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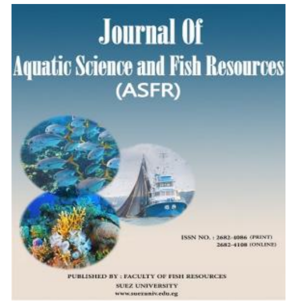

Immobilization of Cd (II) using *Pistia stratiotes* L.(Araceae) biomaterial: optimization study using statistical design

Alaa M. Younis[1], Mohamed A. Aly-Eldeen[2] and El-Sayed Nafea[1]

1- Aquatic Environment Department, Faculty of Fish Resources, Suez University, Egypt.

2 - National Institute of Oceanography and Fisheries, Egypt.

ABSTRACT

In the current study the free floating macrophyte *Pistia stratiotes* L. belonging to family (Araceae) powder was used as bioadsorbent material for immobilization of Cadmium from contaminated water samples. Design experiment was employed to reduce the number of trials and examine the effect of different controlling parameters at the same time on cadmium adsorption process. From the results we observed that the size of powdered materials were ranged from 116.6 -187.5 nanometers. The maximum removal percent (92 %) was recorded at pH 8, 190 min. of contact time, 28.75 mg/l of Cadmium concentration and 3.875 g of adsorbent material.

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1. Introduction

Aquatic pollution caused by heavy metal represents an important environmental problem worldwide due to its toxic effects (Khalil et al., 2013; Nafea and Zyada, 2015; Nafea, 2016; Younis, 2018; Nafea, 2019a; Nafea, 2019b; Soliman et al., 2018; Soliman et al., 2019). Heavy metals are widely used in industrial sectors such as mining, fertilizers, medicine and agriculture, because of their unique properties. Release of these metals into aquatic environment resulted in a great risk on aquatic environment and subsequently, on the human health (Younis and Nafea 2012; Khalil et al., 2013). Recent developments in environmental quality standards highlight the need for development and improvement the wastewater treatment especially in the industrial sectors that discharge toxic heavy metal in their effluents (Abd El-Ghaffar et al., 2009). Heavy metal ions of more concern are cadmium, arsenic, lead, mercury and chromium as they pose a serious threat to human health and living organisms (Kumar and Chawla, 2014).

* Corresponding authors at: Elsayed M.ali Nafea.
E-mail addresses: Elsayed.nafea@suezuniv.edu.eg
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Aquatic pollution by cadmium is of considerable concern, as this metal has found widespread uses in batteries, galvanized pipes, pigments, metal plating, alloys, phosphate fertilizers, paints, combustion of fossil fuels and discharge from metal refineries (Lambert et al. 2007; John and Santhi 1994). Because of its toxicity characteristics, the maximum permissible concentration of cadmium for drinking water is 0.003 mg/L as per the guidelines of World Health Organization (WHO 2008; Kumar and Chawla, 2014)

Removal of toxic metals from wastewater has been a crucial issue related to public health. Many studies have been focused on the removal of toxic pollutants from wastewater such as precipitation, reverse osmosis, exchange, coagulation-sedimentation and adsorption onto different materials (El-Said et al., 2018; Hua et al., 2012; Younis et al., 2014; Shreadah et al., 2019; Younis et al., 2019; Younis et al., 2020). Surface adsorption has been considered as an important basis, sustainable and more efficient in the removal of toxic metals from aqueous solution (Redha, 2020), particularly for cadmium because it is easy to operate, economical and highly efficient in wastewater treatment (Kumar and Chawla, 2014)

Biosorption is the first stage in metal accumulation. It involves adsorption of metal onto the surface of adsorbent material (Tobin et al., 1994). Many plants can be employed in this process, including Spirulina maxima (Gong et. al., 2005), Rice Husk (El-said, 2010), salvinia natans (Somnath and Sunil, 2005; Aly-Eldeen and Egorv, 2012), Ulva fasciata (El-Naggar et al., 2018), Chlorella vulgaris (Kumar et. al., 2018), Curtobacterium sp. (Xingjie et. al., 2018), Cladophora green algea (Bahaa et. al., 2019), Cystoseria indica (Khajavian et al., 2019), and Padina gymnospora (Hussein et. al., 2019).

Many plants can thrive in the metal enriched environments and some of them can accumulate very high concentrations of toxic metals in their tissues up to high levels, which are essential for their growth and development. These metals include As, Mg, Fe, Mn, Pb, Zn, Cu, Mo , Ni, Cr and Co . However, excessive accumulation of these heavy metals can be toxic to some plants.

Pistia stratiotes L**.** is a free floating aquatic macrophytes belonging to family Araceae, present and grow in fresh and brackish shallow water with high nutrient contents, where it can absorb and accumulate heavy metals and elements in its tissues by high rate (Rhizofiltration).

Because of water lettuce *(Pistia stratiotes* L. family araceae) has a wide geographic distribution, rapid growth rate, it may adversely affect multiple uses of water resources, for example, obstructing irrigation and drainage channels, hindering navigation and recreational aquatic activities and compromising fishing activity specially in northern delta lakes in Egypt (Nafea.2019b), so that it is considered to be a weed by various authors. Also, from the viewpoint of biomass large-scale production, it is economically interesting to use its biomass as biosorbents materials which in turn help to solve the previous stated problems (Henry-Silva & Camargo, 2006; Pott and Pott 2002).

Pistia stratiotes L. could be used as a Bio-materials for removing of phenolic compounds and heavy metals from wastewater as a sustainable treatment strategy for the polluted wastewater in the laboratory as a trial to be used in a wide scale treatment strategy.

The factorial experimental design methodology involves changing all variables at the same time at each trial. The reason for this is that variables can influence each other, and the ideal value for one of them can depend on the values of the others. This interaction between variables is a frequent phenomenon (Montgomery, 1997). Also in the nature, the real effluent samples are affected by different factors at the same time, so that using of such design is a powerful tool for process optimization (Brasil, *et al*., 2006).

Factorial design was employed in the current study to avoid the traditional one factor effect at a time of experiment. Common statistical tools, such as analysis of variance, *F*-test, the Student's *t*-test, and lack of fit, were used to define the most important variables affecting the metal removal efficiency (Montgomery, 1997).

Thus, the objective of this study was to evaluate the biosorption efficiency of the dried biomass of water lettuce (*Pistia stratiotes L*.) for cadmium removal from contaminated water using statistical programs.

Materials and Methods

Chemicals

A stock solution (250 mg/L) of cadmium chloride was prepared in deionized water. Fresh working solutions (5-100 mg/L) were prepared immediately before use.

Preparation of dried *Pistia stratiotes*:

Pistia stratiotes L.was collected from Egyptian Northern Delta Lake, named El-Manzala lake, washed using stream water, then by sterile distilled water to remove all debris and then dried at room temperature. Before using, the dried *Pistia stratiotes* was grinding down using a planetary ball mill.

Scanning electron microscopy:

The micrograph and particle size of Pistia stratiotes powder were observed by a scanning electron microscope (JSM-5300 – JEOL).

Batch experiments using experimental designing

Here, a commercially available software programs were used (Design Expert, Version 10.0.0.3, Stat-Ease Inc, and Minneapolis, MN and Minitab 17). The experimental design chosen was Response Surface Methodology (RSM), central composite. The investigated variables were coded as; pH (A), Contact time (B) min, Initial dye concentration (C) mg/l, and adsorbent dosage (D) g (Table 1). The optimization process was studied by varying the selected factor at these levels: +2, +1, -1, -2 with 6 central points (0) to give a total of 30 experimental runs (Table 2). Statistical analyses were performed using the same software. Experiments were performed in duplicate and results were averaged.

Table 1: factors and levels used in the biosorption of Cd using *Pistia stratiotes* dried biomass

	Symbol	-2	-1	0	1	2
pH	A	2	4	6	8	10
time (min.)	B	10	70	130	190	250
Conc. (mg/l)	C	5	28.75	52.5	76.25	100
Dose (g)	D	0.5	1.625	2.75	3.875	5

Table 2: experimental factorial design for the biosorption process using *Pistia stratiotes* dried biomass

Run order	A	B	C	D
1	1	1	1	-1
2	0	2	0	0
3	0	0	-2	0
4	0	0	0	0
5	0	-2	0	0
6	-2	0	0	0
7	1	-1	-1	1
8	1	1	1	1
9	-1	-1	-1	-1
10	0	0	0	0
11	0	0	0	0
12	0	0	0	0
13	1	-1	1	1
14	1	-1	-1	-1
15	1	-1	1	-1
16	0	0	2	0
17	0	0	0	2
18	-1	-1	1	-1
19	0	0	0	-2
20	-1	-1	-1	1
21	-1	-1	1	1
22	1	1	-1	-1
23	0	0	0	0
24	-1	1	-1	1
25	2	0	0	0
26	-1	1	-1	-1
27	-1	1	1	1
28	-1	1	1	-1
29	1	1	-1	1
30	0	0	0	0

After equilibrium was reached, the residual supernatants were filtered through 0.45 µm membranes. The initial and equilibrium metal concentrations were measured using of atomic absorption spectrophotometer (AAS, Model AA-6800 Shimadzu)

The removal efficiency of cadmium was calculated according to the following equation

. $$R(\%) = (C_i - C_f)/C_i \times 100 \quad (1)$$

Where R: metal removal efficiency (%), C_i and C_f are initial and equilibrium metal concentration (mg/l), respectively.

25 It and discussion:

The *Pistia stratiotes* L fresh materials was collected from Lake Manzala water with the following characteristics; pH is 7.3, water conductivity 3100 mmhos/cm, dissolved oxygen 7.1 mg/l and biological oxygen demand 13 mg/l with water depth 110 cm.

Characterization of adsorbent material:

Aquatic Macrophytes are considered as one of the most important biosorbents material (Shanab et al., 2012).

The surface morphology of the prepared *Pistia stratiotes* L. powder was revealed through the SEM image shown in Figure (1), it shows that the pours of *Pistia stratiotes* surface were in irregular distribution. From SEM images it is confirmed that the particles having size in between 116.6 -187.5 nanometers and this confirm the nanosize of adsorbent powdered materials.

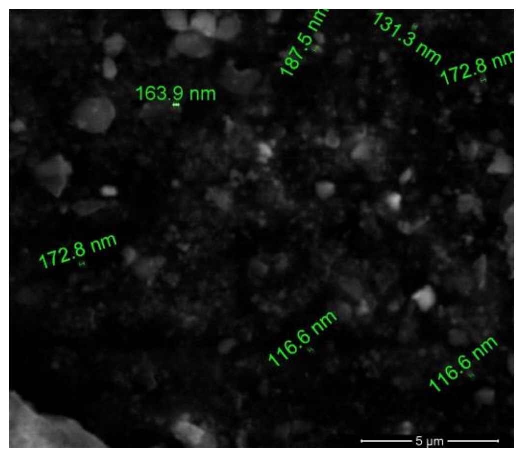


Fig. (1): SEM image of Pistia stratiotes powder

Batch Adsorption Separation using statistical model

Most optimization studies in the treatment experiments involve variation of one factor at a time, while keeping all other factors constant. Additionally, designing the experiments using the factorial designs, enable all factors to vary simultaneously. This helps in determining the main and interactive effects of the test variables as indicated by (Khuri and Cornell, 1987; Montgomery, 1991).

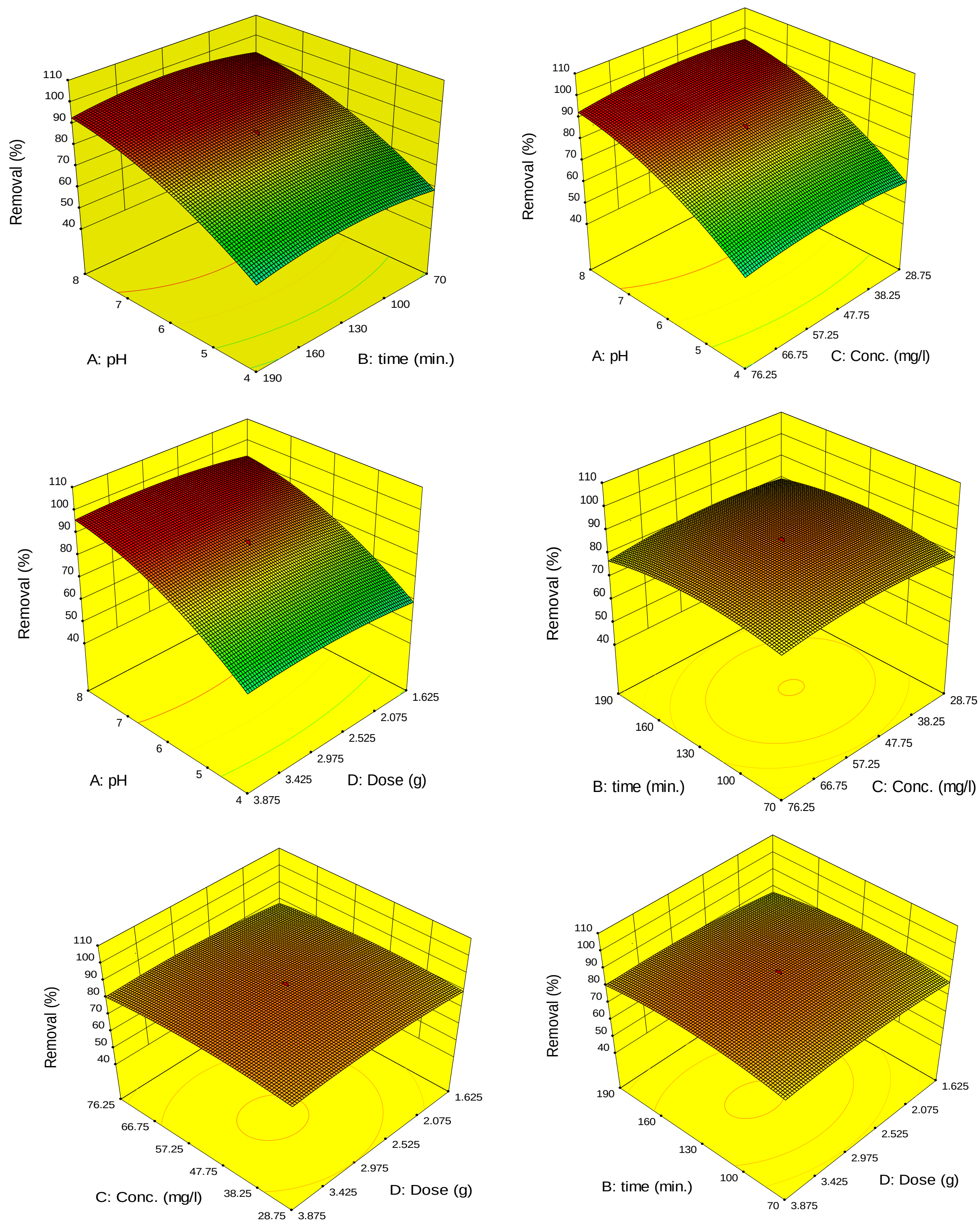
The statistical analyses of the current study revealed that the model is significant at 0.03% level of probability (F= 7). In this case pH, $(pH)^2$ and (Time) are significant model terms.

The interaction effects of these variables are described in three-dimensional representations (Fig.2).

Using the experimental design, the final equation in terms of coded factors is:

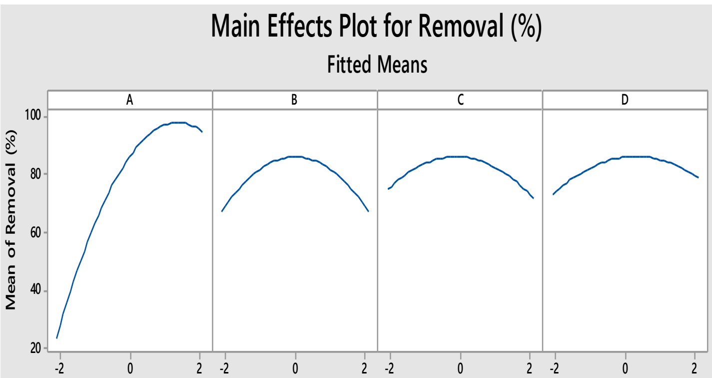
Biosorption of Cd (%) = $86.00 + 17.02\ A - 8.333{*}10^{-3}\ B - 0.76\ C + 1.35\ D + 0.60\ AB - 0.27\ AC + 0.26\ AD - 0.49\ BC + 0.58\ BD + 0.18\ CD - 6.13\ A^2 - 4.27\ B^2 - 2.94\ C^2 - 2.34\ D^2$

Fig.2: 3-D response surface plot of Removal (%) as a function of the interaction effect between different parameters affecting metal removal process



From the above figure (Fig. 2) we observed that removal efficiencies are highly affected by the pH values compared to other variables that controlled the removal process and this also confirmed from the Minitab figure (Fig. 3).

Fig.3: Main effect of different factors on the removal process.



It is well known that the solubility of metal and its concentrations on the functional group of the cell wall of the biosorbent are pH dependent, consequently, the pH is considered as the most important parameter that could affect the biosorption process (Gaur and Dhankhar, 2009; King et. al., 2007). The pH of the aqueous solution may affect both binding sites of *Pistia stratiotes* surface and the aqueous solution chemistry itself (Ai et al., 2010).

Removal efficiencies of cadmium using *Pistia stratiotes* powder were ranged from 41.20 % to 92.00 %. The higher efficiencies of plant biomass may be explained by the presence of various functional groups on the surface of the biomass, for example, carboxyl, protein, and lignin groups, which form complexes with metal ions (Fleck et al., 2013). This was confirmed from previous study that was conducted by Lima et al., 2013. They resulted that, dried biomass of *Pistia stratiotes* has different functional groups such phosphate, amide, hydroxyl, as carboxyl, and inorganic ions such as Ca^{2+}, Mg^{2+}, etc.

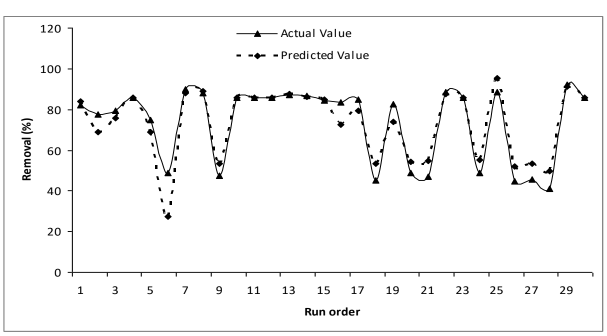


Fig. 4: Comparison between the experimental and predicted values of Cd removal (%) using dried biomass of *Pistia stratiotes.*

The efficiency of fresh plants *Pistia stratiotes* L. as a Bio-materials for removing of heavy metals from wastewater as a sustainable treatment strategy for the polluted waste water is very high and could be used in a wide scale treatment strategy (Nafea, 2019a and b).

The comparison between Actual and predicted values of removal efficiencies according to suggested model are shown in figure 4. From this figure, we observed that there is a good agreement between predicted and actual values, confirming that the RSM model effectively used to optimize the removal process.

Conclusions

From environmental and economic viewpoints, Bio-adsorption of heavy metal using the aquatic plants is a matter of great interest worldwide. In the current study, *Pistia stratiotes* powder was used to remove cadmium from contaminated water. The obtained results from the design showed that pH play an important role compared with other factors that affecting adsorption process.

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