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Original Article

Evaluation of Bio-Membrane System Efficiency Optimized With Nanotechnology for Treatment of Pulp and Paper Industry Wastewater

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Abstract
Background & Aims: The membrane adsorption bioreactor (MABR) process is the integration of biological treatment and membrane technology. Accordingly, in this study, an MABR was employed for the pulp and paper industry wastewater treatment.
Materials and Methods: The purchased powdered activated carbon (PAC) was added to the system as an adsorbent which improved the flux of the membrane.
Results: Based on the obtained results, the organic compounds were successfully removed by the average removal of 62% and 86% without and with an adsorbent, respectively. Moreover, the activated sludge was prepared from the Babol-Toyoor Slaughterhouse wastewater treatment, and adding the PAC to the activated sludge led to the better performance of the MABR system by providing a proper condition for microorganism growth. Monitoring the mixed liquid suspended solids during the process demonstrated that increasing mixed liquor suspended solids (MLSS) increased the contaminant removal rate.
Conclusion: Overall, the presence of PAC could prevent microorganisms from accumulating on the membrane surface.
Keywords: Thylakoid membrane proteins, Carbon, Chemistry, Nanotechnology, Water purification

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1. Introduction

Water pollution implies the chemical or microbial contamination of lakes, rivers, oceans, and groundwater. When pollutants, without the treatment of harmful materials, are directly or indirectly discharged into the water, water contamination would increase, and water pollution will affect plants and living organisms. Water is crucial for life, and most chemical reactions occur in the aqueous environment. Water has special and fundamental properties in regulating nature and protecting it against sudden climate changes; it is converted into wastewater after various actions (e.g., agricultural, industrial, and the like). Some strategies and solutions must be adopted to prevent water and environmental contamination by this waste [1].

Human activities, especially industrial ones, produce wastewater and pollutant gases that enter the environment in various ways. Some natural species in the environment can refine part of them and convert them to compatible compounds and materials through natural processes. Environmental factors such as light exposure, heat, and activity of living things are the cause of processes such as chemical, biochemical, photochemical, surface adsorption, and gas transfer reactions which can purify the contaminants. It is worth mentioning that if ecosystems are exposed to more pollutants than their natural purification capacity, environmental equilibrium conditions will be in crisis [2]. Nevertheless, wastewater can be used in some fields after treatment. For instance, utilizing industrial wastewater in agriculture can have several benefits; it is a good alternative to high-quality consuming water in agriculture, and the nutrients in the wastewater will reduce the need for fertilizing plants. Moreover, wastewater is known as a cheap and reliable source (permanent accessibility) in most large and industrial cities. However, due to the presence of some pollutants (heavy elements) and their highly damaging effects on the environment, utilizing this wastewater in agriculture is not as simple as using ordinary water; hence, it requires several management measures [3].

Generally, industrial wastewater arises from the use of water in industrial activities during various stages of production and is occasionally the most dangerous type of wastewater. Due to the diverse chemicals and methods of production in the industry, the quality of industrial wastewater greatly depends on the type of industry. For

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instance, the pulp and paper industry, in which water consumption is high, produces wastewater containing large amounts of organic and inorganic materials, organic halogen derivatives, chemical detergents, acidic and base compounds, heavy metals, furans, dioxins, cyanides, sulfides, and dichloro glycosides. Discharging this wastewater into the surface water causes fish mortality, immunosuppression, anemia, and change in hematological parameters, and the like [4] Furthermore, this type of wastewater produces sludge and foam and has thermal effects on the environment [5]. Industrial wastewater is usually divided into three groups, including wastewater related to a production line or industrial process, wastewater associated with the water treatment plant, blowdown of the boiler and cooling tower, and utilities. The third group includes wastewater related to the washing of tanks, enclosures, and the like, which is similar to production line effluent. The nature of the mentioned contaminants is sometimes different, thus industrial wastewater treatment methods are different [6]. In the case of industrial wastewater, the concentration of effluent is usually measured by contaminants. However, most industrial effluents are also identified through contaminants created by organic materials (soluble or insoluble) that are the most important indicators of contamination. Two commonly used methods are biochemical oxygen demand (BOD) and chemical oxygen demand (COD), and the degree and severity of industrial wastewater contamination can be estimated through these methods [7]. In addition to the aforementioned contaminants, many other compounds are found in industrial effluents, which are occasionally hazardous. Therefore, the first step in industrial wastewater treatment is to perform a qualitative analysis and identify the type of effluent contamination. Next, the wastewater treatment system is designed and implemented according to physical, chemical, and biological treatment methods based on the type of present pollution in the wastewater [8]. The production of pulp and paper requires significant consumption of process water and energy in the form of steam and electricity. As a result, the most important environmental issues associated with pulp and paper production are the emission of pollutants into water and air, and energy consumption. The significant amount of residue and wastewater is also gradually increasing environmental concerns [9]. The process of paper production has varied phases, making it one of the largest water-consuming and subsequently wastewater-producing industries in the world alongside the automotive, oil, petrochemical, and steel industries [10]. In addition, this wastewater has high BOD, COD, pH, suspended materials, color, and turbidity. The three categories of the main pollutants in the paper industry are suspended organic matter, dyes, and inorganic solids [11]. Despite the high diversity of pollutants and their complex structure, many of the compounds present in the water are poorly soluble, thus their brownish-red color and resistance to purification methods, especially the biological treatment, pose many problems for treatment processes. Moreover, chlorinated compounds and dyes are among the main concerns in wastewater treatment. These factors have turned the wastewater of the paper industry into one of the most difficult industrial treatment processes associated with complexity in the applied processes [12,13]. Due to the low biodegradability of many colors, the bio-treatment of effluents is not always effective. Therefore, in particular, to remove dyes, various chemicals and adsorbents are directly added to the activated sludge system [14,15]. In general, there are various methods for the decontamination of industrial wastewater, including chemical oxidation, coagulation and flocculation, electrochemical methods, ion exchange, adsorption process, membrane processes, chemical recovery, and biological treatment. Nevertheless, the high volume of paper industry wastewater and in particular, the presence of some special compounds make it difficult to apply some of these methods for the treatment and decolorization of this type of effluent [16,17]. The physical-chemical treatment of wastewater includes preliminary treatments such as coagulation-flotation, sedimentation, and sludge displacement. Secondary and advanced treatments such as bio-filters or activated sludge can also be used in later stages [18,19]. Bohdziewicz et al researched the field of the membrane bioreactor on utilization and design concentrations for landfill leachate wastewater treatment using an anaerobic submerged membrane bioreactor. The results indicated that the COD removal rate was higher than 90% at 2 days hydraulic retention time (HRT), and nitrification reached 95% [20]. Yu et al focused on the bioreactor membrane absorption system that used submerged membranes in the filtration system, which was designed to purify potable water during 30-minute HRT. The activated carbon adsorbent was also employed at a dose of 8 mg/L. The system had three units, including ultrafiltration membranes for retention, a biological unit using micro-organisms, and surface adsorption using activated carbon [21]. Further, the evaluation of the MBR filtration system was performed using a new flat plate membrane with a COD removal efficiency of up to 96% and nitrogen of up to 54% [22]. Moreover, numerous studies [23-28] evaluated parameters such as organic charge rate, solid retention time, feed-to-microorganism ratio (F/M), and membrane function. However, the pulp and paper industry wastewater treatment has undergone no comprehensive study with an affordable process.

Hence, in this study, the state-of-the-art membrane adsorption bioreactor (MABR) process was utilized for the treatment of the pulp industry wastewater of the Babol-Kenar industrial estate, Mazandaran, Iran. This process is the incorporation of the biological treatment of activated sludge and separation with membrane technology in which powdered activated carbon (PAC) was used as an adsorbent to improve the treatment process and increase flux through the membrane. It should be mentioned that the investigations were performed at the Nanotechnology Institute (Babol Noshirvani University of Technology, Babol, Iran).

2. Materials and Methods

2.1. Materials

The provided wastewater in this study was from the pulp and paper industry in the Babol-Kenar industrial estate, Mazandaran, Iran. Overall, 20 L of wastewater were gathered from the output of the pre-treatment section of the pulp industry in each step; it remained at 4°C to maintain its physical and chemical properties. The activated sludge was prepared from the Babol-Toyoor Slaughterhouse wastewater treatment plant. The physical and chemical properties of pulp and paper wastewater are tabulated in Table 1. The employed membrane in this study was provided by Nanotechnology Research Institute, Babol, Iran. Sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4) were provided from Merck for pH adjustment and regeneration process.

2.2. Methods

2.2.1. Characterization

The operational parameters such as COD and pH were determined to represent wastewater characteristics. The characteristics of the provided activated sludge are presented in Table 2. Based on the results, mixed liquor suspended solid (MLSS) and COD were 2440 mg/L and 100 mg/L, respectively.

The standard test method [29] was the base of all the analytical methods. The samples were taken at specific times. The closed reflux method after filtration was performed to determine COD, in which Whatman filter papers (0.47 µ Millipore) method 5220D was used [29]. Further, the pH meter electrode (AD 1200 BENCH TOP) was employed to measure the pH of the samples. The inline conductivity was measured via the standard method 2510. As mentioned earlier, the MLSS was measured through the filtration of samples with 0.47 µ Millipore paper. An oven was applied at 105°C for drying the samples in order to reach constant weight [30]. The distilled water and PAC were provided from laboratory distillation equipment and Sigma-Aldrich, respectively.

To prevent the shock and consequently the mortality of microorganisms, adapted sludge with wastewater is needed in the treatment process of the wastewater of the pulp and paper industry by activated sludge. The COD and MLSS parameters were measured based on standard methods to control the process. Then, glucose was added to the reactor as a substrate for the easier adaptation of the biomass and wastewater. During this procedure, the COD, MLSS, and pH were computed, and the COD produced by extra glucose was removed from the COD removal efficiency calculation.

The PAC concentration in batch experiments was in the range of 0.5-4 g/L, and an experiment was conducted in the absence of PAC. The COD removal was measured in the reactor; subsequently, the experiments were performed under aeration at optimum HRT. PAC was also regenerated by soaking it in H_2SO_4 for 12 hours while stirring, followed by vacuum filtration. The pH of the filtrate was maintained by rinsing filtered carbon with deionized water until the pH was stable. The samples were dried in a 105°C oven for 24 hours [31]. In addition, to calculate the regeneration efficiency (RE) using Eq. (1), COD removal was measured before and after regeneration [32].

$$RE = \frac{COD_{after}}{COD_{before}} \times 100\% \quad \text{(Eq.1)}$$

Table 1. The Characteristics of the pulp industry wastewater

Parameter	Unit	Value
COD	mg/L	3371
BOD_5	mg/L	1490
pH	-	6.58
Conductivity	µs/cm	3470
TDS	mg/L	2082
TSS	mg/L	3430
TKN	mg/L	6.8

Note. COD: Chemical oxygen demand; BOD: Biochemical oxygen demand; TDS: Total dissolved solid; TSS: Total suspended solids; TKN: Total Kjeldahl nitrogen.

Table 2. The activated sludge characteristics

Parameter	Unit	Value
COD	mg/L	100
MLSS	mg/L	2440
pH	-	7.6
TDS	mg/L	1080

Note. COD: Chemical oxygen demand; MLSS: Mixed liquor suspended solid; TDS: Total dissolved solid.

2.2.2. MABR process

The MABR system comprising the ultra-filtration (UF) process was the pulp industry wastewater treatment equipment. The purchased UF membrane was cut out in the proper shape, dimensions, and structure for use in the module. The membrane was sectioned to the same size as the modules (12 × 20 cm); then, the membrane was placed on the module with two-part epoxy adhesive. In this study, plate-and-frame modules and the module body are made of hollow rectangle plexiglass. Furthermore, there are some grooves (with 0.2 mm deep) on the frame for permeate spacer placement. The flow carrier tube which has been attached to a pneumatique valve applied the suction through the vacuum pump. A cubic tank with a total volume of 10 L was the aeration tank in this process. An air compressor with two diffuser stones which supplied a constant flow rate of 5.5 L/min was equipped with the system. Moreover, this aeration system acts as an anti-fouling agent by applying shear stress to the surface of the membrane. The membrane efficiency was evaluated by the retention and flow rate of the membrane.

3. Results and Discussion

3.1. Acclimatization of activated sludge with pulp industry wastewater

Figure 1 shows the COD and MLSS changes in terms of time. In the adaptation method, first, the activated sludge was aerated for one day; then, 500 mL of pre-treated wastewater was added to the reactor. For the formed biomass in the sludge to be able to tolerate the wastewater with the high organic load, the volume of the wastewater was slightly increased each day. The described adaptation process was performed for 10 days. The capability of the pulp wastewater treatment and efficiency of the COD removal of microorganisms tended toward constant values after 7 days. The constancy of COD removal (64%) and biomass concentration (5140 mg/L) for a few days indicated that the microorganisms are adapted to the wastewater and consumed as a substrate or a new nutrient source.

3.2. Investigation of HRT

The treatment of pulp industry wastewater via activated sludge aerobic process was investigated in 12, 24, and 48 hours of HRT. Figure 2 displays the COD removal efficiency in different retention times. As shown, as the HRT increases from 24 to 48 hours, the COD removal has no significant increase. After investigating the repeatability of the obtained removal rate in each HRT, 24 hours was considered as the optimum HRT.

3.3. The Optimum Adsorbent Dose

The COD removal rate is depicted in Figure 3. The rate of COD removal has been demonstrated in terms of various

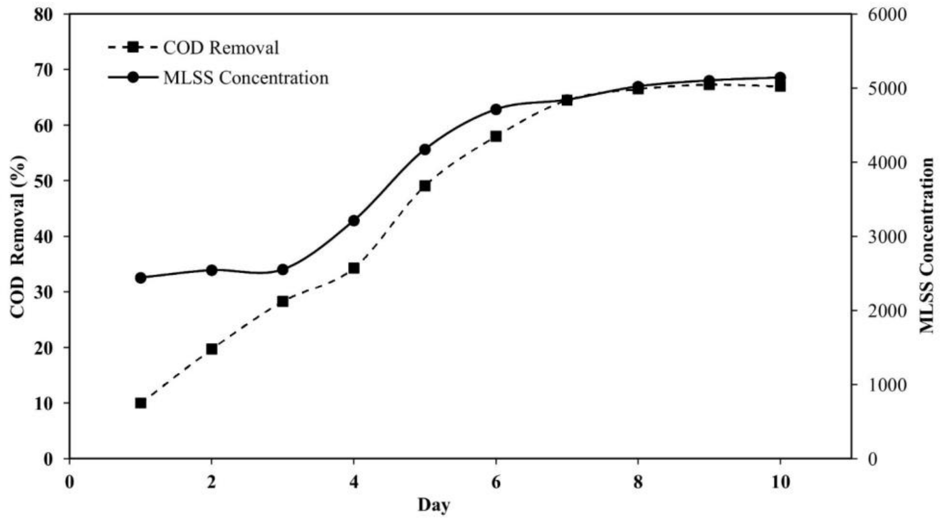

Figure 1. Changes in the COD removal and MLSS concentration during the acclimatization process. *Note*. COD: Chemical oxygen demand; MLSS: Mixed liquor suspended solid

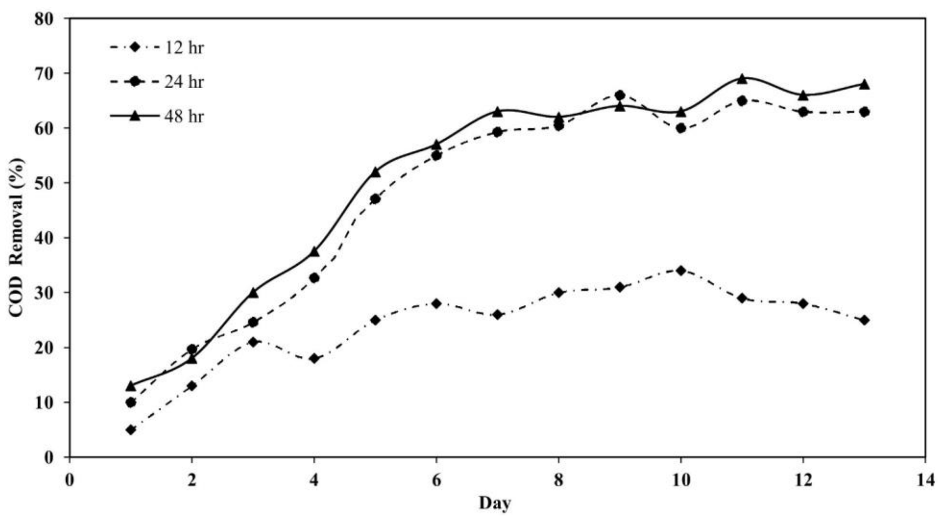

Figure 2. HRT effects on the COD removal. *Note*. HRT: Hydraulic retention time; COD: Chemical oxygen demand

amounts of activated carbon adsorbents. According to Figure 3, the COD removal percentage increases from 8% to 23% while the adsorbent dose changes from 0.5 g/L to 4 g/L. The existence of the PAC in the bioreactor provides a growth bed for microorganisms, and this growth causes an increase in MLSS and consequently improves the COD removal rate. Additionally, PAC improves the bio-oxidation of the organic component, but decreases the total growth of sludge, thus reducing the oxidation of organic substances [33]. Although the COD removal increases with the growth of the adsorbent dose, this increase is not significant; hence, 2.5 g/L was determined as the optimum adsorbent dose.

3.4. MABR process: investigation of effective factors and operational parameters

The membrane water flux is one of the most important indicators for membrane performance analysis in the MABR system. In addition, analyses such as COD and MLSS are performed for a more precise investigation of the performance of this system. For this purpose, a bioreactor with 16 L of volume includes a plate-and-frame module with 0.033 m^2 of effective surface which has been vertically placed in the bioreactor. Further, an aeration system (air pump and stone diffuser) supplied the oxygen demand of the aerobic-activated sludge process.

Figure 4 illustrates the changes in the membrane flux in two situations (in the presence of activated carbon and without the adsorbent). As shown, comparing the membrane fluxes of two bioreactors indicates that the flux of membranes decreased gradually. Furthermore, the decline of this flux is sharper in the early hours; however, the flux reduction trend is almost constant after a while. Moreover, in the bioreactor containing the activated carbon adsorbent, the flux drop is far less; it can be due to the formation of another bed for the growth of microorganisms. The presence of a variety of organic and inorganic salts in the pulp industry wastewater, as well as

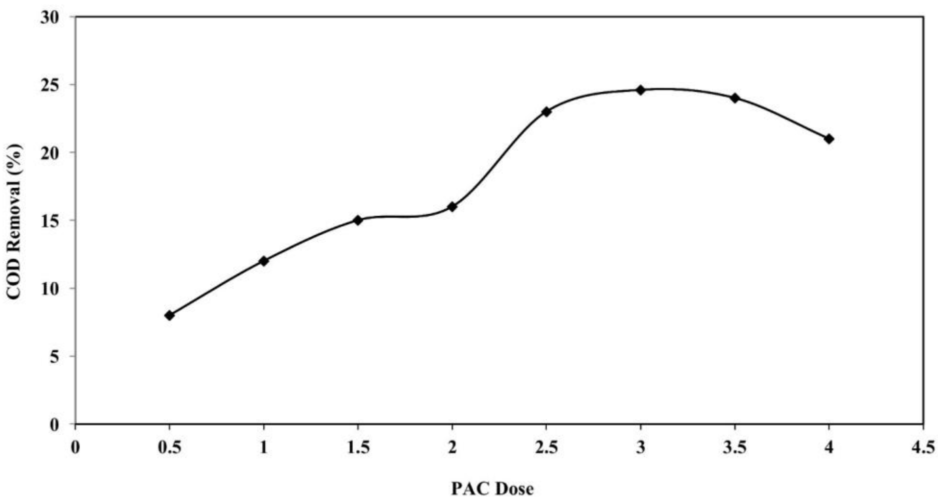

Figure 3. COD removal at different PAC doses. *Note*. COD: Chemical oxygen demand; PAC: Powdered activated carbon

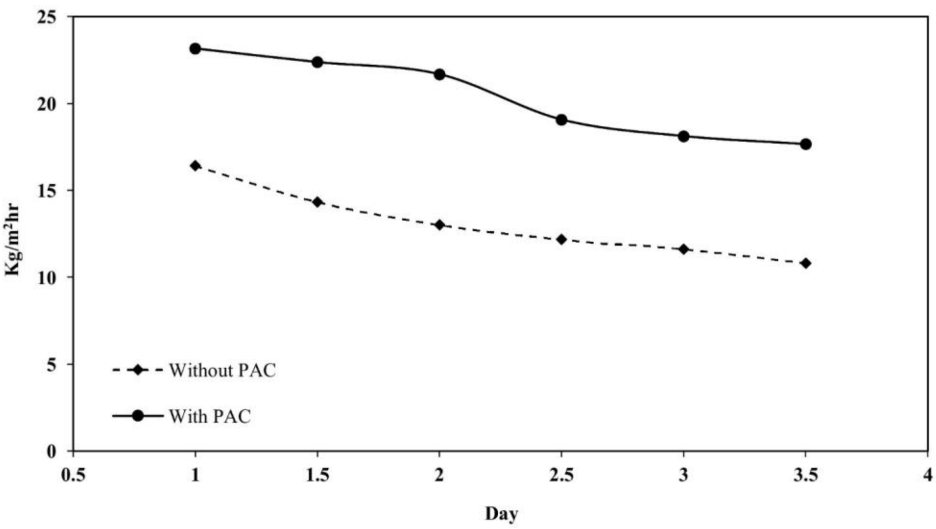

Figure 4. The flow rate in the presence and absence of the adsorbent

the presence of a variety of bacterial agents in activated sludge, strongly influences the tendency for membrane fouling. Therefore, all three types of organic, inorganic, and biological fouling simultaneously occur on the membrane surface, and the severity of this fouling increases over time. As the bioreactor activity increases, the intensity of this fouling increases, leading to a reduction in the flux. This implies that the thickness of the biofilm layer formed on the membrane surface and the mass transfer resistance increase, ultimately reducing the membrane flux. The air compressor creates a turbulent stream; then, the PACs in the flow collide with the membrane and prevent microorganism accumulation on the surface. Hence, the existence of PAC in the process inhibits the fouling and flux drop of the membrane, improving membrane performance in the treatment system.

3.5. COD removal and MLSS content in MABR

Figure 5 shows the COD removal and the MLSS changes in both bioreactors with and without activated carbon adsorbents during the 3.5 days of the MABR process. Based on the obtained results, COD removal increased in both systems, but this increase was greater in the adsorbent-containing system. Thus, the presence of activated carbon as an adsorbent is the cause of the increase in the concentration of MLSS, thus removing COD. One of the benefits of using adsorbent is the enhancement of the MLSS of the system. Not all MLSS increases are solely due to the presence of microorganisms; some of them are related to the number of adsorbents. Furthermore, activated carbon affects the microorganisms, and then the membrane increases their efficiency. In addition to improving the performance of the MABR system, it improves the anti-fouling properties of the membrane, which has an effective role in both the flux and the retention of the membrane. In the reactor containing PAC, the highest MLSS reached 7320 mg/L during the treatment period. Adsorbents provide the growth medium for microorganisms, leading to the reaction between microorganisms and organic matter on their surface.

Five sequential regeneration cycle studies were performed to test the ability of regenerated PAC to be reused as an adsorbent in the MABR process and the experimental findings are depicted in Figure 6. Based on the findings, as the number of cycles increased, the

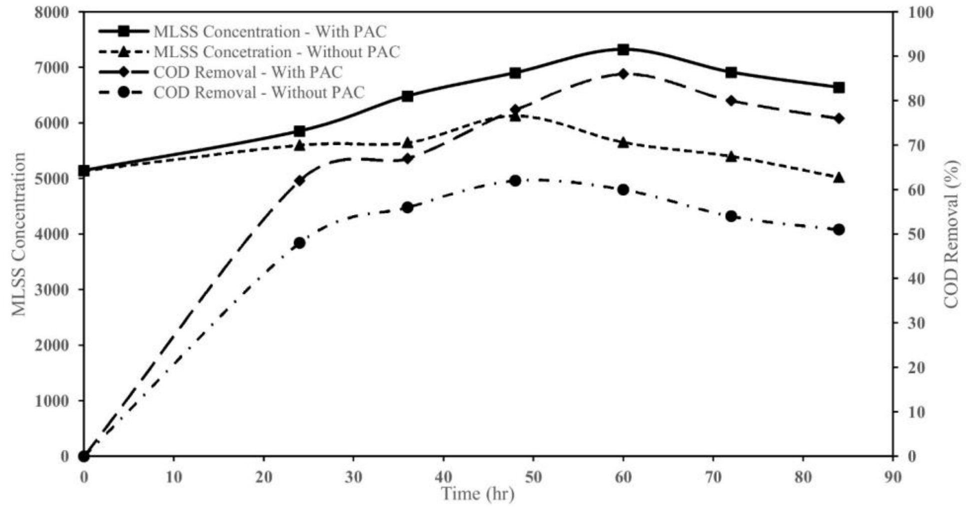

Figure 5. The effect of adsorbent on the COD removal and MLSS concentration. *Note*. COD: Chemical oxygen demand; MLSS: Mixed liquor suspended solid

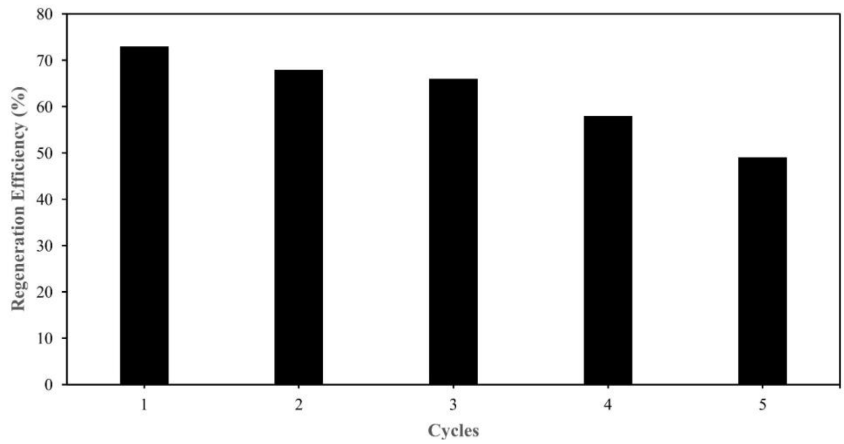

Figure 6. The influence of regeneration cycles on the regeneration efficiency of PAC. *Note*. PAC: Powdered activated carbon

Table 3. Effluent quality with and without PAC

Parameter	Unit	Without PAC	With PAC
COD	mg/L	510	302
BOD_5	mg/L	220	130
pH	-	6.8	6.71
Conductivity	μs/cm	1056	381
TDS	mg/L	634	227
TSS	mg/L	720	343
TKN	mg/L	-	-

Note. PAC: Powdered activated carbon; COD: Chemical oxygen demand; BOD: Biochemical oxygen demand; TDS: Total dissolved solid; TSS: Total suspended solids; TKN: Total Kjeldahl nitrogen.

regeneration efficiency of PAC was reduced due to the accumulation of organic compounds from pulp and paper wastewater on the surface of the activated carbon [34].

In addition, to understand the effect of PAC in the MABR process, the effluent quality characteristics with and without PAC are provided in Table 3.

4. Conclusion

In this study, the MABR was employed to improve the effluent outlet quality. In comparison with the classical biological systems, MABRs represented higher potential in treating pulp and paper industry wastewater. Due to the presence of PAC as an adsorbent, the membrane flux and the accumulation of aerobic microorganisms increased in the bioreactor, while the fouling represented a decrease. Eventually, it was revealed that MABR treatment can enhance COD removal by increasing the MLSS.

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Competing Interests

The authors declare that they have no known competing financial interests, direct or indirect, or personal relationships that could have appeared to influence the work reported in this paper.

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