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PROCEEDINGS

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SEMINAR NASIONAL TEKNIK INDUSTRI

TEMA:
***"Peluang dan Tantangan Globalisasi, Industrialisasi dan kelestarian lingkungan untuk meningkatkan kualitas* hidup Masyarakat"**

Editor :
Dr. M. Sayuti, ST.,M.Sc.Eng
Fatimah, ST.,MT
Ir. Amri, MT
Diana Khairani Sofyan, ST.,MT
Syarifuddin, ST., MT

Jurusan Teknik Industri
Fakultas Teknik, Universitas Malikussaleh
Lhokseumawe-Aceh

PROCEEDINGS SEMINAR NASIONAL TEKNIK INDUSTRI [SNTI] 2013

"Peluang dan Tantangan Globalisasi, Industrialisasi dan kelestarian lingkungan untuk meningkatkan kualitas hidup Masyarakat"

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Jurusan Teknik Industri
Fakultas Teknik
Universitas Malikussaleh
Jl. Medan-Banda Aceh, Reuleut, Aceh Utara
E-mail : snti2013@yahoo.com

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KATA PENGANTAR

Assalamu'alaikum Warahmatullaahi Wabarakaatuh

Puji syukur kehadirat Allah SWT atas segala rahmat dan hidayah-Nya sehingga *Proceedings* Seminar Nasional Teknik Industri [SNTI] 2013 dengan tema ""Peluang dan Tantangan Globalisasi, Industrialisasi dan kelestarian lingkungan untuk meningkatkan kualitas hidup Masyarakat" " yang diselenggarakan oleh Jurusan Teknik Industri Fakultas Teknik Universitas Malikussaleh pada 28-29 Agustus 2013 dapat kami selesaikan. Penyusunan *Proceedings* ini dimaksudkan agar masyarakat luas dapat mengetahui berbagai informasi terkait dengan penyelenggaraan Seminar Nasional tersebut. Informasi yang disajikan dalam Proceedings ini meliputi:

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3. Sambutan Dekan Fakultas Teknik
4. Sambutan dan Pembukaan oleh Rektor Universitas Malikussaleh
5. Keynote I
6. Keynote II
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Ucapan terimakasih dan penghargaan yang setinggi-tingginya kami sampaikan kepada Pembicara Utama, Bapak/Ibu Pemakalah dan Peserta yang telah menyumbangkan pemikirannya dalam acara Seminar Nasional Teknik Industri 2013 ini. Tak lupa juga terimakasih yang sedalam-dalamnya kepada Sponsor dan semua pihak yang telah memberikan dukungan bagi terselenggaranya Seminar Nasional Teknik Industri 2013 ini dan atas tersusunnya proceedings ini.

Akhir kata semoga *Proceedings* ini dapat memberikan manfaat bagi semua pihak khususnya untuk Keberlanjutan dan Peningkatan Daya Saing Industri Nasional.

Wassalam

Lhokseumawe, 28 Agustus 2013
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MANAJEMEN SUPPLY CHAIN

TEKNOLOGI PRODUKSI DAN PROSES LANJUTAN

MEMBRANE ULTRAFILTRATION FOR AMMONIUM NITROGEN REMOVAL: USE OF RESPONSE SURFACE METHODOLOGY TO IMPROVE UNDERSTANDING OF PROCESS PERFORMANCE AND OPTIMIZATION

Erna Yuliwati[1*] dan Amrifan Saladin Mohruni[2]

[1]*Department of Industrial Engineering,Faculty of Engineering*
University of Bina Darma, 30251 Palembang, Indonesia,
Tel. +62 (711) 515-679; Fax: +62 (711) 518-000
[2]*Departmentof Mechanical Engineering, Faculty of Engineering*
University of Sriwijaya, Sumatera Selatan, Indonesia,
Tel. +62 (711) 580272
Email:erna_yuliwati@mail.binadarma.ac.id***

Abstrak

The aim of this study was to remove of ammonium nitrogen (NH_3-N) from refinery wastewater that was conducted by submerged membrane ultrafiltration.The experimental set-up comprised mainly of submerged membrane ultrafiltration reservoir, circulation pump, and aerator was used throughout investigation, which operated at vacuum pressure. Deposition and accumulation of suspended solids on membrane surface were prohibited with continuously air bubble flow rate that plays the main role in this filtration process. Responsesurface methodology (RSM) wasused for modelling and optimizing the process, and to gain a better understanding of the process performance. Quartic model was used as the experimental design. The factors studied were mixed liquor suspended solid (MLSS), air bubble flow rate (ABFR), and hydraulic retention time (HRT). Using RSM the retention of NH_3-N was maximized while optimizing the air bubble flow rate. Response surface plots improved also the understanding of the factors effect on permeate flux. The optimized conditions obtained for fluks of 148 L/m^3h and NH_3-N removal of 92.89 *% with an experimental design were MLSS concentration* 4.50*mg/L, aerator flow rate 2.25 ml/min, and HRT 276.93 min.*

Keywords: *Membrane ultrafiltration, Response Surface Methodology, Ammonium Nitrogen, Air Bubble Flow Rate*

Introduction

The environment is becoming more polluted because of the discharge of various wastes. There are more industries as a consequence of developed modern way of life. Worldwide, the oil industry, vital for modern industry, generates a vast amount of wastewater that is highly contaminating and difficult to treat. Thus, strict discharge standards for refineries have been implemented in many countries. The national primary regulatory of discharged standard in Malaysia (Standard B; December 10, 2011) has set discharged limits for constituents shown in Table 1. To attain these limits, the refinery wastewater treatment plants need additional tanks and equipment, but in many cases the refineries are located in populous areas with little space for expansion. Technologies that can treat large quantities of wastewater with relatively small requirements are, therefore, of particular importance. The developed submerged membrane technology is able to completely retain biomass and operate with high suspended solids concentration.

Table 1.Composition of refinery wastewater and the national discharge standards for refinery wastewater [1].

Constituent, unit	Influent	National primary discharged standard (P.U. (A) 434, Standard B, December 10, 2011)
pH	6.7	5.5 – 9.0
Oil and grease, mg/L	17.0	10.0
COD, mg/L	555.0	400.0
NH3-N, mg/L	29.1	20.0
Suspended Solid, mg/L	213.0	100.0
Chlorine free, mg/L	4.6	2.0
Sulfide, mg/L	2.5	0.5

Submerged membrane is now widely used in water and wastewater treatment due to its high packing density and ease of module manufacture and operation [2]. The removal of organic wastes from wastewater is, therefore, becoming increasingly important, and submerged ultrafiltration finds its application in this area. The direct immersion of hollow fiber membranes was assembled in the feed reservoir with withdrawal of liquid through the fibers by the application of a vacuum on the outlet of the fiber lumen. There has been increasing attention to the application of refinery effluent in petroleum industry since a few years ago because refinery wastewaters were characterized by presence of several aromatic hydrocarbons and inorganic substances such as, COD, TOC, sulfide, ammonia nitrogen, and total suspended solid (TSS) [3].

Traditionally, the study membrane ultrafiltration has been conducted using the one variable at a time approach, where the effect of each factor is investigated separately. However, this approach implies a large amount of experiments and may often important conclusions about the effect of one experimental variable when the level of another variable is changed (i.e. interactions effects). Further, the use of statistical methods such as response surface methodology (RSM) overcomes the limitations of the one-variable at a time approach. RSM is an efficient statistical tool, which is used for modelling and optimization of several process variables [4].

In this study, RSM was used for the optimization of NH_3-N removal from separation of refinery wastewater synthetic which carried out using modified PVDF membrane. The effect of aeration, HRT, pH and MLSSof feed solution on NH_3-Nremoval was studied with several apparatus.

Experimental

Materials. Commercial PVDF polymer pellets (Kynar®740) were supplied by Arkema Inc. Philadelphia, USA. N,N-dimethylacetamide (DMAc, Aldrich Chemical) (synthesis grade, Merck, >99%) was used as solvent without further purification. Lithium chloride monohydrate (LiOH. H_2O) (Sigma Aldrich) and titanium dioxide (TiO_2) (Sigma Aldrich) were used as inorganic additives.The used membrane properties was tabulated in Table 2[5].

Table 2. Membrane Properties

Parameter	Membrane
Outer diameter (mm)	1.1
Inner diameter (mm)	0.55
Pore size (nm)	34.05
Contact angle (^{o})	54
Pure water flux (L m^{-2} h^{-1})	82.95 at 250 mmHg

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Experimental set-up and operational conditions. Figure 1 illustrates the flow pattern of air bubble within the submerged UF system. Two bundles of modified PVDF hollow fibers with total effective area of approximately 184 cm^2were immersed in the membrane reservoir and a constant TMP was maintained to pressurize wastewater from outside to inside of hollow fibers. All filtrations were conducted at room temperature and vacuum condition created using a peristaltic pump (Master flex model 7553-79, Cole Palmer). The permeate flow rate was continually recorded using flowmeter. The volume of the water permeation collected was determined using a graduated cylinder. After completing filtration, the membrane surface was cleaned with a soft sponge to remove the particle-packed layer which might form during filtration.

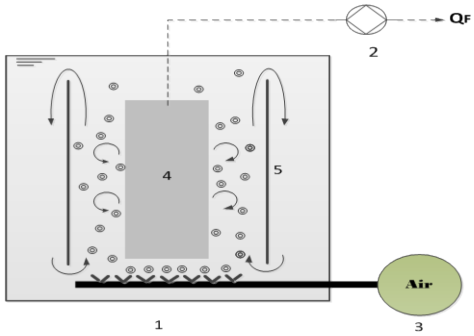

Fig.1. Schematic representation of the air bubble upflow stream in submerged ultrafiltration system: membrane reservoir (1); peristaltic pump (2); aerator (3); membrane bundles (4) and partitioned glass (5).

Analytical method. Field emission scanning electron microscope (JEOL JSM-6700F) was used to examine the morphology of the PVDF hollow fiber membrane prepared. The FESEM micrographs of cross-section of fiber membranes were taken at various magnifications.
Membrane was tested with a self-made U-shape membrane bundle. Pure water permeation rate was measured after the steady state was reached, using the following equation

$$F = \frac{V}{At} \quad (1)$$

where$F$ is the pure water flux (l/m^2 h), V is the permeate volume (l), A is the membrane surface area (m^2), and t is the time (h).

NH_3-N concentrations were measured using a spectrophotometer (DR5000, HACH) in accordance to the standard procedures. During the operation with high organic loading rates, the parameters were evaluated daily and sampling was carried out three times a week again. The NH_3-N removal efficiencies are calculated with Eq.(2):

$$NH_3\text{-}N\ removal\ (\%) = \frac{NH3-N_0 - NH3-N}{NH3-N_0} \times 100 \quad (2)$$

where NH_3-$N_o$and NH_3-N are the initial concentration of synthetic refinery wastewater and the concentration of permeate produced.

Response Surface Methodology (RSM). RSM is derived from mathematical and statistical technique. It can be used for studying the effect of several factors at different level and their influence on each other. Based on the center composite design (CCD) of RSM, the Design Expert 8.0.5.2 software is used for the statitical design of

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experiments and data analysis in duplicate. The four factors were made of air bubble flow rate(ABFR) (x_1), hydraulic retention time (HRT) (x_2), mixed liquor suspended solid concentration (MLSS) (x_3), and pH (x_4), were used to optimizes NH_3-N removal in submerged membrane process. Model fitting to equation of up to the fourth-order polynomial was performed to determine the goodness-of-fit. The responses were fitted to the variables by multiple regression. The minimum and maximum range of variables were investigated and the full experimental plan with respect to their values in actual and coded form was listed in Table 3.

Table 3. Independent variables and limit level for response surface study

Variables	Unit	Symbols		Levels				
		uncoded	coded	-2	-1	0	+1	+2
air bubble flow rate	min/L	x_1	X_1	0.3	1.2	2.1	3.0	3.9
HRT	min	x_2	X_2	120	180	240	300	360
MLSS	mg/L	x_3	X_3	1.5	3.0	4.5	6.0	7.5
pH	pH	x_4	X_4	3.5	5.0	6.5	8.0	9.5

In RSM, a model with the form of Eq. (3) is fitted to experimental data and, by optimization methods, the coefficients for the model are calculated. To identify the right model that can fit the data, it can be started with the simplest model forms like first- and second-degree Scheffe's polynomial [8]. After testing these models for adequacy of fit, they were augmented to simplex centroid and special quartic models by adding the appropriate terms. In this study, the quartic model used for predicting the optimal point is as follows

$$y(x) = \sum_{i=1}^{N} e_i x_i^4 + + \sum_{ij(i<j)}^{N} e_{ij} x_i^3 x_j + \sum_{ij(i<j)}^{N} e_i x_i^2 x_j^2 + \sum_{ijk(i<j<k)}^{N} e_{ijk} x_i^2 x_j x_k + \sum_{ijkl(i<j<k<l)}^{N} e_{ijkl} x_i x_j x_k x_l \qquad (3)$$

where y is the response variable, e_i, e_{ij}, e_{ijk}, and e_{ijkl} are the polynomial coefficients of the model, x_i, x_j, x_k and x_l are the coded levels of the independent variables [6].

All these coefficient variables are analysed by multiple regression analysis and response contour plot are generated using the software Design-Expert. Validity of the selected model used for optimizing the process parameters has to be tested using analysis of variance (ANOVA) that is determined by performing Fisher's statistical test. In particular, the proportion of variance exhibited by the multiple coefficient of determination R^2 should be close to 1 as this would demonstrate better correlation between the experiment and the predicted values [7,8]. Moreover, a good model must be significant based on F-value and P-value as opposed to the lack of fit (insignificant).

Results and discussion

Morphology observation. Figure 2 shows the cross-section of the PVDF hollow fiber membrane with additives (TiO_2 and LiCl). The cross-sectionalimage of the hollow fiber indicates that the finger-like macrovoids extendes from both inside and outside of the hollow fiber to a sponge-like intermediate layer. This result is possibly attributed to the porous structure and higher hydrophilicity of TiO_2 nanoparticles than PVDF polymer. LiCl may be associated to the formation of a complex with DMAc, and also cross-linkingof the polymer by lithium ions. The PVDF complex will cause the presence of hydroxyl ions at the membrane surface, which enhances hydrophilicity [9-11]. The details of the membrane fabrication process and its properties determination procedure could be found in previous study [12].

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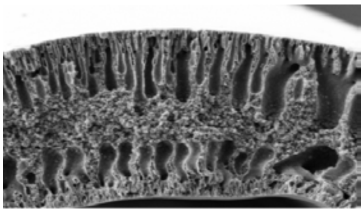

Fig. 2. Cross sectional (Mag. 500x)images of the PVDF hollow fiber membranes.

Statistical analysis. In the present work, the relationship between four factors (ABFR, HRT, MLSS concentration, and pH) and two responses (TSS and NH_3-N removal efficiencies) for submerged hollow fiber membrane is analyzed using RSM. The adequacy of the RSM is justified through analysis of variant (ANOVA). ANOVA is a statistical technique that subdivides the total variation in a set of data into component parts associated with the spesific sources of variances for the purpose of testing hypotheses on the parameters of the models[8]. ANOVA of these models have demonstrated that the model is highly significant effects and interactions of ABFR, HRT, MLSS, and pH on NH_3-N removal of filtered refinery wastewater. The backward elimination procedure was employed to eradicate the insignificant terms and ANOVA results of this backward quadratic model. The values determine the rank of significance's degree. The confidence level of ANOVA analysis of NH_3-N removal response, as shown in Table 4, which was greater than 80% ($P<0.05$) for NH_3-N response while F-value and P-value of the model were 1975.47 and 0.0001 respectively. This indicatess also that the estimated model fits the experimental data adequately. It was further shown that the main effect of ABFR (x_1), HRT (x_2), MLSS (x_3), and pH (x_4) and more level interactions of x_1, x_2, x_3, x_4, x_1x_2, x_1x_3, x_1x_4, x_2x_3, x_2x_4, x_3x_4, x_1^2, x_2^2, x_3^2, x_4^2, $x_1x_2x_3$, $x_1x_2x_4$, $x_1x_3x_4$, $x_2x_3x_4$, $x_1^2x_2$, $x_1^2x_3$, $x_1^2x_4$ $x_1x_2^2$ and $x_1x_2x_3x_4$ were significant model terms (factors).

Effects of ABFR on NH_3-N removal. The following fitted regression models (equations in terms of coded values for the regressors) are used to quantitatively investigate the effects of ABFR, HRT,MLSS, and pH on the characterization of the submerged membrane process for NH_3-N removal efficiencies. According to the sequential model sum of squares, the models were selected based on the highest-order polynomials where the additional terms were significant. An empirical relationship between the responses and the variables was expressed by the following equationsof fourth degree. The effects of the independent variables on the dependent variables, i.e. NH_3-N (y_1) removal efficiencies, is evaluated using approximating functions Eq. (4).

$$y_1 = 90.39 + 5.14\, x_1 + 6.56\, x_2 + 0.40\, x_3 + 10.88\, x_4 + 0.16\, x_1x_2 + 0.13\, x_1x_3 - 1.10\, x_1x_4 + 0.50\, x_2x_3 - 1.63\, x_3x_4 - 3.86\, x_1^2 - 2.64\, x_2^2 + 1.11\, x_3^2 - 6.29\, x_4^2 - 0.41\, x_1x_2x_3 - 0.35\, x_2x_3x_4 - 0.68\, x_1x_3x_4 - 0.12\, x_2x_3x_4 - 4.46\, x_1^2x_2 + 3.45\, x_1^2x_3 - 5.06\, x_1^2x_4 - 3.46\, x_1x_2^2 - 0.81\, x_1x_2x_3x_4 \quad (4)$$

where y_1 is defined as the NH_3-N removal in the permeate solution and x_1, x_2, x_3 and x_4 represent normalized ABFR, HRT, MLSS, and pH.

Figs.3 shows 3-D graphs of NH_3-N removal. It is clearly shown by Fig. 3a that the slight increment of NH_3-N removal occurs monotonously with the increase of ABFR at HRT = 180 min.On the other hand,,NH_3-N removal shows a maximum at ABFR of 2.25 mL/min when HRT is maintained at 300 min. Similar trend was illustrated in Fig. 3b, i.e. NH_3-N removal increased with an increase inABFR from 1.2 mL/min to 2.25 mL/min, and then decreased with further increase in ABFR when pH was 8.00. The reason for the presence of a critical ABFR value has already been given, while discussing its effect on

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TSS removal.The ABFR must be carefully controlled to maintain adequate expansion and liquid-liquid mass transfer while minimizing shear effects. Rosenberger *et al*. (2002) alsomentioned that the smaller particle size in aerated submerged ultrafiltration was mainly due to violent turbulence that aeration produced under membrane bundles [13].In Fig. 3d NH_3-N removal decreased slightly, at pH 8.00, when MLSS concentration increased from 3.00 g/L to 6.00 g/L. Both Figs. 5b and 5d show a strong effect of pH increase from 5 to 8. According to Fig.3cNH_3-N became the highest at the highest HRT value of 300 min. This was true at both the lowest (3.00 g/L) and the highest (6.00 g/L) of MLSS concentration.The above observation was consistent with the conclusion made by Bai and Tien(2005) [14].

It can thus be concluded that NH_3-N removal increased with increase in HRT and pHand decrease in MLSS concentration. ABFR must be carrefully controlled to maintain adequate expansion and mass transfer while minimizing shear effects near the optimum value. The concentration polarization on the membrane surface was also one of the factors, as has been observed at low ABFR. It should however be noted that there are other reasons for the high NH_3-N removal observed. The nitrogen compounds are adsorbed to the deposited matters thatare retained by the membrane in filtration process. Besides, the biomass also assimilates organic nitrogen. This high removal value was also possible due to nitrification reactions that occurred in the reservoir where ammonium was highly soluble in water. The ammoniumions thus formed can be readily reduced to nitrite and nitrate. Pulefou*et al*., (2008) observed that the percentage of ammonia removal increased with increasing alkalinity of dissolved ammonium in water, as it is known that ammonium ions are formed with increasing alkalinity (pH 8.00) [15]. Maximum NH_3-N removal efficiencies as well as operating variables at optimized values for treatment of refinery wastewater are listed in Table 5.

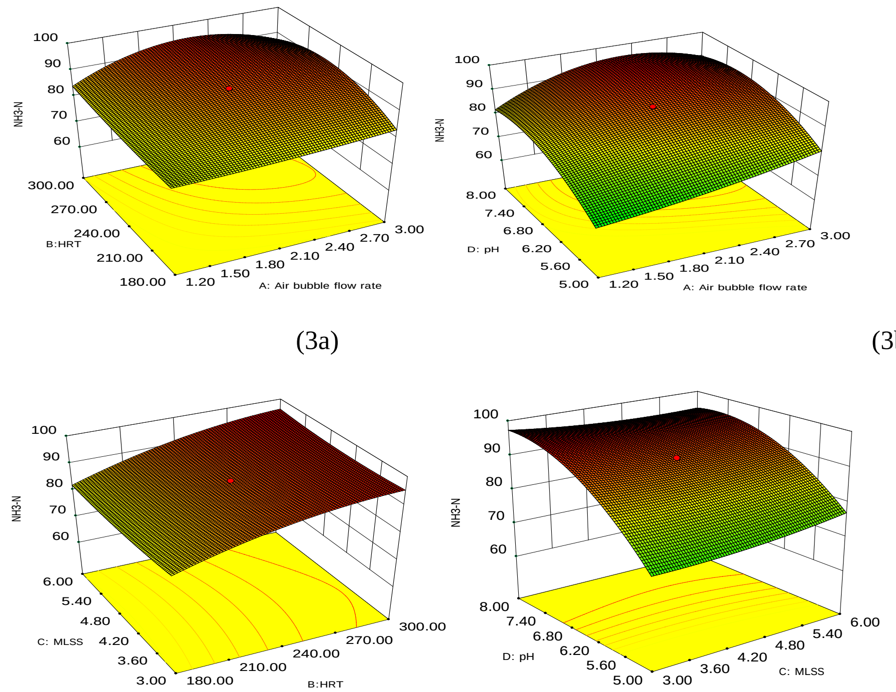
Table 4.Anova for response surface backward quartic model, Response: NH_3-N removal.

Source	Sum of squares	Degree of freedom	Mean square	F-value	Prob>F
Model	3874.01	23	168.44	1975.47	< 0.0001[a]
A	211.70	1	211.70	2482.85	<0.0001
B	344.22	1	344.22	4037.15	<0.0001
C	1.26	1	1.26	14.77	0.0184
D	946.51	1	946.51	11100.98	<0.0001
AB	0.42	1	0.42	4.88	0.0917
AC	0.28	1	0.28	3.32	0.1423
AD	19.44	1	19.44	228.01	0.0001
BC	4.09	1	4.09	48.00	0.0023
BD	4.08	1	4.08	47.86	0.0023
CD	42.55	1	42.55	499.06	<0.0001
A^2	356.95	1	356.95	4186.40	<0.0001
B^2	167.22	1	167.22	1961.23	<0.0001
C^2	29.66	1	29.66	347.85	<0.0001
D^2	950.46	1	950.46	11147.29	<0.0001
ABC	2.73	1	2.73	32.06	0.0048
ABD	2.01	1	2.01	23.52	0.0083
ACD	7.48	1	7.48	87.76	0.0007
BCD	0.23	1	0.23	2.68	0.1768
A^2B	106.04	1	106.04	1243.65	<0.0001
A^2C	63.58	1	63.58	745.66	<0.0001
A^2D	136.58	1	136.58	1601.86	<0.0001
AB^2	63.95	1	63.95	750.08	<0.0001
ABCD	10.41	1	10.41	122.15	0.0004
Residual	0.34	4	0.085		
Lack of Fit	0.21	1	0.21	4.55	0.1227[b]
Pure error	0.14	3	0.045		
Cor Total	3874.35	27			
Std. Dev.	0.29		R^2		0.9999
Mean	80.38		Adjusted R^2		0.9994

[a]Significant
[b]Not significant
Values of 'Prob>F' less than 0.0500 indicate model terms are significant

Table 5. Optimum process conditions (factors) for maximum response results with standard deviation (S.D.)

Factors	Optimum value (S.D.)
y_1 (NH_3-N removal efficiency,%)	92.89(0.29)
x_1 (Aeration flow rate, ml/min)	2.25
x_2 (HRT, min)	276.93
x_3 (MLSS, g/L)	4.50
x_4 (pH)	6.50



(3a) (3b)

(3c) (3d)

Fig.3. 3-D plot of NH_3-N from the model equation of effect the condition process: (a) ABFR-HRT, (b) ABFR-pH, (c) HRT-MLSS, (d) MLSS-pH.

Conclusions

Response surface methodology was used to find the optimal process parameters in suspended solid and ammonia nitrogen removal for refinery wastewater treatment. This statistical analysis shows that all the variables set in the preparation of the model, within the tested boundaries of the model, have significant effect on the model. Four process parameters, such as air bubble flow rate, hydraulic retention time, mixed liquor suspended solids and pH solution influence NH_3-N removal efficiencies. The optimal filtration conditions for maximum removal efficiencies of NH_3-N (92.89 %)found by RSM are satisfied at ABFR of 2.25 ml/min, HRT of 276.93 min, MLSS concentration of 4.50 g/L, and pH of 6.50.

In addition, it can be concluded that the four parameters tested have significant effect on NH_3-N removal, is borne out by the statistical analysis (ANOVA) of R^2 value were 0.9999, respectively. It has been concluded that a mathematical approach is useful for picturing the theoritical has been testified to be a powerful tool in studying submerged ultrafiltration process.

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