DETERMINATION OF SORPTION CAPACITY OF SORBENT BASED ON SILICA GEL

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Abstract. *In this research work, sorbents were synthesized based on non-covalent immobilization of diphenyl carbazone on silica gel matrix. Copper (II) ions were absorbed into the obtained sorbent, and physio-chemical and also elemental analysis methods were used to determine its sorption capacity as well. The full paper is designed based on the results of spectrophotometer and X-ray fluorescence spectrum analysis.*

Keywords: *EMC-30PC-UV Spectrophotometer, EDX-8100 X-ray fluorescence, silica gel, diphenyl carbazone, Cu (II) ion.*

INTRODUCTION

On a global scale, scientific and research work is being carried out aimed at immobilizing ligands containing nitrogen, phosphorus, and sulfur into organic polymer and mineral matrices in order to obtain selective and effective complex-forming sorbents [1]. In many research studies, a special emphasis has been given to find selective sorbents by the method of chemical modification of silica gel [2]. Lately, the global production of synthetic sorbents has doubled compared to the previous decade [3].

This is a matter of great concern due to copper pollution. It's high toxicity and non-biodegradability. According to previous literature report [4] it has already establsihed that Cu(II) is a human carcinogen. That's why, it should be needed to develop effective methods to remove Cu^{2+}. Many processes, such as chemical precipitation, ion exchange, reverse osmosis, and adsorption, have been employed to remove Cu^{2+} from solutions.

In fact Cu(II) is widely used in many industries. In the meantime there are many pragmatic processes that can be used to remove heavy metal ions and other contaminants from aqueous solutions, the most common for Cu(II) and other soft metal ions being oxygen and nitrogen containing ligands. are sorbents obtained on the basis of immobilization in the matrix [4].

Silica gel is entensively used in many chemical processes to provide new technical applications. Moreover, silica gel is an amorphous inorganic polymer composed of internal siloxane groups (Si-O-Si) with silanol groups (Si-OH) dispersed on the surface [5].

In our paper, a new solid-phase extractant diphenylcarbazone-reacted silica gel was synthesized and also characterized by spectroscopic techniques. This solid phase extractant was found to be stable in 1-6 mol l^{-1} HCl or H_2SO_4 as well as common organic solvents.

The resulting sorbent selectively could able to separate and enriche several metal ions along with similar properties, such as Hg (II), Cd (II), Ni (II), Co (II), Mn (II), Pb (II) and Zn (II). The results revealed that this new solid extractant has good stability and it can also be reused frequently without reducing its extraction percentage.

It was noted that a micro-column packed with diphenylcarbazone-functionalized silica gel was used for the determination of mercury in real samples by online solid-phase extraction and flow injection spectrophotometry [6].

We clearly studied the noncovalent stability of Arsenico I, Alizarin Red, and Xylenol Orange diphenyl carbazone by adding to silica xeroxels and modified the silica gel method to determine fluoride and chloride ions through solid phase spectrophotometry, and test methods were also developed. Moreover, the reactions of immobilized reagents with aluminum (III), zirconium (IV) and mercury (II) were studied.

The feasibility study was exercised by using immobilized reagent-metal ion-halide ion systems for the determination of halide ions and finally it has evaluated. Particularly, Indicator powders have been proposed for the detection of 0.5-10 mg/l fluoride ions and 1-30 mg/l chloride ions, and indicator tubes have been developed for the detection of 20-200 mg/l chloride ions. The amount of halide ions in Narzan mineral water was determined [7].

In addition, that the sorption of 1,5-diphenylcarbazone on highly dispersed silica from toluene and acetone-hexane (1:4) solutions was also studied. By spectroscopic method the absorption nature of the binding of the reagent to the surface of silica gel was determined as well. Typically, a solid-phase reagent was proposed for the determination of molybdenum (VI) up to 0.025 and 0.05 μg/L by absorption-diffuse-reflection and visual test, respectively, and it was further used for the determination of molybdenum in sea salt and pharmaceutical preparation [8].

EXPERIMENTAL PART

The main objective of our study is to explore the absorption efficiency of sorbontes containing N and O derived from silica gel and diphenyl carbazone (DK). The absorption capacity of the synthesized SG-DK01, SG-DK02 and SG-DK04 sorbents was determined by an EMC-30 PC-UV spectrophotometer at a wavelength of 602 nm, and the amount of Cu (II) absorbed by the sorbents from the $CuSO_4$ solution was also measured. Elemental analysis was performed by X-ray fluorescence analysis.

10 g of silica gel grains were measured on an analytical scale and later it has washed with distilled water and dried in a oven at high temperature for 2 hours. in order to wash off the additional substances and reagents on its surface.

Diphenylcarbazone containing O and N adonating atoms lignad, was dissolved in an organic solvent and 3 different molar concentration solutions wuth maintaing 20 ml were prepared and later 2 grams of dried silica gel grains were added to each solution for three days. As a result of noncovalent immobilization of silica gel with new solid phase sorbents named SG-DK01, SG-DK02 and SG-DK04 were obtained. The products were dark brown, reddish solids, non-volatile.

The sorbents were produced by environmentally friendly modification methods have been investigated for the removal of copper ions from aqueous media. For the particular testing, 50 mg of SG-DK01, SG-DK02, and SG-DK04 sorbents were added to a 0.1N solution of CuSO4 salt in 3 test tubes, respectively. Afterwards, a spectrophotometric analysis method was used to determine the concentration pattern of Cu ions in the solution.

A standard solution of Cu2+ was prepared (listed in Table:1) and the sorption capacity of the prepared sorbents was determined using the EMC-30PC-UV Spectrophotometer (Table:2).

Table 1:

Preparation of standard solution of Cu^{2+}.

№	0.1 M solution of CuSO4 V(ml)	H_2O V(ml)	25% solution of NH_4OH V(ml)	Solution concentration (Cm)	Optical density (Abc)
1.	1	8	1	0,01	0,1235
2.	2	7	1	0,02	0,247
3.	3	6	1	0.03	0,6151
4.	4	5	1	0.04	0,8984
5.	5	4	1	0.05	0,1898
6.	6	3	1	0.06	0,4445

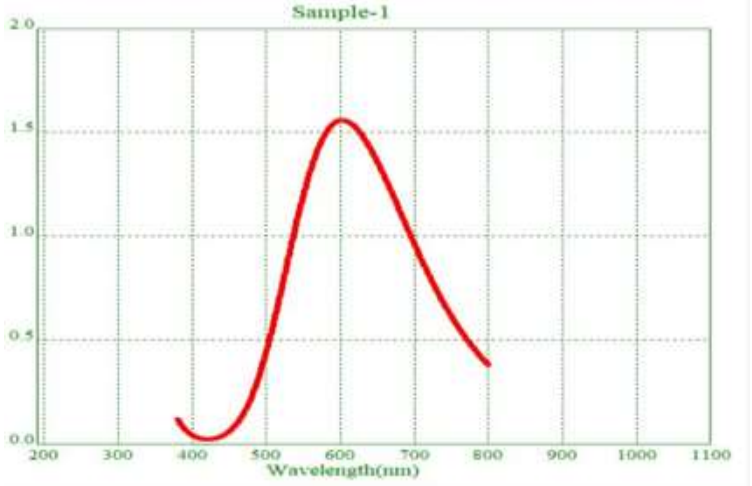

Figure 1. Spectrum of the maximum light absorption wavelength in the determination of Cu^{2+} ion in the form of an ammonia complex.

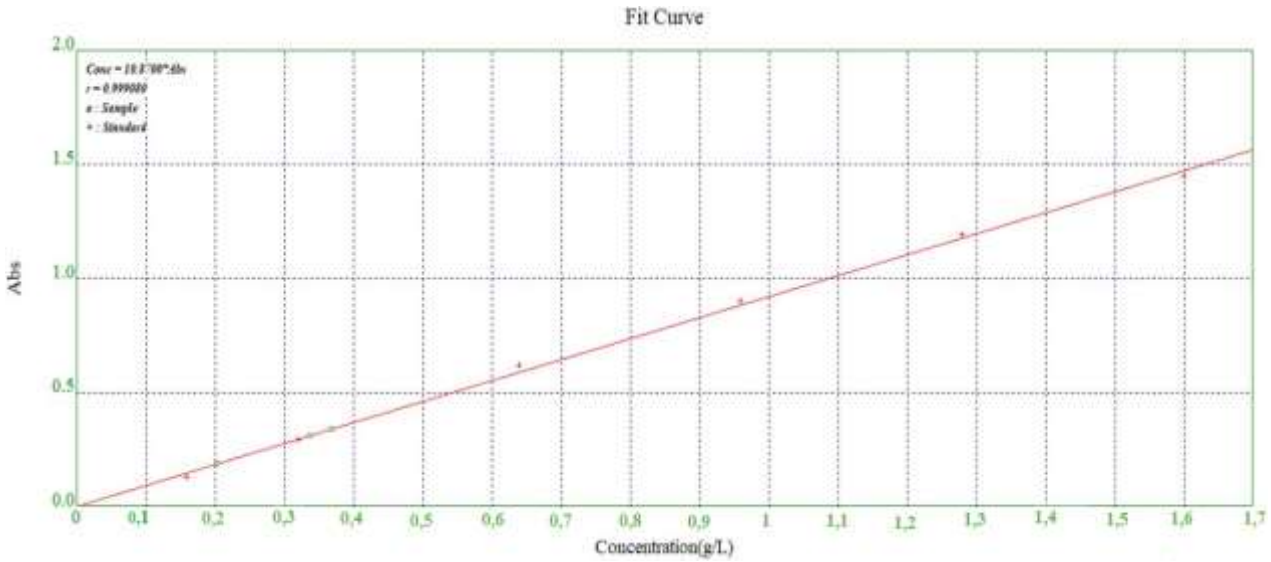

Figure 2. Graded graph for the determination of Cu^{2+} ion in the form of an ammonia complex.

Table 2

The sorption capacity of the synthesized sorbent was determined

Solution sample	Normal concentration solution - C_1	Solution of unknown concentration - C_2	SEC (static exchange capacity)
1	0,1	0,08	2
2	0,1	0,07	3
3	0,1	0,085	1,5

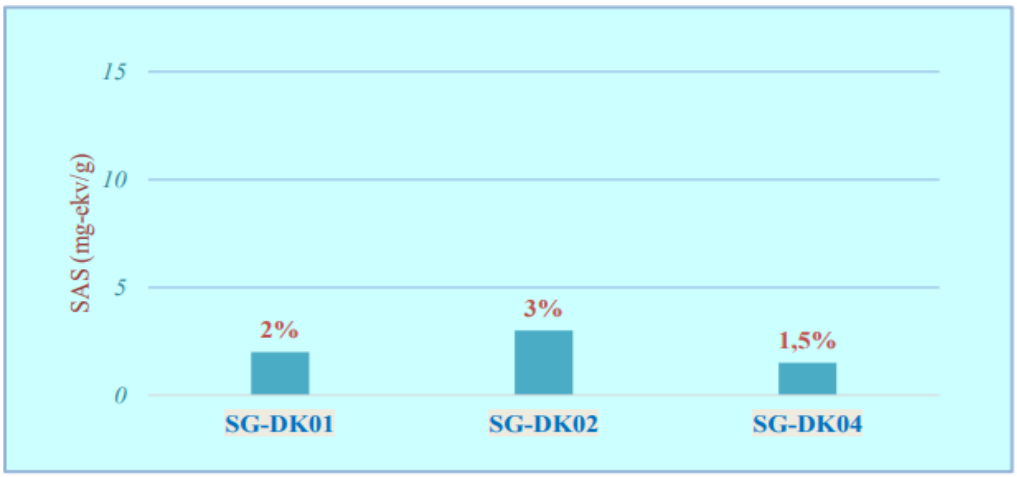

Figure 3. SEC indicators of the obtained sorbents

It has cleary revealed that the sorption capacity of our SG-DK02 sorbent was higher than the other two sorbents, i.e. 3 mg-ekv/g.

The elemental analysis was used by X-ray fluorescence (EDX-8100) to determine Cu^{2+} ions contained in copper (II) sulfate solution of prepared sorbents. The essence of this method was to determine the elemental composition of the sample. In addition that the elemental composition of SG-DK01 and SG-DK02 sorbents with higher SEC index was examined by X-ray fluorescence spectrometric analysis.

We have calculated the percentage of copper ions absorbed by our SG-DK01 sorbent by mass and also found that the percentage of copper ions was 9.665 in the SG-DK02 sorbent. We found that SG-DK02 sorbent has a higher sorption capacity than SG-DK01 brand sorbent.

Quantitative Result

Analyte	Result	[3-sigma]	Proc.-Calc. Line		Int.(cps/uA)
Si	86.796 %	[1.692]	Quan-FP	SiKa	2.3813
Cu	9.550 %	[0.050]	Quan-FP	CuKa	85.1298
S	2.796 %	[0.416]	Quan-FP	S Ka	0.0120
Ca	0.656 %	[0.050]	Quan-FP	CaKa	0.1208
Ti	0.101 %	[0.020]	Quan-FP	TiKa	0.1323
Fe	0.097 %	[0.003]	Quan-FP	FeKa	0.2327
Br	0.003 %	[0.001]	Quan-FP	BrKa	0.0053

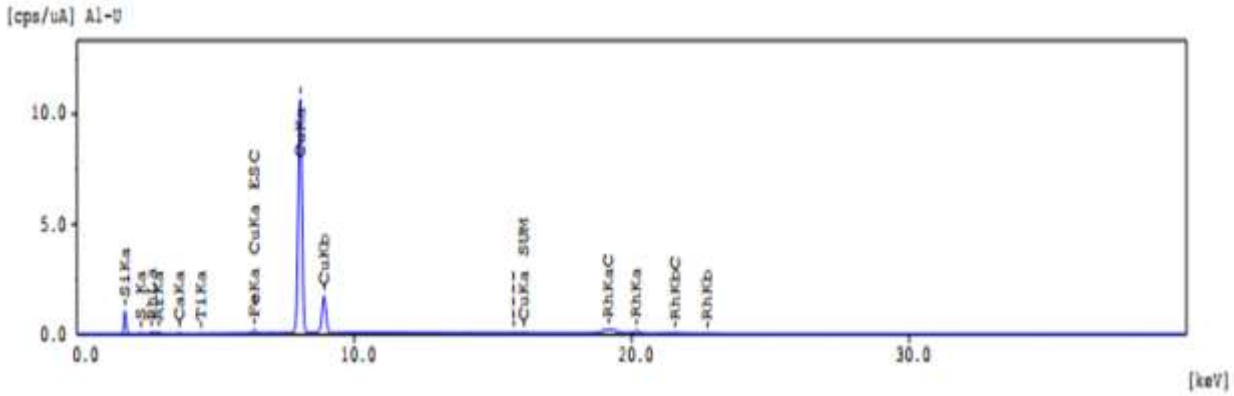

Figure 4. X-ray fluorescence spectrum analysis of the complex compound formed by SG-DK01 sorbent with Cu

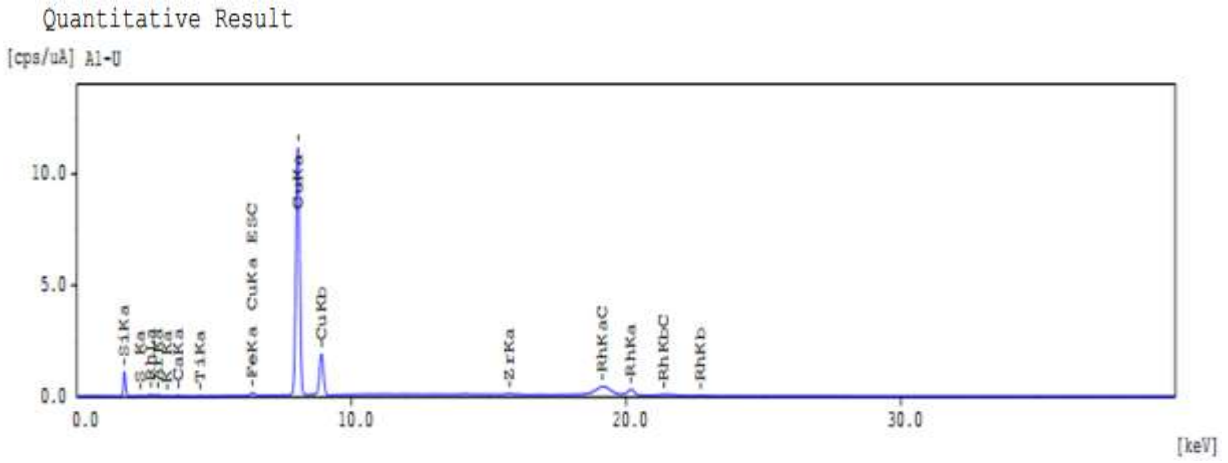

Figure-5. X-ray fluorescence spectrum analysis of SG-DK02 sorbent complex with Cu

CONCLUSION

There is an increasing interest in improving the metal exchange capacity of silica gel by modifying its surface. The present research work has aimed at solvent-free modification of the silica gel surface using diphenylcarbazone as an organic ligand. The major target of this work is to determine the sorption capacity of SG-DK01, SG-DK02 and SG-DK04 sorbents by the EMC-30PC-UV Spectrophotometer. In our experiment, the SEC index of SG-DK02 sorbent was 3 mg-eq/g.

Specially, the composition of complex compounds of the obtained sorbents with metal ions was checked by X-ray fluorescence spectrometric analysis. The amount of absorbed copper ions in the sorbent was 9,550% in 1 mg SG-DK01 sorbent, and also 9,665% Cu in SG-DK02. This showed that our SG-DK02 sorbent has a higher sorption capacity compared to SG-DK01 brand sorbent and the expected result was achieved as well.

It can be concluded that ligands containing oxygen and nitrogen atoms are noncovalently (physically) immobilized in the silica gel matrix, they can react easily with copper and also form stable complex compounds.

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