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Application of Factorial Design in Adsorption Studies of Silver Ions with Multi-Walled Carbon Nanotubes

Feza Geyikçi

Ondokuz Mayıs University, Department of Chemical Engineering, 55139, Samsun, TURKEY

Abstract: The adsorption-related applications of CNTs to solve environmental pollution problems have received considerable attention in recent years. Carbon nanotubes are strong materials with the capacity to form covalent bonds. These features can be improved with various modifications. The use of CNTs in the adsorption of heavy metals has become widespread. MWCNT-Metal pairs can be important for many different applications. The aim of this study is to investigate the Ag(I) ions adsorption capacity of MWCNTs from aqueous solutions using the experimental design method. A full factorial design was employed to optimize the adsorption of Ag(I) ions onto multi-walled carbon nanotubes (MWCNT). The effects of process parameters such as the initial Ag(I) concentrations (10 and 100 mg/L), pH (2 and 8) and multi-walled carbon nanotubes dosage (0.1 and 1.0 g/L) on Ag(I) adsorption were investigated on the adsorption process. Under acidic conditions, the removal efficiency of silver ions was found to be high by using MWCNTs. According to the results of factorial design calculations, the maximum adsorption capacity was found as 0.998 mg/g.

Keywords: Multi-walled carbon nanotubes, full factorial design, Ag(I) adsorption.

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Corresponding author. E-mail: fezag@omu.edu.tr, Tel: +90 (362) 312 1919-1343

INTRODUCTION

Heavy metals are among the most common pollutants found in industrial effluents. The presence of excess amounts of these metals in the environment is of great concern due to their accumulated toxicity in living organisms and threatening human life and the environment. Among the heavy metals such as lead, copper, cadmium, zinc, silver, and nickel, the least harmful metal is silver. Silver is one of the transition metals found in nature in the form of compounds. It is often found combined in copper or lead mineralization (1–3). Its main industrial use is as a silver halide compound in the production of photographic film. Other industrial uses include electrical conductivity, thermal conductivities, photosensitivity, battery catalysts, and mirrors. Silver (Ag) as one of the precious metals has prominent ductility and antibacterial properties, and the free silver ion is dangerous for sensitive aquatic plants and representative species of invertebrates (4–6).

While the adsorption method is used for the recovery or uptake of silver from aqueous media, methods such as precipitation, ion exchange, membrane processes, solvent extraction, electro coagulation, coagulation-flocculation, adsorption, reductive exchange and electrolytic recovery are also applied. Adsorption is known as a method used in the recovery process and synthesis of new material types. Adsorption processes using activated carbon and carbon nanotubes have been widely proposed and used for the removal of different (organic and inorganic) pollutants from waters. This process is generally applied method to protect water resources (7).Apart from the purification method, this method can be applied for the synthesis of materials with many different properties. Cost-effective materials that have been investigated for their potential use as adsorbents for heavy metal uptake include agricultural waste, food organic waste, algae, fungi, bacteria, fly ash, red mud, industry waste, fly ash, phosphogypsum bentonite

limestone waste materials as refuse concrete, zeolite and others (8–11).
In recent years, adsorption studies with different carbon nanotubes have attracted attention. Carbon nanotubes (CNTs) are one of the most commonly used building blocks of nanotechnology. CNTs had been discovered by Iijima, and has led research to new area in many interdisciplinary investigations as the advantages of CNTs are unique structural, electronic, optoelectronic, semiconductor, mechanical, thermal, chemical, and physical properties. CNTs are new generation materials with the potential to be used in water pollution control. The USEPA has identified CNTs as a key material to be used in environmental applications such as treatment and remediation. In a previous study, CNTs were oxidized with nitric acid and then treated as adsorbent for various heavy metal ions including Cu^{2+}, Zn^{2+}, Pb^{2+}, Cd^{2+}, Co^{2+} from wastewater (12). Since then CNTs have been gaining increasing recognition for their adsorption capabilities. This is mainly due to their extremely small size, uniform pore distribution and large specific surface area (13–15). CNTs can be classified into two main types: single wall carbon nanotubes (SWCNT) and multi-wall carbon nanotubes (MWCNT). MWCNTs provide high electrical conductivity. There are two main methods to modify MWCNTs for further adsorption applications, including noncovalent modification and covalent modification (12). MWCNTs have high electrical conductivity, a large specific area, and exceptional chemical inertness, also making them ideal for use in energy storage devices (16). MWCNT/Metal nanohybrids can be used in the synthesis of polyalcohols. The PtCoNi/MWCNTs nanocatalyst was used for the hydrogenation of furfural (17). Due to their large specific surface area, small size, and hollow and layered structures, MWCNTs have been proven to possess great potential as superior adsorbents for removing many kinds of organic and inorganic contaminants including 1,2-dichlorobenzene, trihalomethanes, microcystins, fluoride, lead, arsenate, cationic dyes, and ureamic toxins (18,19). In another study, ZIF-8/MWCNTs-COOH was synthesized as a nanoadsorbent in the removal of dyestuff (Congo Red) and antibiotic (tetracycline) wastes (20). Because of their accessible availability and inexpensiveness, MWCNTs have been widely used in many studies. They have excellent advantages such as distinctive hollow structure, chemical stability, and large specific surface area. MWCNTs were modified with carboxyl and MoS_2 and used for adsorption of Ag(I) ions. The maximum adsorption capacity of the MoS_2/MWCNTs nanohybrid for Ag(I) was 601.97 mg/g. This result was obtained with the activated MWCNTs and no other study related to Ag(I) adsorption of MWCNTs was found in the literature. However, the studies on the adsorption of heavy metals with MWCNTs are still very limited in the literature. In this study, the evaluation of the best adsorption conditions of Ag (I) ions onto MWCNTs has been made using a 2^3 factorial design method. The main objectives of this research are as follows: (1) To analyze the effect of controllable factors in the adsorption process with experimental data; (2) To determine the changing experimental conditions for the adsorption of Ag(I) onto MWCNTs; (3) To create a model equation for adsorption efficiency using the MiniTab 20 Software. Experiments were performed as a function of pH, adsorbent dosage and initial Ag(I) ions concentration of the solution.

MATERIAL AND METHODS

Materials and Reagents

MWCNTs synthesized by the catalytic vapor decomposition method were purchased from Chengdu Organic Chemicals Co. Ltd. The specific properties of MWCNTs are presented in Table 1. The morphology of MWCNTs are given by scanning electron microscopy (SEM, Zeiss Supra 50VP). SEM images (Figure. 1) show that carbon materials have very distinct morphological features. In Figure 1, the presence of characteristic helical MWCNTs were observed. The micrographs of MWCNTs are evidence of the adsorption process of MWCNTs with Ag(I). The pure MWCNTs were given in Figure 1 and have their outer diameter in the between 8 and 15 nm with tubular structures.

Table 1: The specific properties of MWCNTs.

Outer Diameter (nm)	8-15
Length	50 μm
Specific Surface Area	>233 m^2/g
Ash	<1.5 wt%
EC	>10^2 s/cm
Purity	>95 wt %

All chemicals were purchased in analytical purity and were used without further purification. All the plastic and glassware were cleaned by soaking in dilute HNO_3 (1:9 v/v) and were rinsed with distilled water prior to use. To determine the effect of pH, adsorbent dosage and initial metal concentration on Ag(I) ions adsorption efficiency, batch experiments were conducted. A stock solution that consisted of 1000 mg/L of the Ag(I) ions was prepared and diluted according to initial concentrations. The pH of the solutions was adjusted with HNO_3 (Merck) or NaOH (Merck) solutions by using WTW 330 pHmeter with a combined pH electrode. The samples were continuously agitated with a speed of 175 rpm at a room temperature for 1 h.

The maximum adsorption capacity at equilibrium q_e (mg/g), was calculated by (Eq.1):

$$q_e = \frac{(C_o - C_e)V}{m} \quad (1)$$

where C_o is the initial concentration of silver solution (mg/L), C_e is the equilibrium concentration of silver solution (mg/L), V is volume of silver solution (mL), and m is the adsorbent weight (g).

The adsorption percentage of silver was calculated by the difference of initial concentration using the equation (Equation 2) expressed as follows:

$$R = \frac{C_o - C_e}{C_o} x100 \quad (2)$$

where C_o is the initial concentration of silver solution (mg/L), C_e is the equilibrium concentration of silver solution (mg/L), and R is the retention of Ag(I) ions in % of the added amount.

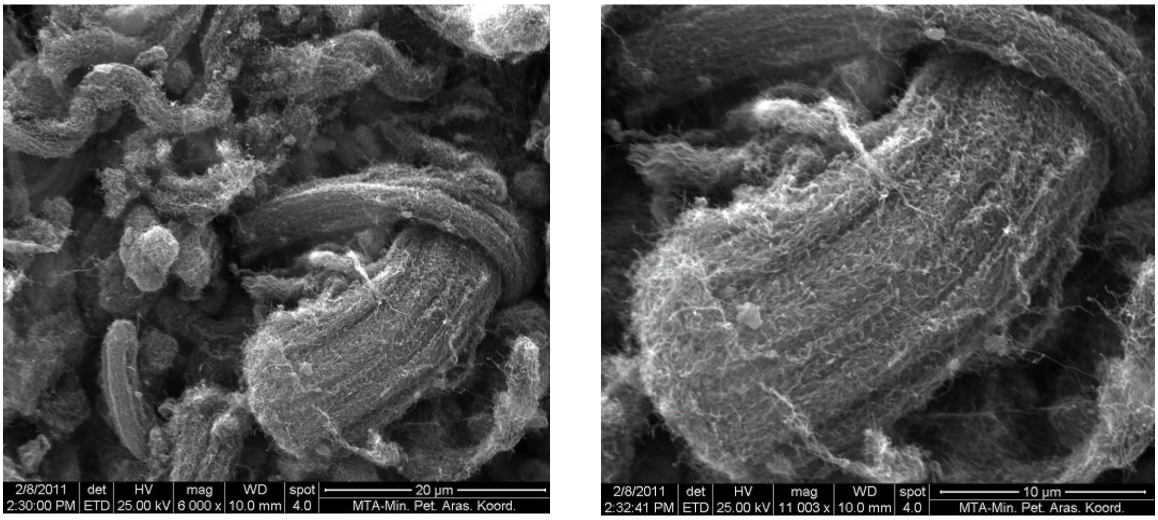

Figure 1: (Left) and (right) Some SEM images of helical multi-walled carbon nanotubes.

Factorial Experimental Design

Experimental factorial design is employed, to reduce, the number of experiment cost, time, to find best overall optimization of the experimental conditions. The design determines that factors have important effects on a response as well as how the effect of one factor varies with the level of the other factors. The number of experimental runs at b levels is b^k, where k is the number of factors and have successfully applied by many researchers. Today, the most widely used kind of experimental design, to estimate main effects as well as interaction effects, is the 2^p factorial design in which each variable is investigated at two levels (21,22).

The 2^3 factorial experiments were applied in two levels high and low. The low and high levels of the factors were listed in the Table 2. These levels of the factors were determined by considering some preliminary experiments. The experiments were carried out two parallel and adsorption efficiencies (%) was determined as average of experiments. The order in which the experiments were made was randomized to avoid systematic errors. The results of experiments data were analyzed with the Minitab 16 software, and the main effects and interactions between factors were determined. The cubic diagram levels for high and low of three factors, pH, adsorbent dosage, and initial concentration, is given in Figure 2.

Table 2: Factors and levels used in the factorial design.

Factor	Coded Symbol	Low Level (-1)	High Level (+1)
pH	*A*	2.0	8.0
Adsorbent Dosage (g/L)	*B*	0.1	1.0
Initial Concentration (mg/L)	*C*	10	100

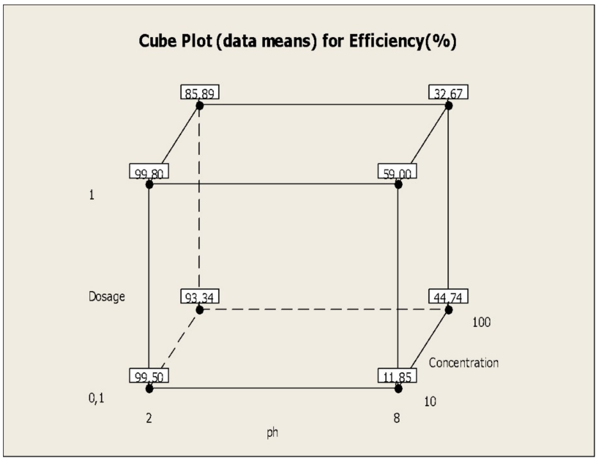

Figure 2: Cubic plots for efficiency (%).

RESULTS AND DISCUSSION

The SEM images of the MWCNTs and Ag(I) ions after adsorption were presented in Figure 3. The factorial design matrix and efficiency calculated in each factorial experiment is shown in Table 3, with the low (−1) and high (+1) levels as specified in Table 2. Figure 2 illustrates the mean of the experimental results for the respective low and high levels of pH, adsorbent dosage, and initial concentration. Factors that influence the adsorbed quantity of Ag(I) ions adsorbed onto MWCNTs were evaluated by using factorial plots: main effect and interaction effect. The SEM images of MWCNTs/Ag(I) given in Figure 3 were taken under acidic conditions (pH 2). After adsorption, it was seen that Ag(I) uptake in macrostructure of MWCNTs. This heterogeneous structure is characteristic and contains plenty of micropores and cavities where adsorption of Ag(I) occurs.

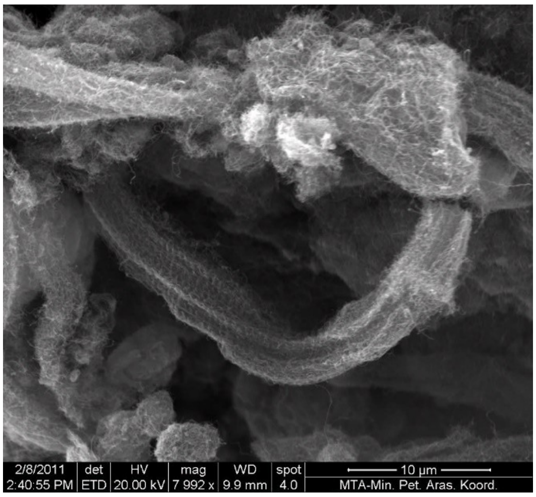

Figure 3: Scanning electron microscope (SEM) images of Multi-walled Carbon Nanotubes/Ag(I).

Table 3: Design matrix and the results of the 2^3 full factorial design.

Run No.	Factor			*Efficiency (%)*		
	A	B	C	Replicate		Average
				I	II	
1	-1	-1	-1	99.50	99.50	99.50
2	+1	-1	-1	11.90	11.80	11.85
3	-1	+1	-1	99.80	99.80	99.80
4	+1	+1	-1	59.60	58.40	59.00
5	-1	-1	+1	93.23	93.45	93.34
6	+1	-1	+1	44.80	44.68	44.74
7	-1	+1	+1	85.90	85.88	85.89
8	+1	+1	+1	32.70	32.64	32.67

The results of main effect and interaction effect were given as graphs in Figure 4 and 5. The main effects of each parameter on the silver adsorption are shown in Figure 4. The main effects plots were generated to represent the results of regression analysis. It shows only the factors that were significant at the 95% confidence interval. The interaction effects of each parameter on the silver adsorption are shown in Figure 5.

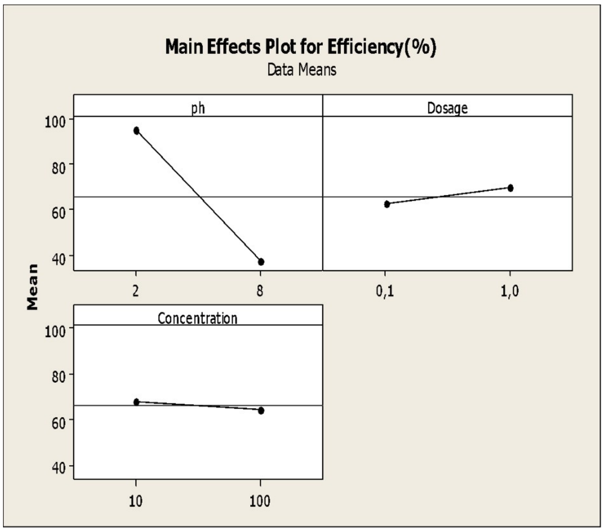

Figure 4: Main effects plot for efficiency.

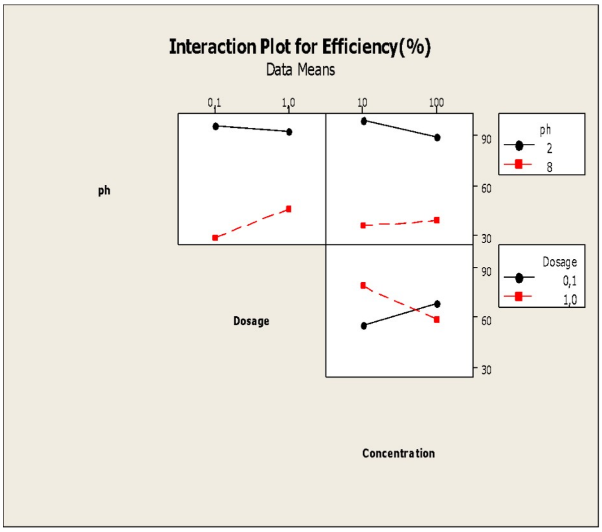

Figure 5: Interaction plot for efficiency.

The graphs of interaction effects explain the interaction effects between experimental parameters. According to the Figure 5, the strongest interaction is between adsorbent dosage and initial concentration. Because the lines are not parallel to each other, there is little interaction between pH and adsorbent dosage, and pH and initial concentration. But these interactions are not strong. Under acidic conditions, the Ag(I) uptake efficiency onto MWCNTs was found high due to favorable electrostatic interactions (23).

Significant levels of ANOVA and P-value were used to check the significance of the effect on % efficiency. The significance of the regression coefficients was determined by applying a Student's t-test. All effects were significant with 95% confidence level. The adequacy of model was evaluated in terms of the analysis of variance (ANOVA) (24). Coefficients of the model, standard deviation of each coefficient were presented for the full 2^3 factorial designs in Table 4. In addition, the model presented an adjusted square correlation coefficient R^2 (adj) of 99.98%, fitting the statistical model quite well. In this way, the Ag(I) ions uptake by MWCNTs could be expressed using the following equation:

R (%) = 65.85 – 28.78 x pH + 3.49 Dosage – 1.69 Concentration + 5.28 pH x Dosage + 3.3 pH x Concentration – 8.37 Dosage x Concentration – 6.43 x pH x Dosage x Concentration

Table 5 shows the sum of squares being used to estimate the factors effect and the *F*-ratios, which are defined as the ratio of respective mean-square-effect to the mean-square-error. The significance of these effects was evaluated using the t-test, and had a significance level of 5%; i.e., with a confidence level of 95%. The *R*-squared statistic indicated that the first-order model explained 99.99% of *R*’s variability to the rejection of null hypothesis, it appears that the main effect of each factor and the interaction effects were statistically significant: P<0.05. The results revealed that the studied factors (A, B and C), their 2-way interaction (AB, AC and BC) and 3-way interaction (ABC) were statistically significant to *R (%)*.

It was found that experimental design approach can predict equilibrium adsorption behavior for MWCNT and Ag(I). The adsorption capacity determined from the 2^3 factorial design onto MWCNT was compared with uptake different heavy metal ions and results was given in the Table 6. The performance of MWCNTs for the removal of miscellaneous heavy metal has been demonstrated, and varying values of the adsorption efficiency (%) have been reported in the literature. In this work, Ag(I) uptake by MWCNT was the greatest with a maximum value of 0.998 mg/g, while maximum Ag(I) uptake capacity of MoS_2/MWCNTs was 601.97 mg/g.

Table 4: Estimated Effects and Coefficients.

Estimated Effects and Coefficients for Efficiency(%) (coded units)

Term	Effect	Coef	SE	Coef	T	P
Constant	65.85	0.07697	855.47	0.000		
pH	-57.57	-28.78	0.07697	-373.94	0.000	
Dosage	6.98	3.49	0.07697	45.36	0.000	
Concentration	-3.38	-1.69	0.07697	-21.94	0.000	
pH*Dosage	10.56	5.28	0.07697	68.58	0.000	
pH*Concentration	6.66	3.33	0.07697	43.25	0.000	
Dosage*Concentration	-16.74	-8.37	0.07697	-108.75	0.000	
pH*Dosage*Concentration	-12.87	-6.43	0.07697	-83.58	0.000	

S = 0,307896 PRESS = 3,0336

R-Sq = 100,00% R-Sq(pred) = 99,98% R-Sq(adj) = 99,99%

Table 5: Analysis of Variance.

Analysis of Variance for Efficiency(%) (coded units)

Source	DF	Seq	SS	Adj	SS	Adj	MS	F	P
Main	Effects	3	13496.7	13496.7	4498.9	47456.82	0.000		
	pH	1	13256.1	13256.1	13256.1	139831.94	0.000		
	Dosage	1	195.0	195.0	195.0	2057.19	0.000		
	Concentration	1	45.6	45.6	45.6	481.33	0.000		
2-Way	Interactions	3	1744.4	1744.4	581.5	6133.54	0.000		
	pH*Dosage	1	445.8	445.8	445.8	4702.99	0.000		
	pH*Concentration	1	177.3	177.3	177.3	1870.14	0.000		
	Dosage*Concentration	1	1121.2	1121.2	1121.2	11827.48	0.000		
3-Way	Interactions	1	662.3	662.3	662.3	6986.18	0.000		
	pH*Dosage*Concentration	1	662.3	662.3	662.3	6986.18	0.000		
Residual	Error	8	0.8	0.8	0.1				
	Pure	Error	8	0.8	0.8	0.1			
Total	15	15904.1							

Table 6: Comparison of adsorption efficiency (%) for the adsorption of heavy metal ions by MWCNTs.

Adsorbent	**Adsorbates**	**Adsorptivity (%)**	**References**
MWCNTs	Ag^{1+}	99.80	Recent study
MWCNTs	Cd^{2+},	10.80	(25)
MWCNTs	Mn^{2+}	4.80	(26)
MWCNTs	Ni^{2+}	49.00	(27)
MWCNTs	Pb^{2+}	97.06	(28)
MWCNTs	Co^{2+}	2.77	(29)

CONCLUSION

The results of present study clearly show that MWCNTs is effective in adsorption of Ag(I) ions. Adsorption efficiency was achieved up to 99.50 and 11.80 (%) Ag(I) ions from aqueous solutions in 1 h contact with initial concentration 10 and 100 mg/L, respectively. The adsorption of Ag(I) onto MWCNTs was characterized by SEM analysis. The factorial design method was employed for estimating the optimum values of pH, adsorbent dose, and initial metal ions concentration. The statistical model and ANOVA results determined that pH was the most efficient factor in the adsorption process. Then pH-adsorbent dosage interaction and pH-initial concentration interaction, were also found highly significant. When the results were examined, the highest efficiency was obtained at pH 2. In the first stage of research, it have been defined the most significant parameters, that influence adsorption process and was possible to make a first optimization of the process using a screening design of the adsorption process. MWCNTs has a potential to be used as an effective adsorbent for uptake of Ag(I) ions from aqueous media. The MWCNTs/Ag(I) binary structure has been supported by experimental results, which has the potential to be used in many areas. One of the important results is that the MWCNTs/Ag couple obtained by adsorption can be suggested as a catalyst for different reactions. In addition, the high capacity of MWCNTs to hold Ag(I) ions gives hope that they can be used in the industry.

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