

Investigation of nickel removal using poly(amidoamine) generation 4 dendrimer (PAMAM G4) from aqueous solutions

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Running title: Nickel removal using PAMAM generation 4 dendrimer.

ABSTRACT

Dendrimers are branched molecules that have medical, pharmaceutical, and environmental applications. Dendrimers of polyamidoamine (PAMAM) with functional groups of hydroxyl and amine are useful for making complex of heavy metals. The aim of this experimental study is to utilize dendrimer PAMAM G4 in removing nickel. FTIR and DLS analyses were conducted for determining the structure of the dendrimer. The efficiency of the process was examined under different conditions including different pH values, namely, 3, 7, and 11, the concentration of the dendrimer adsorbent (0.5-2.5 g/L), the nickel concentration (5-25 mg/L), and different reaction times (5-60 min). The data analysis was carried out using with SPSS. With the increase in the reaction time at constant values of the dendrimer (1g/L), the nickel removal ($C=20$ mg/L), at the pH values of 3, 7, and 11, increased from 7 to 18.5, from 45 to 69, and from 54 to 73, respectively. With the increase in the concentration of nickel from 5 to 25 mg/L, the removal efficiency at pH=11 decreased from 97 to 59%. The optimal value of the dendrimer for removing nickel was obtained as 1.5 g/L. The adsorption reaction followed Freundlich isotherm ($R^2=0.95$). Through optimizing different conditions, it is possible to use this adsorbent as a method with suitable effectiveness in removing heavy metals including nickel from industrial wastewaters.

Keywords: dendrimer; industrial wastewater; nanotechnology; nickel.

INTRODUCTION

The majority of heavy metals interfere with biological reactions of living cells and disrupt their metabolic activities. One of the important heavy metals is nickel, found in the forms of bivalent and trivalent oxide and hydroxide in nature (Samarghandi et al., 2011). Nickel is widely

used in the electroplating industry for the protection of iron and steel (Boujelben et al., 2009; Malakootian et al., 2012). Precipitation, adsorption on active carbon, ion exchange, and membrane separation are the conventional methods for removing nickel and other heavy metals (Boujelben et al., 2009; Kabra et al., 2008; Rajic et al., 2010; Barakat et al., 2013). Although these methods are considered efficient methods for removing metal ions, the high costs of operation and lack of reusability, membrane fouling, and increased secondary contamination are some of the constraints of these methods (Holister et al., 2003). Dendrimers are branched molecules that include a central section attached to tree-like branches, which are attached in a replicated form (Peng et al., 2009; Walington, 2005). Every group of these particles are very similar to each other in terms of size, shape, length of branches, particle density, the functional groups of the particle's surface, and the general structure of the particle (both the internal and external structure (Walington, 2005)). These particles are able to accommodate different molecules among their branches and entrap them entirely, thereby protecting them against external factors (Peng et al., 2009).

Many groups of the dendrimer family are commercially available. The most important include poly(amidoamine) dendrimers and polypropylene imines (Gajjar et al., 2015; Liu et al., 1999). Polyamidoamine dendrimers with functional groups of hydroxyl and amine are the most suitable option for applications of complex making. These dendrimers, due to their effectiveness, possess various applications. One of the key characteristics of this dendrimer is its ability in chelating metal ions from aqueous solutions (Gajjar et al., 2015). The existence of numerous and fully known functional groups across the surface of these spherical particles changes them into suitable carriers for attachment of different ligands (Liu et al., 1999). In dendrimers, as the generation grows, the number of terminal functional groups doubles (Ratanarat et al., 2003).

Kaczorowska et al. (2009) used polyamidoamine dendrimer for separating gold, copper, zinc, iron II, and iron III and achieved acceptable results (Kaczorowska Cooper et al., 2009). Fang et al. (2005) used dendrimer modified with magnetic nanoparticles for destabilizing protein compounds (Fang et al., 2005). Ming Chu et al. (2011) employed dendrimers mixed with nanoparticles for the removal of zinc from aqueous solutions. Since the PAMAM dendrimer is regarded as an adsorbent, the aim of this research was to apply polyamidoamine dendrimer in removing nickel from aqueous solutions.

MATERIALS AND METHODS

The present experimental research was conducted at laboratory scale using Jar device. Polyamidoamine G4 dendrimer was purchased from the Institute for Color Science and Technology (ICST), which had a molecular weight of 14215 g/mol, a density of 0.813 g/cm³, 64 terminal amine groups, and 250 amine groups in total. Other materials were purchased from Sigma-Aldrich. The structural schema of the dendrimer G4 and its 64 amine functional groups are provided in Figure 1. Fourier transform infrared spectroscopy (FTIR) analysis and dynamic light scattering (DLS) were conducted for determining the properties of the PAMAM G4 dendrimer. The degree of nickel removal from the aqueous solutions using the dendrimer was investigated. All of the methods in preparation of solutions and the relevant experiments were carried out based on «Standard Methods for the Examination of Water and Wastewater» instructions (APH., 1981).

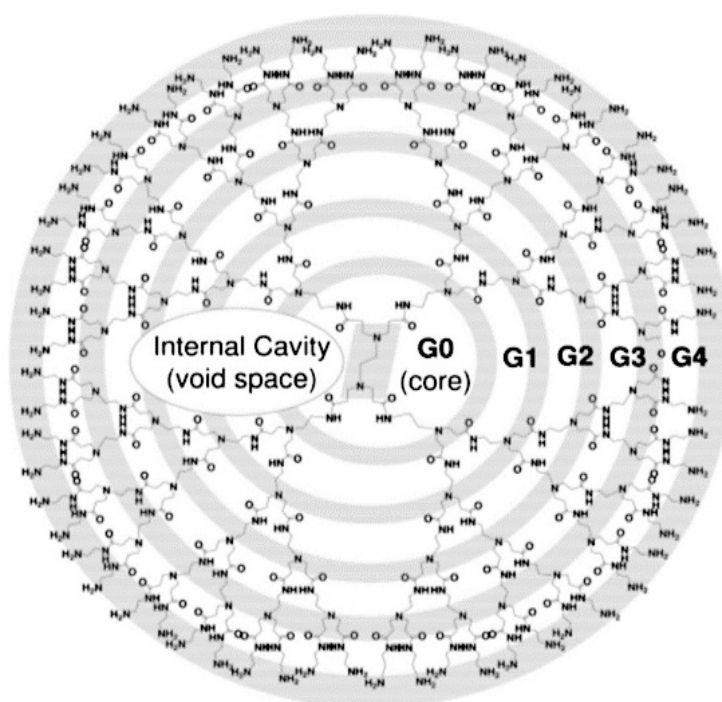


Figure 1. The structure of the PAMAM G4 dendrimer with 64 amine groups.

The experiments were carried out in the laboratory of environmental health engineering Research Centre at Kerman University of Medical Sciences. A stock solution of 1000 ppm of nickel was prepared by dissolving sufficient amounts of nickel nitrate in deionized distilled water, and its concentrations of 525- mg/L were prepared. The process efficiency was examined at three pH values, namely, 3, 7, and 11 at different times (560- min). The pH values of the aqueous solutions were adjusted using 1 M NaOH and H₂SO₄ solutions. All variables were optimized in a similar way (full factorial method). Based on the variables present in the design of the experiments, with the change of one variable and keeping the other variables constant, the number of the necessary samples in this study was 45. In order to minimize errors, the experiments were replicated twice. The temperature throughout the experiments was kept at 253^o±C. The samples were taken out in order to determine the residual nickel concentration within certain time periods (560- min), and the removal efficiency (%R) was obtained according to Equation (1).

$$R(\%) = \frac{(C_0 - C)}{C_0} \times 100 \quad (1)$$

where C_0 is the initial concentration of nickel (mg/L) and C is the concentration of nickel after the experiments. In order to separate the complex developed between the metal and dendrimer, a membrane filter 0.45 micron was used. Measurements of the concentration of nickel were carried out using an atomic absorption device (model: Philips PU 9400).

The equilibrium studies were performed by a solution containing 20 mg/L of nickel and different values of dendrimer (0.52.5- g/L) at optimal pH, and the adsorption capacity was obtained by Equation (2) (Samadi et al. 2009).

$$q_e = \frac{(C_0 - C_e)V}{M} \quad (2)$$

where, according to Equation (1), q_e represents the amount of the compound adsorbed per mass unit of the adsorbent body in terms of mg/g, C_0 represents the initial concentration of nickel in the solution, C_e shows the equilibrium concentration of the adsorbed compound in the solution after adsorption in terms of mg/L, V is the volume of the solution in terms of L, and M is the mass of the adsorbent in terms of g. Adsorption isotherms are used to define the compound adsorbed mass per mass unit of the adsorbent. Freundlich and Langmuir isotherm models are extensively used for this purpose (Wahaab. 2010). The linear equation of Langmuir isotherm is in the form of Equation (3).

$$\frac{C_e}{q_e} = \frac{1}{q_m k} + \frac{1}{q_m} C_e \quad (3)$$

where q_e represents the amount of the adsorbed fraction per mass unit of the adsorbent in terms of mg/g, C_e shows the equilibrium concentration of the compound in solution following the adsorption in terms of mg/L, q_m is the adsorption capacity, and k is the Langmuir constant.

The Freundlich adsorption isotherm is given by Equation (4).

$$q_e = k C_e^{\frac{1}{n}} \quad (4)$$

which is linearized in the form of Equation (5).

$$\log q_e = \log k + \frac{1}{n} \log C_e \quad (5)$$

where C_e is the equilibrium concentration in terms of mg/L, q_e represents the adsorption capacity at the time of equilibrium (mg/g), and n and k are the Freundlich constants.

The obtained data were analysed by SPSS 16 and for plotting the diagrams, Microsoft excel 2010 was used. A T-test was employed for investigating the difference of the concentration before and after the reaction.

RESULTS AND DISCUSSION

Figures 2(a) and 2(b) represent the FTIR and DLS analysis of the PAMAM G4 dendrimer, respectively. The interaction of the infrared radiation with the sample containing PAMAM G4 dendrimer causes altered vibrational energy of the bond in its molecules and is a suitable method for detecting functional groups and molecular structure. According to FTIR analysis depicted in Figure 2(a), the peaks at nearly 1649 and 1728 cm^{-1} illustrate the stretching band of carbonyl group (C=O). The band at nearly 3250 cm^{-1} in the PAMAM analysing eventuated from the stretching band of the amine I and the intense absorbance at nearly 1633 and 1544 cm^{-1} are specificity of the stretching band of primary and secondary amine groups. The peaks specificity of C-N groups is observed at nearly 1098 and 1027 cm^{-1} . Based on Figure 2b, the mean size of the dendrimer particles was around 4.5 nm.

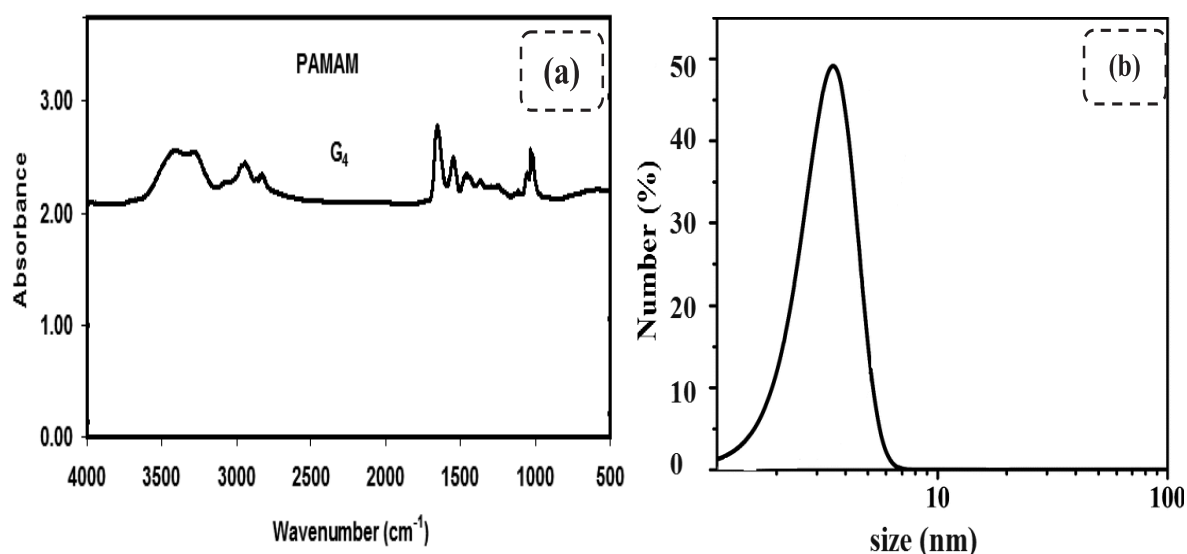


Figure 2. (a) FTIR analysis and (b) DLS of PAMAM G₄ dendrimer.

Figure 3 demonstrates the findings obtained from the investigation of the effect of reaction time on the degree of nickel removal by the PAMAM G₄ dendrimer. With the increase in the reaction time, at constant values of the dendrimer (1g/L), the degree of nickel removal (at constant concentration of 20 mg/L) at the pH values 3, 7, and 11 has increased from 7 to 18.5, 45 to 69, and 54 to 73, respectively ($p < 0.001$). According to the investigation of the results obtained from the reaction time, it was observed that, with the increase in the reaction time, the efficiency of nickel removal increased. This increased efficiency has a positive slope until 30 min. From 30 to 60 min, due to saturation of the adsorption sites with nickel ions, it remains almost constant. Therefore, the duration of 30 min is considered as the optimal contact time. According to these results, elevation of pH results increased removal efficiency by PAMAM G₄ dendrimer, owing to the effect of the adsorbent charge and ionization degree of the adsorbent. Regarding the findings of Zhang et al. (2014) about the removal of metal ions, for example, copper, cadmium, and manganese, using the composite of polyamidoamine and graphene oxide (GO/PAMAMs), with the elevation of pH from 2 to 5, the adsorption capacity of the metal ions increased. The reason of the decrease of the degree of removal at the foregoing pH values is the development of the concentration of the H³O⁺ ions, causing diminished adsorption sites. On the other hand, with the increase in the pH value, the degree of protonation of the amine groups declines, thereby increasing the chelating power of these amine groups for metal ions (Zhang et al., 2014; Niu et al., 2013). At low pH values, many of the amine groups changing to a protonated form causing development of extra positive charge on the adsorbent, whereby creating a powerful electrical repulsion force that inhibits the contact of the metal and adsorbent. Therefore, the adsorption capacity drops (Niu et al., 2013). Therefore, a pH value of 11 is considered as the optimal pH.

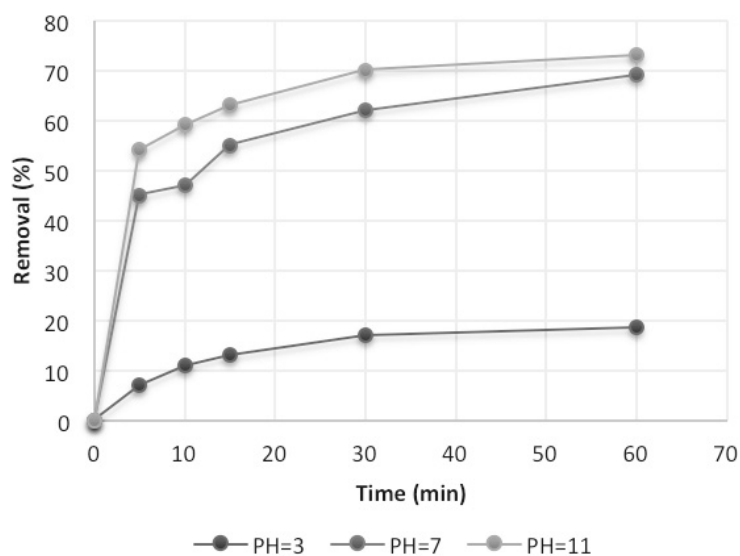


Figure 3. The effect of reaction time on the efficiency of the nickel removal (initial concentration of nickel: 20 mg/L and the concentration of dendrimer: 1 g/L).

The results obtained from the present investigation of the effect of the initial concentration of nickel on the efficiency of its removal by PAMAM G4 dendrimer are presented in Figure 4. With the increase in the concentration of nickel from 5 to 25 mg/L, the removal efficiency at a pH value of 11 has decreased from 97 to 59%. At pH values of 3 and 7, the degree of removal declined from 44 and 82 to 52%, respectively. This reduction in the efficiency had a significant difference across various concentrations ($p < 0.001$). With the increase in the concentration of nickel in the aqueous solution, the removal efficiency of this metal declined. According to the findings of Barakat et al. (2013), the increase in the concentration of the metal ions of copper, chromium, and nickel resulted in a decrease in removal efficiency by PAMAM dendrimer and titanium dioxide composite. This was due to the saturation of adsorption sites on the dendrimer (Barakat et al. 2013).

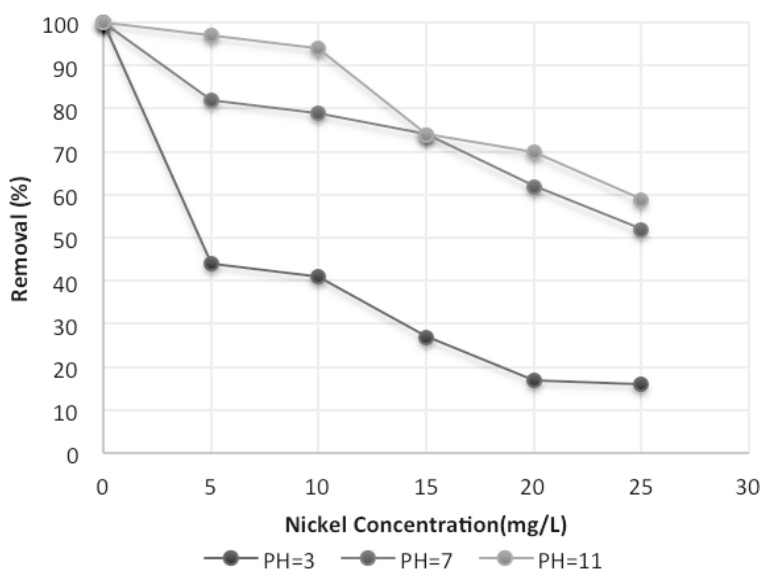


Figure 4. The effect of the initial concentration of nickel on the removal efficiency (time: 30 min, dendrimer concentration: 1 g/L).

The effect of different concentrations of PAMAM G4 dendrimer is presented in Figure 5. With the increase in the concentration of the dendrimer from 0.5 to 2.5 g/L at different pH values, namely, 3, 7, and 11, and at a constant value of nickel (20 mg/L), the removal efficiency increased such that, at a pH value of 11, it has risen from 44 to 83%. At the pH values of 3 and 7, with the increase in the concentration of the dendrimer, the degree of removal has increased from 12 to 30 and 48 to 78, respectively. Since the degree of nickel removal at dendrimer values above 1.5 g/L did not change significantly, the optimal value of this adsorbent for removing nickel is chosen as 1.5 g/L.

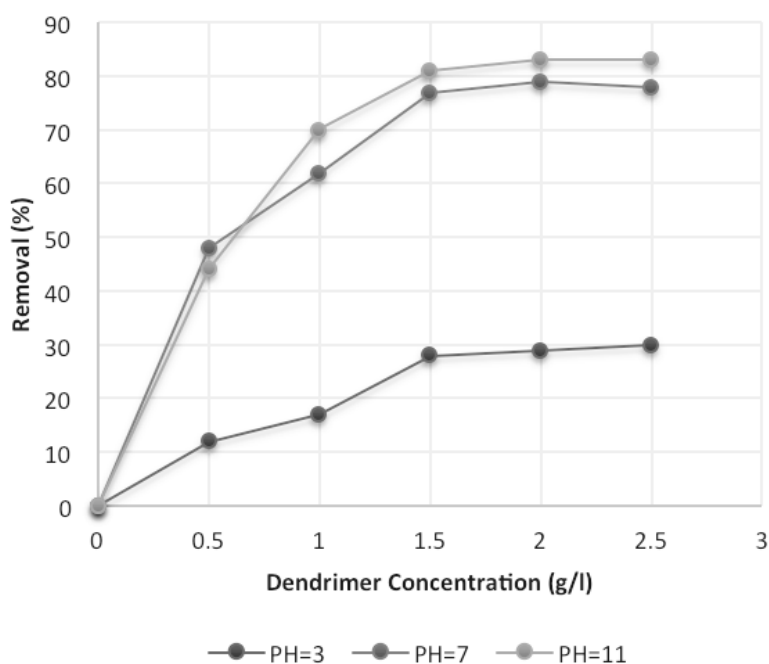


Figure 5. The effect of the dosage of PAMAM dendrimer in removing nickel at different pHs (retention time: 30 min, initial concentration of the nickel: 20 mg/L).

With the increase in the value of the adsorbent, due to elevation of the number of adsorption sites, there are larger numbers of ligands to create a complex with the PAMAM dendrimer, resulting in increased efficiency of removal of metal ions from aqueous solutions. The optimal value of PAMAM dendrimer was obtained as 1.5 g/L. The results are congruent with the findings obtained by Shahbazi et al. (2011) who worked on the adsorption of metal ions of lead, copper, and cadmium by dendrimers with SBA-15 functional group. In their research, the amount of the optimal adsorbent was obtained as 1 g/L, where the maximum removal efficiency of lead, copper, and cadmium was obtained as 97, 93, and 53% (Shahbazi et al., 2011).

Various mathematical models have been used for describing the adsorption balance studies of contaminants on the surfaces of solid materials. Amongst them are the Freundlich and Langmuir adsorption models. In the Langmuir model, it is assumed that the adsorption of metal ions takes place on a homogeneous surface (monolayer), and there is no interaction amongst the adsorbed species. In the Freundlich adsorption model, it is assumed that adsorption of metal ions occurs on a heterogeneous surface of the adsorbent. Table 1 provides the findings related to the adsorption

isotherm of nickel by the PAMAM G4 dendrimer. Adsorption isotherms for PAMAM G4 dendrimer are provided in Figure 6. By comparing the determination coefficient (R^2) of these diagrams with each other, it is possible to determine the model related to this adsorbent in removing nickel. As can be observed from Figure 6, the Freundlich adsorption isotherm is considered a better model for describing adsorption of nickel by PAMAM G4 dendrimer ($R^2=0.95$).

Table 1. The findings associated with the adsorption isotherm of nickel by PAMAM G4 dendrimer under optimal conditions (t: 253 \pm C, C_i : 20 mg/L, pH=11, T: 30 min).

M(g)	C_i	C_e	$X=(C_i - C_e)$	$\eta\% = \left(\frac{C_i - C_e}{C_i}\right) * 100$	$q_e=(X/M)$	$1/q_e$	$1/C_e$	$\text{Log}q_e$	$\text{log}C_e$
0.5	20	11.2	8.8	44	17.6	0.057	0.089	1.24	1.049
1	20	6	14	70	14	0.071	0.166	1.14	0.78
1.5	20	3.8	16.2	81	10.8	0.092	0.26	1.033	0.58
2	20	3.4	16.6	83	8.3	0.12	0.29	0.919	0.53
2.5	20	3.4	16.6	83	6.64	0.15	0.29	0.82	0.32

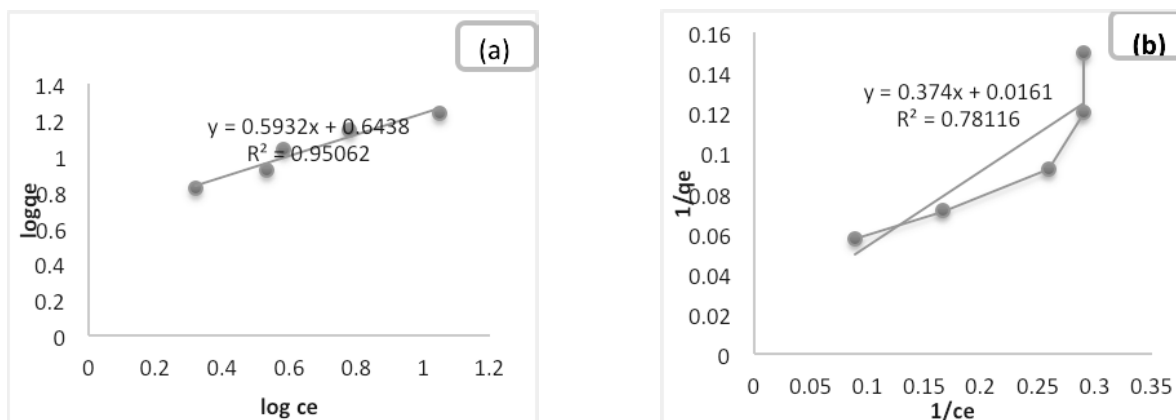


Figure 6. Freundlich adsorption isotherm (a) and Langmuir adsorption isotherm (b) by PAMAM G4 dendrimer in removing nickel.

CONCLUSIONS

The aim of this research was to investigate the removal efficiency of nickel by PAMAM G4 dendrimer. The degree of nickel removal grew with the increase in the pH value. The optimal time for the maximum degree of removal was obtained as 30 minutes. Elevation of nickel concentration in aqueous solutions, due to saturation of the capacity of adsorption sites, leads to decreased efficiency of the process. On the other hand, the increase in the amount of the dendrimer adsorbent resulted in increased removal efficiency of nickel from aqueous solutions owing to the increased number of adsorption sites and magnification of the surface.

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دراسة عن إزالة النيكل باستخدام دندر يمرات أميدو أمين المتعدد من الجيل الرابع (PAMAM G4) من المحاليل المائية

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الخلاصة

الدندريمرات هي جزيئات متفرعة لها استخدامات طبية وصيدلانية وبيئية. إن خليط دندريمرات PAMAM مع مجموعات فعالة من الهيدروكسيل والأمين يكون مفيداً في صناعة مركب من المعادن الثقيلة. والهدف من هذه الدراسة التجريبية هو استخدام دندريمرات PAMAM G4 في إزالة النيكل. تم إجراء تحليلات FTIR وDLS لتحديد بنية الدندريمر. تم اختبار كفاءة العملية تحت ظروف مختلفة بما في ذلك استخدام محاليل ذات قيم مختلفة من pH؛ بمعنى 3، 7 و11. وكان تركيز الدندريمر الممتز (0.5-2.5 جم / لتر)، وكان تركيز النيكل (25-5 ملجم / لتر)، وأزمنة التفاعل المختلفة (5-60 دقيقة). تم تحليل البيانات باستخدام SPSS. ومع زيادة زمن التفاعل عند قيم ثابتة للدندريمر بمقدار (1 جم / لتر)، ارتفعت كفاءة إزالة النيكل (C= 20 mg / L) من 7 إلى 18.5، ومن 45 إلى 69، ومن 54 إلى 73، على التوالي عند قيم pH 3 و7 و11. ومع زيادة تركيز النيكل من 5 إلى 25 ملجم / لتر، انخفضت كفاءة الإزالة عند قيم pH = 11 من 97% إلى 59%. تم الحصول على القيمة المثلى للدندريمر لإزالة النيكل عند 1.5 جرام / لتر. وكان تفاعل الامتزاز من خلال تطبيق نموذج Freundlich isotherm ($R^2=0.95$). من الممكن استخدام هذا الممتز كطريقة ذات فعالية مناسبة في إزالة المعادن الثقيلة بما في ذلك النيكل من مياه الصرف الصناعية عند تحسين الظروف المختلفة.