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Sorption of Cr(VI) by Amberlite XAD-7 resin impregnated with brilliant green and its determination by quercetin as a selective spectrophotometric reagent

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ABSTRACT

A new chelating polymeric sorbent as an extractant-impregnated resin (EIR) has been developed using brilliant green (BG) and Amberlite XAD-7 resin. The BG-impregnated resin showed superior binding affinity for Cr(VI) in the presence of many co-existing ions and no considerable interference was observed. The influence of various physicochemical parameters on the recovery of Cr(VI) were optimized by both static and dynamic methods. The Langmuir adsorption isotherm gave a satisfactory fit of the equilibrium data. The kinetic studies performed for Cr(VI) sorption revealed that <45 min was sufficient for reaching equilibrium metal ion sorption. A preconcentration factor of 100 was found for the column-mode extraction. The spectrophotometric determination of eluted Cr(VI) was carried out using quercetin as a selective reagent. The calibration graphs were linear in the range 5.0×10^{-8} to 4.0×10^{-7} M with a detection limit of 8×10^{-9} M. The proposed method has been successfully employed for the analysis of natural water. The recoveries for the Cr(VI) amounts spiked to the samples were >93%, which confirmed accuracy of the measurements.

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1. Introduction

The determination of low level of Cr(VI) in environmental samples and its removal from the water by using an inexpensive processing are very important. Several methods have been described for the separation and determination of Cr(VI) ions including, solvent extraction [1], coprecipitation [2], and solid phase extraction [3-6]. Solid phase extraction is one of the wellknown preconcentration/separation techniques for this purpose [7-10]. In this technique, various reagents used as chelating agents are grafted onto appropriate solid supports, such as Amberlite XAD series, which have been successfully used in most studies in our working group [11-13]. Among the various methods of solid sorbent preparation, impregnating methods have been more developed in the last decade [12-16]. The impregnation method is free from difficulties encountered in chemically linking a chelating reagent to a support matrix. In addition, there is a wide choice of reagents for desired selectivity [17-20].

Brilliant green (BG) is one of the commonly known cationic dye (structure is shown in Fig. 1(a)) used for various purposes,

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e.g. biological stain [21], veterinary medicine [22], an additive to poultry feed to inhibit propagation of mold [23], intestinal parasites and fungus [24]. It is also extensively used in textile dying and paper printing [25]. Recently, adsorption of BG on a series of organic/inorganic substances was characterized for various purposes, e.g. removal of BG from aqueous solution [26–28], preparation of optical pH sensor [29], separation/preconcentration of uranyl ions [30].

In the light of the requirements for the search of a simple and inexpensive method for separation/preconcentration of Cr(VI) followed by their individual elution and determination, this work has been designed to prepare a selective anion-exchange resin containing BG via the impregnation in/on Amberlite XAD-7, a copolymer backbone for the selective uptake of Cr(VI) from weakly acidic solutions. Subsequently, enabling to determine Cr(VI) eluted from the resin by a simple spectrophotometric method has targeted to use quercetin, a natural antioxidant reagent, which was used previously as a selective agent for the determination of Cr(VI) in natural water samples [31].

2. Experimental

2.1. Reagents

All the chemical reagents were of analytical reagent grade and supplied by Merck (Darmstadt, Germany). Amberlite XAD-7

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Fig. 1. Chemical structure of brilliant green (BG).

(20–50 mesh, surface area: $450 \text{ m}^2 \text{ g}^{-1}$, pore diameter: 90 Å) was obtained from Fluka (Switzerland). Double distilled and deionized fresh water was used in all the experiments. BG solutions were prepared by dissolving the appropriate amounts in 100 mL of methanol. Quercetin solution $(1.0 \times 10^{-3} \text{ M})$ was prepared by dissolving the appropriate amount in 100 mL of n-amyl alcohol. All of these solutions were stored in refrigerator at 4°C. Cr(VI) solution $(1 \times 10^{-2} \text{ M})$ was prepared by dissolving appropriate amounts of K₂Cr₂O₇ in distilled water and the prepared solution was standardized by iodometric titration. Working solutions were papered by diluting the stock solution. A solution with concentration of 0.1 M respect to NH₃ and NaCl was prepared as the eluent. The following solutions with equal concentrations of 1 M were used for buffering the experimental solutions at the pH of interest: HCl/KCl for pH 1-2; formic acid/sodium formate for pH 2-4; acetic acid/sodium acetate for pH 4-6.

2.2. Apparatus

Spectrophotometric Cr(VI) determinations with quercetin were performed by use of a Shimadzu model UV-1601PC spectrophotometer equipped with quartz cuvettes of 1 cm thickness. A Corning model 130 pH-meter was used for pH measurement. The flow of liquids through the short column was controlled with an Eyela SMP-23 peristaltic pump and a 6-port valve (V-451, Upchurch). Nitrogen analysis of the prepared impregnated resin was carried out using a Gerhardt micro Kjeldahl apparatus. A Sartorius membrane filter of pore size 0.45 μ m was used for filtration of the natural water samples.

2.3. Preparation of the EIR

In order to remove each type of impurity, which may be found with the fabricated beads, before the impregnation, the Amberlite XAD-7 resin was treated with 1:1 methanol-water solution containing 6 M HCl for 12 h. Then, the resin was thoroughly rinsed with doubly distilled water and placed into a drying oven at 50°C for 30 min. To prepare the impregnated resin, portions of Amberlite XAD-7 (1g of dry resin) was transferred into a series of glassstoppered bottles containing 200 mL of BG solutions with different concentrations. The mixtures were slowly shaken for 6 h to complete the impregnation process. After that, they were placed into a drying oven to remove the solvents at the ambient temperature. Each type of the resin beads was then transferred to a porous filter and washed successively with HCl and large amounts of distilled water until none amount of BG was detected in the filtrate, spectrophotometrically. Finally, the impregnated resins were dried at 50 °C and weighed. The amount of BG impregnated on/in the resin bead was determined from the amount of weight change in resin. To protect the prepared EIR from damage, it was kept in a stoppered dark glass bottle under the distilled water content.

2.4. Sorption equilibrium procedure

The sorption isotherms of Cr(VI) ions on/in the EIR were obtained using the batch technique at the pH of maximum uptake. For this purpose, aliquots of 100-mL of the buffered solutions (pH 3.0) containing Cr(VI) with the concentration range of 1.0×10^{-4} to 5.0×10^{-4} M were placed in a series of conical flasks and 0.05 g of the EIR was added to each of them. The mixtures were placed in a constant temperature shaker and were shaken at 298 ± 1 K for 45 min. After that, portions of 5-mL of the supernatants were withdrawn and subjected to the determination process of Cr(VI) as discussed at the following.

2.5. Sorption rate procedure

A series of fixed weighed portions (0.0500 g) of the EIRs were immersed into aliquots of 100-mL of Cr(VI) buffered solutions (pH 3.0) with concentration of 2.0×10^{-5} M at $25 \,^{\circ}$ C. The mixtures were stirred mechanically for a pre-determined time interval at a fixed speed (220 rpm). The EIR amount, contact time and temperature of sorption were carefully controlled. At the appointed time, each one of the mixtures was rapidly filtrated using a porous filter and the SIR was treated with 4.5 mL of the eluent solution. After that, the filtrate eluent was acidified with 0.5 mL 1 M HCl and subjected to the determination process of Cr(VI) as discussed at the following. The optimum contact period was taken as the time at which maximum Cr(VI) sorption took place.

2.6. Metal extraction procedure

0.5 g of EIR was packed into a column with an internal diameter of 0.4 cm. The ends were fitted with glass wool to keep the EIRs inside of the column. The bed length of resin in the column was about 75 mm. Working solutions containing Cr(VI) with the concentration exceeding the detection limit prepared in which the pH and ionic strength were respectively adjusted to 3.0 and 0.01 M using the formic acid and ammonium formate solutions. Periodically, aliquot of 500-mL of the prepared solutions was passed through the column at a flow rate of 6 mL min⁻¹. After washing the column with 10 mL of distilled water to remove free matrix substances, the Cr(VI) content sorbed on the EIR was desorbed using 4.5 mL of the eluent solution at a flow rate of 1 mL min⁻¹. The eluent was transferred to a 10-mL screw cap test tube, then 0.5 mL of 1 M HCl and 5 mL of the quercetin solution were added to it. The tube was capped and shaken for 20 min. After centrifugating the tube contents and separating the aqueous and organic phases, aliquot of the aqueous phase was withdrawn and subjected to the spectrophotometric measurement at the maximum wavelengths of 292 nm against a reagent blank prepared by the same manner.

3. Results and discussion

3.1. Preparation and characterization of the EIR

As pointed out above, there is no report referring to use BG as an extractant for Cr(VI) ion recovery. In the current study, BG was impregnated on/in Amberlite XAD-7, which is an inert polymeric substance based on acrylic ester, hydrophobic, continuous pore phase with a weak dipole moment of 1.8. It is used for removal of organic pollutants form aqueous wastes, ground water and vapor streams. It has been also used in the impregnation procedures for preparation of various impregnated resin employed for selective separation of certain metal ions [17,20,31–34].

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