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POST-TREATMENT OF PALM OIL MILL EFFLUENT USING ZEOLITE AND WASTEWATER

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ABSTRACT

The palm oil mill effluent (POME) is one of the most important ecosystems hazard and can become a crucial environmental burden if discharged without any treatment to nature. The present study aimed to develop a fast method for post-treatment of POME. To enhance treatment process, the domestic wastewater (DWW) and zeolite were added to the sequencing batch reactor (SBR) as the available microbial source and new adsorbent, respectively. The results indicate that the chemical oxygen demand (COD), biochemical oxygen demand (BOD), total suspended solids (TSS), ammonia nitrogen (AN) and colour removal rates were in the range of 95.34%-98.31%, 88.79%-91.44%, 95.47%-98.95%, 96.19%-98.30% and 56.94%-81.64%, respectively. Moreover, SBR with both DWW and zeolite addition was able to remove high percentage of all pollution compared to only DWW addition (with a removal percentage of 77% TSS, 74% COD, 76% colour and 90% AN) or zeolite (23% TSS, 10% COD, 9.6% colour and 80% AN). The response surface methodology (RSM) was used to elucidate response surface and optimise the independent variables. The highest desirability of POME treatment (0.988) was achieved in optimum operation conditions. Under these conditions, COD, BOD, colour, AN and TSS removal rates were 96.80%, 90.1%, 69.90%, 98.20% and 97.20%, respectively.

Keywords: zeolite, wastewater, palm oil mill effluent, adsorption, sequencing batch reactor.

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INTRODUCTION

The industry of palm oil has rapidly developed over the last decades. In Malaysia, the palm oil is considered as a major part of agro-economy with a production of 39% in the world (Moradi *et al*., 2015; Wong *et al*., 2015). However, a great amount of palm oil mill effluent (POME) is generated due to high production of palm oil. Ng *et al*. (2012) indicated that with production of 94 million tonnes of fresh fruit bunch (FFB), more than 60 million tonnes POME is produced. The POME with high total solids (43 635 mg litre^{-1}), average chemical oxygen demand (COD) and biochemical oxygen demand (BOD) of 70 000 and 30 000 mg litre^{-1}, respectively can be considered as the main reason for ecosystem and environmental hazard if it is discharged without any treatment (Chan *et al*., 2012; Ma *et al*., 1993). Liew *et al*. (2015) showed that COD, BOD, total suspended solids (TSS), and colour are the most serious contaminants in POME. A range of BOD/COD ratio between 0.29 and 0.62 indicates that the POME is a highly polluted agro-industry

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wastewater that should be treated using biological methods (Mohan and Karthikeyan, 1997; Chan *et al*., 2010; Vijayaraghavan *et al*., 2007; Metcalf and Eddy, 2014; Khemkhao *et al*., 2015).

Generally, several treatment systems have been applied to improve POME quality such as sequencing batch reactor (SBR), up-flow anaerobic sludge bioreactor, added chemical and biochemical productions, enhanced waste stabilisation ponds (aerated lagoon), membrane bioreactor, waste stabilisation pond, extended aeration activated sludge and membrane filters (Aziz *et al*., 2011a, b). However, the open tank digester and ponding methods are the most popular and commonly used systems to treat POME. Although the ponding systems were suggested as the cost-effective techniques for treatment of POME, several disadvantages were observed in these systems such as the big size of the digester, off odour, long treatment duration, insufficient effluent quality, colour, large variation in effluent quality, and large footprint. In order to overcome the problems and improve POME treatment, a high-rate anaerobic bioreactor system has been recommended with smaller foot print and high efficiency such as up-flow anaerobic sludge bioreactor (Najafpour *et al*., 2006), improved anaerobic baffled bioreactor (Setiadi and Husaini, 1996; Faisal and Unno, 2001) and anaerobic fluidised bed bioreactor (Borja and Banks, 1995). Yacob *et al*. (2006) reported that compared to conventional methods, the anaerobic bioreactors remove more pollution at shorter hydraulic retention time and provide better treatment efficiencies.

Although, the anaerobic pond is one of the most commonly used methods for POME treatment, the effluent of this method can hardly fulfil the standard level of discharge defined by the Malaysian Department of Environment (DOE) (Chan *et al*., 2010). In order to meet the DOE levels, an appropriate post-treatment is essential for POME discharge. Ho and Tan (1983) reported that the aerobic technologies should be applied as a post-treatment system to reduce pollution of POME within the standards rate. Nasrullah *et al*. (2017) proposed a post-treatment method for colour removal of POME using electrocoagulation. They reported that this method was effective and slightly expensive for POME treatment. Moreover, phytoremediation was recommended as another laboratory-scale and plant-based post-treatment that is often used for anaerobically treated and diluted POME (Darajeh *et al*., 2014). For treatment of the industrial and municipal wastewater, the anaerobic-aerobic processes using reactors have been recommended as an effective method with less sludge production, low energy and higher treatment efficiency (Jenícek *et al*., 1999; Garbossa *et al*., 2005; Chan *et al*., 2010; 2011). Although, the conventional anaerobic-aerobic methods have been able to successfully treat the POME, their low organic loading rate (OLR), long hydraulic retention time (HRT), and large space requirement are the disadvantage of these methods. Chou *et al.* (2016) reported that only a few studies are available in literature related to aerobic post-treatment of anaerobically digested POME and more effort is required to understand the performance of these treatments.

Since the conventional method have no high potential to treat POME, various methods have been proposed to solve this problem such as adsorption, advanced oxidation processes, membrane filtration and coagulation/flocculation. Recently, SBR with a simple configuration of the tank was recommended as a new system with the low-space requirement and effective technique for treatment of industrial and municipal wastewater (Aziz *et al*., 2012). Ahmad *et al*. (2003) indicated that flocculation is one of the effective method to remove turbidity in POME. Moussavi *et al.* (2011) showed that zeolite has high ability in coagulation and flocculation of suspended particle. Recently, SBR with a simple configuration of the tank was recommended as a new system with the low-space requirement and effective technique for treatment of industrial and municipal wastewater (Aziz *et al*., 2012). Compared to other biological treatment methods, the SBR has greater process flexibility to treat wastewater and leachate (Lim *et al*., 2014; Mojiri *et al*., 2014). This treatment is highly recommended as a cost-effective eco-friendly green technology. As shown in *Table 1*, the SBR with high efficiency in COD, TSS, and BOD_5 removal has been employed for POME treatment in previous studies. Moreover, the SBR with activated sludge has been used frequently in a laboratory scale for POME aerobic treatment (Chin *et al*., 1987; Ma and Ong, 1988; Chin *et al*., 2013; Chou *et al*., 2016). Although the efficiency of the SBR method is close to DOE standards, operational cost, activated sludge requirement, and extended period of treatment are drawbacks of this system for POME treatment (Chan *et al*., 2011). Therefore, a cost-effective and fast method is necessary to improve removal efficiency of aerobic SBR system.

The raw POME, acidic media with a temperature of 80ºC-90ºC (Chin *et al*., 2013), will cause low content of microbial population. However, the low biodegradability of raw POME makes the digestion process longer and decreases pollutant removal efficiency (Oswal *et al*., 2002). Zahrim *et al*. (2009) indicated that the environmental adaptation of microorganisms supplied from activated sludge is a time-consuming process with an average of 60 days. Moreover, low efficiency and high energy consumption were reported when extending the period of treatment (Fun *et al*., 2007; Vijayaraghavan *et al*., 2007; Chou *et al*.,

TABLE 1. EFFECTIVENESS OF THE SBR TREATMENT FOR POME

Parameters	Fu (2007)	Chin and Ma (1987)	Zahrim *et al.* (2009)	Chan (2010)	Chan *et al.* (2011)	Chou *et al.* (2016)
Post-treatment	SBR activated sludge	SBR activated sludge	SBR activated sludge	SBR activated sludge	SBR activated sludge	SBR activated sludge*
Pre-treatment	Anaerobic digested	Adsorbent pre-treated	Anaerobic digested	Anaerobic digested	Anaerobic digested	Anaerobic digested
Influent COD (mg $litre^{-1}$)	-	1 550	1 141	13 650	13 532	10 030
Influent BOD_5 (mg $litre^{-1}$)	-	700	-	1 355	1 355	-
Biodegradability	-	0.45	-	0.1	0.1	-
COD removal (%)	82	31-50	70	95-96	63-86	75-93
BOD_5 removal (%)	-	50-70	-	97-98	65-87	-
TSS removal (%)	62	-	-	98-99	79.2-89.1	81-95
Colour removal (%)	-	50	41	-	-	-
Temperature (°C)	-	42	-	28	50	30
pH	-	-	7.7 - 8.3	7.4	7.4	7.7
Treatment time	14 days	25 days	60 days	30 days	> 11 days	7.2-18 days

Note: *Equal inoculum used as activated sludge.
SBR - sequencing batch reactor; COD - chemical oxygen demand; BOD - biochemical oxygen demand; TSS - total suspended solids; POME - palm oil mill effluent.

2016). Many studies reported that a cost-effective natural adsorbent can be used for enhancing the efficiency of POME decontamination and biological treatment of domestic wastewater (DWW) (Aziz *et al*., 2011b; 2012; Ismail *et al*., 2013). The combination of municipal wastewater for enhancing microbial degradation of pollutants, such as BOD_5, COD, TSS and AN, is reported frequently for landfill leachate (Mojiri *et al*., 2014; Aziz *et al*., 2011a, b). The POME is a non-toxic agricultural effluent with an extremely high concentration of BOD_5, COD, TSS and colour and low concentration of microbial community. Therefore, municipal or DWW as a low cost material with high microbial source can be used for the treatment of POME. However, a gap of knowledge can still be founded in the literature, particularly to decrease time and cost of POME treatment in SBR system as well as activated sludge requirement (Mansor *et al*., 2017).

The aim of this study is to develop a fast method to treat POME and improve removal efficiency of aerobic SBR technique. The DWW and zeolite were employed as an available microbial source and new adsorbent, respectively to enhance the SBR treatment process. To achieve a high efficiency of aeration, a new aeration system was developed for the SBR through a couple bulb bottom with opposite direction of aeration. In this reactor, the couple bottom aeration was employed for both mixer and oxygen supplier. The removal of COD, BOD, TSS, AN and colour were measured to evaluate removal efficiency of the suggested technique. The central composite design (CCD) and response surface methodology (RSM) were used to elucidate response surface and optimise the independent variables including contact time, aeration rate and DWW/POME ratio, as well as their dependent variables.

MATERIALS AND METHODS

POME Sampling

In the present study, eight raw POME samples were collected during 15 October 2014 to 18 May 2015 from an anaerobically ponding treatment system of United Oil Palm (UOP) located at 100° 30′ 27.90″ E and 5° 9′ 13.63″ N. As shown in *Figure 1*, the pond number 8 which is located at the end of the POME treatment system (before algae pond), was selected for the data sampling. The collected samples were transported to a cooling room with temperatures of 4°C to minimise the chemical and biological reactions. The characteristics of the collected POME are listed in *Table 2*.

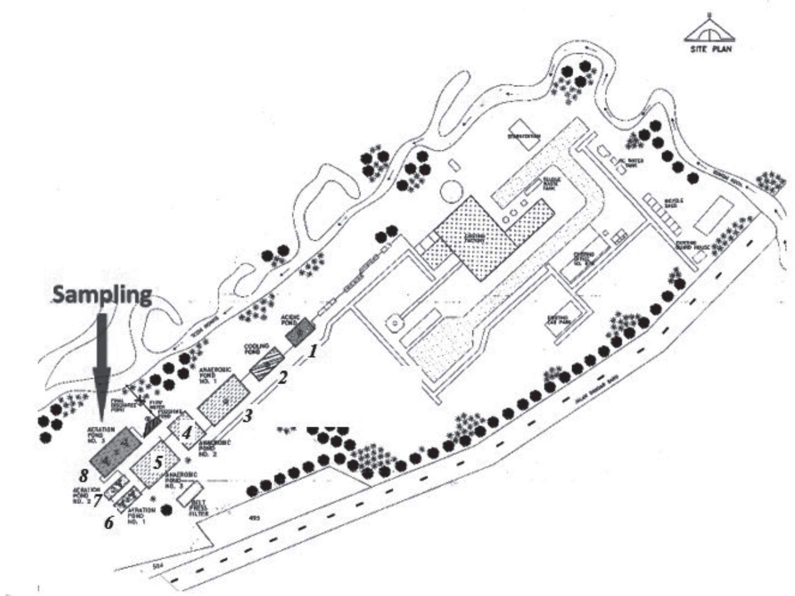


Figure 1. Anaerobically treated palm oil mill effluent (POME) sampling (pond number).

TABLE 2. CHARACTERISTICS OF POME, DWW AND STANDARD DISCHARGE LIMIT FOR POME

Parameters	POME			DWW			Standard discharge limit for POME
	Min	Max	Ave	Min	Max	Ave	
Temperature	28.5	32	30.2	29	31	29.3	45.0
pH	6.7	7.6	7.32	6.6	7	6.8	5.0-9.0
Total suspended solids (mg litre^{-1})	2 890	4 570	4 310	4.7	5.2	5.0	200.0
Colour (ADMI)	1 750	3 350	2 550	68	75	77	100.0
BOD_5 (mg litre^{-1})	995	1 685	1 230	91	102	100	20.0
COD (mg litre^{-1})	8 225	13 555	11 090	225	260	240	50*
BOD_5/COD	0.1	0.116	0.11	0.38	0.46	0.41	-
Total phosphorus (mg litre^{-1})	97	153	124.5	7.2	9.8	9	-
Ammonia nitrogen (AN) (mg litre^{-1})	82	107	94.5	11	21	14	10.0
Total nitrogen (mg litre^{-1})	275	440	360	16.8	30.5	22.9	10.0
Total iron (mg litre^{-1})	0.34	0.55	0.4	1.39	1.55	1.4	-
Total manganese (mg litre^{-1})	135	153	146.5	5.5	5.8	5.7	-
Total calcium (mg litre^{-1})	38	43	40.9	18.5	23.2	22.1	-
Turbidity (NTU)	4362	790	6012	8	13.3	11.9	-

Note: *Requirement set by the Malaysia Sewage and Industrial Effluent Discharge Standard, Deparment of Environment (DOE).
POME - palm oil mill effluent; DWW - domestic wastewater; BOD - biochemical oxygen demand; Ave - average; ADMI - American Dye Manufacturers Institut; COD - chemical oxygen demand; NTU - nephelometric turbidity units.

Domestic Wastewater (DWW) Sampling

DWW was collected from the Indah Water Konsortium Regional Treatment Plant located at 100° 27′ 10.8″ E and 5° 20′ 25.5″ N. The samples were directly send to a cool room (4°C) and kept in a white high density polyethylene (HDPE) container. Before using the samples, they were shaken until their temperature reached the laboratory temperature (31.2°C). The characteristics of DWW and POME standards discharge suggested by DOE is shown in *Table 2*.

Reactor Characteristics

In this research, a new SBR (new-SBR) was developed to improved removal efficiency of traditional reactors. The new-SBR was composed of a transparent column and a plexiglas plate to observe the settling process. To achieve a high efficiency of aeration, a novel system was developed for aeration of reactors through a couple bulb bottom with opposite direction of aeration. In the new-SBR, the couple bottom aeration was used for both mixer and oxygen supplier (*Figure 2*). No need for any mixing system can be considered as a novelty of the new-SBR. Twenty of the new-SBR reactor were created for experiments and they run at the same time under the same environmental conditions. Aeration was prepared using two air pumps (air volume, 60 litre min^{-1}; LP-60A Model, Yasunaga, Air Pump Inc., China) where each pump was connected to 10 new-SBR using a valve. To adjust the flow rate, a manual air flow meter was used. Easy application and can be simultaneously run is the attraction of this new system.

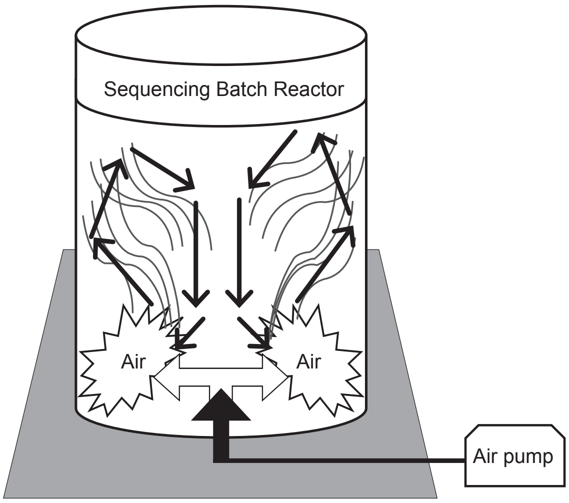


Figure 2. New-sequencing batch reactor without a mixer system.

Zeolite Preparation

In the present study, powdered natural zeolite (PNZ) was used as an adsorbent in the new-SBR. The sieves No. 100 and 200 were chosen for all experiment to provide the suitable powder PNZ in range of 75-150 μm (Mojiri *et al*., 2014). To prepare zeolite for each treatment, at first, 15 g litre^{-1} of zeolite was dried in the oven for 24 hr at 105±2°C. *Table 3* shows the features of the PNZ with the autosorb test. *Figure 3* and *Table 4* indicate the results of the X-ray fluorescence (XRF) and scanning electron microscope (SEM) analyses for PNZ, respectively. The SEM affords a magnified 3D view of natural zeolite surface with a great depth of focus. The SEM images from the surface of zeolite with magnified of 10 000 and 20 000 is shown in *Figure 3*. The pores on the surface of zeolite is a good reason for low density of zeolite and indicates that it is a suitable adsorbent with high-quality.

The energy dispersive X-ray spectroscopy (EDS) was carried out to illustrate the structural compound and elements existing in the zeolite. *Table 5* shows the weight and atomic percentage of natural zeolite elements before treatment. The result indicates that zeolite is an aluminosilicate mineral with a low carbon content in its structure. Furthermore, the Si/Al with a value of 3.41 (19.52/5.71=3.41, *Table 5*) is within the range of 2.7 to 5.3, therefore the natural zeolite applied in this study can be considered as Clinoptilolite (Kesraoui-Okui *et al*., 1994). The percentages of the different structural elements of the zeolite were also determined from the spectrum diagram in *Figure 4*. The spectrum of the natural zeolite shows sharp peaks without shoulders and separated elements in a thin basement line. The results indicate that the carbon content in the zeolite is very low with a little impurity of unknown elements.

TABLE 3. POWDERED NATURAL ZEOLITE CHARACTERISTICS

Parameters	Unit	Value
Single point	$m^2 g^{-1}$	23.76
Multi point BET	$m^2 g^{-1}$	23.88
Langmuir surface area	$m^2 g^{-1}$	36.10
T method micro pore surface area	$m^2 g^{-1}$	10.03
T method external surface area	$m^2 g^{-1}$	13.85
Total pore volume for pore	ml g^{-1}	0.0052

Note: BET – Brunauer-Emmet-Teller.

TABLE 4. RESULTS OF X-RAY FLUORESCENCE (XRF) FOR ZEOLITE

Compounds	Percentage
Silicon dioxide (SiO_2)	73.30
Aluminium oxide (Al_2O_3)	16.69
Calcium oxide (CaO)	2.59
Potassium oxide (K_2O)	2.54
Iron (III) oxide (Fe_2O_3)	2.17
Sodium oxide (Na_2O)	0.52
Magnesium oxide (MgO)	1.53
Titanium dioxide (TiO_2)	0.24
Manganese oxide (MnO)	0.10
Others	0.32

TABLE 5. WEIGHT AND THE ATOMIC PERCENTAGE OF NATURAL ZEOLITE ELEMENTS (SEM-EDS)

Element	Weight % before treatment	Atomic % before treatment
Oxygen (O)	56.72 ± 0.72	66.94
Silicon (Si)	29.04 ± 0.67	19.52
Carbon (C)	3.92 ± 0.44	6.16
Aluminium (Al)	8.16 ± 0.42	5.71
Magnesium (Mg)	2.15 ± 0.26	1.67
Total	100.00	100.00

Note: SEM – scanning electron microscope; EDS – energy dispersive X-ray spectroscopy.

Analytical Method

In this study, the standard methods of the American Public Health Association (APHA) 2017 were used for all tests of wastewater and water. A Multiprobe system of YSI 556 was used to record the total dissolved solids (TDS, mg litre^{-1}), pH, and dissolved oxygen (DO, mg litre^{-1}).

A spectrophotometer (DR 2800, 2100 N and DR 2500 HACH) was used to determine the suspended solids (mg litre^{-1}), contents of colour (Pt. Co.), total phosphorus (mg litre^{-1}), total nitrogen (mg litre^{-1}), AN (mg litre^{-1}), nitrate (mg litre^{-1}), nitrite (mg litre^{-1}), and COD (mg litre^{-1}). The inductively coupled plasma (ICP Varian, OES 715) was utilised to determine the concentration of metallic elements, such as iron (Fe, mg litre^{-1}), magnesium (Mg, mg litre^{-1}), and calcium ($CaCO_3$, mg litre^{-1}).

The New-SBR Operation

The new-SBR system involves the following phases: fill, react, settling, and draw and idle. The durations of fill and mix (20 min), settling (156 min), and draw and idle (8 min) were considered to be fixed in all of the experiments. Various aeration rates of 0.5, 4 and 7.5 litres min^{-1}; different duration for contact times (2 hr, 12 hr and 22 hr); and different rates of DWW to POME (DWW/POME) such as 20%, 50%, and 80% were applied to evaluate treatment of the new-SBR. Twenty Plexiglas beakers with a working volume of 1 litre and a final volume of 1700 ml were used to avoid errors caused by operational or environmental factors. As mentioned, the developed new-SBR was used in all of the 20 reactors. Based on the preliminary experiments and before aeration, 15 g litre^{-1} PNZ was used in each new-SBR for the adsorption of pollutants. The pollution parameters such as BOD, TSS, COD, AN and colour were measured after and before the treatment process. The Equation (1) was used to determine the removal efficiency of the new-SBR systems:

$$Removal\ (\%) = \frac{C_i - C_f}{C_i} \times 100 \qquad \text{Equation (1)}$$

where C_i is initial and C_f is final concentration of pollution parameters.

Data Analysis and Experimental Design

To determine the optimum conditions for the independent parameters and demonstrate the nature of the response surface in the experimental design, both CCD and RSM were utilised in this study. The Design Expert software was used to estimated CCD

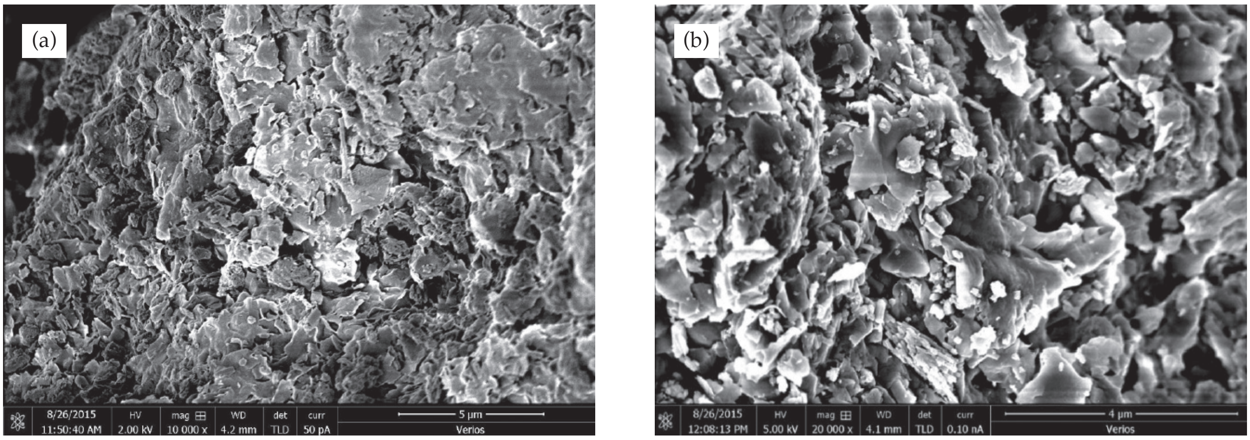


Figure 3. Scanning electron microscope (SEM) images from the surface of zeolite with magnified of (a) 10 000 and (b) 20 000.

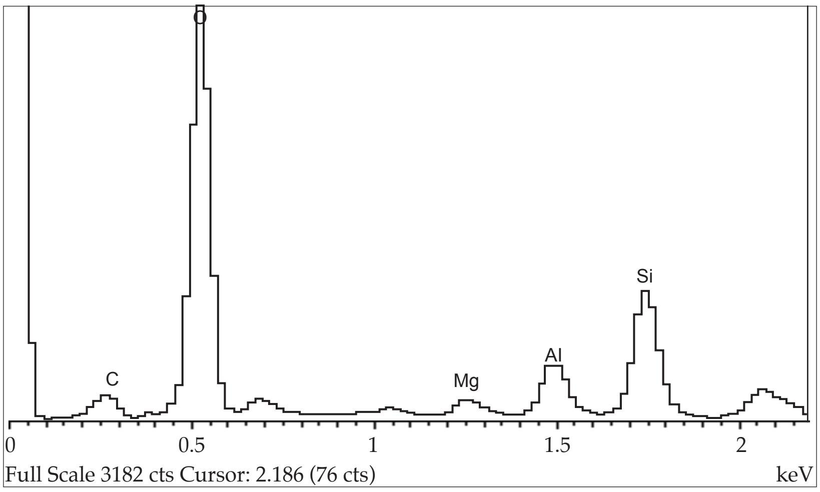


Figure 4. Natural zeolite spectrum (energy dispersive X-ray spectroscopy of the plotted area) before treatment.

and RSM. The polynomial equation with second-order, as expressed in Equation (2), was selected to evaluate the system performance:

$$Y = \beta_0 + \Sigma_{i=1}^{k} \beta_i X_i + \Sigma_{i=1}^{k} \beta_{ii} X^2 + \Sigma_{i i<j}^{k} \Sigma_{j}^{k} \beta_{ij} X_i X_j + \cdots + e \quad \text{Equation (2)}$$

where Y represents the response; the variables are represented by X_i and X_j; β_0 represents a constant coefficient; numbers of studied factors are represented by k; the linear quadratic and second-order coefficients are represented by β_j, β_{ij}, and β_{ii}; and error is represented by e. The provided results were evaluated through analysis of variance (ANOVA) technique. As mentioned, k is the number of variables; thus, k^2 is equal to the factorial point supported by a centre point and $2k$ axial points. To fit the second-order polynomial models and calculate the experimental error, six replicates were used at the central points. Three levels were chosen to evaluate the three operating variables namely, high (+1), medium (0), and low (−1). The RSM and CCD were applied to determine the best value of responses and for optimising the appropriate circumstances of the operation. The contact time (2 hr, 12 hr and 22 hr), aeration rate (0.5, 4 and 7.5 litres min^{-1}), and DWW/POME ratio (20%, 50%, and 80%; V/V) were chosen as independent variables and their response (COD, BOD, TSS, AN and colour) were selected as dependent variables in this study (*Table 6*).

RESULTS AND DISCUSSION

COD, AN, Colour and BOD Removal

Table 2 indicates the maximum, minimum and average values of measured parameters of POME, DWW and standard discharge limit suggested by DOE. Understanding the characteristics of pollutants is crucial in the selection of treatment method. According to Ho and Tan (1983), centrifuge technique (depending on gravity force and operation time) can reduce the total amount of POME pollutants, such as TSS, BOD_5, COD, total nitrogen and AN. Consequently, POME contains high concentrations of real colour particles. Therefore, the parameters of BOD, COD, AN and colour are chosen for discussion in this section. The POME contains high concentrations of TSS (4310 mg litre^{-1}) COD (11 090 mg litre^{-1}) and high intensive colour (2550 Pt. Co.). The value of BOD_5 (1230 mg litre^{-1}) and BOD_5/COD (0.11) show the low biodegradability in the POME (*Table 2*). Metcalf and Eddy (2014) reported that for a wastewater with biodegradability index (BOD_5/COD ratio) bigger than 0.5, the wastewater can be considered for biological treatment. These results indicated that the anaerobic treatment of POME still contains high pollution and this method can hardly reach to the standard level of discharge defined by DOE. Therefore, an appropriate post-treatment is essential in order to meet the DOE levels. Previous

TABLE 6. EXPERIMENTAL VARIABLES AND RESULTS FOR THE NEW- SEQUENCING BATCH REACTOR (SBR)

Run No.	Aeration* (litre min^{-1})	Aeration duration (hr)	DWW/ POME (%)	TSS removal (%)	COD removal (%)	BOD removal (%)	Colour removal (%)	Ammonia removal (%)	BOD/COD
1	0.5	2	20	98.21	95.91	90.16	64.66	98.2	0.94
2	4.0	22	50	98.37	97.07	91.44	73.05	98.09	0.94
3	4.0	12	50	96.61	96.67	90.56	71.88	98.09	0.94
4	7.5	12	50	96.14	96.35	89.47	66.70	98.20	0.93
5	0.5	22	80	98.95	98.31	90.08	81.64	96.93	0.92
6	7.5	22	20	98.86	95.92	89.98	69.52	97.98	0.94
7	4.0	12	50	96.79	96.48	90.03	65.84	97.88	0.93
8	4.0	12	80	98.90	98.23	89.67	80.43	98.09	0.91
9	4.0	12	50	98.23	96.40	89.10	67.17	98.29	0.92
10	0.5	2	80	95.87	96.32	89.51	59.60	97.98	0.93
11	7.5	2	20	96.31	95.34	90.01	56.94	97.67	0.94
12	7.5	22	80	98.32	98.10	90.69	75.25	98.30	0.92
13	0.5	22	20	97.91	95.70	89.30	57.52	97.21	0.93
14	4.0	12	50	96.12	96.37	88.79	64.70	98.20	0.92
15	7.5	2	80	98.32	98.07	90.29	80.70	98.20	0.92
16	0.5	12	50	95.47	96.33	89.46	62.66	97.67	0.93
17	4.0	12	20	97.74	97.85	89.52	79.05	97.98	0.91
18	4.0	2	50	97.88	96.50	89.20	67.25	96.19	0.92
19	4.0	12	50	97.23	96.74	88.95	70.39	97.67	0.92
20	4.0	12	50	97.09	96.91	89.57	74.15	98.09	0.92

Note: *Before aeration, 15 g litre^{-1} zeolite (based on the volume of POME) was used in each new-SBR.
DWW - domestic wastewater; POME – palm oil mill effluent; TSS - total suspended solids; BOD - biochemical oxygen demand; COD – chemical oxygen demand.

studies indicated that high concentration of COD, NH_3-N and low biodegradability index decreased the efficiency of SBR (Aziz *et al*., 2011; Kamarudzaman *et al*., 2011). However, adding absorbent into the SBR can efficiently remove pollutants at an improved rate (Neczaj *et al*., 2007).

In this study, the PNZ and wastewater were used as a cost-effective biological co-treatment materials to improve the efficiency of the new-SBR and to decrease the environmental impacts of discharging caused by POME. *Table 6* shows the removal efficiency of the new-SBR for the different variable of POME. As mentioned, before aeration and for the adsorption of pollutants, 15 g litre^{-1} PNZ was used in the new-SBR. The results are reported based on different aeration rate, aeration duration and percentage of DWW to POME (DWW/POME). As shown in *Table 6*, the variation of COD removal is between 95.34% and 98.31%. The highest removal of COD (98.31%) was achieved under the contact time of 22 hr, aeration rate of 0.5 litre min^{-1} and DWW/POME ratio of 80%. The minimum COD removal (95.34%) was at 7.5 litres min^{-1} aeration rate, 2 hr contact time, and 20% of DWW/POME. The 2D contour plot and 3D response surface plot for COD removal are illustrated in *Figure 5*. An optimal COD removal (98.362%) was provided at the aeration rate of 2.930 litres min^{-1}, contact time of 13.617 hr, and DWW/POME ratio of 78.476%. The results are consistent with those in previous studies (Aziz *et al*., 2011; Mojiri *et al*., 2014).

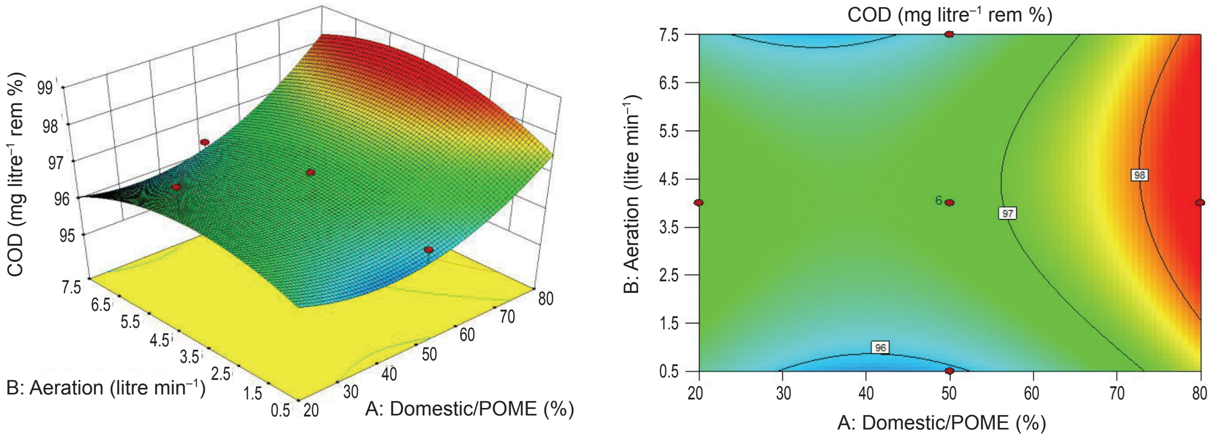
In the conventional biological treatment of wastewater, the AN is a significant inhibitor compound. Ammonia decreases biodegradability ratio and prohibits microbial growth. Furthermore, eutrophication accelerates and DO decreases with increasing concentration of AN (Li *et al*., 1999). Aziz *et al*. (2013) showed that most AN can be removed biologically. A combination of biological degradation and zeolite adsorption was used in the new-SBR for AN removal through microbial biofilm formation on the surface of the zeolite. As shown in *Table 6*, the ammonia removal increased from 96.19%-98.30%. The lowest ammonia removal occurred under contact time of 2.0 hr, the aeration rate of 4.0 litres min^{-1}, and DWW/POME ratio of 50%. The highest ammonia removal was observed under the following conditions, 7.5 litres min^{-1} aeration rate, 22 hr contact time, and 80% DWW/POME.

Figure 6 shows the 3D response surface plot for AN removal. The results showed that the new-SBR system can reach to the optimum removal efficiency of ammonia (98.33%) at a contact time of 18.32 hr, an aeration rate of 4.81 litres min^{-1}, and DWW/POME ratio of 76.97%. This finding means that the optimal desirable operation can be achieved through POME treatment in the new-SBR system under the aforementioned operation conditions.

In medium strength wastewater, approximately 75% of suspended solids and 40% of filterable solids are the organic materials (Metcalf and Eddy, 2014). The major organic compounds present in wastewater, detergents, carbohydrates, greases and oil, and proteins. Drinan and Spellman (2012) reported that around 30% of the organic compounds are not biodegradable. The POME is a high-strength colloidal agroindustry effluent with a high value of TSS (4310 mg litre^{-1}, *Table 2*), then a chemical adsorption is necessary to remove TSS. Chemical adsorption plays a supplementary role in microbial growth and adsorption, and zeolite is a promising adsorbent for suspended solids. According to Montalvo *et al*. (2012), a 3D structure of zeolite presents a high specific surface area (SSA). Erdem *et al*. (2004) reported that the zeolite with 41.5% porosity, 2.27 g cm^{-3} appearance density and 1.32 g cm^{-3} weight per unit volume is recognised as a suitable adsorbent with high hollow micropores and macropores. These properties provide a high surface for the monolayer adsorption of organic pollutants and a cation exchangeable capacity that can act as a capable chemical adsorption of macro and micro elements. *Table 6* indicates that the efficiency of the new-SBR with zeolite and DWW in TSS removal increased from 95.47% (contact time of 12 hr, aeration flow of 0.5 litre min^{-1}, and DWW/POME ratio of 50%) to 98.95% (contact time of 22 hr, aeration rate of 0.5 litre min^{-1} and DWW/POME ratio of 80%).

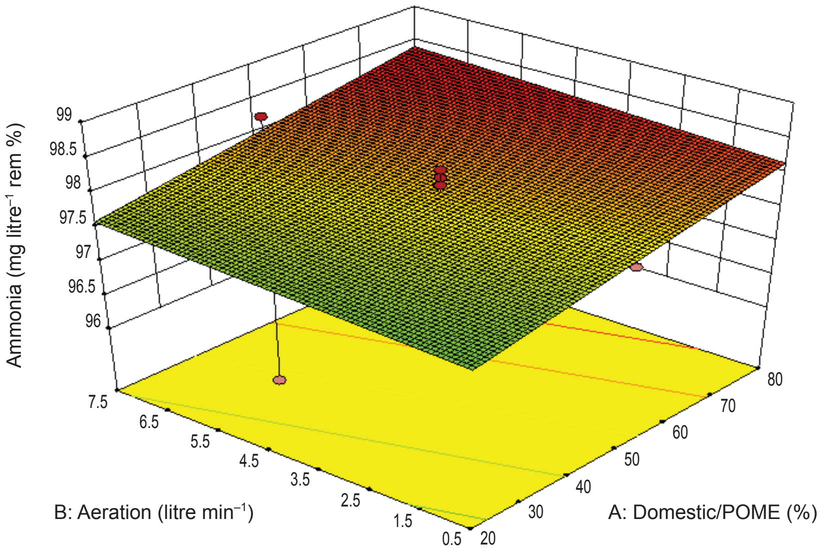
TSS removal and biodegradability index (BOD_5/COD ratio) are bigger than 95% and 0.5, respectively for all cases in *Table 6*. High BOD_5/COD increases the percentage of degradation and functions as an acceptable indicator of enhanced TSS removal as an organic pollutant. Therefore, the efficiency of the new-SBR in POME treatment increases by enhancing POME biodegradability. Results of TSS removal indicated that the combination of raw DWW as a microbial biodegradation supplier in the presence of zeolites for coagulation is an effective treatment method for the decontamination of biodegradable and non-biodegradable suspended solids. *Figure 7* shows the 2D contour plot and 3D response surface plot for TSS removal. The optimum TSS removal (99.16%) was observed at DWW/POME of 22.06%, aeration rate of 5.16 litres min^{-1} and a contact time of 21.6 hr. This result means that the new-SBR system could reach the highest achievable desirability under the aforementioned conditions.

Table 2 shows that collected POME from UOP contained a high intensity of colour (2550 Pt. Co.). According to Liew *et al.* (2015), colour is a critical pollutant in POME. Several methods have been applied to remove colour such as, the SBR systems (Zahrim *et al*., 2009), anaerobic reactions (Zhang *et al*., 2008), banana peel (Mohammed and Chong, 2014), boiler fly ash (Igwe *et al*., 2010) and activated carbon (Zahrim *et al*., 2009). The low declorisation efficiency



Note: POME - palm oil mill effluent; COD - chemical oxygen demand.

Figure 5. The 3D surface plots and 2D contour plot for COD removal.



Note: POME – palm oil mill effluent.

Figure 6. The 3D surface plots of ammonia nitrogen (AN) removal.

and high-cost treatments are the two main problems that limit the applications of commonly used methods. The biological and adsorption phenomena are the main factors influencing colour removal. The zeolite contains a high SSA which provides a specific suitable surface for microbial film forming (adsorbing and biodegradation). Micropores and macropores could be considered as suitable spaces for colour particles absorbing. For colour removal of landfill leachate, the aerobic SBR with adsorbent and wastewater as the augmentations was recommended as a cost-effective method (Aziz *et al*., 2011a). *Table 6* illustrates that in the new-SBR system, the minimum and maximum colour removal are 56.94% (contact time of 2 hr, DWW/POME of 20%, and aeration rate of 7.5 litres min^{-1}) and 81.64% (contact time of 22 hr, DWW/POME of 80%, and aeration rate of 0.5 litre min^{-1}) respectively.

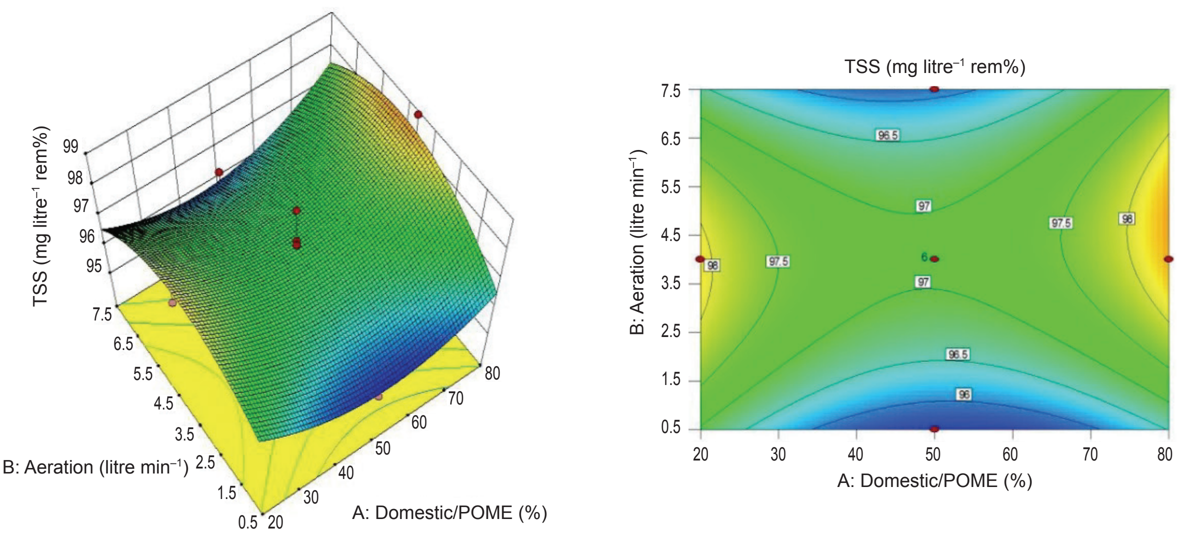
Based on the Brunauer-Emmet-Teller (BET) test, the SSA of zeolite were decreased significantly after treatment process which may illustrate the action of zeolite as a suitable adsorbent. Moreover, the SEM-EDX indicates that natural zeolite fully covered by pollutants after the treatment process. Therefore, the suggested new-SBR can be employed as a cost effective declorisation system because of the positive effect of zeolite and DWW that enhances biological treatment. The POME treatment in the new-SBR could reach to optimum colour removal at DWW/POME ratio of 77.296%, aeration rate of 4.298 litres min^{-1} and contact time of 15.506 hr. Under these conditions, optimum decolourisation was observed to be 82.028%.

Estimation of BOD in the wastewater is one of the best methods to determine organic content. Gerardi (2011) showed that reducing oil, fats and grease decreases BOD significantly. The BOD adsorption is recognised as particle diffusion-controlled mechanism and can be used to treat POME. In the new-SBR, the BOD removal varied from 88.79%-91.44% (*Table 6*). The lowest BOD removal was observed at a DWW/POME of 50%, contact time of 12 hr, and aeration rate of 4 litres min^{-1} while the highest one occurred at a DWW/POME of 50%, contact time of 22 hr and aeration rate of 4 litres min^{-1}. The results indicate that there is no significant different between the highest and lowest BOD removal efficiencies (88.79%-91.44%). It seems that the presence of DWW even in very low concentration provides high performance of BOD removal. For Run 1 (*Table 6*), although the DWW is low (DWW/POME=20%), the BOD removal is 90.16%. More researches required to specifying the minimum DWW as augmentation for POME treatment in the aerobic new-SBR system. The duration of aeration is another parameter which directly effects on BOD removal. A comparison between Run 12 and 15 with a same DWW/POME of 80% and aeration of 7.5 litres min^{-1} indicates that BOD removal increases from 90.29%-90.96% with increase in the aeration contact time from 2.0-22 hr, respectively. Furthermore, a comparison between Run 5 and 12 shows that BOD removal enhances by raising the aeration rate. Therefore, high aeration rate and long contact time did improve BOD removal efficiency through the new-SBR systems. Similar results were reported by Sahu *et al*. (2009). The RSM and CCD analysis indicate that the optimum removal efficiency of BOD (90.67%) was attained at 6.74 litres min^{-1} aeration rate, 1.99 hr contact time, and 66.39% DWW/POME ratio.

Microbiological treatment can be determined from biodegradability index (BOD/COD ratio). However, decontamination of wastewater increases with enhancing biodegradability. As shown in *Table 2*, by considering the value of BOD_5/COD as the biodegradability index in POME (0.11) and DWW (0.41), DWW is more biodegradable in comparison with anaerobically treated POME. After treatment of POME in the new-SBR, the results indicate that the ratio of BOD_5/COD is bigger than 0.9 for all cases (*Table 6*). It can be concluded the new-SBR with DWW and zeolite as the augmentations was achieved to successfully improve the biodegradability of POME. A 3D surface plots and 2D contour of POME biodegradability is shown in *Figure 8*. The optimum POME biodegradability was obtained for 12 hr contact time, 4 litres min^{-1} aeration and the DWW/POME of 80%.

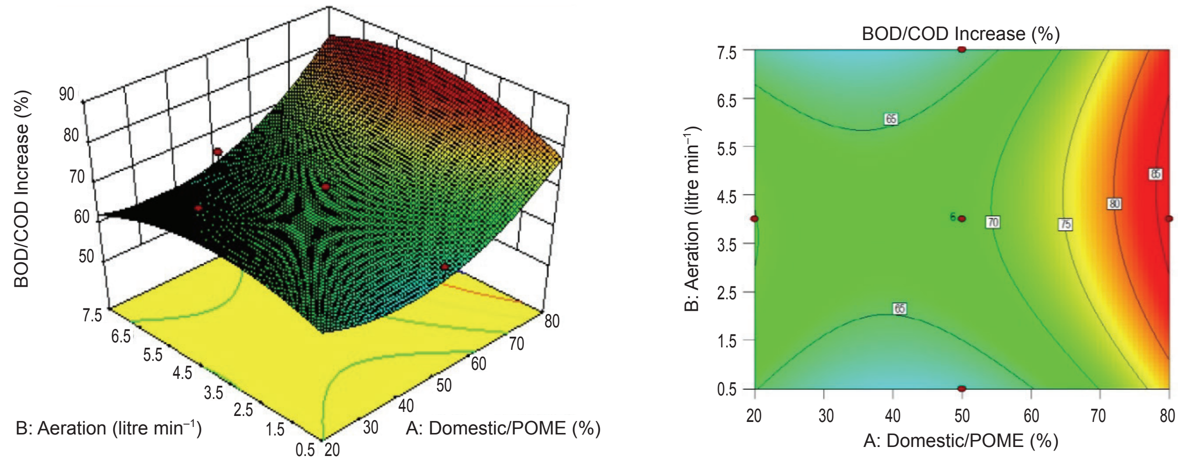
Evaluation of the New-SBR with Zeolite and DWW

The aim of this part of the research is to identify the most influenced augmentation for POME treatment in the new-SBR system. As shown in *Table 7*, several experiments were conducted to evaluate removal efficiency of the new-SBR for TSS, COD, colour and AN. Previous studies reported that augmentation of natural adsorbents can enhance pollutant removal efficiency (Halim *et al*., 2010; Kalló, 2001; Shavandi *et al*., 2012). Moreover, Vijayaraghavan *et al*. (2007) found that microbial communities exert a positive effect on POME treatment. Therefore, different conditions were considered to compare the ability of the new-SBR to improve POME such as 1) the blank new-SBR without any extra material as a case for control, 2) the new-SBR with adding zeolite, 3) the new-SBR with adding DWW and 4) the new-SBR with adding both zeolite and DWW. The experiments were performed under the similar optimum conditions and three replication runs were conducted to provide actual assessment.



Note: POME – palm oil mill effluent; TSS – total suspended solids.

Figure 7. The 3D surface plots and 2D contour plot for TSS removal.



Note: POME – palm oil mill effluent; BOD - biochemical oxygen demand.

Figure 8. The 3D surface plots and 2D contour plot for POME biodegradability.

TABLE 7. REMOVAL POME COMPONENTS IN THE NEW-SBR UNDER DIFFERENT CONDITION

Parameters	POME (control) removal (%)	Zeolite + POME removal (%)	DWW+ POME removal (%)	Zeolite + DWW+ POME removal (%)
TSS (mg litre^{-1})	-22	23	77	98.95
COD (mg litre^{-1})	9	10	74	98.31
Colour (Pt. Co.)	-30	9.6	76	81.64
AN (mg litre^{-1})	69	80	90	98.30

Note: POME - palm oil mill effluent; SBR - sequencing batch reactor; DWW - domestic wastewater; TSS – total suspended solids; COD – chemical oxygen demand; AN – ammonia nitrogen.

Table 2 illustrates that the anaerobic treatment of POME did not successfully removed colour (2550 Pt. Co.), AN (94.5 mg litre^{-1}), COD (11 090 mg litre^{-1}) and TSS (4310 mg litre^{-1}). As shown in *Table 7*, the negative values of TSS (-22) and colour (-30) show that the TSS and colour were increased in the POME due to strong mixing power of the aeration system on the suspended solids. However, in other cases, the presence of adsorbent (natural zeolite) and microbial degradation source (DWW) is a reason to increase removal efficiency of TSS (from -22% in POME to 23% in PMOE + zeolite, 77% in PMOE + DWW and 98.9% in PMOE + zeolite+ DWW) and colour (from -30% in POME to 9.6% in PMOE + zeolite, 76% in PMOE + DWW and 81.6% in PMOE + zeolite + DWW). The results indicate that removal of TSS, AN, COD and colour in the new-SBR with DWW (POME + DWW) is higher than those in both blank new-SBR (POME) and the new-SBR with zeolite (POME + zeolite). Therefore, DWW has a significantly higher influence on POME decontamination compared to the zeolite. The results indicate that the new-SBR with augmentation of DWW and zeolite (POME + DWW + zeolite) was able to remove a high percentage of all pollutants compared to the new-SBR with the only DWW. In sum, the removal efficiency of the new-SBR systems can be sorted as the following order: DWW + POME + zeolite > POME + DWW > POME + zeolite > POME. In can be concluded that using both zeolite and DWW improves biodegradability and shortens treatment duration of POME treatment.

Optimisation of Experimental Conditions and Statistical Analysis

In this section, the RSM and CCD were employed to determine the optimum conditions of the independent factors. In this analysis, DWW/POME ratio, aeration rate (litre min^{-1}), and the contact time (hr) were chosen as independent variables. To conduct a sufficient analysis for the aerobic method of the new-SBR, five dependent variables of TSS, BOD, COD, AN and colour were considered as responses. The effects of operational factors and responses of RSM modeling are presented in *Table 8*. The RSM results illustrated the specific effect of the independent parameters as well as the influence of the interactive effects on the selected responses. These results indicated that DWW/POME ratio has a significant positive effect on COD, AN and colour removal with P-value < 0.05 and a low effect on BOD_5 (P-value= 0.5681) and TSS (P-value= 0.6456). Moreover, a P-value of 0.0007 for BOD_5/COD indicates that POME biodegradability is significantly affected by DWW/POME.

TABLE 8. RESPONSES AND THE EFFECTS OF OPERATIONAL FACTORS FOR RSM MODELING

Responses	Intercept	A*	B	C	AB	AC	BC	A^2	B^2	C^2
TSS	97.0674	0.1330	0.1540	0.5820	0.3463	0.1038	-0.0288	1.1691	-1.3459	0.9741
P-value**		0.6456	0.5951	0.0648	0.2954	0.7476	0.9288	0.0537	0.0306	0.0986
BOD_5	89.5549	0.1270	0.1930	0.2320	0.1075	0.2325	0.0825	-0.0423	-0.1723	0.6827
P-value		0.5681	0.3908	0.3063	0.6645	0.3566	0.7387	0.9200	0.6835	0.1271
COD	96.7792	0.8310	0.1210	0.2960	0.2363	0.2063	-0.1463	0.9845	-0.7155	-0.2705
P-value		0.0007	0.4959	0.1145	0.2453	0.3066	0.4625	0.0130	0.0532	0.4268
Colour	69.4014	4.9930	2.3030	2.7830	1.3038	1.3938	-0.9713	8.0191	-7.0409	-1.5709
P-value		0.0337	0.2830	0.2004	0.5783	0.5528	0.6778	0.0651	0.0989	0.6934
AN	97.8850	0.4000	0.1260	0.1050	-	-	-	-	-	-
P-value		0.0096	0.3680	0.4514	-	-	-	-	-	-
BOD_5/COD	68.6273	8.2000	0.6000	2.2000	2.0000	1.2500	-1.7500	9.6818	-6.3182	-4.3182
P-value		0.0007	0.7318	0.2253	0.3181	0.5262	0.3795	0.0138	0.0802	0.2130

Note: RSM - response surface methodology.
*A - MWW/POME ratio, B - aeration rate and C - contact time.
**P-value < 0.05 - significant and P-value > 0.05 - no significant.
TSS - total suspended solids; COD - chemical oxygen demand; AN - ammonia nitrogen; BOD - biochemical oxygen demand.

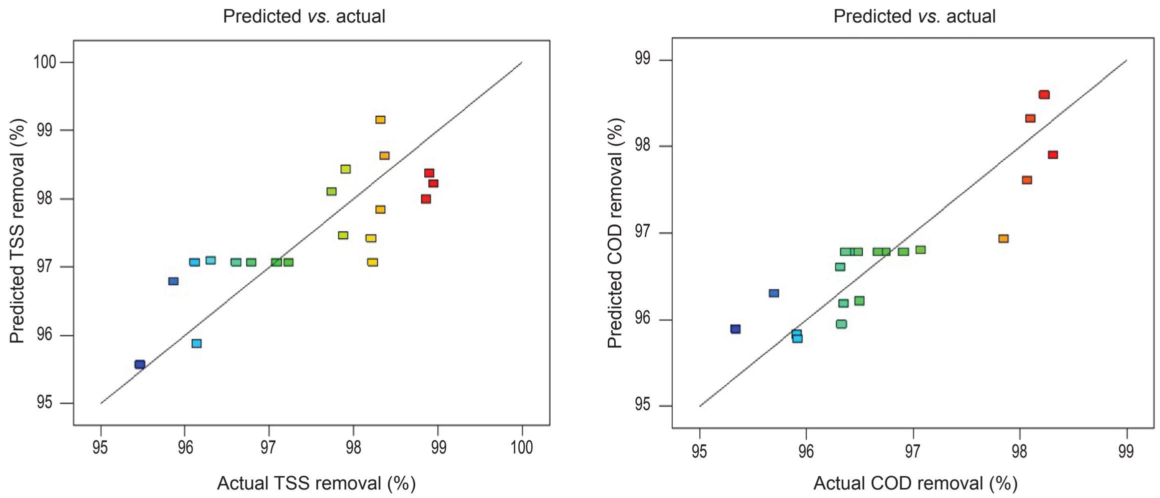
TABLE 9. DEVELOPED EQUATIONS AND ANOVA RESULTS FOR RESPONSE PARAMETERS

Responses*	Modified equations with significant terms**	Prob.***	R^2	Adjusted R^2	Adequate precision	SD	CV	Press****
TSS	$99.47+0.80A-0.18C-7.14AC-0.11A^2+9.5C^2$	0.24	0.71	0.66	3.53	1.16	1.19	121.3
BOD_5	$90.15+0.09A+-0.17C+2.86AC+-0.02A^2-+6C^2$	0.67	0.54	0.45	2.72	0.74	0.82	23.5
COD	$96.07+0.63A+0.09C-6.42AC-0.08A^2+-2.62C^2$	0.35	0.54	0.45	5.81	0.89	0.92	15.12
Colour	$65.61+6.04A+0.88C-0.02AC-0.711A^2-0.03C^2$	0.44	0.68	0.62	3.26	9.08	13.02	7 472.5
AN	$95.61-0.06A-0.06C+3.57AC+0.02A^2+4.18C^2$	0.10	0.82	0.77	5.18	0.5	0.51	12.11
BOD_5/COD	$63.05+5.04A+1.65C-0.05AC-0.65A^2-0.06C^2$	0.18	0.86	0.83	5.53	6.97	10.15	3 026

Note: *All removal units are mg $litre^{-1}$ except BOD_5/COD (unit less).
**In final equations, where A - DWW/POME (%); B - the aeration rate (litre min^{-1}); C - contact time (hr).
***Prob. - the probability of error; R^2 - coefficient of determination; SD - standard deviation; CV - coefficient of variance;
****Press - predicted residual error sum of square. ANOVA - analysis of variance.
TSS - total suspended solids; COD - chemical oxygen demand; AN - ammonia nitrogen; BOD - biochemical oxygen demand.

The second-order functions of pollutant removal including the results of ANOVA analysis are shown in *Table 9*. These equations express the relationship between independent parameters and the reduction of TSS, BOD, COD, AN, colour and BOD_5/COD. It was noted that the regression model for the reduction of pollutant was significant at a confidence level of 95% ($p < 0.05$) with R^2 equal to 0.86, 0.82, 0.71, 0.68 0.54, 0.54 and for BOD_5/COD, AN, TSS, colour, BOD and COD respectively. The coefficient of determination (R^2) indicates that although the regression model was able to successfully predict BOD_5/COD (R^2=0.86) and AN (R^2=0.82), other techniques such as soft computing methods can be recommended to prodive higher accuracy for other parameters (Zakaria *et al*., 2010; Mohammadpour *et al*., 2014; 2018; 2019; Ghani and Mohammadpour, 2015; Mohammadpour, 2017). A comparison between actual and predicted TSS and COD removal is illustrated in *Figure 9*.

Finding the optimum point of each element (aeration rate, contact time and DWW/POME ratio) could produce a clear picture for achieving the highest performance of decontamination. Based on the predicted model, the highest desirability of treatment (0.988) could be achieved in optimum operation conditions (contact time of 17.9 hr, DWW/POME ratio of 58.7%, and aeration rate of 6.85 litre min^{-1}). Under these conditions, COD, BOD, colour, AN and TSS removal rates were 96.80%, 90.1%, 69.90%, 98.20% and 97.20%, respectively. This research highlights that the recommended system can be successfully used as a cost-effective methods for post-treatment of POME. This system is able to highly remove the pollution within a short time (less than a day). Therefore, it can be commonly used as a rapid, economic and highly reliable technique to remove pollution and treat wastewater at any aquatic system worldwide.



Note: TSS - total suspended solids; COD - chemical oxygen demand.

Figure 9. Experimental vs. *predicted (a) TSS removal; (b) COD removal.*

CONCLUSION

The POME with high pollution components can be considered as an ecosystem and environmental hazard if it is discharged without any treatment to the environment. SBR is recommended to biologically treat wastewater and POME. In this study, a novel and cost-effective aeration system namely the new-SBR with opposite direction of aeration was developed to achieve a high efficiency of aeration in SBR. The DWW and zeolite were employed as an available microbial source and new adsorbent, respectively to improve the new-SBR treatment duration. A high pollution removal was obtained using the new-SBR. The COD, BOD, TSS, AN and colour removal rates were found in the range of 95.34%-98.31%, 88.79%-91.44%, 95.47%-98.95%, 96.19%-98.30% and 56.94%-81.64%, respectively. Several extra experiments have been conducted with and without zeolite and DWW to determine the ability of these materials in POME treatment. The results indicate that the new-SBR with adding both DWW and zeolite (POME+DWW+zeolite) is able to remove a high percentage of all pollution in compare to the new-SBR with only DWW or zeolite. In sum, the removal efficiency of the new-SBR systems can be sorted as the following order: POME + DWW + zeolite > POME +DWW > POME + zeolite > POME. It can be concluded that using both zeolite and DWW improves biodegradability and shortens treatment duration of POME treatment. The RSM and CCD methodology have been used to determine the optimum values of the independent parameters, including contact time (hr), aeration rate (litre min^{-1}), and ratio of domestic wastewater to POME (DWW/POME; v/v), as well as their dependent parameters (COD, BOD, colour, AN and TSS). The highest desirability of POME treatment (0.988) was achieved in optimum operation conditions (DWW/POME ratio of 58.7%, aeration rate of 6.85 litre min^{-1}, and contact time of 17.9 hr). Under these conditions, COD, BOD, colour, AN and TSS removal rates were 96.80%, 90.1%, 69.90%, 98.20% and 97.20%, respectively. Finally, an ANOVA analysis indicated that DWW/POME ratio has a significant positive effect on COD, AN and colour removal and low effect on BOD_5 and TSS removal.

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