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Short Communication Electrochemical Treatment of Landfill Leachate to Remove Chromium (VI) using Ni₃N and NiO NPs anodes

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This study focused on electrocoagulation treatment of leachate in solid waste landfills to remove Cr(VI) on nickel nitride (Ni₃N) and nickel oxide (NiO) nanoparticles (NPs) anodes. NiO NPs and Ni₃N anodes were synthesized by electrodeposition and hydrothermal methods, respectively. The structural characterization using SEM and XRD showed the smooth surface Ni₃N was grown in hexagonal crystal structure and morphology NiO NPs contained the high porous and well-distributed nanoparticle in facecentered cubic phase. The Zeta potential studies illustrated the higher zeta potential of NiO NPs than Ni₃N. Electrocoagulation studies indicated that complete removal of 1000 mg/l of potassium dichromate pH 5.1 as model solution at a current density of 0.2 A was obtained after 85 and 50 minutes treatment on Ni₃N and NiO NPs anodes, respectively. The 70% treatments were achieved at first 25 and first 15 minutes of the process for Ni₃N and NiO NPs anodes, respectively. In the same electrocoagulation condition, the complete removal of 982 mg/l of Cr(VI) in prepared leachate as real sample was obtained after 110 and 65 minutes on Ni₃N and NiO NPs anodes, respectively. The comparison of the obtained results in this study with other reported removal Cr(VI) by electrocoagulation technique showed that the high removal rate was obtained on NiO NPs anode due to stronger electrostatic interaction with Cr(VI) due to higher porosity and higher effective surface area, as a result, it supplies more active sites of Ni³⁺ on surface.

Keywords: Electrocoagulation treatment; Leachate; Solid waste landfill; Cr(VI); NiO nanoparticles; Ni₃N anode

1. INTRODUCTION

Hexavalent chromium (Cr(VI)) is the most applicant chemical compound for synthesizing chromium-based materials in industries such as chromium oxide, chromium trioxide, chromic acid,

sodium dichromate, etc [1]. The anticorrosive and chemical properties of Cr(VI) made its compounds as the beneficial materials in textile, dye and pigment, paint, inks, wood preservation, coating, metallurgy, plastics, leather tanning, insecticide and paper industrials [2]. Thus, more than 20 million tons of Cr(VI) have been produced in the world which make it a great concern in waste management [3]. Improper management in the use, collection, storage and burial of toxic industrial waste has caused these compounds to contaminate the soil, air and water sources [4]. Therefore, Cr(VI) compounds have been found in drinking water and public water systems [5, 6].

All Cr(VI) compounds are toxic, corrosive, and carcinogenic. Many studies have been shown the exposure Cr(VI) compounds lead to increased incidence of asthma, damage to the nasal epithelia and skin, mutagenic damage, and lung cancer [7]. Problematic exposure has been frequently reported for workers in chromate-related industries such as chromate products factories and metallurgies.

Therefore, many researchers have been conducted for remediate Cr(VI) compounds in soil, ground water and drinking water through reduction of toxicity, removal technologies and containment technologies which contained reverse osmosis, absorption, ion exchange, oxidation, flocculation, flotation, membrane filtration, electrocoagulation and electrochemical methods [8]. Among them, electrocoagulation as an effective treatment technique showed the great removal efficiency for removing Cr(VI) [9-12]. Moreover, this technique as an easily handled process shows the low sludge production and low maintenance costs. Many studies have been performed using Al and Fe electrodes in electrocoagulation treatment due to low cost and frequently. The number of studies on different materials as electrodes in electrocoagulation treatment has been limited [13-16]. Therefore, this study was conducted on electrocoagulation treatment of leachate in solid waste landfills to remove Cr(VI) on Ni₃N and NiO NPs anodes.

2. EXPERIMENT

All analytical-grade chemicals with purity >99% were purchased from Xilong Scientific Co., Ltd., China. All solutions were prepared with deionized (DI) water. For synthesis the NiO NPs [17], the aquatic mixture of 1M nickel chloride hexahydrate and 1M potassium chloride were prepared in equal volume ratio as electrodeposition electrolyte under magnet stirring for 1 hour at room temperature. Next, the 0.2 wt% boric acid and 0.2 wt% sodium dodecyl sulfate were added to prepare electrolyte. The electrochemical cell contained a Pt plate as the anode and high-purity Ni foam as cathode and the substrate for NiO NPs deposition. Electrodeposition was carried out on potentiostat (CS350, Wuhan Corrtest Instruments Corp., Ltd., China) at a current density of 1 A/cm² for 25 minutes. As-prepared Ni NPs is an active metal layer that reacts with oxygen in air and creates a tenacious surface oxide layer.

 Ni_3N was prepared by a hydrothermal method using 0.5M Ni(NO₃)₂.6H₂O (97%,Sigma-Aldrich) and 0.5M urea as nickel and nitrogen precursors, respectively. The 20ml of ethylene glycol was ultrasonically added to the mixture of precursors. The mixture was transferred to Teflon-lined stainless-steel autoclave and heated at 130°C for 5 hours. Then, the cooled product rinsed with DI water and ethanol, respectively. The Ni foam was immersed in the resulting suspension for 24 hours. After then, To obtain Ni(OH)₂, the immersed Ni foam in suspension was transferred to the oven and dried at 45°C

for 7 hours. To achieve the Ni_3N , the obtained $Ni(OH)_2$ was placed in a ceramic boat and heated at $300^{\circ}C$ for 2 hours under an NH_3 atmosphere.

The SLVs were recorded using potentiostat/galvanostat (Autolab, Netherlands) in a conventional three electrode cell which contained the Ni₃N and NiO NPs as working electrode, Pt plate as counter and Ag/AgCl as the reference electrode. Scanning electron microscopy (SEM) and X-ray diffractometer (XRD, Bruker D2 Phaser, Beerlika, USA) were used to study the morphology and crystalline structure of the samples, respectively. Zeta potential of samples was measured using electrophoretic light scattering (Zeta sizer nano series, Malvern Instruments Ltd., Malvern, UK).

The leachate samples were collected from Xi'an city landfill, which is located in Shaanxi Province, China. The samples were collected into glass bottles and transferred in a cold store room at 4°C. Prior to any experiment, the collected samples were taken out from the cold store room and left to reach the room temperature. Then, the leachate was pumped into the anaerobic digester reactor to treat biodegradable organic compounds according to Kheradmand et al. [18]. After then, landfill leachate was treated by membrane bioreactor and nanofiltration to filter bio-effluent and rejection of ions and molecules with molecular weight greater than 200 g/mol according to Mohammad et al. [19]. The reverse osmosis process was performed to rejection of recalcitrant pollutants and inorganic salts according to Linde et al. [20]. The physical parameters of leachate were measured in situ at the sampling point by using a multiparameter probe (HI 8424, HANNA Instruments, Sarmeola di Rubano PD, Italy). Atomic absorption spectroscopy (Shelton, CT, USA) was applied to analysis of Heavy metals. NH₃-N level was measured using the spectrophotometer (DR2500, Hach-Lange, Germany). BOD and COD levels in leachate were determined by using APHA Standard method [21]. The properties of leachate samples is shown in Table 1. 1g/l NaCl solution was utilized as the supporting electrolyte during electrocoagulation treatment. As observed from Table 1, the Cr(VI) concentration in landfill leachate is 982 mg/l. accordingly, electrocoagulation experiments with model solution of potassium dichromate were prepared in concentration of 1000 mg/l.

Prior to the experiments, electrocoagulation reactors were washed with distilled water. 100 ml of leachate sample was transferred into the 150ml electrocoagulation reactor which contained an electrolytic cell with graphite as cathode and Ni₃N and NiO NPs electrode as anodes with dimensions of $15 \times 4 \times 0.2$ cm³. The electrodes were immersed in the electrolytic cell solution with inter-electrode distance of 2 cm. The power of electrocoagulation process was performed at adjusted voltage of 1.2V which was provided from DC power supply (Topward DC 3306D, maximum output 30 V and 6 A) under galvanostatic mode for 60 minutes. The pH of solutions was adjusted by HCl and NaOH.

Property	value
BOD	104.9 mg l ⁻¹
COD	110.7 mg l ⁻¹
Cr(VI)	982 mg/l
pH	5.1
conductivity	17.3 mS/cm

Table 1. Characteristics of landfill concentrates

3. RESULTS AND DISCUSSION

3.1 Structural and electrochemical characterization of Ni₃N and NiO NPs

The SEM images of synthesized Ni₃N and NiO NPs are displayed in Figure 1. The SEM image of Ni₃N shows the smooth surface. SEM image of NiO NPs shows the high porosity and clearly well-distributed nanoparticle with an average diameter of 90 nm in surface. The high porosity and presence of sharp tips of nanoparticles on NiO NPs toward the Ni₃N reveal that NiO NPs provide a large number of adsorption sites for hydroxide and Cr(VI).



Figure 1. SEM images of synthesized (a) Ni₃N and (b) NiO NPs.



Figure 2. XRD patterns of the powders of synthesized Ni₃N and NiO NPs.

The crystalline structure of the synthesized Ni_3N and NiO NPs are shown in XRD patterns of Figure 2. XRD pattern of NiO NPs shows three diffraction peaks at 37.11°, 43.44° and 63.75°, which illustrated to present of the (111), (002) and (022) planes of face-centered cubic phase NiO, respectively (JCPDS Card No. 47-1049). As observed from XRD pattern of Ni_3N , there are diffraction peaks of

hexagonal structure of Ni₃N at 39.22°, 42.15°, 44.69° and 58.77°, which related to the (110), (002), (111) and (112) planes, respectively (JCPDS Card No. 10-0280).

Results of Zeta potential measurements using electrophoretic light scattering is shown in Figure 3a. As observed, the peak of zeta potential of NiO NPs and Ni₃N are recorded at 42.03 mV and 26.32 mV in pH 5.1, respectively, which indicated that higher zeta potential is belonging to NiO NPs which corresponds to stronger electrostatic interaction with Cr(VI) because of higher porosity and higher effective surface area, as the consequence it supplies more active sites of Ni³⁺ on surface [22, 23]. Therefore, the NiO NPs can electrostatically interact with anions that can be a useful electrode for removal of Cr(VI) from waste water through electrocoagulation treatment. The pore structure of NiO NPs can greatly accelerate the rate of permeation and soak of aqueous solution into electrode surfaces [24]. Furthermore, more active sites on the surface can improve the adsorption efficiency [25].



Figure 3. (a) Results of zeta potential, and (b) the recorded LSVs in 0.1 M NaCl aqueous solution at scan rate of 10 mV/s for NiO NPs and Ni₃N at pH 5.1

The removal of Cr(VI) by electrocoagulation technique was performed on adsorption, and electrochemical reduction process at anode which conducted on following reactions [26]:

$Ni + 2OH^- \rightarrow \beta - Ni(OH)_2 + 2e^-$				
β -Ni(OH) ₂ + OH ⁻ $\rightarrow \beta$ -NiOOH + H ₂ O + e ⁻	(2)			
Hydrogen evolution: $2H_2O + 2e \rightarrow H_2 + 2OH$	(3)			
Cr(VI) reduction: $HCrO_4^- + 7H^+ + 3e^- \rightarrow Cr^{3+} + 4H_2O$	(4)			

For electrochemical characterization of NiO NPs and Ni₃N samples, the recorded LSVs in 0.1 M NaCl pH 5.1 at scan rate of 10 mV/s are shown in Figure 3b which indicated the better electrocatalytic performances NiO NPs due to unfilled d orbitals, positive charges of Ni²⁺, higher electrical conductivity Ni³⁺ and its stronger electrostatic interaction with OH⁻[27, 28]. These can facilitate the adsorption,

complexation, and electrochemical reduction process. Moreover, nanoporous structured electrodes provide fast charge transfer processes [9, 29-31].

The adsorption properties of NiO NPs and Ni₃N anodes were investigated in batch adsorption to remove Cr(VI). Figure 4a exhibits the removal capacity (q₁) of Cr(VI) onto NiO NPs and Ni₃N adsorbents with 30 mg/l as initial concentration of potassium dichromate solution. As observed, the adsorption equilibrium of Cr(VI) on NiO NPs and Ni₃N anodes are obtained at 30 minutes. The stronger binding sites on NiO NPs anode can be rapidly occupied by the adsorbates [32]. After 30 minutes, The binding strength decreases gradually with increasing the occupied sites [33]. The aqueous Cr ions would generate monolayer surface complexes on anode to occupy the available reactive sites [34]. Figure 4b shows the fit of these experimental data with pseudo-second-order kinetic model which reveals that the obtained rate constant values (k) are 0.0163 and 0.0133 g/mg.min, equilibrium adsorption capacity (q_e) values are 20.99 and 23.28 mg/g, and the initial adsorption rates values (kq_e²) are 7.18 and 7.21 mg/g.min for NiO NPs and Ni₃N, respectively. Thus, NiO NPs show the better Cr(VI) adsorption performance. Studies have been shown that the most important operative parameters of the nanomaterials for treatment of heavy metals are adsorption capacity and removal efficiency due to higher specific surface area and strong binding affinity and high porosity of nanostructured anode [33-35].



Figure 4. (a) The removal capacity (q_t) of Cr(VI) onto NiO NPs and Ni₃N adsorbents with 30 mg/l as initial concentration of potassium dichromate solution pH 5.1 and (b) linear plots of fit of experimental data with pseudo-second-order kinetic model.

3.2 Electrocoagulation study of Ni₃N and NiO NPs anodes for remove Cr(VI) of prepared leachate

Figure 5a demonstrates the removal efficiencies of 1000 mg/l of potassium dichromate pH 5.1 as model solution vs. electrocoagulation time at a current density of 0.2 A. it is shown that complete removal are obtained after 85 and 50 minutes treatment on Ni₃N and NiO NPs anodes, respectively. The rates of Cr(VI) removal are different for both of anodes. 70% treatments are observed at first 25 minutes and first 15 minutes for Ni₃N and NiO NPs anodes, respectively, and the final 30% treatments are obtained at lower rates which associated with the saturation the active site on both of electrodes. These

trends for removal rates are observed for 982 mg/l of Cr(VI) in prepared leachate as a real sample in Figure 5b. As seen, Ni₃N anode shows 100% removal efficiencies after 110 minutes electrocoagulation treatment in pH 5.1. In addition, NiO NPs electrode displays the higher removal rate than other electrodes with 100% removal efficiency after 65 minutes electrocoagulation treatment in pH 5.1, that it can be related to the presence of NiO and its enhanced electrostatic interaction with OH⁻. In addition, it facilitates the subsequent reactions (1 to 4) and rapid reduction of suspended particles of Cr(VI) [36]. The lower removal rate for real samples is related to presence of other materials in prepared sample of leachate in solid waste landfills. Table 2 shows the comparison the obtained results of in this study with other reported removal Cr(VI) by electrocoagulation technique. As observed, the fast removal rate is obtained for treatment of 1000 mg/l of Cr(VI) using NiO NPs electrode due to nanostructured morphology and high isoelectric point of NiO electrode that enhance its absorbance ability to Cr groups [37].



Figure 5. Removal efficiency vs. electrocoagulation time at a current density of 0.2 A in pH 5.1 onto NiO NPs and Ni₃N electrodes for treatment of (a) 1000 mg/l of potassium dichromate as model solution and (b) 982 mg/l of Cr(VI) in prepared leachate sample.

Table 2. Comparison the obtained results of removal Cr(VI) by electrocoagulation technique.

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Anode	cathode	Initial	Current	pН	Treatment	Removal	Ref.
		concentration	(A)		time	efficiency	
		(mg/l)			(minute)	(%)	
Fe	Fe	10	0.05	-	45	100	[38]
Al	graphite	188.8	1.5	7	10	32.20	[39]
steel	steel	1000	1	6	60	100	[40]
Fe	Fe	750	0.013	7	25	100	[41]
3D hierarchically porous	Pt	20	0.01	4	20	99.5	[15]
NiO/Ni foam							
NiO NPs	graphite	1000	0.2	5.1	50	100	This
							work
Ni ₃ N	graphite	1000	0.2	5.1	85	100	This
							work



Figure 6. (a) pH effect (current density of 0.2 A) and (b) current density effect (pH 4) on the removal efficiency of Cr(VI) onto NiO NPs and Ni₃N electrodes with 1000 mg/l as initial concentration of Cr(VI).

pH has a significant effect on the electrocoagulation process performance. Depending on the electrode material and initial pH of electrolyte in the electrocoagulation cell, the pH of the electrolyte changes during the treatment [42]. In order to determine the effect of pH on chromium removal, measurements were performed on solutions with adjusted pH values of 2, 4, 6, 8, 10, and 12 during the electrocoagulation process. The results in Figure 6a represent that acidic mediums have the shortest electrocoagulation after 60 minutes for removal of chromium from solution. For pH values of 2, 4, 6, 8, 10, and 12, the removal efficiencies of chromium on NiO NPs are obtained 100, 97.87, 95.03, 88.02, 77.91 and 71.02, respectively, after 60 minutes electrocoagulation treatment. For Ni₃N, the removal efficiencies of chromium are achieved 85.54, 82.70, 79.86, 68.33, 62.57 and 63.02 for 2, 4, 6, 8, 10, and 12, respectively, after 60 minutes electrocoagulation treatment. Accordingly, pH 4 can be considered as optimum pH for chromium removal because of its close value to pH of leachate and shorter electrocoagulation time that it associated the presence of high hydrogen ion concentration in acidic medium and reduction the hexavalent chromium ions to trivalent chromium ions [43].

Another important parameter in time and energy consumption of electrocoagulation treatment is current density. Figure 6b depicts Cr(VI) removal efficiency of prepared solutions pH 4 on the both of electrodes at current densities from 0.05 A to 1.5 A. As observed, with increasing the value of current density, the removal efficiency is increased. The complete removal is observed after 60 minutes at a current density of 0.5 A on NiO NPs electrode. For current densities from 0.05 A to 0.2 A, the removal efficiency is sharp with increasing the value of current density but for current densities more than 0.2 A, the removal efficiency is slightly increased with increasing the value of current density which indicated to the minimum current density with best removal rate is achieved at 0.2 A.

4. CONCLUSION

This study presented the electrochemical treatment of leachate in solid waste landfills to effectively remove chromium on Ni₃N and NiO NPs anodes through electrocoagulation technique.

NiONPs and Ni_3N were prepared by electrodeposition and hydrothermal method, respectively. The structural studies showed the smooth surface of Ni₃N was synthesized in hexagonal structure and the surface morphology of NiO NPs displayed the high porous and well-distributed nanoparticle in facecentered cubic phase. The results of Zeta potential measurements indicated that higher zeta potential was belonging to NiO NPs. Electrocoagulation studies showed that complete removal of 1000 mg/l of potassium dichromate pH 5.1 as model solution at a current density of 0.2 A was obtained after 85 and 50 minutes treatment on Ni₃N and NiO NPs anodes, respectively. The 70% treatments were observed at first 25 minutes and first 15 minutes of the process for Ni₃N and NiO NPs anodes, respectively. In the same electrocoagulation condition, the complete removal of 982 mg/l of Cr(VI) in prepared leachate as a real sample was obtained after 110 and 65 minutes on Ni₃N and NiO NPs anodes, respectively. The lower removal rate for real samples can be related to presence of other materials in prepared samples of leachate in solid waste landfills. The comparison the obtained results of in this study with other reported removal Cr(VI) by electrocoagulation technique revealed the fast removal rate for treatment of 1000 mg/l of Cr(VI) using NiO NPs electrode due to stronger electrostatic interaction with Cr(VI) because of higher porosity and higher effective surface area, as the consequence it supplies more active sites of Ni³⁺ on surface. Study of the pH and current density effects showed that acidic medium and 0.2 A had the high treatment rate of Cr(VI).

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