Application of Taguchi L32 orthogonal array design to optimize copper biosorption by using Spagnum moss

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ABSTRACT

In this work, Taguchi L32 experimental design was applied to optimize biosorption of Cu²⁺ ions by an easily available biosorbent, Spagnum moss. With this aim, batch biosorption tests were performed to achieve targeted experimental design with five factors (concentration, pH, biosorbent dosage, temperature and agitation time) at two different levels. Optimal experimental conditions were determined by calculated signal-to-noise ratios. “Higher is better” approach was followed to calculate signal-to-noise ratios, as it was aimed to obtain high metal removal efficiencies. The impact ratios of factors were determined by the model. Within the study, Cu²⁺ biosorption efficiencies were also predicted by using Taguchi method. Results of the model showed that experimental and predicted values were close to each other demonstrating the success of Taguchi approach. Furthermore, thermodynamic, isotherm and kinetic studies were performed to explain the biosorption mechanism. Calculated thermodynamic parameters were in good accordance with the results of Taguchi model.

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1. Introduction

In last several decades, biosorption has gained significant attention as an efficient alternative method for removal of heavy metals from wastewater effluents. Low-cost biomasses of vegetal or microbial origin such as bacteria, fungi, yeast, algae (Wang and Chen, 2006; Duan et al., 2003) provide remarkable economical benefits in biosorption processes. There are numerous types of biomaterials readily available in the nature so researches have studied to find out the most efficient and appropriate biomass type. Brown algae (Davis et al., 2003), white-rot fungi (Yetiş et al., 1998), sugar peel pulp pectin gels (Mota et al., 2009), green algae waste biomass (Bugarina and Bugarina, 2012), peanut shell (White-Krowiat et al., 2011), marine bacterium (Yer et al., 2005), citrus peels (Schiewer and Parib, 2009), thermoophilic bacteria (Özdemir et al., 2009) are some of the numerous biosorbent types used to remove heavy metals from aqueous solutions.

Selection of appropriate biomaterial plays an important role in efficiency of biosorption processes. Besides this, determination of optimum process conditions (pH of solution, biosorbent dosage, agitation period, temperature etc.) is also mandatory to achieve maximum biosorption capacity. In conventional experimental studies, generally one parameter is varied keeping other parameters constant to determine the optimum conditions (Al-Qadah, 2006; Ibrahim, 2011; Njoku, 2014). This conventional approach requires numerous time-consuming tests which increase the research costs remarkably. The conventional approach also precludes investigating interactive effects between the relevant process parameters.

Optimization methods can be used in biosorption studies in order to determine the specific design parameters which could yield the highest biosorption capacity (White-Krowiat et al., 2014). Multi-variate statistics techniques such as Box–Behnken (Bash et al., 2006; Kousha et al., 2012), central composite surface design (Hocan et al., 2009; Ranjan et al., 2009), Dohlerit design (Zeligharni et al., 2008) and full factorial design (Yama et al., 2007) have been used in biosorption studies to reduce the number of experiments and describe the impacts of independent process variables.

Taguchi statistical design method, developed by Genichi Taguchi can also be applied to biosorption data with the aim of optimizing process variables easily, cheaper and faster (Chary and Dashtdar, 2012). By using this method, impacts of several factors can be determined simultaneously (Idris et al., 2002). When compared with the other optimization methods, Taguchi may provide some superiority; for e.g. it gives a better graphic visualization to determine the optimum condition compared to response surface methodology. Furthermore, Taguchi method demands less number of experiments to find the optimum condition than RSM (Sažd et al., 2013).

In this work, Taguchi design method was used to optimize copper biosorption by using Spagnum moss. With this aim, firstly
L2⁰ orthogonal array was created and impacts of process variables (pH, biosorbent dosage, metal concentration, agitation time and temperature) on biosorption capacity were analyzed. Furthermore, copper uptake capacity of *Sphagnum* moss was predicted by applying Taguchi method. Within the paper, experimental data were also applied to the selected isotherm, kinetic and thermodynamic equations to investigate biosorption mechanisms.

2. Materials and methods

2.1. Experimental procedure

2.1.1. Instrumentation

In the study, copper analyses were carried out using a HACH DR 2000 model spectrophotometer according to Bicinchoninate Method (Method no. 8506). NIVE model shaker was used for batch biosorption experiments. pH adjustments were performed by using TESTO model pH-meter.

2.1.2. Chemicals

Copper (II) nitrate trihydrate (CuN₂O₆.3H₂O) was used in biosorption experiments. pH adjustments were performed using 0.1 N hydrochloric acid (HCl) and 0.1 N sodium hydroxide (NaOH). All reagents were used in analytical grade quality and purchased commercially.

2.1.3. The used biosorbent

*Sphagnum* moss was chosen as a biosorbent to remove copper ions from aqueous media as different types of moss have already been found to be capable of removing heavy metals from aqueous solution (Zhang and Banks, 2006; Ho and McKay, 2009). It is also abundant and locally available in the northern region of Korea.

The collected moss was separated from other flora, and washed thoroughly in order to remove impurities. Then the biomass was dried at 70°C for 48 h and finally it was sieved through 1 mm pore size. FTIR technique was used to characterize the presence of specific chemical groups in the biosorbent. With this aim, Perkin Elmer Spectrum One FTIR spectrometer was used.

2.1.4. General procedure

In this study, batch biosorption technique has been used. Samples with different metal concentrations have been prepared by diluting 1000 mg/L of Cu²⁺ stock solution, 0.1 N HCl and 0.1 N NaOH solutions were used for pH adjustments. The volume of the samples was decided as 50 mL. Biosorbent dosages have been varied in the range of 0.3-0.6 g.

Samples with different biosorbent contents were agitated on the shaker at predetermined experimental conditions and the biosorption was separated by filtering at the end of agitation period. The remaining copper concentration was analyzed by spectrophotometer.

The biosorbed heavy metal amount ($q_e$) per unit biomass was calculated as according to the following equation:

$$ q_e = \frac{(C_0 - C_f)W}{m} $$

where $C_0$ is the initial copper concentration, $C_f$ is the concentration of heavy metal at equilibrium (mg/L), $m$ is the biosorbent amount (g) and $V$ is the solution volume (L).

2.2. Design of experiments process

The Taguchi method is an effective design of experiments (DOE) approach. This tool can significantly minimize the costs and time of experiments (Zolghadri et al., 2011; Al et al., 2013). Selection of control factors is an important stage of Taguchi applications and design of the factors is generally set by the experimental experience (Kim et al., 2005). As known, pH, biosorbent dosage, metal concentration, agitation time and temperature are among the most important factors in adsorption processes (Shah and Dogan, 1998; Zhaghamzadeh et al., 2013). In this study, all factors were chosen as input variables and their impacts on response variable ($q_e$) were investigated. Two levels were selected for each of the studied factors (initial copper concentration: 50 and 90 mg/L, pH: 5 and 9, biosorbent dosage: 0.3 and 0.6 g, temperature: 22 and 33°C, agitation time: 25 and 90 min).

In Taguchi statistical method, different special designs named as "orthogonal arrays" are used to study entire parameter space (Taguchi, 1990). In the study, L2⁰ orthogonal array was chosen to determine the optimum conditions. Coded and uncoded data were used in Taguchi’s L2⁰ experimental design (Table 1).

Deviation between desired and experimental value is evaluated by a loss function. Signal-to-noise (SN) ratio is required to measure the quality characteristics deviating from the desired values (Yang and Tung, 1998). There are three different types of SN ratios (lower is better – LB, nominal is better – NB and larger or higher is better – HB) in Taguchi applications (Zolghehri et al., 2011). In this study, higher is better (HB) approach was chosen at this process was designed to determine the higher metal removal.

The signal-to-noise ratio for HB characteristics was calculated by using the following equation:

$$ \frac{S}{N} = 10 \log_{10} \left( \frac{1}{n} \sum_{i=1}^{n} y_i^{-2} \right) $$

where $n$ is the number of repetitions under the same experimental conditions, and $y_i$ represents the measured results for biosorption efficiency.

Optimization process and required calculations were performed by using Minitab 16 software package and Microsoft Excel 2007.

3. Results and discussions

3.1. Fourier transform infrared spectral analysis of the biosorbent

The FTIR spectral analysis is an important tool used to determine the characteristic functional groups, which are responsible for biosorption process (Nadeem et al., 2008). In the study, FTIR analyses have been performed before and after copper biosorption in order to compare the functional groups of the biomass.

Peaks were obtained at 3429.6, 2920.5, 1633.4, 1426.7, 1373.6, 1248.2 and 1032.7 cm⁻¹ in FTIR spectrum of the native *Sphagnum* moss. The strong band at 3429.6 cm⁻¹ may be due to N–H and O–H stretching whereas the peak at 2920.5 cm⁻¹ may be due to C–H stretching vibration. The peak at 1633.4 cm⁻¹ may attribute to functional groups such as C=O, C=C, or C=O, and C=N. The band at 1032.7 cm⁻¹ is assigned to stretching of R–C=H groups whereas the band at 1373.6 is assigned to stretching of R=C=H, C–O and C–N groups (Pan et al., 2007).

FTIR spectrum of the copper loaded *Sphagnum* moss showed peaks at 3425.7, 2917.1, 1629.6, 1425.3, 1372.9, 1245.1 and 1033.1 cm⁻¹. Comparing the results of FTIR analyses performed before and after biosorption, it can be concluded that functional
groups such as N-H, O-H, C=H, C≡C, C=O and C≡N are the main biosorption sites for copper uptake on Sphagnum moss.

### 3.2. Results of Taguchi method

The main goal of the study was to determine the optimum experimental conditions which provide the highest metal biosorption. With this aim, Taguchi method was designed by 96 experiments in 32 plots with three replications. In Taguchi method, relationships between the levels should be clearly defined to determine the optimum conditions. Experimental conditions providing higher signal-to-noise ratio were decided to be optimal. In this scope, signal-to-noise response table was prepared for L12 experimental design (Table 2).

In Table 2, bold written numeric values denote the optimum levels of the studied factors. In the response table, \( \Delta \) values were used to determine the impacts of the factors on removal efficiency. These values are calculated from the difference between the signal-to-noise ratios of the levels. Considering the obtained \( \Delta \) values, biosorbent dosage was found to be the most effective factor whereas contact time and concentration were relatively less effective. Impact ratios of the factors were calculated as 7.25%, 29.77%, 38.17%, 20.99% and 3.82% for concentration, pH, biosorbent dosage, temperature and agitation time, respectively.

Obtained results should be in good accordance with the results in response table. S/N (HB type) ratios were calculated according to Eq. (2). Experimental conditions providing the highest S/N ratio are as 2→1→2→1→2. All responses within the system are in good accordance with the responses. Minitab graph of S/N ratios representing optimum conditions is also given in the paper (Fig. 1).

All of these experimental design analysis showed that it is required to study under optimum pH conditions to provide high removal efficiencies. Furthermore, increasing the biosorbent dosage provides significant increase in the removal efficiencies. As lower temperature level was found to be optimum, the process is thought to be in exothermic nature. Second levels of contact time and concentration were recommended in order to obtain higher biosorption efficiency.

In the study, Taguchi method is also used as a predictive model. Copper biosorption efficiencies were predicted by using experimental data (Fig. 2).

As seen from Fig. 2, remarkable correlation \( R^2 = 0.93 \) was obtained between predicted and actual efficiencies. This result demonstrates the prediction success of Taguchi method. Higher correlation \( R^2 = 1 \) can also be obtained by increasing repetition number.

### 3.3. Thermodynamic studies

In biosorption studies, thermodynamic studies should be performed to evaluate the feasibility of the biosorption process (Ghao et al., 2010; Gupta et al., 2012). With this aim, free energy change \( (\Delta G) \), isosteric enthalpy change \( (\Delta H) \), and entropy change \( (\Delta S) \) were calculated in the study. Following equations were used:
for thermodynamic analyses:

\[ K_c = \frac{C_{\text{eq}}}{C_c} \]  
\[ \Delta G = -RT \ln K_c \]  
\[ \Delta G = \Delta H - T \Delta S \]  
\[ \ln K_c = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \]

In these equations, \( K_c \) represents the equilibrium constant, \( C_{\text{eq}} \) shows the amount of heavy metal adsorbed on Sphagnum moss at equilibrium (mg/g) and \( C_c \) is equilibrium concentration of Cu\(^{2+}\) (mg/L). \( R \) is the universal gas constant (J/mol K) and \( T \) is absolute temperature (K).

Thermodynamic values have been calculated from van’t Hoff plot (Fig. 3).

\( \Delta G \) and \( \Delta S \) were determined from the slope and intercept of the plot between \( \ln K_c \) versus \( 1/T \) respectively.

Calculated \( \Delta G \), \( \Delta H \) and \( \Delta S \) values are summarized at Table 3. As seen from the table, negative \( \Delta G \) values were determined for all of the studied temperature conditions. The negative values of free energy change show spontaneous characteristics of the biosorption process (Ghaedi et al., 2010). The calculated negative value of \( \Delta H \) is indicative of an exothermic process. Furthermore, physical biosorption process is thought to be dominant as calculated \( \Delta H \) exists in the range of 10–40 kJ/mol (Özbay et al., 2013). And finally, negative \( \Delta S \) value indicates regularity of the solute molecules during the biosorption process.

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>( \Delta G ) (kJ/mol)</th>
<th>( \Delta H ) (kJ/mol)</th>
<th>( \Delta S ) (kJ/mol K)</th>
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<tr>
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</tr>
<tr>
<td>333</td>
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<td>-31.29</td>
<td>-72.57</td>
</tr>
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</table>

* Measured between 293 and 333 K.

3.4. Isotherm studies

Langmuir and Freundlich isotherm models were applied by using experimental data in order to examine the reaction of copper ions with Sphagnum moss.
3.4.1. Langmuir isotherm

The Langmuir isotherm model assumes that biosorption process occurs at specific homogeneous surfaces. According to this model, the interaction between the biosorbed molecules is negligible (Alyan and Veli, 2008).

The following linear equation can be used to express Langmuir isotherm model:

\[ \frac{C_e}{q_e} = \frac{1}{kV_m + C_e} \]

\[ q_e = \frac{C_e}{V_m} \]

(7)

In the equation, \( q_e \) is the biosorbed metal amount per unit biomass, \( V_m \) is the monolayer biosorption capacity, \( k \) is the equilibrium constant and \( C_e \) is the equilibrium concentration of the solution (mg/L).

For this study, Langmuir isotherm equation was obtained as

\[ \frac{C_e}{q_e} = 0.4386 + 0.833C_e \]

(8)

Langmuir isotherm curve is found to be highly linear (\( R^2 = 0.99 \)) for biosorption of copper by Sphagnum moss. Related isotherm parameters (\( V_m \) and \( k \)) were determined from the intercept and slope of the Langmuir plot (Table 4).

3.4.2. Freundlich isotherm

Freundlich isotherm assumes a heterogeneous surface with a non-uniform distribution of biosorption heat. This isotherm can be expressed as

\[ q_e = K_f C_e^{1/n} \]

where \( K_f \) is the Freundlich constant (mg/g) and \( 1/n \) is the adsorption intensity.

For this study, Freundlich equations was obtained as

\[ q_e = 2.72C_e^{0.38} \]

(9)

\[ (10) \]

Table 4

<table>
<thead>
<tr>
<th>Langmuir</th>
<th>Freundlich</th>
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<td>( V_m )</td>
<td>( k )</td>
</tr>
<tr>
<td>12.00</td>
<td>0.19</td>
</tr>
</tbody>
</table>

\[ \text{In} q_e = -0.3t \text{versus time for biosorption of } Cu^{2+} \text{ ions by Sphagnum moss (pH 5, biosorbent dosage 0.5 g, agitaiton speed 200 rpm, temperature 22°C).} \]

\[ \text{In} q_e = -0.3t \text{versus time for biosorption of } Cu^{2+} \text{ ions by Sphagnum moss (pH 5, biosorbent dosage 0.5 g, agitaiton speed 200 rpm, temperature 22°C).} \]

The \( R^2 \) value was determined as 0.77 for the Freundlich isotherm. Calculated \( K_f \) and \( 1/n \) values are given in Table 4.

1/n values change in the range from 0 to 1 in favorable biosorption studies (Alyan and Veli, 2008). The calculated 1/n value (0.38) indicates the suitability of the performed biosorption process.

Considering the calculated \( R^2 \) values, it is concluded that the biosorption process is in good agreement with the Langmuir isotherm rather than the Freundlich isotherm. This result demonstrates the monolayer coverage of copper ions on the surface of the biomass.

3.5. Kinetic studies

Kinetic models were also applied to experimental data. By this way, chemical reaction mechanism, diffusion control capacity and mass transfer can be understood better (Özcan et al., 2013). In this study, the initial \( Cu^{2+} \) concentrations have been determined as 50, 80, and 100 mg/L for kinetic modeling.

3.5.1. Pseudo-first-order model

Firstly pseudo-first-order model was used to explain the biosorption capacity of Sphagnum moss for copper ions. Following equation was used with this aim (Iagergren, 1898):

\[ \ln(q_e - q_t) = \ln(q_e) - k_1 t \]

(11)

where \( k_1 \) shows rate constant of the first order reaction kinetics, \( q_e \) and \( q_t \) are the amount of heavy metal adsorbed at time \( t \) and saturation (mg/g), respectively. \( k_1 \) can be determined from the plot of \( \ln(q_e - q_t) \) versus time.

\( q_{\text{calculated}} \) values were determined from the intersection points of the curves (Fig. 4).

3.5.2. Pseudo-second-order model

The pseudo-second-order kinetic model can be explained by the following formula (Ho and McKay, 1998):

\[ \frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \]

(12)

In the equation \( k_2 \) explains the pseudo-second-order rate constant, \( k_2 \) and \( q_e \) can be determined from the plots of \( t/q_t \) versus time (Fig. 5).

All calculated kinetic parameters have been summarized in Table 5. The biosorption process has followed the pseudo-second-order kinetic model considering closer \( q_{\text{experimental}} \) and \( q_{\text{calculated}} \) values and higher \( R^2 \).

Results obtained from the performed isotherm and kinetic studies showed the appropriateness of Sphagnum moss for
biosorption of copper ions from aqueous solutions. In Table 6, metal uptake capacities of different biosorbents have been compared.

4. Conclusion

The major aim of this study was to determine optimum experimental conditions for biosorption of copper by Sphagnum moss. With this aim, we have applied Taguchi L$_{25}$(L) orthogonal array by using input variables (pH, biosorbent dosage, metal concentration, temperature and agitation time) and response variable (biosorption efficiency). After determining the design of experiments, S/N ratios were calculated by the model in order to obtain the impact ratios of the input parameters. Biosorbent dosage was found to be the most effective factor with 38.17% impact ratio. Another aim of the work was to investigate usability of Taguchi method for prediction of copper biosorption. Obtained high correlation between the predicted and actual biosorption efficiencies ($R^2=0.933$) indicated the appropriateness of the developed model. Beside Taguchi applications, conventional biosorption studies (isotherm, kinetic and thermodynamic) were also performed. Results of the isotherm studies showed that the biosorption process is in good agreement with the Langmuir isotherm ($R^2=0.991$) rather than the Freundlich isotherm ($R^2=0.77$). Pseudo-second-order reaction kinetics has provided a realistic description for biosorption of copper considering closer experimental and calculated values of $q_e$. Correlation coefficients were also higher in pseudo-second-order kinetics. Finally, results of the thermodynamic studies demonstrated that the biosorption process has shown spontaneous, exothermic and regular characteristics.

Acknowledgment

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.fexenv2014.06.018.

References


Table 5

<table>
<thead>
<tr>
<th>Initial heavy metal concentration (mg/L)</th>
<th>$q_{	ext{experimental}}$ (mg/g)</th>
<th>$q_{	ext{pseudo-first-order}}$ (mg/g)</th>
<th>$K_1$ (1/min)</th>
<th>$q_{	ext{pseudo-second-order}}$ (mg/g)</th>
<th>$K_2$ (mg/g min$^{-1}$)</th>
<th>$q_{	ext{experimental}}$ (mg/g)</th>
<th>$q_{	ext{pseudo-second-order}}$ (mg/g)</th>
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Table 6

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<tr>
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<td>Cd</td>
<td>6.97</td>
<td>Ibrahim et al., 2006</td>
</tr>
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<td>Acrea catechu</td>
<td>Cd</td>
<td>10.66</td>
<td>Chakraverty et al., 2010</td>
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<td>Calophyllum inophyllum</td>
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<td>4.86</td>
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<td>12.98</td>
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<td>Sargassum sp.</td>
<td>Cd</td>
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<td>M. hemsleyi</td>
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<td>Pseudomonas putida</td>
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