

AYUNI YUSTIRA¹
HAFIFUDDIN ARIF¹
ZAHEDI ZAHEDI²
FACHRUR RAZI³

¹Department of Chemical Engineering, Faculty of Engineering Universitas Sumatera Utara Medan, Indonesia

²Department of Mathematics, Faculty of Mathematics and Natural Sciences Universitas Sumatera Utara Medan, Indonesia

³Department of Civil Engineering, Faculty of Engineering Universitas Al-Washliyah Medan, Indonesia

SCIENTIFIC PAPER

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INFLUENCE OF ACTIVATORS ON SPENT COFFEE GROUNDS AS BIOSORBENT FOR CHROMIUM

Article Highlights

- Characteristic Adsorbent of Spent Coffee Ground (SCG)
- Determination of the best activator for iodine absorption
- Determination of pH of Cr solution
- Determination of contact time against the percentage of removal
- Determination of concentration to the percentage of Cr(VI) removal

Abstract

Global production of Cr(VI) can reach 44 million metric tons annually, of which 49% is released into the environment. Developments in the industry are to blame for this. There are numerous ways to defend against the damaging impact that heavy metals have on the environment. The presence of Cr(VI) causes environmental problems because it can endanger the physical and ecosystem balance of aquatic fauna in river flows. Exposure to Cr(VI) causes health problems such as liver, lung, and kidney damage. Adsorption is a straightforward, affordable, and user-friendly technique. The adsorbent utilized is derived from biomass waste, specifically spent coffee ground (SCG), but its potential as an adsorbent still needs to be developed. The purpose of this work was to examine the impact of activators on activated SCG and its capacity to adsorb Cr(VI) compounds. Nitric and bromide acid were used to activate 25 g of SCG for 1, 2, and 3 hours. Adsorbent-to-activator ratios were 1:4, 1:5, and 1:6. Cr(VI) solution was started at a concentration of 100 mg/L, and the adsorption process was stirred at 300 rpm. In the adsorption process, a ratio of 1:6 was best, along with an HNO₃ solution (pH 4) as the best activator, a contact time of 120 minutes, and an initial Cr(VI) concentration of 20 mg/L. OH is the major functional group of both adsorbents.

Keywords: Cr(VI) removal; nitric acid activator; bromide acid activator; spent coffee grounds bio-waste.

Wastewater containing heavy metals, such as Cr(VI) ions, which are a source of environmental contamination, is produced by mining, electrical equipment, leather tanning, and electroplating companies, or so-called anthropogenic activities [1]. Because heavy metals cannot break down

they will be hazardous if they contaminate water. The overall annual output of chromium is 44 million metric tons, of which 49% is generated in Finland, Brazil, Zimbabwe, Kazakhstan, India, South Africa, Turkey, and Zimbabwe. Additionally, more than 170.000 tons of Cr(VI) are released into the environment annually [2,3].

The development of industrialization, urbanization, and globalization causes water pollution. Polluted water contains Cr(VI) ions. Hexavalent chromium is toxic and can be easily found in river flows. The presence of Cr(VI) ions causes environmental problems because they can endanger the physical and ecosystem balance of aquatic fauna in river flows [4,5]. Long-term exposure to high concentrations of Cr(VI) triggers liver, lung, and kidney damage. Meanwhile,

Correspondence: A. Yustira, Department of Chemical Engineering, Faculty of Engineering Universitas Sumatera Utara Medan 20155, Indonesia.
E-mail: ayuni@usu.ac.id
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short-term exposure to Cr(VI) causes skin irritation. The maximum tolerable concentration of Cr(VI) set by the US Protection Agency (EPA) is 100 mg/L [6,7].

Certain heavy metal species are present in soil, water, and air streams. These species include Pb, Cu, Cd, Hg, As, Ni, and Cr. This is a result of rapid industrial development and poses a risk to human health as well as the environment at the moment [8,9]. The adsorption process is one of many strategies that can be employed to guard against the damaging effects of heavy metals on the environment. In theory, this strategy is straightforward, affordable, and simple to implement [10].

Research is currently focused on using biomass waste, such as potato skins [11], *Lathyrus sativus* husk [12], corncob [13], and garlic straw [14], to create a bio-adsorbent. Though coffee is the second most significant commodity in the world and the largest agricultural product after petroleum [15], the use of spent coffee ground (SCG), one of the biomass wastes, as an adsorbent is still neglected [16].

SCG is the residue left over after brewing coffee grinds [17]. If not properly treated, SCG frequently ends up as waste that pollutes the environment. Compared to the process, which requires high working temperatures, processing SCG as an adsorbent chemically with an activator has a number of advantages, including direct processing without treatment, operating at ambient temperature, and using less energy [18].

The selectivity and adsorption affinity of SCG are lower, and regeneration is inefficient due to the complexity of the polymers of SCG. However, several modification methods, such as physical, mechanical, and chemical, make SCG more efficient in the adsorption process. Therefore, the primary objective of this study was to improve the efficacy of modified SCG for Cr(VI) adsorption by altering the type of activator, adjusting the activator ratio, and varying the activation time. SCG was activated with solutions of nitric and bromide acids by opening the pores of SCG.

MATERIAL AND METHODS

Preparation and characterization of SCG

A total of 2 kg of SCG was obtained from Cafetaria Jl. Karya Waisata, Medan Johor, North Sumatra. The SCG was first washed with hot water (preheated to 100 °C) and then rinsed with clean water. Next, the SCG was dried in an oven at 55 °C for 24 hours. To make 1.000 ml of a synthetic Cr(VI) solution, 0.283 g of potassium dichromate ($K_2Cr_2O_7$) (Pudak. P.a $\geq 99.8\%$) was dissolved in water. Nitric (65%, Emsure) and

bromic (48%, Chemie) acids were used as activators for adsorbent activation using the batch technique.

The purity of all the substances utilized was analytical. Fourier transform infrared (FTIR) was used to identify functional groups of the adsorbent material. FTIR wave readings were carried out in the middle spectrum region (mid-infrared) in the wavelength range of 400–4000 cm^{-1} . The results of the FTIR test were in the form of a graph between the wavelength (λ) on the x-axis and the amount of light reflected or the percentage (%) transmittance on the y-axis. FTIR test results were analyzed by looking at specific peaks at certain wavelengths. Each specific peak indicates a functional type in the adsorbent compound. Scanning electron microscope (SEM) analysis was carried out to determine the surface structure and particle size of each adsorbent. The SEM tool used was JSM-6510 LA, 20 kV 57 PA. The iodine number was carried out to determine the ability of each adsorbent activated by nitric or bromide acids to adsorb Cr(VI), where KI solution was used as an iodine solvent. To determine the iodine concentration, the iodometric titration method was used. The iodine number was measured for both the pre and post-activation states of SCG.

Activation procedure

Distilled water was used three times to get rid of contaminants that stuck to the SCG collected from the café. Next, the SCG is dried for 24 hours at a steady weight in an oven (Mettler, UF110) set to 55 °C. Following the process of drying, The SCG is activated with an activator of nitric and bromide acids (0,1 N), activation time of 1, 2, and 3 hours, and an activator to the adsorbent ratio of 1:4, 1:5, and 1:6. After that, the treated adsorbent was dried for two hours at 110 °C using Whatman No. 40 filter paper to remove it from the acid solution and wash it with distilled water until the pH was neutral [19].

Adsorption experiment

The adsorbent was activated using inorganic acids, and the adsorption test was conducted at room temperature using the batch method. In a 250 mL glass beaker, up to 25 g of adsorbent was combined with a synthetic Cr(VI) solution. A hotplate magnetic stirrer 79 min^{-1} was used to stir the mixture under the following adsorption working parameters: 300 rpm stirring, pH 1–5, and 60–140 minutes of contact time. After filtering the mixture, an atomic adsorption spectrometer (AAS-7000) was used to measure the concentration of the Cr(VI) synthesis solution before and after adsorption. The determination of Cr(VI) removal was calculated using the following equation [17]:

$$\text{Removal}(\%) = \frac{C_i - C_e}{C_i} \cdot 100 \quad (1)$$

where C_i is the initial concentration of the solution Cr(VI) (mg/L) and C_e is the equilibrium concentration of the solution Cr(VI) (mg/L).

RESULTS AND DISCUSSION

Adsorbent characteristics

Pore structure analysis using scanning electron microscope (SEM)

Figure 1 shows the findings of the SEM analysis for the SCG activated by nitric and bromide acids. They indicate a particle shape change, with the surface having large holes and a uniformly rough texture. It can be believed that the activators like strong acids can facilitate the release of contaminants from the SCG material and the dehydration process in the activation process, hence enabling the formation of larger holes.

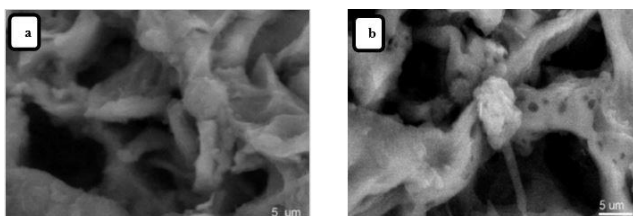


Figure 1. SEM micrographs of the surface morphology of SCG activated with (a) nitric acid, and (b) bromide acid.

Surface functional group analysis of SCG by FTIR

FTIR is employed in the absorption process to identify the adsorbent's functional groups that can interact with the adsorbate [20].

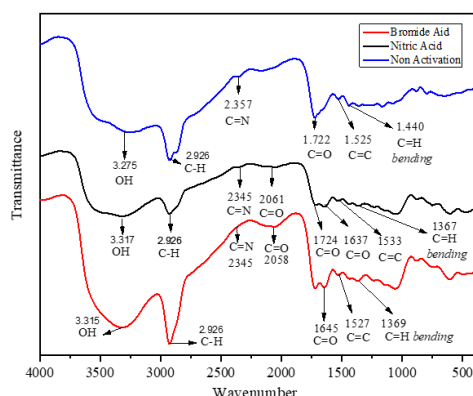


Figure 2. FTIR spectra of activated and non-activated SCG.

FTIR spectra of SCG activated by bromide or nitric acid are shown in Figure 2. The results of the infrared (IR) analysis produce the broadest absorption peaks with a spectrum value of 3316 cm^{-1} for activators

bromide and nitric acids as big 3318 cm^{-1} as well as indicated by the presence of compounds O-H (Hydrogen bonds, alcohols, and phenols). The presence of compound elements C-H (alkanes) for activator bromide and nitric acids identified at the adsorption peak $2926,01 \text{ cm}^{-1}$, adsorption peak 2345 cm^{-1} identified compounds C=N that is a triple bond. In the SCG activation process, bonds C=O were identified at the absorption peaks 2058 cm^{-1} and 1645 cm^{-1} for the bromide acid-activated SCG and 2062 cm^{-1} , 1724 cm^{-1} , and 1636 cm^{-1} for the nitric acid-activated SCG. The bond strain C=O can be associated with amino acids and derivatives of xanthene in the form of caffeine and carboxylic acids from pectin, esters, and aldehydes, which in the adsorption process can participate [18].

The existence of aromatic ring compounds (C=C) was discovered at the absorption peak 1369 cm^{-1} for bromide acid and 1368 cm^{-1} for nitric acid, which indicated the C-H bending. The peaks of bromide acid and nitric acids are at 2058 cm^{-1} and 2062 cm^{-1} , respectively. The molecules C-O (carboxyl) detected by activator Bromide acid have an absorption peak of 1056.99 cm^{-1} .

The functional groups of SCG were activated using inorganic acids, so the FTIR analysis was utilized to identify the compound elements that contained C=N (triple bond), OH, C-H (alkanes), C=O, aromatic ring (C=C), C-H bending, and C-O (carboxyl). In addition, with a different and increasing number of OH intensities in the activated adsorbent, the OH group is an ideal active site, and at the same time, indicates that the resulting adsorbent is negatively charged. The research results obtained agree with Imessaoudene *et al.* [21] who used SCG in adsorbing cobalt.

The activator's ability to adsorb iodine

The iodimetric titration method is used to determine the adsorption capacity and quality of adsorbent in adsorbing the adsorbate, where the adsorbed amount of iodine serves as an indicator. SCG can adsorb iodine because it contains OH compounds. When SCG is activated using nitric or bromide acid, the intensity of OH compounds in SCG continues to increase, whereas the OH group becomes an ideal active site for the adsorption of iodine by SCG. Figure 3 shows that the adsorption value of iodine differs for two adsorbents due to differences in the ability of solutions or activators to facilitate the pyrolytic decomposition process for the formation of structures related to pores and the surface area of the adsorbent. The adsorption capacity of iodine can be greatly impacted by hydration and pyrolytic breakdown that occur in the adsorbent material [22]. The surface area of the adsorbent is also related to the ability of each activator to increase the

porosity characteristics of the adsorbent so that the iodine adsorption capacity increases significantly [23].

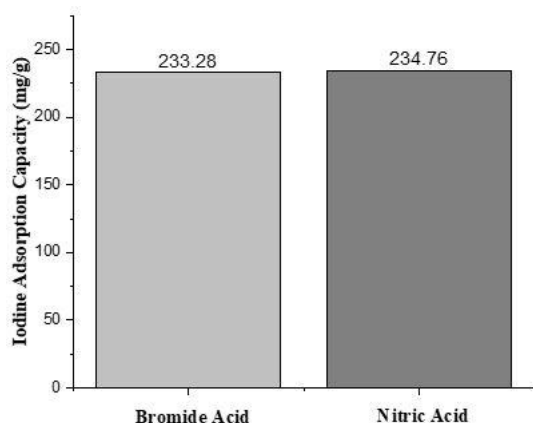


Figure 3. The activator's ability to adsorb iodine.

Determination of the best activator

The adsorption atom spectrometer (AAS-7000) can be used to examine the initial and final concentrations of the Cr(VI) solution. In the adsorption procedure, 25 mL of Cr(VI) solution at a concentration of 100 mg/L, 60 minutes of contact time, and 300 rpm stirring rate were utilized. There is a considerable variation in the percentage of each adsorbent activated with inorganic acids; bromide and nitric acids have the highest percentages of elimination Cr(VI) at 86.2% and 86.7%, respectively. This is ascribed to the resultant pores, so the surface area becomes more open, impacting the adsorption; on the other hand, the activating ingredient of the inorganic acid utilized can volatile compounds and remove adhering impurities or metal linked to the adsorbent. [22,23].

Effect of pH on Cr(VI) removal

The force of attraction between the adsorbent and the adsorbate is a mechanism of adsorption. The pH variation used in this experiment was 1–5. This aims to determine the best pH conditions for the adsorption of Cr(VI) using an adsorbent activated by bromide and nitric acids. The concentration of the solution was 100 mg/L, the adsorbent dose was 25 g, the contact time was 60 minutes, and the stirring rate was 300 rpm.

Figure 5 shows the removal of Cr(VI) using an adsorbent activated with inorganic acids. The maximum removals of Cr(VI) were 98.9% and 98.4% for the nitric and bromide acid-activated SCGs, respectively, at pH 4. At pH 5, the percentage of Cr(VI) removal decreases because when the acidic pH comes into contact with lignocellulose materials, it will reduce Cr(VI) to Cr(III). Adsorption of Cr(VI) ions using coffee grounds adsorbent is more beneficial at low pH because at low pH chromium reduction occurs, namely a reaction that binds protons so that it can reduce Cr(VI)

to Cr(III) [24]. Maximum Cr(VI) removal efficiency can be achieved at low pH due to the presence of various forms of chromium because it can reduce the SCG surface charge [25].

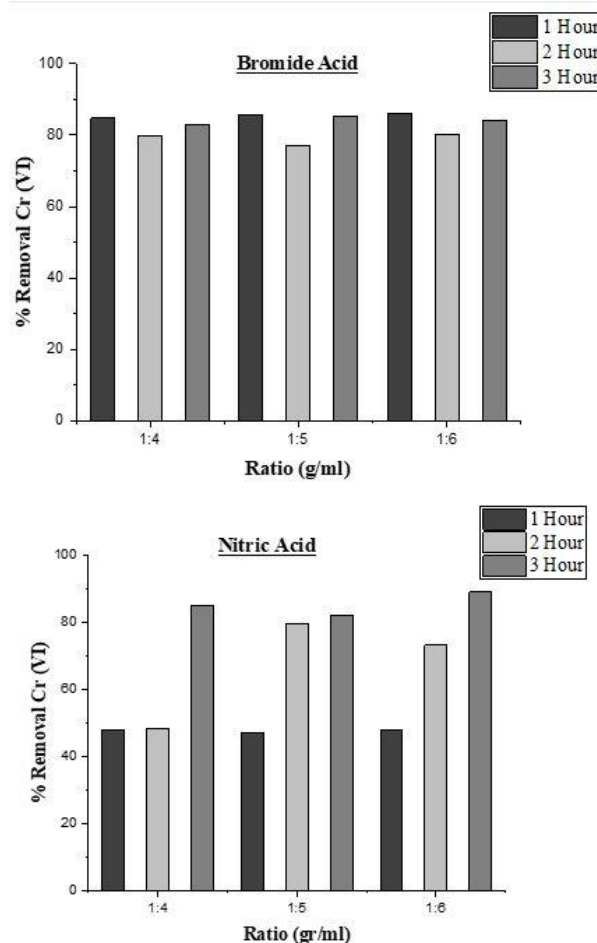


Figure 4. Uptake (percentage of removal) of Cr(VI) by investigated materials.

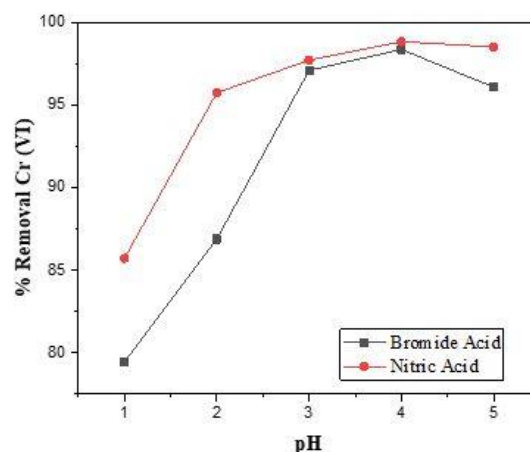


Figure 5. The influence of working pH on Cr(VI) removal.

Effect of contact time on Cr(VI) removal

Another crucial factor in the adsorption process is the contact time, which is utilized to calculate the ideal

mixing [26]. The time variable (60–140 minutes), the adsorbent weight (25 g/L), the initial Cr(VI) concentration (100 mg/L), pH 4 (obtained from earlier investigations), and the stirring rate (300 rpm) were the working parameters.

With an optimal contact time of 120 minutes, the maximum percentages of Cr(VI) removal obtained were 97.9% and 94.9% for the nitric and bromide acid-activated (Figure 6). SCG, as an adsorbent, demonstrated a significant increase in the percentage of removal as the contact time increased due to a large number of active sites participating in chromate ion adsorption, which will be gradually used up as the contact time is longer [20].

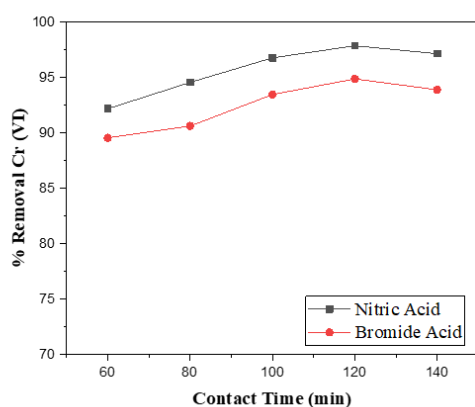


Figure 6. The influence of contact time between adsorbent and pollutant on Cr(VI) adsorption.

Effect of the initial concentration of Cr(VI) on the percentage removal

The operating conditions used in the experiment to determine the effect of the initial Cr(VI) concentration on the percentage of Cr(VI) removal were obtained from earlier experiments. These conditions included a 120-minute contact time, an adsorbent weight of 25 g/L, a pH of 4, and a stirring rate of 300 rpm. The Cr(VI) concentration employed in this experiment was varied at 20, 50, 80, 110, and 140 mg/L.

As the initial Cr(VI) concentration increased, the percentage of removal Cr(VI) dropped. It is depicted in Figure 7, showing that 20 mg/L is the ideal concentration for the adsorbents activated with nitric or bromide acid. This is attributed to the availability of a larger surface area of the adsorbent at low concentrations that might accelerate the adsorption [27]. On the other hand, the percentage of removal is low at higher concentrations, as the sites undergo saturation, allowing for the maximal adsorption of metal ions [28].

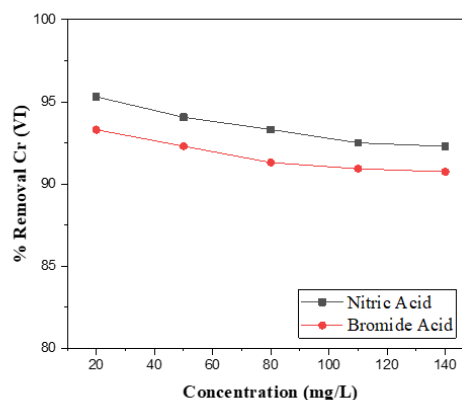


Figure 7. The influence of the initial concentration of Cr(VI) on the percentage of its removal.

CONCLUSION

In this study, the ability of SCG to absorb Cr(VI) as a pollutant was investigated. The effectiveness of nitric and bromic acids as SCG activators was also investigated. SCG was initially activated with nitric and bromide acids for three hours. The SCG activated with nitric acid provided the best iodine adsorption results by showing a removal percentage of 88.9% (an adsorption value of 235 mg/g) at an adsorbent-to-activator ratio of 1:6. Based on the SEM examination results, the adsorbent activated with nitric acid had a larger surface texture and more cavities compared to the adsorbent activated with bromic acid. The most dominant functional group of the two adsorbents is the OH group. In testing the adsorption process, the best operating conditions were determined, namely pH 4, contact time 120 minutes, and solution concentration 20 mg/L.

SCG is used as an adsorbent because it, as a biomass waste, is easy to obtain. However, the use of SCG as an adsorbent requires further activation to increase the adsorption capability. Therefore, it has not been used more widely in wastewater treatment processes. The application of SCG-based adsorbents in wastewater treatment has challenges because SCG adsorbents require the use of strong acid solutions, such as nitric and bromide acids, which are not safe and environmentally friendly. Potential limitations or obstacles in the use of strong acid as an activator can be overcome using an alternative activator that is more environmentally friendly or an adsorbent of natural origin with a higher percentage of adsorption without activation. Future potential or findings that are in line with the use of SCG are that it has been widely used as an adsorbent of contaminants in the form of dyes, Pb and Cr(III), active carbon precursors, and for composting.

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AYUNI YUSTIRA¹
HAFIFUDDIN ARIF¹
ZAHEDI ZAHEDI²
FACHRUR RAZI³

¹Department of Chemical
Engineering, Faculty of
Engineering Universitas
Sumatera Utara Medan,
Indonesia

²Department of Mathematics,
Faculty of Mathematics and
Natural Sciences Universitas
Sumatera Utara Medan,
Indonesia

³Department of Civil
Engineering, Faculty of
Engineering Universitas Al-
Washliyah Medan, Indonesia

NAUČNI RAD

UTICAJ AKTIVATORA NA OTPADNI TALOG KAFE KAO BIOSORBENTA ZA HROM

Globalna proizvodnja Cr(VI) može dostići 44 miliona metričkih tona godišnje, od čega se 49% ispušta u životnu sredinu za šta je kriv razvoj industrije. Prisustvo Cr(VI) izaziva ekološke probleme jer može ugroziti fizičku i ekosistemsku ravnotežu vodene faune u rečnim tokovima. Izloženost Cr(VI) izaziva zdravstvene probleme, kao što su oštećenje jetre, pluća i bubrega. Postoje brojni načini za odbranu od štetnog uticaja koji teški metali imaju na životnu sredinu. Adsorpcija je jednostavna, pristupačna i laka tehnika. Adsorbent korišćen u ovom radu je dobijen iz otpada biomase, tj. korišćene mlevene kafe (SCG). Njegov potencijal kao adsorbenta tek treba da se razvije. Svrha ovog rada je bila da se ispita uticaj aktivatora na SCG i njegov kapacitet da adsorbuje Cr(VI) jedinjenja. Azotna i bromidna kiselina su korišćene za aktiviranje 25 g SCG tokom 1, 2 i 3 sata. Odnosi adsorbenta i aktivatora bili su 1:4, 1:5 i 1:6. Početna koncentracija rastvora Cr(VI) je 100 mg/l, a brzina mešanja 300 o/min. Najbolji rezultati adsorpcije postignuti su sa rastvorom HNO₃ (pH 4) kao boljim aktivatorom pri odnosu 1:6, vremenom kontakta 120 minuta i početnoj koncentraciji Cr(VI) 20 mg/l. OH je glavna funkcionalna grupa oba adsorbenta.

Ključne reči: uklanjanje Cr(VI); azotna kiselina; bromidna kiselina; otpadni talog kafe.