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Full Length Research Paper

Adsorption of heavy metal from landfill leachate by wasted biosolids

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The adsorption capacity of wasted solids that contained dead fungal biomass (*Phanerochaete chrysosporium*) was studied to remove cadmium, copper, zinc and iron from synthetic water and leachate. The biomass was produced due to the experiments conducted for bioconversion of wastewater for lignin peroxydase production in the laboratory. In the screening experiments, the maximum cadmium (Cd) adsorption from synthetic water was 28.81% at 18 h. Meanwhile, adsorption of copper (Cu), zinc (Zn) and iron (Fe) reached maximum condition after 5 h with 41.29, 58.94 and 52.03% removal efficiency, respectively. However, the concentration of Cd, Cu and Zn was not detected in the leachate but Fe was found to be in high concentration (184 mg/L) in raw leachate collected from a municipal landfill site. Therefore, the effects of biomass dosage, contact time, pH and agitation speed were observed for optimal adsorption of iron from leachate. Optimum removal of iron from leachate was 45.56% in every 1 L of leachate after 1/50 dilutions. The optimized biomass dosage, contact time, pH and agitation speed were 750 mg/L, 4 h, pH 5, and 150 rpm, respectively. The results of this study indicated that the biomass generated from the laboratory experiments could be used, before discarding, to remove iron from leachate which is one of the main problems at the landfill sites.

Key words: Adsorption, biomass waste, heavy metal, synthetic water, leachate.

INTRODUCTION

Healthy living is somehow related to good quality of water. Quality of water is deteriorating at every inhabited part of the globe, due to various anthropogenic activities (Nery and Bonotto, 2011). Heavy metals (Pb, Zn, Cu, Ni, Cd, Cr, As, Hg, etc.) generated from various human activities are increasing toxicity in the surface and ground water resources (Cuevas et al., 2011; Satyawali et al., 2011). Leachate from the landfill sites is one of the pollutants, which affects both sources. Leachate is considered as one of the highly polluted wastewater, which may not be suitable for direct biological treatment due to presence of toxic elements (Evanko and Dzombak, 1997).

Chemical precipitation treatment (Meunier et al., 2006) and some other methods such as ion exchange (Fiskum et al., 2005) and reverse osmosis (Chianese et al., 1999) processes are often required to treat leachate, which are costly and usually not environmentally friendly solution (Kurniawan et al., 2006). Removal of cadmium and cyanide from effluents through electro dialysis, removal of cadmium by adsorption on granular activated carbon (GAC) are few of the effective but expensive methods of heavy metals removal for the industrial applications (Li et al., 2003). The costs for these types of operations are relatively high as compared to the current requirements of treatments needed in this country. For example, treatment by water hyacinth consumes a vast amount of land spaces due to the large water surface area needed to grow the plants. Adsorption of the pollutants from leachate could be potential solutions for the problems associated with it. Generally, adsorption is a process that occurs when the heavy metal ions, referred to as adsorbate, accumulates on the surface of an adsorbent, forming a molecular or atomic film (Masters, 1998).

Aluminium salts (El Samrani et al., 2008) and synthetic

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or natural polymers (Chang et al., 2009) are frequently used as primary adsorbent. These elements are added into water to accomplish adsorption where it turns the small particles into large flocs to be readily removed in the subsequent process (Masters, 1998). A significant portion of the toxic metals can be removed by chemical precipitation and adsorption. However, the usage of aluminium salt have some drawbacks such as higher Al content in the water, reduction in water flow by formation of hydrous Al precipitation, effect in the disinfection process due to flocks of Al (Masters, 1998). Furthermore, operational cost of treatment and waste disposal can increase due to production of large volumes of gelatinous sludge caused by the aluminium salts. Activated materials, polymers and most of the commercial adsorbents are still costly to treat large volume of leachate produced in tropical countries such as Malaysia. Thus, an immediate alternative and cheap treatment method should be taken into account.

Therefore, the study was aimed to develop alternative process using cheap biomaterials to treat leachate in more environmentally accepted way. Few biomaterials (fungi, yeast and bacteria) have been used for biosorption of pollutants from wastewater. Specifically, fungi from *Fusarium* species have been used as biosorbents, and *Penicillium simplicissimum* has been used to precipitate metals from solution (Burgstaller and Schinner, 1993; Gadd, 1993; White et al., 1997). There is also current interest in the use of these microorganisms for the removal of nitrogen, phosphorus and metals from commercial and municipal waste (Cassidy et al., 1996). Several other species of fungus (*Aspergillus niger*, *Phanerochaete chrysosporium*, etc.) have been potential for the treatment of leachate.

Biosorption of pollutants through wasted biomass would provide a cheaper solution for leachate treatment. A dead batch of fungal biomass would provide better heavy metals adsorption advantages in terms of costs, preparations and applications. The usage of dead fungal biomass offers negligible metals toxicity limitations prior to the adsorption process, no requirements for growth media and nutrients, and the dead biomass system can be subjected to conventional theories and mathematical models already in use for traditional adsorption system (Gadd, 1993). Therefore, a fungal biomass could also possibly serve as an economical means for removal/ recovery of metal ions from leachate (Kapoor et al., 1999). As such, this study aimed at searching the potentiality of biomass waste (fungus) to remove pollutants from leachate.

MATERIALS AND METHODS

Synthetic wastewater

Metal salts (copper, zinc and iron) that were dissolved with deionized water were used. All solutions were prepared in the laboratory. Four types of heavy metals solutions (cadmium, copper, zinc and iron) were tested for the biomass adsorption feasibility test. Due to limitation of the HACH spectrophotometer analysis range, synthetic wastewater was prepared with maximum concentrations of 0.3 mg/L cadmium (Cd), 5.0 mg/L copper (Cu), 2.0 mg/L zinc (Zn) and 5.0 mg/L iron (Fe).

The percentage removal of heavy metals from wastewater was calculated by:

$$\text{Removal (\%)} = [(C_i - C_f) / C_i] \times 100 \qquad (1)$$

Where, Ci is the initial concentration of the heavy metals (mg/L) and Cf is the final concentration (mg/L).

Leachate from sanitary landfill

Leachate containing heavy metals (cadmium, copper, zinc, iron) obtained from sanitary landfill managed by Solid Waste Disposal Sdn. Bhd. in Taman Beringin Kuala Lumpur was used. This leachate source represents the Malaysian leachate composition in this experiment.

Biosolids

The dead fungus in the biomass used for the removal of leachate heavy metals was obtained from the laboratory store, which is a waste byproduct of the previous research project, bioconversion of wastewater for lignin peroxydase production (Alam et al., 2003). The desired raw material (dead fungal *Phanerochaete chrysosporium*) was in the form of mixture with other wastes such as municipal wastewater sludge and wheat flour.

The raw material is in bulk form, thus it was grinded into powder form (0.5 mm size) using a grinder. Small size will increase the surface area of the biomass and capability of the heavy metals to be adsorbed. After grinding, it was dried at 100℃ in an oven before it was kept in the desiccators to avoid moisture for further use.

It was initially suspected that the sludge itself contained other various microorganisms even before the dead fungal biomass was applied. Thus, the sludge was initially sterilized to make sure that the sludge did not have any unwanted microorganisms. It was also presumed that the sludge contained traceable amounts of Cu (≈110.0 mg/kg), Cd (≈13.8 mg/kg), Fe (≈15 g/kg) and Zn (≈2.2 g/kg), according to the previous study. Thus, theoretically, iron content in the biosolids used for this study is 15.01 or 0.015 mg iron metals/mg biosolids. This data was taken into consideration when desorption of iron occurred during the duration of the experiment. The amount of adsorbed metal ions per g biomass was obtained by using the following expression:

$$Q = [(C_o - C) V] / M_b \qquad (2)$$

Where, Q is the amount of metal ions adsorbed on the biomass (mg/g), C_o and C are the concentrations of the metal ions in the wastewater (mg/L) initially and after adsorption. V is the volume of the medium (L) and M_b is the amount of the biomass (g).

Statistical analysis

Statistical analysis for iron adsorption was done using Minitab® software Release 14 (Minitab Inc.). The optimization was done using central composite design (CCD) with four factors: Biomass dosing, contact time, pH and agitation speed with five levels, two replication, three center points and one block for all parameters.

The variables and their levels for the central composite experi-

Table 1. Actual values of the factors for CCD with the experimental and predicted values of iron removal efficiency.

Run	Biomass (mg/L)	Contact time (hour)	pH	Agitation speed (rpm)	Residual Fe concentration (mg/L)	Actual removal (%)	Predicted removal (%)
1	500	2	4	100	3.664	0.6	-0.2
2	500	2	4	200	3.652	0.9	-0.6
3	500	2	6	100	3.425	7.1	12.1
4	500	2	6	200	3.210	12.9	14.1
5	500	6	4	100	3.615	1.9	6.4
6	500	6	4	200	3.637	1.3	-0.1
7	500	6	6	100	2.820	23.5	23.6
8	500	6	6	200	2.830	23.2	22.7
9	1000	2	4	100	3.619	1.8	-0.7
10	1000	2	4	200	3.633	1.4	-0.9
11	1000	2	6	100	3.180	13.7	16.2
12	1000	2	6	200	3.300	10.4	13.1
13	1000	6	4	100	3.365	8.7	12.9
14	1000	6	4	200	3.656	0.8	1.3
15	1000	6	6	100	2.760	25.1	33.0
16	1000	6	6	200	2.755	25.2	27.0
17	250	4	5	150	2.925	20.6	20.1
18	1250	4	5	150	1.955	46.9	25.7
19	750	1	5	150	3.175	13.8	5.6
20	750	7	5	150	2.590	29.7	22.3
21	750	4	3	150	3.651	0.9	-1.6
22	750	4	7	150	2.800	24.0	23.4
23	750	4	5	50	2.218	39.8	31.9
24	750	4	5	100	1.245	66.2	45.1
25(C)	750	4	5	150	2.275	38.3	47.9
26(C)	750	4	5	150	1.985	46.1	47.9
27(C)	750	4	5	150	1.960	46.8	47.9

mental designs for iron adsorption are shown in Table 1.

Iron spectrophotometry readings stated that iron ions in the leachate were over the range as compared to the analysis range of the HACH spectrophotometer. Dilution operation with deionized water has been applied with the factor of 1/2, 1/10 and 1/20. Only after the application of dilution with the factor of 1/50, reading of iron concentrations in the leachate was obtained with the average value of 3.685 mg/L. Thus, theoretically, the actual value of iron concentration in the leachate is: 3.685 mg Fe/L x 50 = 184.25 mg Fe/L leachate.

The heavy metals removal efficiency was taken as the dependent variables of response (Y). A second order polynomial equation was then fitted to the data by multiple regression procedure. This result in empirical model related the response measured in the independent variables to the experiment. For a four factors system, the model equation is:

$$Y= \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_3x_3 + \beta_4x_4 + \beta_{11}x_{12} + \beta_{22}x_{22} + \beta_{33}x_{32} + \beta_{44}x_{42} + \beta_{12}x_1x_2 + \beta_{13}x_1x_3 + \beta_{14}x_1x_4 + \beta_{23}x_2x_3 + \beta_{24}x_2x_4 + \beta_{34}x_3x_4 \quad (3)$$

where, Y is the heavy metals removal efficiency (%), predicted response; β_0, intercept; β_1, β_2, β_3, β_4, linear coefficient; β_{11}, β_{22}, β_{33}, β_{44}, squared coefficients; β_{12}, β_{13}, β_{14}, β_{23}, β_{24}, β_{34}, interaction coefficients.

RESULTS AND DISCUSSION

Removal of heavy metals from synthetic wastewater

The concentration (mg/L) of Cd, Cu, Zn and Fe in the untreated wastewater were 0.295, 4.77, 1.985 and 4.67, respectively and the pH were 6.33, 6.2, 5.62 and 6.12, respectively. Although, Mittar et al. (1992) claimed that live *P. chrysosporium* has the ability to adsorb heavy metal ions from wastewaters on its mycelium, but since the fungal biomass in this study was mixed with biosolids and the fungus itself is a dead fungus, a feasibility test was necessary, in order to verify the capability of the dead fungal biomass to adsorb heavy metal ions in leachate. It was observed that cadmium adsorption reached

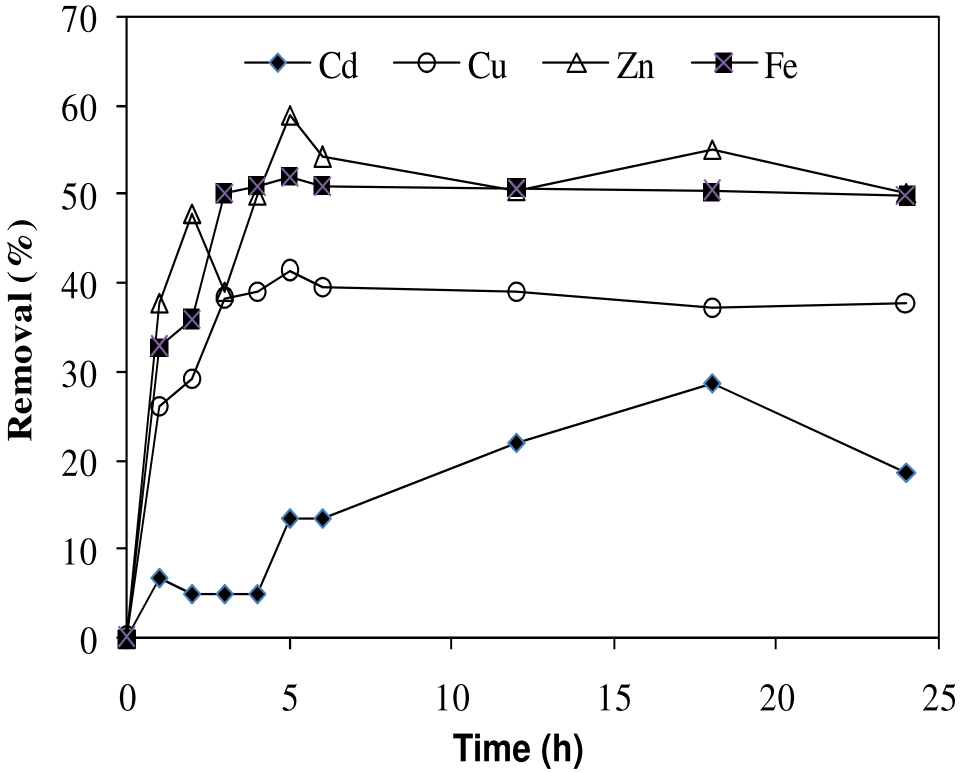


Figure 1. Heavy metal removal from synthetic water.

its optimum after 18 h with 28.81% removal (Figure 1), meanwhile copper, zinc and iron adsorption reached their optimum after 5 h with 41.29, 58.94 and 52.03%, removal respectively.

Conclusively, equilibrium level for the feasibility test was gradually reached between 5 to 6 h for all heavy metals solutions except for cadmium, in which the only possible reason was human operator error. According to Say et al. (2000), after this equilibrium period, the amount of heavy metals ions uptakes did not change significantly with time.

Biomass adsorption capacity for synthetic wastewater

Biosorption capacity of the dead fungal biomass (mg metal ions/g biomass) was observed to increase gradually for all heavy metals solutions. The first 4 h of the cadmium ions experiments showed no obvious change in the adsorption capacity with about 0.004 to 0.006 mg cadmium/g biomass (Figure 2), but it had a significant increment after 5 h of operation with the value of 0.014 mg cadmium/g biomass. Although, the adsorption capacity continued to increase until after 12 and 18 h (0.032 mg cadmium/g biomass), equilibrium or saturation was assumed to occur after 24 h of the process since the adsorption capacity decrease to 0.02 mg cadmium/g biomass.

Except for the cadmium adsorption capacity experiment, it was observed that from all the other experiments, the adsorption capacity from 6 to 24 h were usually lower than their highest peak which was at the 5th hour. It is believed that after 5 h of the experiments, desorption of iron ions occurred since the biosolids itself contains ferrous compound.

Thus, it can be said that the dead fungal biomass in the biosolids has the capability for adsorption of heavy metals ions from the synthetic wastewater and the next step of the experimental method can proceed.

Statistical optimization of heavy metals adsorption

Based on the experimental study (Table 1), it was observed that removal efficiency of the dead fungal biomass generally improved with increasing dosing of the adsorbent, although, some runs show unusual observations. Except for comparison between Run 1 and 9, Run 2 and 10, and Run 4 and 12, all the other experiments comparisons show expected characteristics of the increment in the biomass dosage. It was obvious that from comparison of Run 1 and 9 and Run 2 and 10, it was significantly affected by the low level of pH (pH 4), in which desorption occurred. Thus, we can say that at low pH (pH < 5), increment of biomass dosage will result in desorption.

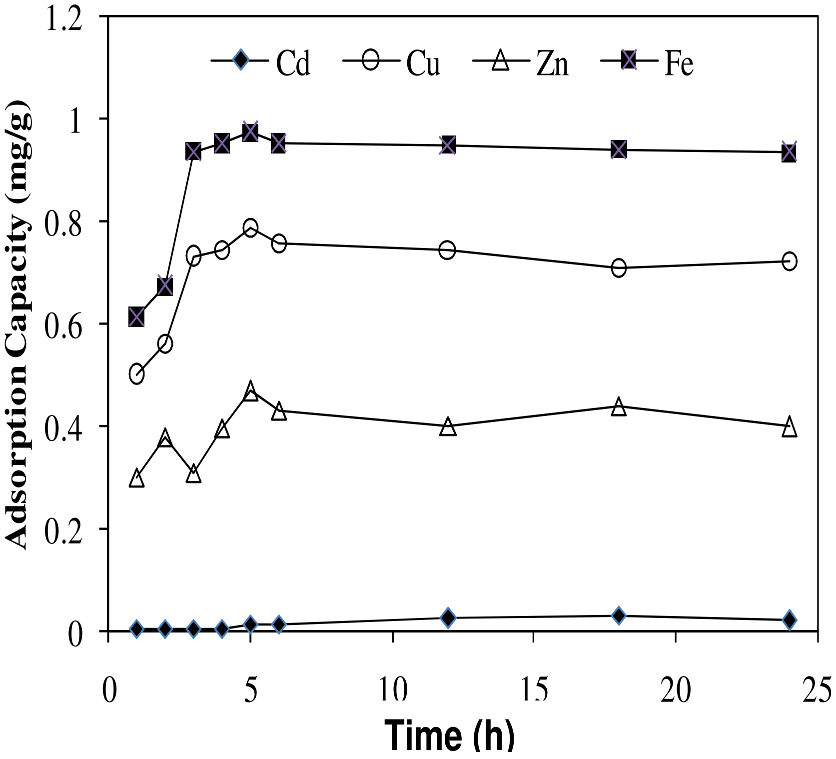


Figure 2. Heavy metal adsorption capacity by biomass.

The value of pH is also an important parameter for adsorption of metal ions from leachate because it affects the solubility of the metal ions, concentration of the counter ions on the functional groups of the adsorbent and the degree of ionization of the adsorbent during reaction. Several researchers have investigated the effect of pH on biosorption of heavy metals by using different kinds of microbial biomass. For example, the biosorption of Cu (II) by non-living *Saccharomyces cerevisiae* was pH dependent and maximum biosorption was obtained in the pH range of 5 to 7 (Huang et al., 1990), and the optimum pH for Cd (II) uptake was 6 for immobilized *Zoogloea ramigera* cells (Park et al., 1999).

It was found from the result (Table 1) that the uptake of ionic Fe depends on pH, where optimal metal removal efficiency occurred at pH 6. The medium pH affects the solubility of metals and the ionization state of the functional groups (carboxylate, phosphate and amino groups) of the fungal cell wall. The carboxylate and phosphate groups carry negative charges that allow the fungal cell wall components to be potent scavengers of cations (Say, 2000), in this case, the iron metals. It was obvious that Runs 1, 2, 6, 9, 10 and 21 it were significantly affected by the low level of pH (pH 3 to 4), in which desorption occurred because their removal percentage turned out to be negative values. Thus, we can say that at low pH (pH < 5), desorption of metal ions was likely to occur when this biosolids were applied for leachate treatment.

The removal of iron metals increased with an optimum condition of 100 rpm with respect to agitation speed. But, theoretically, the increase of the agitation speed should improve the diffusion of Fe ions towards the surface of the adsorbents. The range of contact time was estimated by referring to the feasibility test, which was between 4 to 6 h of operations. The adsorption efficiency was increased for about 40% when the contact time was 7 h. This result is important, as equilibrium time is one of the important parameters for an economical wastewater treatment system.

The experimental results were then analyzed by regression analysis from the Minitab 14 software, which gave the following regression equations of the iron ions removed as a function of biomass dosage (x_1), contact time (x_2), pH (x_3) and agitation speed (x_4).

$$\text{Fe removal (\%)} = -419 + 1.46x_1 + 30.1x_2 + 113x_3 + 0.541x_4 - 0.0100x_{12} - 3.77x_{22} - 11.1x_{32} - 0.00208x_{42} + 0.0265x_1x_2 + 0.0285x_1x_3 - 0.00102x_1x_4 + 0.390x_2x_3 - 0.0073x_2x_4 + 0.0282x_3x_4 \quad (4)$$

The t-value and probability value (p-value) serves as a tool for checking the significance of each of the coefficient. The pattern of interactions between the variables is indicated by these coefficients. Significance of coefficients have been reported to be directly proportional to t-value and inversely to p-value (Kefarov and Akhnazarova, 1982).The larger magnitude of t-test value

Table 2. T- and p-value of the polynomial models for iron adsorption of biomass.

Predictor	Constant	SE coefficient	T	P
Constant	-418.93	69.08	-6.06	0.000
X_1	1.4591	69.08	2.52	0.027
X_2	30.141	0.5797	3.93	0.002
X_3	112.88	7.663	6.52	0.000
X_4	0.5410	17.32	1.70	0.114
X_1X_2	0.02646	0.03658	0.72	0.483
X_1X_3	0.02849	0.07317	0.39	0.704
X_1X_4	-0.001018	0.001463	-0.70	0.500
X_2X_3	0.3901	0.9146	0.43	0.677
X_2X_4	-0.00729	0.01829	-0.40	0.697
X_3X_4	0.02815	0.03658	0.77	0.456

Table 3. ANOVA of polynomial model for iron removal.

Source	DF	SS	Ms	F	P
Regression	14	8250.62	589.33	11.01	0.002
Residual error	12	642.38	53.53		
Total	26	8893.00			

R-Sq = 92.8%, R-Sq(adj) = 84.3%, SS, sum of squares; DF, degree of freedom; MS, mean square.

and smaller p-value indicates the high significance of the corresponding coefficient (Karthikeyan et al., 1996). The variable with low probability levels contribute to the model, whereas the other can be neglected and eliminated from the model. The p-value for the linear and polynomial terms is shown in Table 2. Generally, according to the p-values of Iron adsorption from leachate, it is observed that the coefficient for linear effect of contact time (x_2) and pH (x_3) are the most significant factor since they have low p-values ($p<0.05$). These significant factors are so important where they indicate limiting factors in the sense that even small variations in their values can alter the activity or removal of heavy metals to a considerable extent (Karthikeyan et al., 1996).

The analysis of variance (ANOVA) from the analysis as shown in Table 3 was employed for the determination of significant parameters and to estimate the removal efficiency of heavy metals as a function of biomass dosage (x_1), contact time (x_2), pH (x_3) and agitation speed(x_4). The results reflect the level of confidence (%) with coefficient of determination (R^2) of iron which is92.8%.R^2 resembles the percentage of the total variation explained by the model which suggests a satisfactory adjustment of the quadratic model to the experimental data. As observed, the value of R^2 obtained was quite efficient since it was larger than 0.9. Nevertheless, regardless of this fact, the amount of removal efficiency may still be enhanced if none of the experiment shows unusual observations.

The p-values obtained for adjusted determination coefficient are quite low, indicating its insignificance of the model ($p > 0.05$) (Kefarov and Akhnazarova, 1982; Khuri and Cornell, 1987). The F value is a measure of variation of the data about the mean. The high F-value and very low probability ($p<0.05$) indicates that the present model is in good prediction of the experimental result (Dey et al., 2001). The p-value and F-value are shown in Table 3.

Surface response methodology describes the interaction of the variables in terms of 3-D model to determine the point of optimized condition to maximize the removal of iron metals from leachate. The 3-D surface and contour plots of the interacted parameters such as biomass dosage, contact time, pH and agitation speed, are presented in Figures 3 to 8 where removal efficiency (%) of the iron metal was investigated as a response. The interaction of pH-time, time-biomass, pH-biomass, agitation-biomass, agitation-time and agitation-pH is shown in the Figures 3, 4, 5, 6, 7 and 8, respectively.

After studying the 3-D surface and contour plots of the interacting process parameters, it was found that more than 40% of removal efficiency of iron metal could be achieved under optimal condition.

Conclusions

In this study, dead fungal biomass (*P. chrysosporium*) was successfully used as an adsorbent for biosorption of metal ions from synthetic wastewater and iron from leachate. The mechanism of the heavy metals species

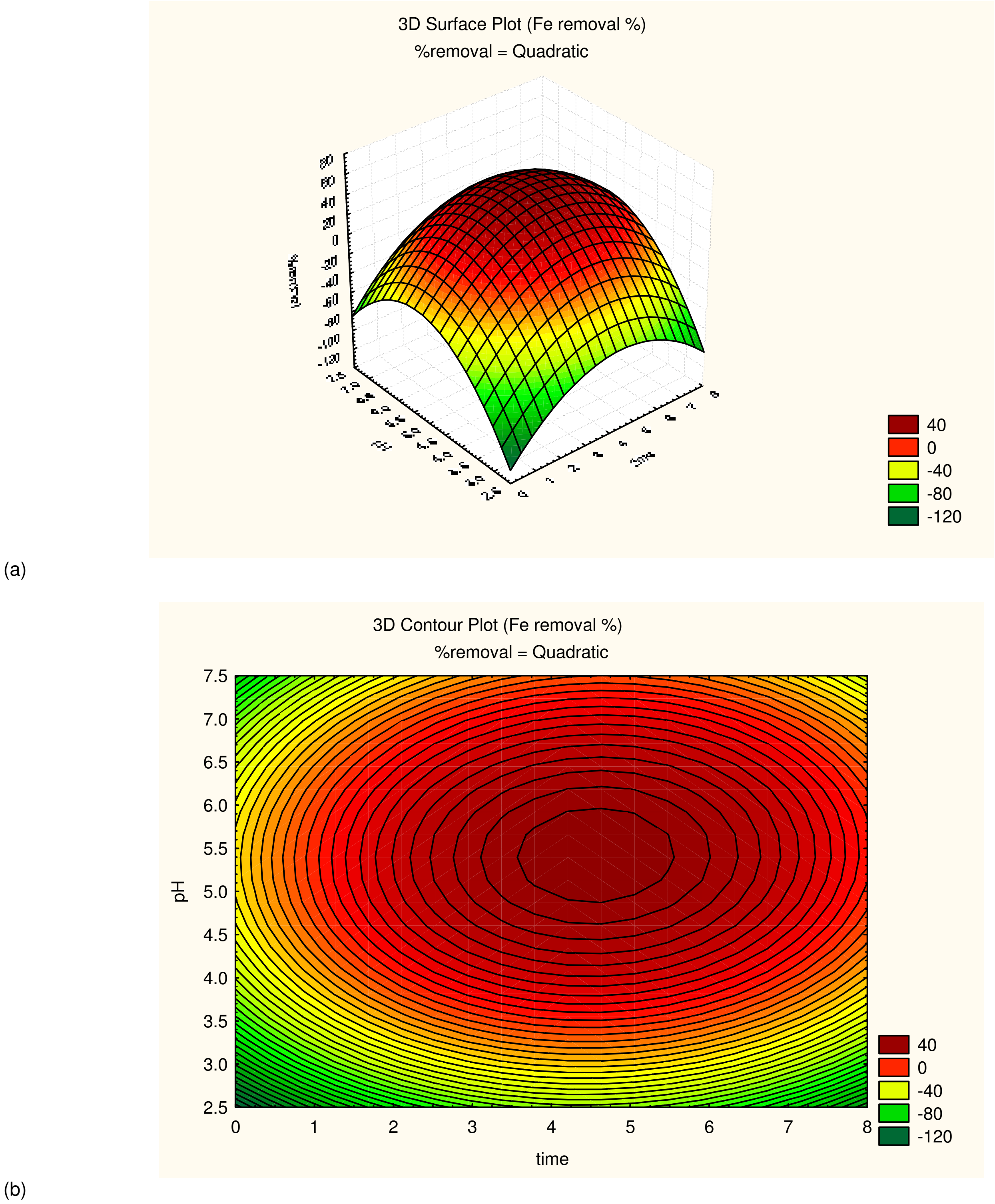


Figure 3. Interaction of contact time and pH during iron removal from leachate: (a) 3-D Surface response; (b) 3-D Contour.

biosorption on the dead fungal biomass depends on the experimental conditions, particularly the biomass dosage, contact time and pH of the solutions. Thus, it can be concluded that all the objectives of this study were met, except for a few and minor errors that occurred, such as fluctuation in analysis data and unusual absence of cadmium, copper and zinc metals in the leachate. Fortunately, this study can be continued but the scope has been narrowed only to iron metals adsorption in leachate. The biomass dosage, contact time, pH and

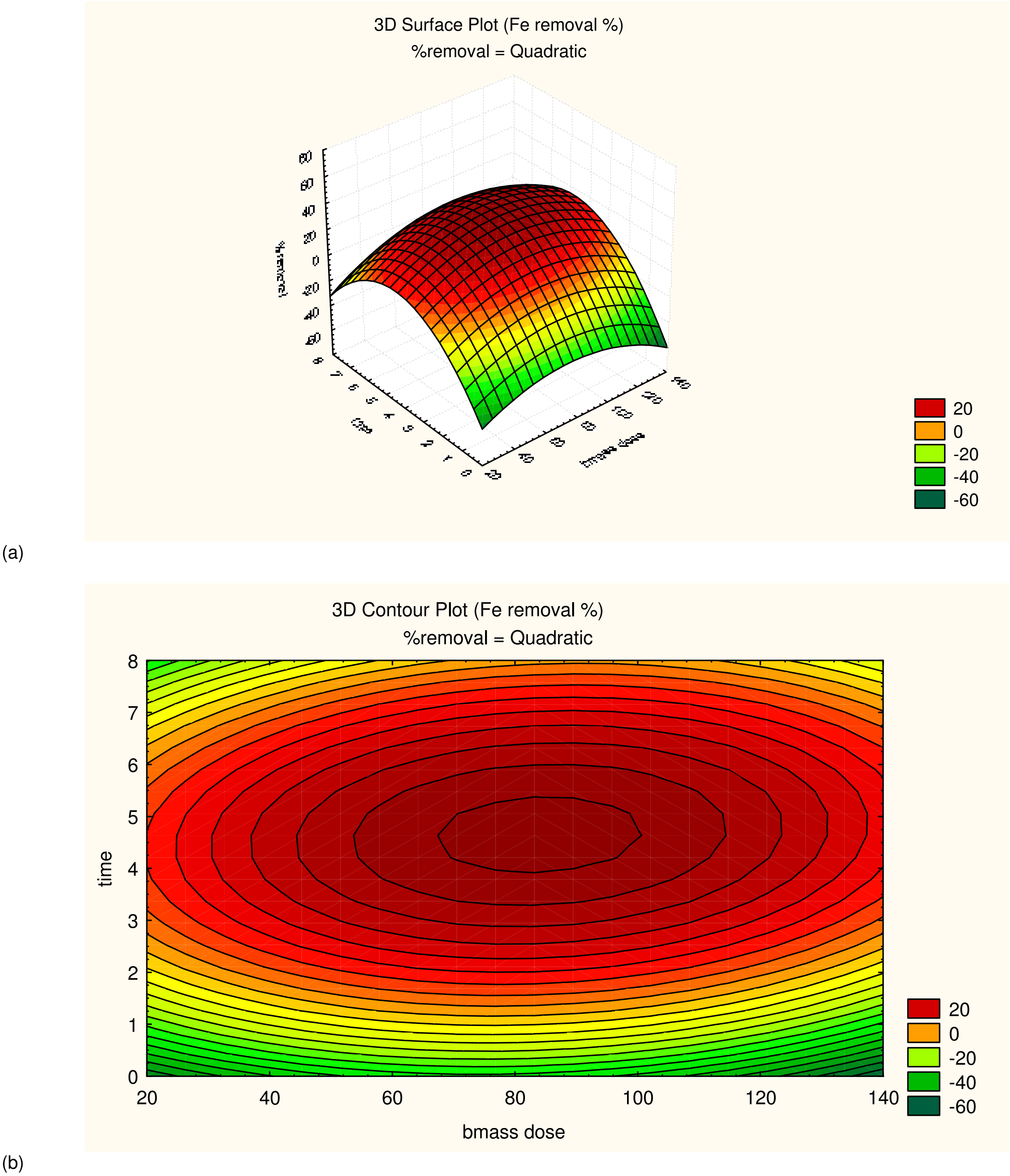
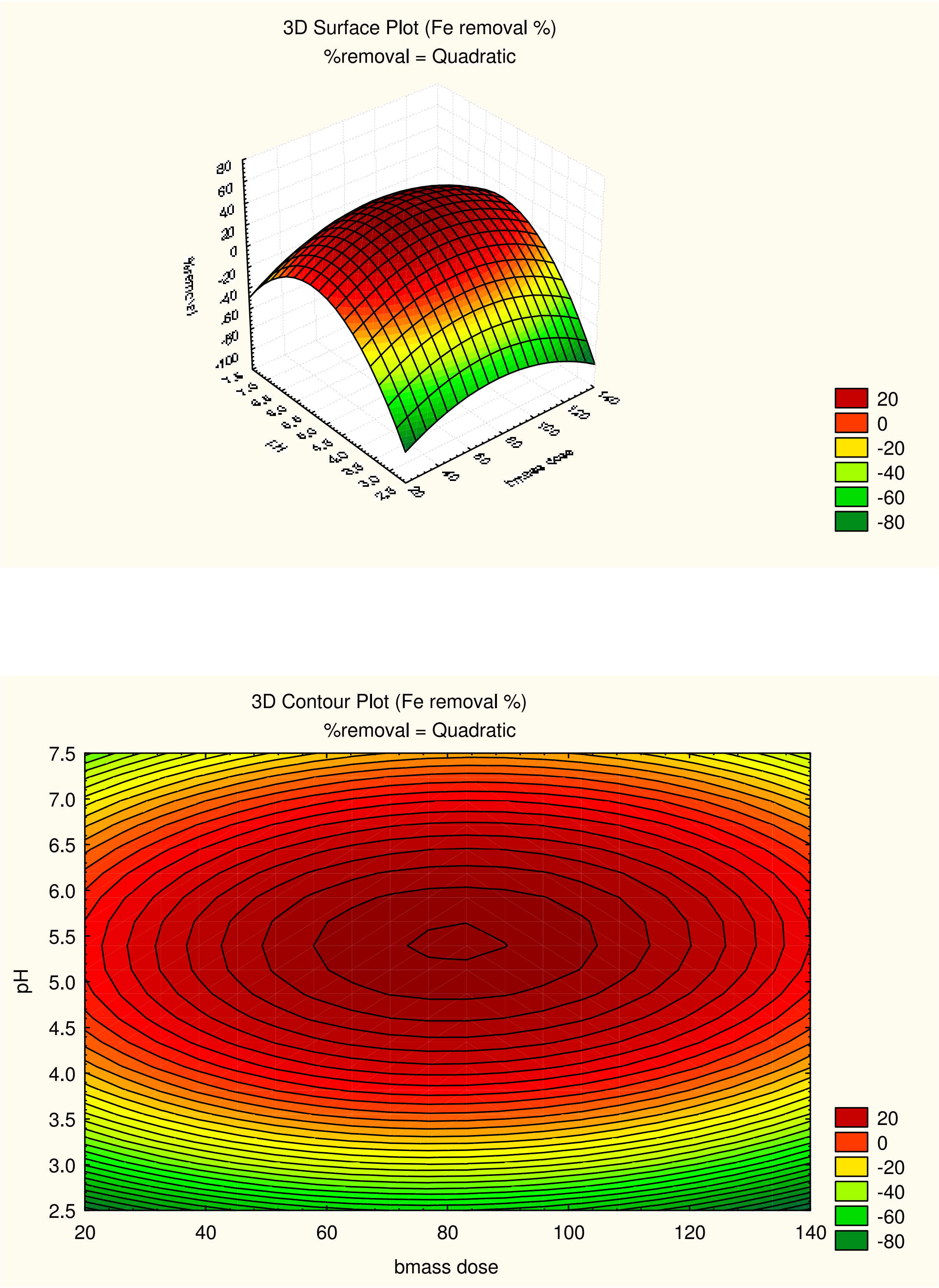


Figure 4. Interaction of contact time and biomass (bmass) dose during iron removal from leachate: (a) 3-D Surface response; (b) 3-D Contour.

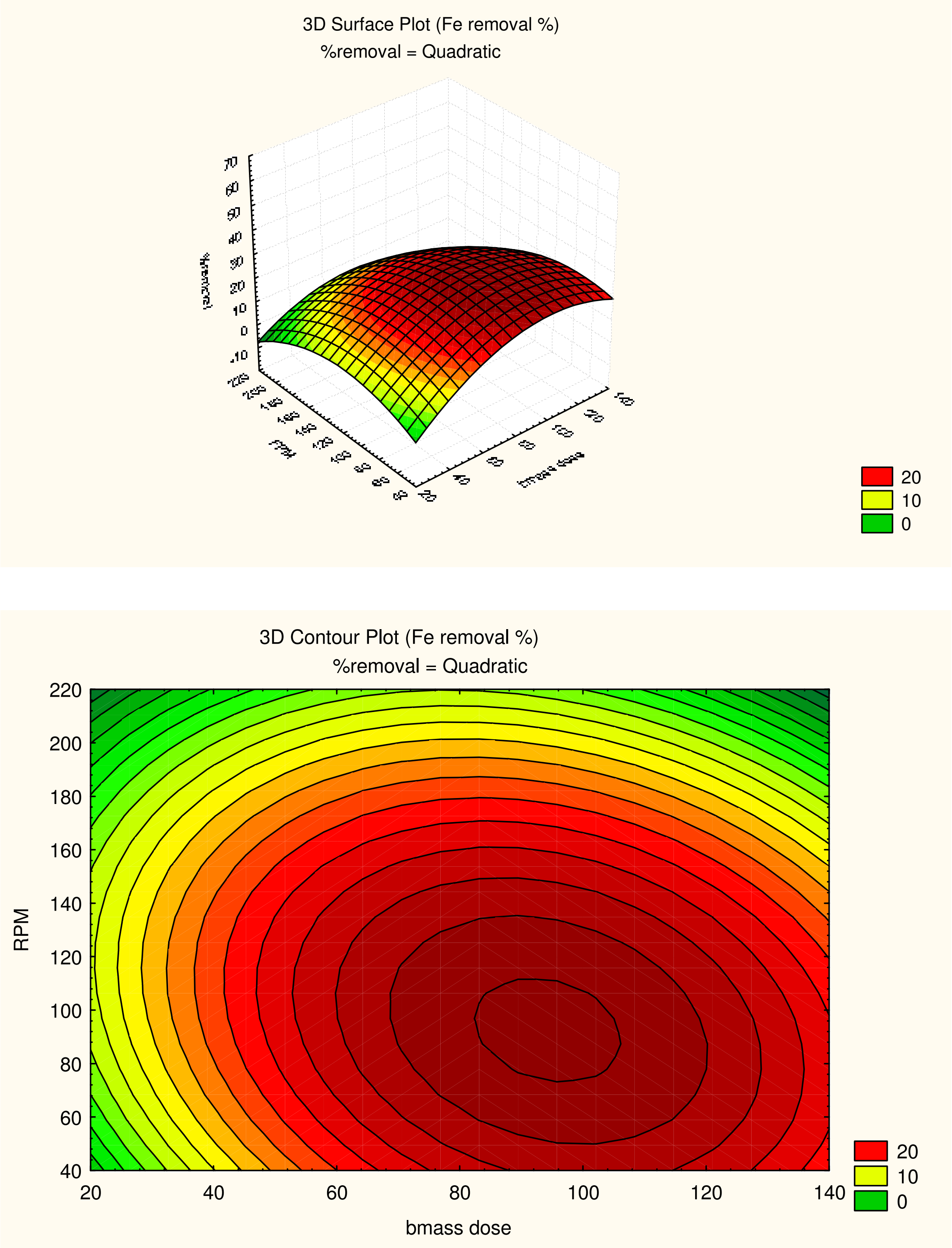
agitation speed was 750 mg/L, 4 h, pH 5 and 150 rpm, respectively with 45.56% removal of iron ions in every 1 L of leachate after 1/50 dilutions as the optimal condition for removal of iron. The highest removal efficiency achieved was 45.5%. In general, this result proves that dead *P. chrysosporium* has a good potential to be used



(a)

(b)

Figure 5. Interaction of pH and biomass (bmass) dose during iron removal from leachate: (a) 3-D Surface response; (b) 3-D Contour.



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(b)

Figure 6. Interaction of agitation (RPM) and biomass (bmass) dose during iron removal from leachate: (a) 3-D Surface response; (b) 3-D Contour.

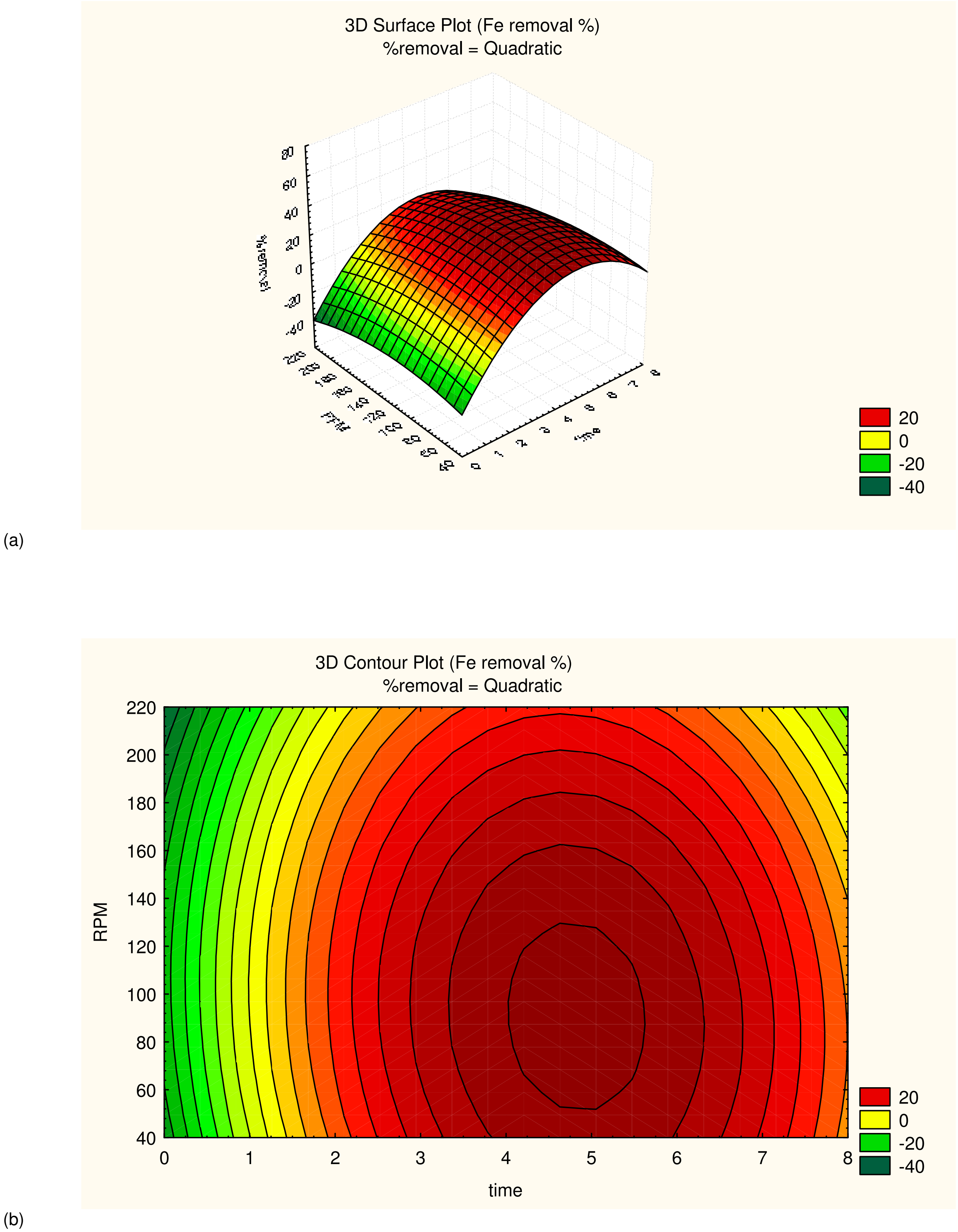
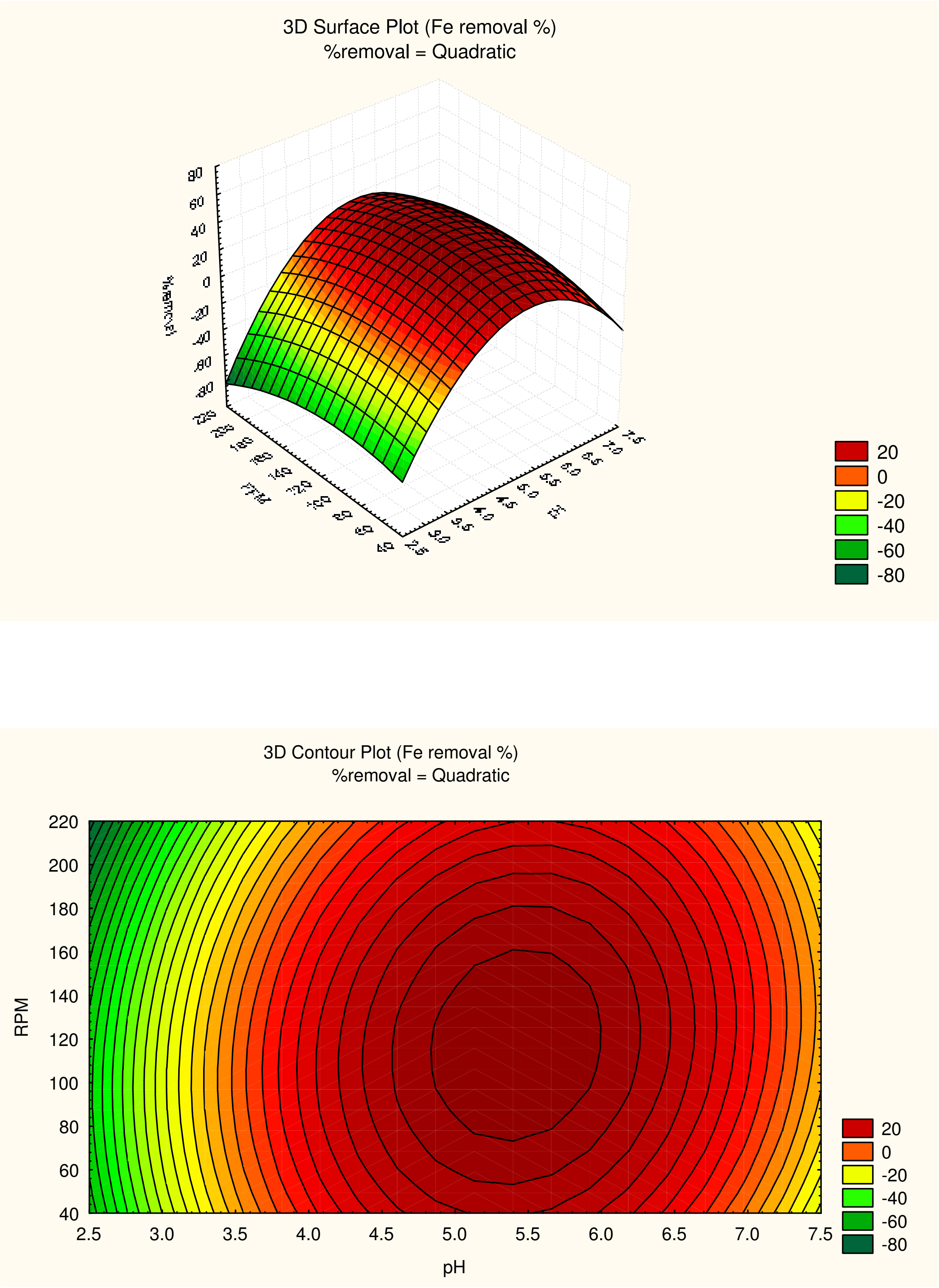


Figure 7. Interaction of agitation (RPM) and time during iron removal from leachate: (a) 3-D Surface response; (b) 3-D Contour.



(a)

(b)

Figure 8. Interaction of agitation (RPM) and pH during iron removal from leachate: (a) 3-D Surface response; (b) 3-D Contour.

as substrate for removal of heavy metals.

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