DOI : [http://doi.org/10.22438/jeb/40/3\(SI\)/Sp-20](http://doi.org/10.22438/jeb/40/3(SI)/Sp-20)

Effects of pentachlorophenol load on PCP, COD and NH₃-N removal in lab-scale multimedia-sequencing batch biofilm reactor treating recycled paper mill wastewater

Paper received: 17. 04. 2018

Revised received: 14. 11.2018

Accepted: 03.01. 2019

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Abstract

Aim: Discharge of improperly treated paper mill wastewater can be dangerous to the environment. The present study aimed to evaluate the effect of PCP load on PCP, chemical oxygen demand and ammoniacal nitrogen removal from recycled paper mill effluent in a lab-scale MM-SBBR system.

Methodology: In this study, the effects of pentachlorophenol (PCP) load were investigated and monitored for a year using lab-scale multimedia-sequencing batch biofilm reactor for the removal of PCP, chemical oxygen demand (COD) and ammoniacal nitrogen (NH₃-N) from recycled paper wastewater. An 18 L reactor composed of Perspex column packed with cylindrical plastic media filled with granular activated carbon (GAC; coconut shells) was used to treat real recycled paper mill effluent with initial COD range of 750–1900 mg l⁻¹ at a fixed hydraulic retention time of 24 hr. Three different PCP concentrations (5, 10 and 50 mg l⁻¹) were spiked in the real effluent to evaluate the reactor's performance.

Results: PCP removal efficiency decreased from 94% to 61% when PCP concentrations were increased from 5 mg l⁻¹ to 50 mg l⁻¹. In addition, more than 88% of COD was removed, and NH₃-N was completely eliminated. Both COD and NH₃-N showed stable removal throughout the study.

Interpretation: The overall performance results confirmed that the combination of biofilm and GAC was the best configuration for wastewater treatment as this combination showed good performance efficiency and stable treatment process under stern organic load fluctuations. Therefore, this hybrid system is recommended for the treatment of pulp and paper mill effluents.

Keywords: Multimedia, Pentachlorophenol, Recycled paper mill wastewater, Sequencing batch biofilm reactor

Citation: Muhamad, M.H., S.R.S. Abdullah, H.A. Hasan and N.I. Ismail: Effects of pentachlorophenol load on PCP, COD and NH₃-N removal in lab-scale multimedia-sequencing batch biofilm reactor treating recycled paper mill wastewater. *J. Environ. Biol.*, **40**, 556-562 (2019).

Introduction

Generation of effluent containing complicated compound mixtures is a result of utilisation of lignocellulosic components of plants and chemicals in the manufacturing process of pulp and paper. Chlorophenols produced during bleaching process become the major environmental pollutants from pulp and paper mill. Pentachlorophenol (PCP) is a type of chlorophenol that has been widely used as wood preservatives, pesticides, herbicides, disinfectants and biocides. The United States Environment Protection Agency has listed PCP as highly toxic to the environment, and the safe permissible limit of PCP in water is $0.30 \mu\text{g l}^{-1}$ (Karn *et al.*, 2010; Elsayed, 2017). Unfortunately, pulp and paper mill effluent still exceed the permissible limit of PCP even after industrial-scale treatment (Raj *et al.*, 2005). Moreover, PCP is difficult to degrade because of its stable aromatic ring system and high chloride content; thus, this compound persists in the environment (Saber and Crawford, 1985; Cho, 2017). Given its persistence and bioaccumulation, PCP has been detected in surface water and sediments, rainwater, drinking water, aquatic organisms, soil and food, as well as in human milk, adipose tissue and urine (ATSDR, 2007). The removal and treatment of PCP is, therefore, of high interest. At present several methods are used for PCP treatment, including sorption (He *et al.*, 2006), biodegradation (Karn *et al.*, 2010), oxidation (Xue *et al.*, 2009), photocatalytic degradation (ThanhThuy *et al.*, 2013) and electrochemical methods (Hanna *et al.*, 2005). Amongst them, biological treatment system is often used for PCP degradation (Karn *et al.*, 2011; Muhammad *et al.*, 2012; De Morais *et al.*, 2014; Vacondio *et al.*, 2015; Wang *et al.*, 2016; Rehman *et al.*, 2017). However, this method alone is insufficient due to low degradation rate (Thompson *et al.*, 2001; Yasin and Usman, 2017). Thus, a more effective method for PCP degradation is needed.

Compared with conventional treatment technology, biofilm-based treatment technology has offered a number of advantages in the wastewater treatment process (Bajaj *et al.*, 2008; Farhadian *et al.*, 2008; Farraji *et al.*, 2017). This kind of treatment technology allows the attachment of microorganisms over solid surfaces, resulting in highly active biomass concentrations and high reaction rates. Moreover, biofilm reactor also offer advantages such as minimal energy and land usage, low sensitivity to toxicity and sludge production, flexible operation and high volumetric loading (Rodgers and Zhan, 2003; De'nan *et al.*, 2017). To increase overall treatment efficiency, the combination of biofilm systems and granular activated carbon (GAC) adsorption has been applied to the treatment of industrial paper mill wastewater (Muhamad *et al.*, 2013; Muhamad *et al.*, 2015; Aziz and Hanafiah, 2017). Adsorption of organic matter is possible in GAC as GAC possesses a highly porous and rough medium, providing an ideal surface for the attachment and growth of active biomass. One of the major limitations of GAC adsorption pertains to organic matter saturation. Once GAC becomes fully

saturated with organic matter, it is considered to be 'exhausted'. GAC exhibits limited service life of approximately 6–12 months (Simpson, 2008). To maximise GAC lifetime, the biofilm should not only prevent GAC from becoming saturated but also regenerate it after a period of net adsorption (Abromaitis *et al.*, 2017). In bioregeneration, microorganisms regenerate the surface of activated carbon using organic substrate as a source of food and energy. This process leads to renewal of adsorptive potential, increased stability of the system and prolonged duration of service by GAC (Nath and Bhakhar, 2010). Thus, this system represents a good option for pulp and paper mill wastewater treatment.

This paper reports the application of sequencing batch biofilm reactor (SBBR) for recycled paper mill wastewater treatment using cylindrical plastic media filled with GAC (coconut shells) as multimedia (MM). The present study aimed to evaluate the effect of PCP load on PCP, chemical oxygen demand and ammoniacal nitrogen removal from recycled paper mill effluent in a lab-scale MM-SBBR system. The novelty of this study is the use of MM, which consists of two types of biofilm supporting media inside the SBBR for the treatment of real recycled paper mill wastewater. Furthermore, very few studies have evaluated the effect of PCP load on SBBR performance in treating real wastewater.

Materials and Methods

Collection of wastewater sample: Waste water samples were collected from Muda Paper Mill Sdn. Bhd., a recycled paper mill located in the western part of Peninsular Malaysia that relies on 100% recovered paper as fibre raw material. Physico-chemically treated effluent discharged from a dissolved air flotation (DAF) unit of an existing wastewater treatment plant at Muda Paper Mill Sdn. Bhd was used in this study. The effluent was collected twice a month and kept in a refrigerator at 4°C for further use. Table 1 presents the characteristics of DAF effluent from wastewater treatment plant.

Biomass: In the lab-scale bioreactors, biomass obtained from activated sludge process of same recycled paper mill served as an inoculum. The biomass was inoculated into the lab-scale bioreactors at 1:10 ratio to the total reactor volume, resulting in an initial mixed liquor suspended solid (SS) concentration of 2000

Table 1: Characteristics of DAF unit effluent

Parameter	Value
pH	6.4 ± 0.3
COD (mg l^{-1})	1048 ± 240
$\text{NH}_3\text{-N}$ (mg l^{-1})	4.1 ± 0.1
$\text{PO}_4^{3-}\text{-P}$ (mg l^{-1})	0.03 ± 0.02
SS (mg l^{-1})	103 ± 43

Values are mean of replicates $\pm$ SD

mg l⁻¹ in the lab-scale bioreactor.

Lab-scale bioreactor and operating schedule: The experiment was conducted using a lab-scale MM-SBBR with a total volume of 18 litre (Fig. 1). The bioreactor was operated over a 400-day period. The operating conditions in the bioreactor experiments are listed in Table 2. The bioreactor column, which composed of Perspex and was packed with cylindrical plastic media filled with GAC, was operated in batch mode under aerobic conditions for the recycled paper wastewater.

The fixed-bed material used was commercially available GAC, a coconut shell-based activated carbon with a density of 560 g l⁻¹. This fixed-bed material served as the adsorption medium and also supported biofilm formation. A diffuser was used to produce fine air bubbles for aeration in the liquid phase over MM. To maintain a dissolved oxygen concentration above 2 mg l⁻¹, sufficient aeration was provided from the bottom of the bioreactor.

A total of 12.6 l of recycled paper wastewater was filled in the bioreactor and operated at an optimal hydraulic retention time of 24 hr consisting of four consecutive phases: filling and reaction (aerated; duration: 23 hr), settling (non-aerated; duration: 0.5 h) and drawing (non-aerated; duration: 0.5 hr). In this study, nutrient addition was required because calculated nitrogen (N) and phosphorus (P) levels of DAF effluent were inadequate for microbial growth (Muhamad et al., 2015). Thus, ammonium sulphate and trisodium phosphate were added. Three different

PCP concentrations (5, 10 and 50 mg l⁻¹) were spiked in the real effluent once a cycle to evaluate the reactor's performance.

Analytical procedure: Physico-chemical analysis of wastewater: The physical and chemical properties of wastewater were characterised. Prior to COD, NH₃-N and PO₄³⁻-P analyses, Whatman-type cellulose nitrate membrane filters (0.45 µm) were used to filter the liquid samples following Hach method using a spectrophotometer (Hach DR3900, USA). SS analysis was performed according to standard methods (APHA, 2005). The pH of water was measured using a portable pH meter (Hach sensION+, USA). A standard curve calibration using HPLC with a Genesis C18 column (250 mm × 4.6 mm, 4 µm) and a UV detector (Agilent 1100 Series, USA) set at 305 nm was used to determine PCP concentration. The mobile phases used for HPLC were 0.01 M phosphoric acid and acetonitrile. The eluent flow rate was 1 ml min⁻¹, and the column temperature was 35 °C. Chromatography was performed with a gradient from ACN:H₃PO₄, 20:80 to 45:55 in 7.5 min and then to 80% ACN after 2 min. The performance of the reactor was evaluated by estimating the substrate (COD, NH₃-N or PCP) removal efficiency of each parameter by comparing the initial substrate concentration in the feed with that in the reactor outlet for each substrate.

Statistical analysis: All experimental data were subjected to analysis of variance (ANOVA) using SPSS program, version 21.0 (SPSS Inc., Chicago, IL). One-way ANOVA test was used to explore the effects of different PCP loads on the performance of

Fig. 1 : Laboratory-scale MM-SBBR system.

Table 2: Operating conditions for bioreactor experiment

Reactor internal diameter (m)	0.24
Height of the reactor (m)	0.4
H/D ratio	1.7
Total volume of the reactor (l)	18
Total working volume (l)	16
Supported media	MM=GAC+plastic media
MM filling ratio (%)	20
Volume occupied by MM (l)	3.2
Wastewater volume (l)	12.6
Inoculated biomass volume (l)	1.8
Initial MLSS conc. (additional biomass) (mg l ⁻¹)	2000

bioreactor with a Post hoc multiple comparison of means using Tukey's method. Statistical significance was considered at $p \leq 0.05$.

Results and Discussion

Start-up of Lab-Scale MM-SBBR: The PCP, COD and NH₃-N concentrations in the influent and effluent of lab-scale MM-SBBR were measured and are shown in Table 3 along with the respective removal percentages. The variations of influent and effluent and their removal efficiency are shown in Fig. 2–4. During reactor start-up (day 0–97), the wastewater was fed into the lab-scale reactor to allow biomass adaptation to the environment. During the biomass acclimatisation process, the average COD removal efficiencies were about $97\% \pm 4\%$ (day 0–38) and $97\% \pm 1\%$ (day 39–97), as reported in Table 3. As shown in Fig. 4, the bioreactor was supplied with 50 ± 2 mg l⁻¹ N source between day 0 and day 38 with average NH₃-N removal efficiency up to $60\% \pm 8\%$. To achieve a nutrient-spike with a BOD:N:P ratio of 100:5:1, the bioreactor was fed with 25 ± 1 mg l⁻¹ N source in the following days; afterwards, the NH₃-N concentrations in the influent were insufficiently removed (Metcalf and Eddy, 2003). As shown in Fig. 4, decreasing NH₃-N addition (after 38 days) also decreased the effluent NH₃-N sharply to 0.3 mg l⁻¹ from influent of 26 mg l⁻¹ (99% removal), demonstrating that NH₃-N removal decreased with increasing N load. In this case, a N concentration of not more than 50 mg l⁻¹ in the bioreactor is necessary to obtain sufficient biodegradation. After 38 days, COD and NH₃-N removal efficiencies were consistent daily, indicating a fully acclimatised system. To achieve stable treatment, the biomass was acclimatised for 3 months prior to MM and PCP addition. Initially, the reactor was operated with a PCP load of 5 mg l⁻¹ to test the performance of the bioreactor.

Effects of PCP load on PCP, COD and NH₃-N removal: PCP serves as the reference compound for recalcitrant organics in wastewater. Highly chlorinated organic nature causes this compound to become very resistant to microbial degradation (Rahman *et al.*, 2007; Lijie and Feng, 2018). Fortunately, our lab-scale MM-SBBR was capable of removing this biocide. The effect

of PCP load on PCP removal (day 98–418) is shown in Fig. 2. The PCP loads (in mg l⁻¹) are shown in the figures as boxed sections. PCP levels were measured after media was added to the system (day 98). As shown in Fig. 2, after being treated with the lab-scale MM-SBBR (PCP load of 5 mg l⁻¹), the concentration of PCP was found to be very low in the effluent with a removal percentage between 79% and 100%. Interestingly, from day 98 to day 114, it was almost impossible to detect any PCP compound in the effluent. This phenomenon is possible as GAC and biomass managed to absorb and provide biodegradation platform for PCP degradation (Wilson *et al.*, 1998; Halin *et al.*, 2017).

The easily degradable substances were expected to allow growth of biomass and provide sufficient energy needed to metabolise the non-readily degradable components (Muhamad *et al.*, 2012; Zaidi *et al.*, 2017). The lower PCP concentration in the effluent than in the influent indicates that combination of biofilm and MM had effectively removed the PCP compounds in the MM-SBBR. By increasing PCP load from 5 mg l⁻¹ to 10 mg l⁻¹ (day 198–358), the PCP removal efficiency started to decrease from $94\% \pm 7\%$ to $77\% \pm 11\%$ perhaps due to increased toxicity and decreased biomass growth. A decrease in removal efficiency was observed when PCP load was further increased to 50 mg l⁻¹, indicating that the percentage of PCP removal for this process was dependent on the PCP load. PCP load acts inversely proportional to biodegradation. Statistically, each PCP load showed a significant difference ($p \leq 0.05$) in terms of PCP removal efficiency, as evaluated using Tukey's method as part of one-way ANOVA.

Fig. 2: Variation of influent and effluent PCP and PCP removal efficiency for PCP loads of 5–50 mg l⁻¹.

Table 3: Effects of PCP loads in influent on removal efficiencies of COD, PCP and $\text{NH}_3\text{-N}$

Operational periods (days)	Configuration	Influent			Effluent			Removal		
		COD (mg l^{-1})	PCP (mg l^{-1})	$\text{NH}_3\text{-N}$ (mg l^{-1})	COD (mg l^{-1})	PCP (mg l^{-1})	$\text{NH}_3\text{-N}$ (mg l^{-1})	COD (%)	PCP (%)	$\text{NH}_3\text{-N}$ (%)
0–38	Without EMM	1006 (11)		50 (2)	33 (37)		20 (4)	97 (4)		60 (8)
39–97	Without EMM	1034 (26)		25 (1)	32 (9)		0.3 (0.2)	97 (1)		99 (1)
98–197	With EMM	943 (140)	5	25 (1)	62 (22)	0.3 (0.4)	0.1 (0.2)	93 (3)	94 (7)	99.5 (0.9)
198–358	With EMM	1108 (265)	10	25 (1)	69 (18)	2 (1)	0.1 (0.1)	94 (2)	77 (11)	99.6 (0.6)
359–418	With EMM	1171 (459)	50	25 (1)	110 (28)	19 (4)	0.1 (0.2)	90 (2)	61 (8)	99.7 (0.7)

Values inside parentheses indicate the standard deviation of each mean pollutant concentration.

Fig. 3: Variation of influent and effluent COD and COD removal efficiency during acclimatisation and for PCP loads 5–50 mg l^{-1} .

Fig. 3 depicts the relationship between PCP loads and COD removal. The maximum COD influent concentrations for all samples were less than 1909 mg l^{-1} , and the effluent concentrations were below 152 mg l^{-1} as observed in Fig. 3. At day 98–197 (PCP load of 5 mg l^{-1}), the COD removal efficiency was 88–98% (Fig. 3). At day 198, the PCP load increased to 50 mg l^{-1} , but no significant difference in COD removal was observed as PCP load increased. As shown in Fig. 3, the bioreactor efficiency in reducing COD is remarkable, despite high influent COD. At day 274–289, high carbon source containing 1336–1622 mg COD l^{-1} was supplied into the bioreactor. Fig. 3 shows that the bioreactor was able to endure the increasing COD. Thus, the performance and stability of bioreactor were not affected by high COD levels. Hence, the MM-SBBR was determined to be relevant for the treatment of high CODs (up to 1300 mg l^{-1}). No significant difference in COD removal was observed when increasing PCP load to 50 mg l^{-1} at day 359–418. The experimental data indicate

Fig. 4 : Variation of influent and effluent $\text{NH}_3\text{-N}$ and $\text{NH}_3\text{-N}$ removal efficiency during acclimatisation and for PCP loads of 5–50 mg l^{-1} .

that the reactor performance with respect to COD removal was slightly influenced by PCP load. Although the differences amongst PCP loads regarding COD removal were subtle, the differences between each of the PCP load were statistically significant ($p \leq 0.05$) when analysed using Tukey's method from one-way ANOVA, except for the difference between 5 mg PCP^{-1} and 50 mg PCP^{-1} and also between 10 mg PCP^{-1} and 50 mg PCP^{-1} .

Given that the wastewater contained a limited amount of nutrients, N emissions from the pulp and paper industry were not very significant. These nutrients are important for microbial growth and BOD reduction. Maintaining BOD level within an appropriate range is important. Thus, the addition of external N to the effluents is needed (Sdguide org, 2008). The relationship of PCP load on $\text{NH}_3\text{-N}$ removal is shown in Fig. 4. As shown in figure, the $\text{NH}_3\text{-N}$ concentration was almost completely removed whilst

stabilising $\text{NH}_3\text{-N}$ removal efficiency between 98% and 100% when increasing PCP load from 5 mg l^{-1} to 50 mg l^{-1} . In conclusion, the bioreactor showed excellent $\text{NH}_3\text{-N}$ removal efficiency with increasing PCP load, but the PCP loads could not make any significant change on $\text{NH}_3\text{-N}$ removal in the bioreactor. Statistically, no significant difference ($p \leq 0.05$) in $\text{NH}_3\text{-N}$ removal efficiency was observed amongst the PCP loads, which was confirmed by Tukey's method from one-way ANOVA.

Taken together, our lab-scale MM-SBBR system was able to effectively treat recycled paper industry wastewater under different PCP loads. The PCP removal efficiencies decreased from 94% to 61% when PCP concentrations were increased from 5 mg l^{-1} to 50 mg l^{-1} . The bioreactor was able to cope with the subjected PCP loads and manage to achieve nearly complete $\text{NH}_3\text{-N}$ (96%–100%) and COD (88%–98%) removal during different PCP loads. The stability and efficiency of bioreactor at different PCP loads indicate its potential in the treatment of pulp and paper mill effluents.

Acknowledgment

The authors would like to thank Universiti Kebangsaan Malaysia for supporting this research project under grant ML-2018-003.

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