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OPTIMIZATION OF OPERATING PARAMETERS FOR AGGREGATION UNDER MAGNETIC FIELD BY RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Batch tests were employed to estimate the optimal conditions for improving the settleability of activated sludge through aggregation under magnetic field. A four – factor central composite design (CCD) was employed to find out the interaction effects of the variables while response surface methodology (RSM) was utilized for process optimization. Four independent variables, viz. magnetic field (15.0 – 88.0 mT), exposure time (0.5 – 48.0 hrs), biomass concentration (2000 – 4000 mg l^{-1}) and mixing intensity (200 – 400 rpm) was built to predict the responses. Analysis of variance (ANOVA) was used to evaluate the significance of the independent variables and their interactions. At the optimum conditions of 88.0 mT magnetic field, 16.5 hrs of exposure time, 2800 mg l^{-1} of biomass concentration and 300 rpm of mixing intensity, the aggregation achieved its maximum value by 99.0%. The element analysis showed that the applied magnetic field is potential to enhance the settling property of the activated sludge by improving its aggregation.

Keywords: magnetic field, activated sludge, RSM, aggregation, settleability.

INTRODUCTION

Activated sludge technology has taken a predominat role in municipal and industrial sewage treatment. One of the major problems with this biological technology is the separation and settling problem. Such problem lead to the deterioration of the effluent quality, sludge wash-out and further potential to collapse the overall treatment system performances (Martins *et al.* 2004; Lou and De los Reyes, 2008; Martins *et al.* 2011). Therefore, it is a necessity to investigate for a favorable strategy that able to enhance the property of activated sludge mainly on its settling and separation process.

In recent years, more studies have confirmed that the magnetic field application had many advantages. This application was reported to significantly influence the bacterial activity in heterogeneous sewage, resulted in the enhanced performances of organic compound biodegradations (Sakai *et al.* 1994; Yavuz and Çelebi, 2000; Ji *et al.* 2010; Łebkowska *et al.* 2011). Under low intensity of magnetic field, activated sludge biomass growth and dehydrogenase activity was positively affected (Łebkowska *et al.* 2011). As a result, the 7 mT field reduced the formaldehyde (FA) concentration and COD by 30% and 26%, respectively.

Liu *et al.* (2008) applied magnetic field of ranged 16.8 and 218.0 mT and resulted in significant nutrient removal. It was reported that maximum nitrogen removal occurred at intensity of 60 mT. Apparently, an excessively strong magnetic field could harmed the anammox bacteria while the low field intensity has no or less effect to the bacteria. Thus, it is of great importance to study the variation of magnetic field intensity towards the physical properties of activated sludge.

Hence, the aim of this study is to investigate the influence of magnetic field, exposure time, biomass concentration and mixing intensity on the separation and settling property of activated sludge in terms of aggregation. The operating parameters used in this aggregation enhancement are important and different combinations of these parameters will affect the effectiveness of the enhancement. Traditional method known as the one-factor-at-a-time (OFAT) does not fully explore all phenomena that could possibly occur and may cause misinterpretation of the results (Frigon and Mathews, 1997; Montogomery, 2005; Kusic *et al.* 2010; Dopar *et al.* 2011). Central composite design (CCD) approach was then employed to quantitatively analyze the effects of those factors, the interactions between them and to indicate any correlation between the factors and the response. It is expected that the optimal operating conditions resulting from these batch tests will offer important reference values for later continuous flow experiments.

MATERIALS AND METHODS

Activated Sludge Characteristic

The activated sludge used in this study was obtained from Pulai Emas sewage treatment plant located in Johor, Malaysia. The mixed liquor suspended solids (MLSS) of the activated sludge is ranged between 12000 mg l^{-1} to 23000 mg l^{-1}.

Experimental Design

The magnetic field, exposure time, biomass concentration and mixing intensity were identified as the set of four independent variables that been investigated due to the influence on the aggregation of activated sludge. The CCD method was adopted to decide the number of batch test experiments to be performed for optimization of the process variables. The limits of the variables were coded in ± 1 for factorial points, 0 for

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centre point and ± α for axial points. In this study, α for axial points was set to 1.0 (also known as face-centered) based on consideration that the region of interest is approximately same as the region of operatibility (Kraber, 2002). Thus, the limit for axial points were eventually same as the factorial points. The defined range of process variables with their limits are given in Table-1.

Table-1. Range of process variables and their limits.

Variables	Factorial		Star points*		Centre point
	-1	+1	-1	+1	0
A: Magnetic field (mT)	15.0	88.0	15.0	88.0	51.5
B: Exposure time (hrs)	0.5	48.0	0.5	48.0	24.25
C: Biomass concentration (mg l^{-1})	2000	4000	2000	4000	3000
D: Mixing intensity (rpm)	200	400	200	400	300

* Limit for star points is the same as factorial due to the consideration taken of α = 1.0 (face-centered)

Batch Test

A total volume of 500 mL containing mixture of measured wet volume activated sludge biomass (2000 – 4000 mg l^{-1}) and raw wastewater was mixed in a glass flask that placed in a shaker. The shaker was fabricated to allow an installation of the permanent magnets arranged at all four surfaces of the flask in an alternate order. The magnetic field was produced by the pairs of magnets. Each magnet was a square prism with two faces of 100 mm x 50 mm and a thickness of 5 mm.

Analytical Methods

All tests were conducted according to Standard Methods for the Examination of Water and Wastewater (APHA, 2005) – MLSS with method 2540B. As for aggregation, the parameter was measured in terms of turbidity. After the reaction under magnetic field exposure was stopped, turbidity of 10 mL MLSS with concentration ranged between 20 mg l^{-1} and 40 mg l^{-1} was measured instantaneously and recorded as turbidity at 0 min. The residual turbidity of the supernatant is then measured after 10 min and the value is recorded as final turbidity. The aggregation ability can be expressed in percentage (Ag%) and calculated using equation. (1).

$$Ag\% = [(T_0 - T_i) / T_0] \times 100\% \quad (1)$$

where Ag% is percentage of aggregation of the magnetically-exposed activated sludge; T_0 is turbidity value at 0 min; T_i is turbidity value after 10 min of idle.

RESULTS AND DISCUSSIONS

Factorial Analysis for Aggregation

Summary of the p-values for the main and interaction effects is shown in Table-2. The p-values indicate that magnetic field, exposure time and biomass concentration are significant in improving the aggregation by the estimated positive effect of +7.73, +11.39 and +7.44, respectively.

Table-2. P-values for the estimated main and interaction effects on aggregation.

Variables	p-value
A: Magnetic field	0.000
B: Exposure time	0.000
C: Biomass concentration	0.000
D: Mixing intensity	0.802
A x B	0.000
A x C	0.003
A x D	0.275
B x C	0.001
B x D	0.717
C x D	0.196
A x B x C	0.035
A x B x D	0.802
A x C x D	0.206
B x C x D	0.468
A x B x C x D	0.680

Figure-1 shows the plot of the main effects for the aggregation. Among those three variables, the percentage of aggregation was higher (i.e. 97.3%) during an increased in exposure time. Greater exposure time would ensure the magnetic effect been sustained by particles lasts much longer, therefore enable the improvement in coagulation, aggregation and precipitation (Tombácz *et al.* 1991; Higashitani *et al.* 1992; 1993; Colic and Morse, 1998; 1999). For instance, studies conducted by Xu and Sun (2008) shown an increased by 12% of heavy metal chromium removal prior to the occurred coagulation under weak magnetic field intensity of 6 mT which been continuously exposed within 24 hrs.

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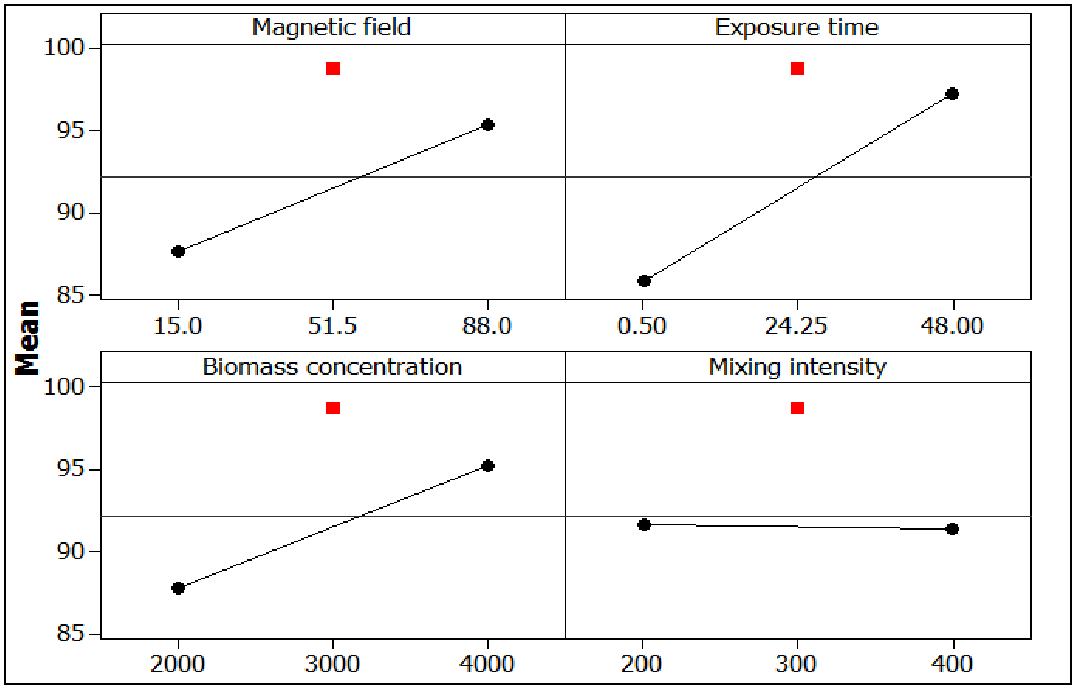

Figure-1. Main effects plot for the aggregation of activated sludge (■ Centre point).

Meanwhile, positive effect indicated by magnetic field has also been agreed by Chin *et al.* (2006). They observed an increase in turbidity removal from 99% to almost 100% when the applied magnetic field was increased from 80 mT to 130 mT. Such observations can be explained in terms of electrostatic attraction arising from the fact that the particles are in high oppositely charged. Under normal condition, the particles are positioned randomly, thus makes it impossible to attach to each other. With the increase in positive and negative charges that resulted due to the increase in magnetic field, the particles could be easily aligned in accordance to their charges. This would favor collisions that can eventually enhance the aggregation performances (Bernardin and Chan, 1991; Vick, 1991).

As for biomass concentration, the positive effect as shown in Figure-1 was due to the reason that more microorganisms are available during the reaction process. The presence of more cell biomass increases the collision among the cells thus potential to cause aggregation. The positive effect on the aggregation given by the increase of biomass concentration can also be explained in terms of the EPS production. More available microorganisms' means more EPS will be produced (Wang *et al.* 2006). The formation of the EPS that covers the cell surface could be regarded as polyelectrolyte adsorbed onto a colloidal particle. The presence of EPS could alter the characteristics of the physicochemical properties of the cell surface which includes the surface charge, surface hydrophobicity and others. These kinds of changes in the bacterial surface properties are important towards improving the aggregation properties of activated sludge (Veiga *et al.* 1997; Liu *et al.* 2004).

Instead of the main effects, the interaction effects which are presented by two-way interactions between the variables have also been observed to be significance. Figure-2 shows the interaction plots between the variables on the aggregation. Literally, interactions between magnetic field and exposure time, magnetic field and biomass concentration, as well as exposure time and biomass concentration are significant with p-values of 0.000, 0.003 and 0.001, respectively.

For the interaction plot between the magnetic field and exposure time, low range of 15.0 mT magnetic field improved the aggregation from 79.2% to 96.2% as the exposure time increased from 0.5 hr to 48.0 hrs. The improvement is about 17% compared to the high range magnetic field of 88.0 mT which only produced about 6% increment. This result can be reasoned in terms of the ability of microorganisms in the activated sludge in sustaining the magnetic effect. In the early stage, higher magnetic field contributed to the higher aggregation compared to the low field intensity. However, throughout increased in exposure time, the aggregation enhancement was significantly progressed at low field intensity because microorganisms are being more active as it sustained greater magnetic effect throughout prolonge exposure. At high magnetic field intensity, microorganisms in the activated sludge may already reach its saturation state in adapting the magnetic field effect, thus resulted to slight aggregation enhancement. According to the previous published researches, microorganisms do have their unique susceptibility level towards the magnetic effect. If the level is exceeded, the microorganisms may show adverse effect to the system performances (Yavuz and Çelebi, 2000; Zaidi *et al.* 2014).

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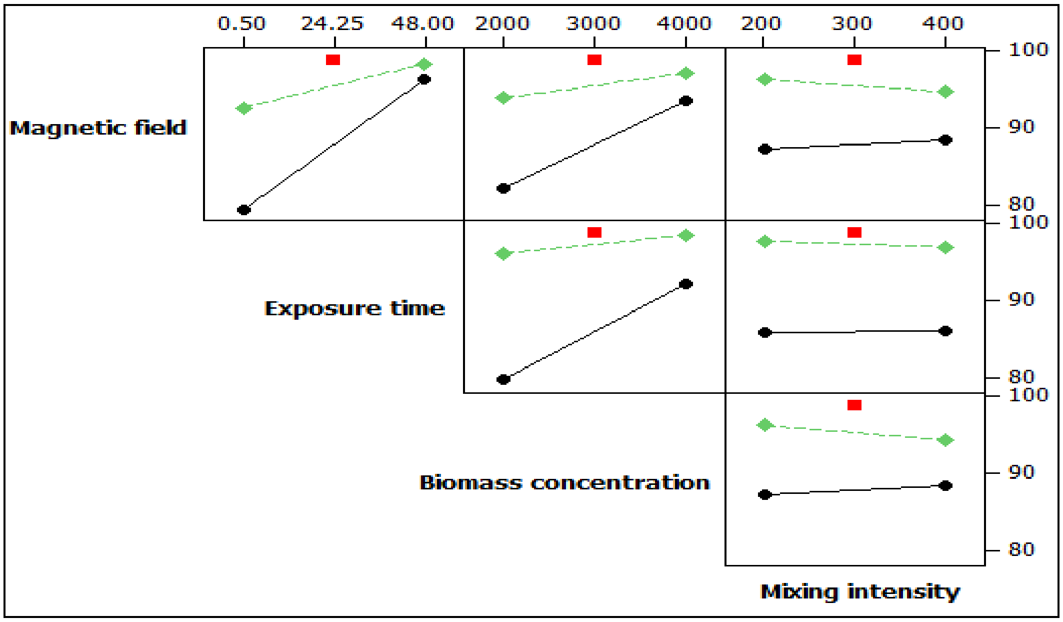

Figure-2. Interaction plot for aggregation (—◆—Low range factors of 15.0 mT, 0.50 hr and 2000 mg l^{-1}; –■– Center points of 51.5 mT, 24.25 hrs and 3000 mg l^{-1}; --◆-- High range factors of 88.0 mT, 48.00 hrs and 4000 mg l^{-1}).

Meanwhile, for the interaction between magnetic field and biomass concentration, the increased in the concentration led to the increase in aggregation at both low and high magnetic field. It was also observed that the aggregation was initially high (93.8%) at high magnetic field even with low biomass concentration. This could be reasoned that high magnetic field of 88.0 mT able to initiate the aggregation occurrence although less microorganisms were available during the reaction period. Such occurrence might be due to the changes occurred in the activated sludge characteristics with regard to the magnetic field exposure. The magnetic field may change hydrophobicity and surface charge properties of the activated sludge flocs. Initially, the conditions of the flocs are in negative charges (Jin *et al.* 2003). With the exposure of magnetic field, the negative charges may be neutralized, thus provide great opportunity for the aggregation to occur. As for low magnetic field, higher the biomass concentration resulted to progress increased of aggregation. This is because the presence of more cell biomass allows the collision among the cells to occur more rapid, thus further potential to cause aggregation.

For the interaction effect between the exposure time and biomass concentration, it can be seen in Figure-2 that there is greater gap of the aggregation performances when low and high exposure time were implemented. At low exposure time (0.5 hr), the aggregation of 2000 mg l^{-1} biomass concentration was very low. Gradual increment was then evidenced when the biomass concentration is increased to 4000 mg l^{-1}. On the other hand, at high exposure time (48.0 hrs), the aggregation was initially high (96.1%) even under low biomass concentration. As the concentration increased, the aggregation was only slightly increased. The result was unsimilar to the result shown by the low exposure time. The reason that can support this occurrence is that higher exposure time have already improved the aggregation during initial stage at low biomass concentration. An increase in the concentration only provides more microorganisms. However, with prolonge of time, there were less available organic compounds thus causing the microorganisms to be starved. As a result, small improvement of the aggregation was evidenced.

Response Surface Analysis for Aggregation

The analysis of variance (ANOVA) for the responses is summarized in Table-3. Generally, the results show that the linear terms of magnetic field, exposure time and biomass concentration, square term of exposure time and interaction terms between magnetic field and exposure time, magnetic field and biomass concentration as well as between exposure time and biomass concentration are significant. The analysed R-squared value of 94.4% indicates the acceptability of the model. Statistical model as shown in equation. (2) was then developed to relate the experimental variables to aggregation.

$$\text{Aggregation} = +98.87 + 3.39*A + 5.89*B + 3.36*C - 0.20*D - 0.021*A^2 - 7.62*B^2 - 0.12*C^2 + 0.46*D^2 - 2.82*A*B - 2.09*A*C - 0.68*A*D - 2.53*B*C - 0.22*B*D - 0.81*C*D$$

Table-3. ANOVA results for aggregation.

Terms	Aggregation (p-values)
A: Magnetic field	< 0.0001
B: Exposure time	< 0.0001
C: Biomass concentration	< 0.0001
D: Mixing intensity	0.7578
A^2	0.9901
B^2	0.0003
C^2	0.9422
D^2	0.7828
A x B	0.0007
A x C	0.0063
A x D	0.3195
B x C	0.0016
B x D	0.7408
C x D	0.2380
R-squared value	94.4%

Figure3a illustrate the relationship between the magnetic field and exposure time on aggregation of the activated sludge with p-value of 0.0007. As shown in the figure, at low magnetic field of 15.0 mT, an increase in exposure time resulted in drastic increment of aggregation from about 83.0% to 98.6%. However, the increment is in concave shape indicated that further increase in exposure time would lead to the decrease in aggregation. This could be due to the ability of microorganisms in sustaining high magnetic effect as a result of longer exposure time. Some of the microorganisms can only sustain low level of magnetic effect. If this level is exceeded, the microorganisms may act adversely, thus show reverse effect to the activated sludge property (Yavuz and Çelebi, 2000; Łebkowska *et al.* 2011; Zaidi *et al.* 2014). Contrary, an increase in the magnetic field shown increment on aggregation (at low exposure time of 0.50 hr) in more saddle shape. This kind of surface plot suggested that the increase was in more progressive manner, thus showing that the aggregation enhancement of activated sludge is more stable under such condition.

Meanwhile, the contour and response surface plot which illustrate the relationship between the aggegation and independent variables of magnetic field and biomass concentration is shown in Figure3b. The figure shows the surface plot that represent the effect of varying magnetic field and biomass concentration at fixed exposure time (24.25 hrs) and mixing intensity (300 rpm). It can be seen that increasing the magnetic field at low biomass concentration level had caused an increase in the aggregation of activated sludge with a high percentage of more than 98.6%. The similar trend is also observed as high biomass concentration was used under the increase of magnetic field. Having conditions at both high magnetic field level and biomass concentration would be the best conditions for achieving maximum percentage of aggregation.

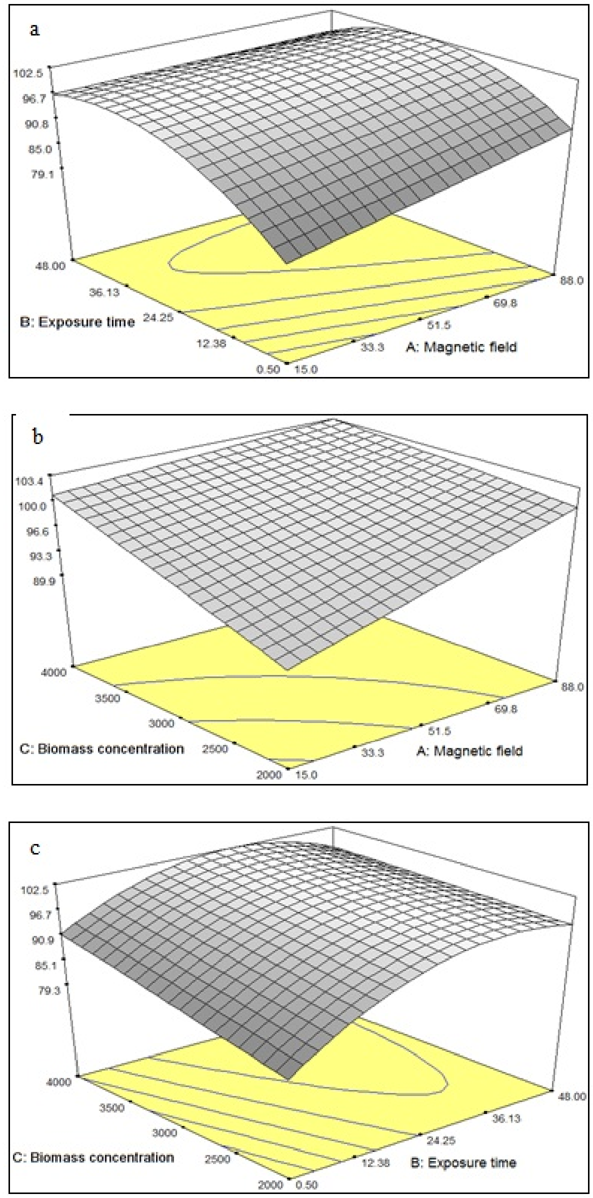

Figure-3. 3-D responce surface plots as a function of a) magnetic field and exposure time b) biomass concentration and magnetic field and c) biomass concentration and exposure time.

Additionally, Figure-3c shows the relationship between aggregation and independent variables of exposure time and biomass concentration. The relationship was significant with p-value of 0.0016. The interaction was observed at constant values of magnetic field (51.5 mT) and mixing intensity (300 rpm) as the hold values. The nearly symmetrical mound shape plot shows that at any biomass concentration level, the percentage of aggregation increases as the exposure time increased from 0.5 hr to about 36.0 hrs. However, as the exposure time further increase from 36.0 hrs to 48.0 hrs, the response started to reduce. The highest percentage of response is 98.6% obtained at exposure time of 36.1 hrs and biomass concentration of about 2500 mg l^{-1}. The point that shows the highest percentage of aggregation was positioned approximately at the middle of nearly symmetrical mound shape plot.

Experimental Condition Optimization

The optimization of experimental conditions was identified by considering whether the percentage of aggregation was higher than the arbitrarily chosen constraint values. According to the model, the predicted optimized conditions occurred at magnetic field of 88.0 mT, 16.5 hrs exposure time, 2800 mg l^{-1} of biomass concentration and at mixing intensity of 300 rpm. These conditions resulted in 99.0% of aggregation. As to verify the obtained optimization result, triplicate batch test experiment was carried out. Average aggregation of 97.8% was recorded against the predicted optimum responses. Although there is difference between the predicted and experimental values of the optimized response, the deviation was still well in agreement (≈ ± 3%).

CONCLUSIONS

The experimental results showed that the magnetically-exposed activated sludge performed much better in terms of aggregation. Under the optimal condition of 88.0 mT magnetic field, 16.5 hrs exposure time, 2800 mg l^{-1} biomass concentration and 300 rpm mixing intensity, the maximum aggregation of 99.0% was obtained. Overall, the element analysis suggested that the applied SMF could enhance the settling property of the activated sludge through the improvement on its aggregation capability.

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