7). It is an understood fact that reverse osmosis is very efficient in removing solids. Solids of organic nature are the reason for fast fouling of the membrane. Hence, pretreatment of the feed water entering an existing RO system can minimize fouling of the membrane and thereby increase the overall recovery rate of the system. The feed water, depending on its source, may contain various concentrations of suspended solids and

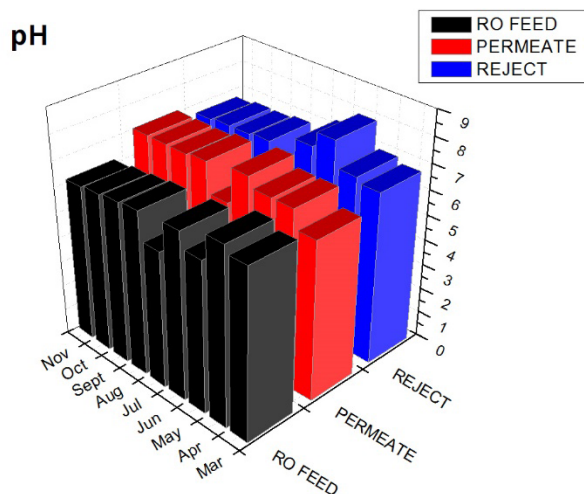


Fig. 6. Trends of pH in reverse osmosis.

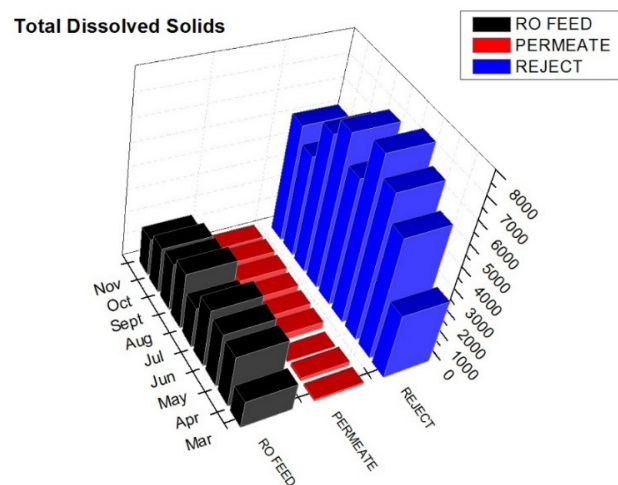


Fig. 7. Reduction in total dissolved solids after treatment in reverse osmosis.

dissolved matter, including both organic and inorganic substances. The size of the membrane fouling is dependent on the complex composition of the feed water and in the present study it was observed to be ranging from 0.0001 to 9.9 micron. The RO after fouling is regenerated using chemical method which includes regeneration by basic, neutral and acidic solutions sequentially.

Suspended particles settle on the membrane surface, increasing friction losses across the system through blocking feed channels. Dissolved solids can also precipitate out of the solution and cause scaling. Water that enters the RO system after pretreatment can reduce the amount of work of the RO pump, thus reducing energy consumption [36].

### 3.4. Chemical oxygen demand

Feed COD values range from 2460 to 5205 mg/L and the permeate values are between 14 and 399 mg/L. Removal percentages of smallest and highest are 85 and 99% respectively (Fig. 8). Preceding studies have shown that the analysis of COD was not affected by the presence of ammonia-nitrogen, meaning that it probably was mainly organic compounds which contributed to the measured COD value.

### 3.5. Ammonia

Feed ammonia values range from 891 to 426 mg/L and the permeate values are between 154 and 76 mg/L (Fig. 9). Ammonia is one among parameters that represent the content of nutrient substances and is widely used in the field of wastewater treatment. Removal percentages of minimum and maximum are 77 and 83% respectively. There are two ways in which nitrogen may be lost in the process: by emission of ammonia to air, and by the transport of ammonia to the permeate.

Emissions of ammonia to the permeate are difficult to avoid as gaseous compounds permeate easily through the membrane, while other losses might be reduced using covered systems. The utmost probable reason for removal of nitrogen would be due to the fact that nitrogen is elimi-

nated mainly through biodegradation by microorganisms and then separation with membrane. Similar results were reported in some previous studies [36–38].

### 3.6. Quality of the permeate and its reuse

Reverse osmosis (RO) membranes have been proven to successfully treat such water and provide water which exceeds reuse quality requirements. From the treatment process it is seen that a superior quality permeate was obtained. The permeate could be discharged to a recipient or be used as cleaning water without any post-treatment. In areas with poor water supplies or low water quality, the permeate might then be used for watering animals, after being neutralized and disinfected with chlorine or ozone [39].

## 4. Conclusions

Few production environments are as exigent and challenging as that of the pharmaceutical industry. The trail level operational studies employed with sequential batch reactor (SBR) combined with reverse osmosis has shown a noble output, where the overall COD and total dissolved solids reduction was shown at 99%, with 83% reduction in ammonia.

In order to enrich the functioning of the reverse osmosis process, it is essential to develop cost-effective pre-treatment systems. Literature review showed that SBR seems to be promising option for the effective treatment of industrial wastewater. In broad-spectrum, it can be concluded that SBR can produce high permeate quality and is capable to be very efficient method for RO pretreatment.

Results from the study indicate that if SBR is used as RO pretreatment, it can be anticipated that the rate of RO membrane fouling will reduce and the life of RO membrane modules will extend. Also effluent water from the SBR has a good quality according to TDS, COD and Ammonia removal during operation. From the study the results are

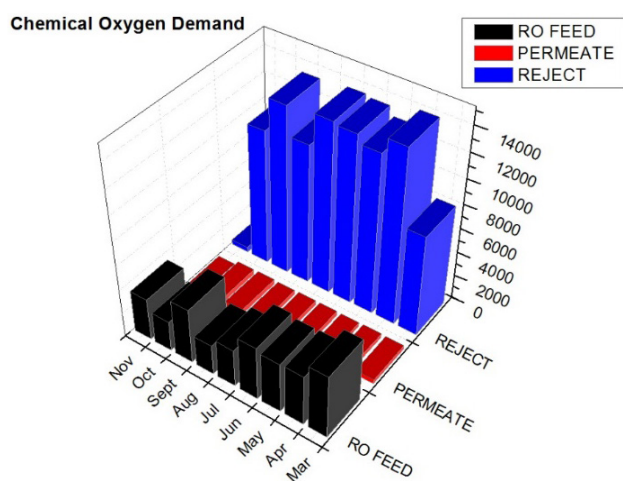


Fig. 8. Reduction in chemical oxygen demand after treatment in reverse osmosis.

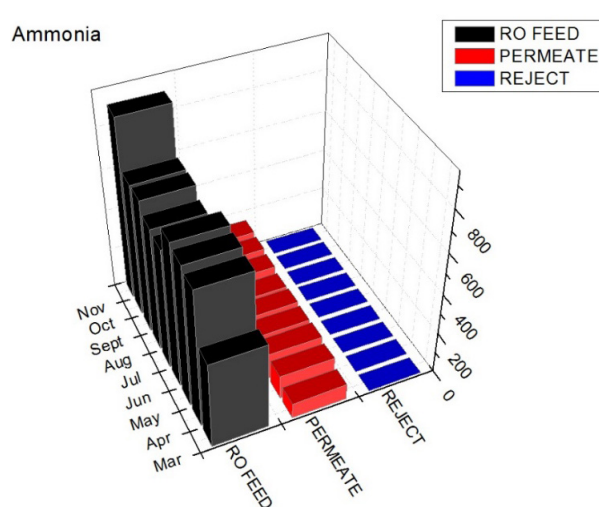


Fig. 9. Reduction in ammonia after treatment in reverse osmosis.

encouraging and all suggestions show that this method is viable for RO pretreatment and water obtained from RO can be reused in different industrial applications. Moreover, the finest quality attained by reverse osmosis permeate, makes this technology the keystone of every zero-discharge approach. Therefore permeate can be reused and thus contribute to reduce the overall water consumption.

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