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Utilization of Adsorbent Based on Rice Straw (*Oryza Sativa*) for Cr(VI) Ions Reduction in Aqueous Solutions

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Abstract. In this study, we utilized an adsorbent based on rice straw for reducing hexavalent chromium ions (Cr(VI)) in an aqueous solution. The rice straw as adsorbent raw material was washed, dried, and powdered. Rice straw powder was heated at 450ºC for 2 hours to obtain rice straw adsorbent. The adsorbent was activated with 1M H_3PO_4 for 4 hours. Characterization of the adsorbent was done using Fourier Transform Infra-Red (FTIR) method. FTIR spectra showed the presence of hydroxy, carboxylic, aromatic, and ether groups on the surface of the rice straw and the made adsorbent. The reduction of Cr(VI) ions in aqueous solutions was carried out using the adsorption batch method. The adsorption process was conducted in various the Cr(VI) solutions pH for 1-5 and variations in contact time for 5-720 minutes. The highest percentage reduction of Cr(VI) reached 66.90%. It has occurred at pH 2 and equilibrium at 600 minutes of contact time.

Introduction

Chromium (Cr) are famous compounds in various industries such as dyes, coating, catalysts, corrosion inhibitors, etc. [1]. This condition makes the main industrial waste contain Cr compounds. Cr compounds in wastewaters is primarily encountered in oxidation stages +3 [Cr(III)] and +6 [Cr(VI)]. Recent research suggests that Cr(VI) compounds are more toxic dan reactive than Cr(III) [2]–[4]. These properties combined with sufficient solubility and mobility of Cr(VI) in the environment, makes the issue of utilization and disposal of chromium-containing waste relevant to many countries. To cover this problem, the possible thing to do is improve the technology for its removal from industrial waste. The most common technology for Cr(VI) removal is adsorption. Adsorption is cost-effective and easy operating method [2]. The important part of adsorption method is the adsorbent used [3]. The conventional choice for adsorbent is commercial activated carbon. Unfortunately, this adsorbent is not cheap due to the high-cost raw materials for production. Therefore, developing low-cost activated carbon from inexpensive and sustainable raw materials is important.

Rice straw is byproducts of rice harvest. Rice straws reached 415,970.61 ton in East Java, Indonesia, in 2018 [5]. Besides being used for material production and as a source of renewable energy, rice straw is often utilized as an adsorbent for reducing heavy metals in aqueous solutions. There are various types of adsorbents that can be obtained from rice straw, such as bio sorbents, nanomaterials, and activated carbon [6]–[9]. Adel et al. made an adsorbent based on heated rice straw at temperatures ranging from 300 to 600°C for 30 to 120 minutes, followed by activation with 1 M HCl and NaOH [10]. The results showed that heating at 500°C for 120 minutes produced adsorbents with the highest adsorption capacity for reducing Pb(II) in aqueous solutions. The heating temperature can affect the functional groups on the adsorbent. Heating above 500°C can increase the amount of aromatic compounds and reduce the polarity of the adsorbent, which has a negative impact on the heavy metals adsorption [11], [12]. Rice straw, which is a lignocellulosic compound, is a suitable raw material for the production of carbon-based adsorbents.

Zhou et al. [13] used activated carbon derived from rice straw, activated with HCl and NaOH, as an adsorbent for the removal of Cr(VI) from aqueous solutions. They found that the optimum condition for Cr(VI) adsorption was pH 2, with equilibrium reached after a contact time of 1440 minutes, resulting in a reduction of 89.90% and 66.55% for HCl and NaOH activators, respectively. Farahmand et al. utilized activated carbon from rice straw with an H_3PO_4 activator for the removal of Ce(IV) [14]. The optimum pH for Ce(IV) adsorption occurred at pH 4, with equilibrium achieved after 500 minutes of contact time. The maximum adsorption capacity for Ce(IV) obtained was 4.13 mg/g. In the research conducted by Sakhiya et al., carbon adsorbent from rice straw was used for the reduction of Pb(II) and Zn(II) metal ions in aqueous solutions [9]. The optimum pH for adsorption of both metals was pH 6, with equilibrium achieved after a contact time of 150 minutes. The adsorption capacity for Pb(II) was found to be 17.93 mg/g, and for Zn(II), it was 25.73 mg/g.

Research on the adsorption of Cr(VI) from aqueous solutions continues to be a topic of investigation. Fan et al. used a mixed adsorbent of rice straw and sewage sludge activated with $ZnCl_2$ to reduce Cr(VI) in aqueous solutions [15]. The optimum pH for adsorption was pH 2, and equilibrium was reached after a contact time of 1440 minutes. The adsorption capacity for Cr(VI) obtained was 138.69 mg/g. Gao et al. employed rice straw bio sorbent activated with $C_4H_6O_6$ for the reduction of Cr(VI) in aqueous solutions [16]. The optimum pH for adsorption was 2, with a contact time ranging from 0 to 3600 minutes. The highest percentage reduction of Cr(VI) occurred at a contact time of 2880 minutes. The study of Ma et al. reported that activated carbon from corn straw, activated with KOH, was used to adsorb Cr(VI) at a capacity of 175.44 mg/g with a contact time of 480 minutes [17]. Despite various studies investigating the reduction of Cr(VI) from aqueous solutions using straw-based adsorbents activated with different agents, it appears that H_3PO_4 has not yet been used as an activator.

The use of H_3PO_4 as an activator can indeed enhance the adsorption capacity of activated carbon obtained from various raw materials. H_3PO_4 plays a crucial role in the pyrolytic transformation of the raw material and contributes to the formation of cross-linked carbonaceous structures [18], [19]. In this study, rice straw adsorbents were made and activated with H_3PO_4 for the reduction of Cr(VI) in aqueous solutions. The research aims to determine the optimum pH and equilibrium contact time for the adsorption process.

Materials and Methods

Materials. The semi-dried rice straw was obtained from local paddy fields in Malang, East Java, Indonesia. Phosphoric acid [H_3PO_4, 85%], chloride acid [HCl, 37%], and sodium hydroxide [NaOH] were purchased from local market. Demineralized water was bought from a local market and used for cleaning and chemical preparation. All chemicals were used without any purification. 1 M H_3PO_4 solution as adsorbent activator and 1 M HCl solution were prepared in Laboratory of Instrumentation and Analytical Sciences, Chemistry Department, Faculty of Sciences and Data Analytics, Institut Teknologi Sepuluh Nopember. The standard solutions of Cr(VI) in various concentration (10-50 mg/L) were prepared by diluting potassium dichromate (VI) [$K_2Cr_2O_7$ (GR)] with demineralized water. The standard solutions were measured by Atomic Absorption Spectrophotometry (AAS, Thermo Fisher) at 357.9 nm to obtain calibration curve. The working solution is wastewater that containing 40 mg/L Cr(VI). The working solution was prepared from dilution of potassium dichromate (VI) [$K_2Cr_2O_7$ (GR)] and demineralized water.

Preparation of Rice Straw Adsorbent. Rice straw was cut into 1-1.5 cm pieces and washed until the rinsed water became clear. The straw was then air-dried at room temperature for approximately 24 hours before being heated in an oven at 105°C for 24 hours. The dried straw was finely ground to obtain a powder, which was subsequently heated to 450°C with a retention time of 2 hours and a heating rate of 10°C/minute, resulting in the rice straw adsorbent [9], [20]. The rice straw adsorbent was washed with 1 M HCl and deionized water until it reached a pH of 6-7, and then it was dried in an oven at 105°C for 12 hours. Prior to use, the rice straw adsorbent was sieved through a 120-mesh sieve to obtain uniform particles with a size below 125 μm and stored in the desiccator [21].

The activated rice straw adsorbent was prepared by mixing it (5 g) with 100 mL of 1 M H_3PO_4 activator solution. The mixture was stirred for 4 hours [10]. Subsequently, it was filtered and washed with demineralized water to obtain a pH of 6-7. Then, it was dried in an oven at 105°C for 12 hours until a constant weight. The activated adsorbent was sieved through a 120-mesh sieve and stored in the desiccator.

Batch Experiments. The batch adsorption method was employed to determine the optimum pH and contact time using an adsorbent dosage of 4 g/L [22]. A total of 0.1 g of rice straw activated carbon was mixed with 25 mL of a wastewater containing 40 mg/L of Cr(VI). The mixture was stirred for a specified period [15]. The mixture was filtered, and the resulting filtrate was analyzed using AAS technique. The removal percentage was calculated using Eq. 1, where C_I refers to the initial concentration of the adsorbate in the solution, and C_0 represents the concentration of the adsorbate after the adsorption process has taken place.

$$Removal\ Percent = \frac{(C_I - C_0)}{C_I} \times 100\% \quad (1)$$

The testing of the adsorbate's pH influence was conducted using a working solution adjusted to pH values of 1, 2, 3, 4, and 5, with each test having a contact time of 100 minutes [23]. The pH variation that resulted in the highest percentage reduction was determined as the optimum pH and will be used in the following experiments. The effect of contact time was conducted by varying the contact time between the adsorbent and adsorbate, starting from 5 minutes and continuing until a constant percentage reduction was obtained. This was done using a working solution containing Cr(VI) at a concentration of 40 mg/L, each at the previously determined optimum pH.

FTIR Characterization. The KBr pellet method was employed for FT-IR characterization. The samples were mixed with KBr at a ratio of 1:100 and finely ground and pulverized. The mixture of powders was then placed into a press apparatus to form pellets, which were subsequently loaded into a sample holder for FT-IR analysis. This analysis was carried out for the following samples: dried rice straw powder, rice straw activated carbon, H_3PO_4-activated rice straw adsorbent, and H_3PO_4-activated rice straw adsorbent after adsorption.

Results and Discussions

Fabrication of Rice Straw Adsorbent. Fig. 1A shows the cut and washed rice straw and the powdered rice straw is depicted in Fig. 1B. The rice straw powder displayed a brown color throughout, indicating the degradation of chlorophyll during the heating process [24]. The heating of the rice straw powder was carried out in an electric furnace at 450°C for 2 hours with a heating rate of 10°C/minute to obtain rice straw adsorbent. The result of this heating process showed a color change in the powder to black, with a small amount of ash on the surface. The formation of ash is attributed to the rice straw powder reacting with oxygen inside the crucible, resulting in complete combustion. The appearance of the rice straw powder after heating can be seen in Fig. 2A. The obtained rice straw adsorbent was washed with 1 M HCl and demineralized water until pH of 6-7 to remove residual ash from carbonization. After activating and sieving, there were no significant differences observed in the rice straw adsorbent except that it appeared darker in color and had uniform particle size, as shown in Fig. 2B.

Fig. 1. Dried rice straw (A) and rice straw powder (B).

Fig. 2. Rice straw adsorbent (A) and the activated rice straw adsorbent (B).

FTIR Characterization. The rice straw powder, rice straw carbon, H_3PO_4-activated rice straw carbon before and after adsorption were characterized its functional groups using FT-IR. Table 1 provides information about the vibrational bands corresponding to the peaks in the spectra. The main peaks that appear indicate the presence of oxygen functional groups such as ether, ketone, phenolic, and alcohol groups. There are changes in the peaks in the spectra of the powder and adsorbent, specifically the disappearance of peaks at wavenumbers 1375 and 2920 cm^{-1}. The absence of the peak at 1375 cm^{-1} is due to dehydration and aromatization resulting from the decomposition and condensation of volatile compounds *[25]*. Meanwhile, no peak of C-H methylene at 2920 cm^{-1} is attributed to the decomposition of C-H bonds during heating to form more stable C=C aromatic bonds at high temperatures. Conversely, a new peak is formed in the adsorbent spectrum at wavenumber 1695 cm^{-1}, indicating the oxidation of surface functional groups of the adsorbent. The increased intensity of the carboxylate peak in the spectrum of the H_3PO_4-activated adsorbent is caused by the protonation of carboxylate groups by the acid activator *[13]*.

Table 1. FT-IR vibration peak.

Wavenumber (cm^{-1})	Vibration	Peak			
		Rice straw powder	Rice straw activated carbon	H_3PO_4-activated rice straw adsorbent	H_3PO_4-activated rice straw adsorbent after adsorption
468	Si-O-Si stretch	✓	✓	✓	✓
800	C-H aromatic	✓	✓	✓	✓
1101	C-O/ C-O-C	✓	✓	✓	✓
1375	C-H aliphatic/ O-H phenolic	✓	×	×	×
1639	C=C aromatic	✓	✓	✓	✓
1695	C=O	×	✓	✓	✓
2060	Overtone aromatic	✓	✓	✓	✓
2920	C-H methylene	✓	×	×	×
3420	O-H stretch	✓	✓	✓	✓

Calibration Curve. The calibration curve is depicted in Fig. 3. The calibration curve exhibits a linear equation, i.e. $y = 0.008x + 0.0114$, with an R^2 value of 0.9968. The R^2 value obtained is above 0.995, which indicates that the calibration curve is acceptable and can be used to calculate the concentration of Cr(VI) in the following tests.

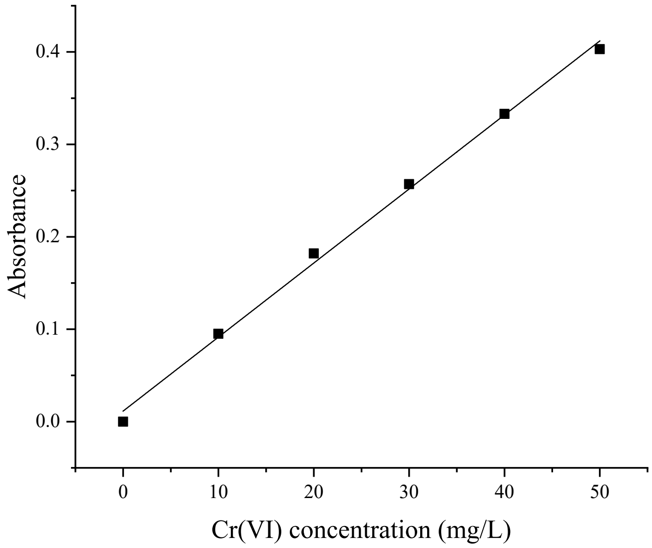

Fig. 3. Calibration curve of the Cr(VI) standard solution.

Effect of pH. The testing of pH variations in the adsorbate was carried out to determine the optimum pH, using a range of pH values: 1, 2, 3, 4, and 5. The results of the removal percentage of Cr(VI) can be found in Fig. 4. The removal percentage of Cr(VI) increases with the acidity of the adsorbate solution, and the highest percentage is achieved at a pH of 2 (43.72%). This is followed by a decrease at pH 1. This can be attributed to the fact that in an acidic environment, the surface of the activated carbon is surrounded by H^+ ions, which facilitate the binding of Cr(VI) anionic, through electrostatic attraction. The more positive the surface of the activated carbon, the easier it is to bind Cr(VI) ions [26]. In addition to electrostatic attraction, the removal mechanism of Cr(VI) in the solution can be attributed to the reduction of Cr(VI) ions to Cr(III) ions. In an acidic environment, Cr(VI) ions exist in the form of $HCrO_4^-$ and $Cr_2O_7^{2-}$, which can be directly reduced to Cr(III) ions by electron-donating groups on the activated carbon [13], [27].

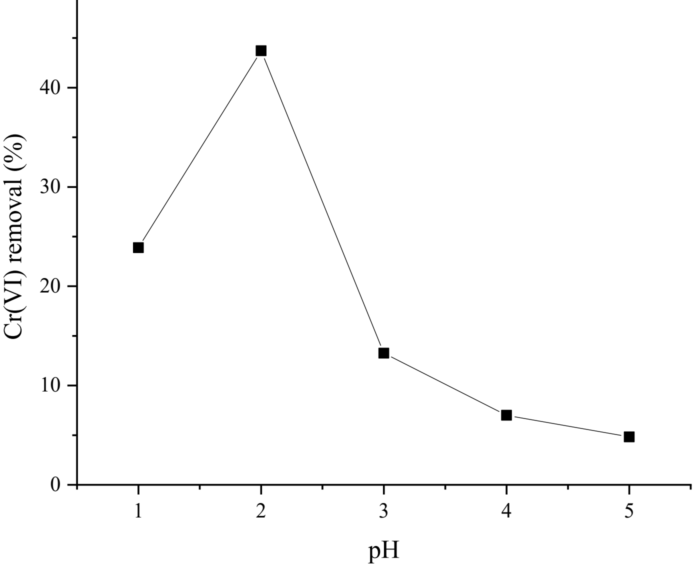

Fig. 4. Graph of the effect of varying pH on the removal percent of Cr(VI) ions.

The reduction reaction of Cr(VI) ions can be seen in Fig. 5. Cr(III) ions, which carry a positive charge, can then be released back into the solution due to electrostatic repulsion from the surface of the activated carbon or adsorbed by the acidic active sites of the activated carbon through cation exchange [12], [15], [28]. At extremely acidic pH levels, the Cr(III) ions released back into the solution may have difficulty reabsorbing onto the surface of the activated carbon due to the excess H^+ ions present on both the carbon surface and in the solution. The results obtained align with the findings of Parlayici and Pehlivan [29], where a decrease in the removal percentage was observed at pH values below 2.

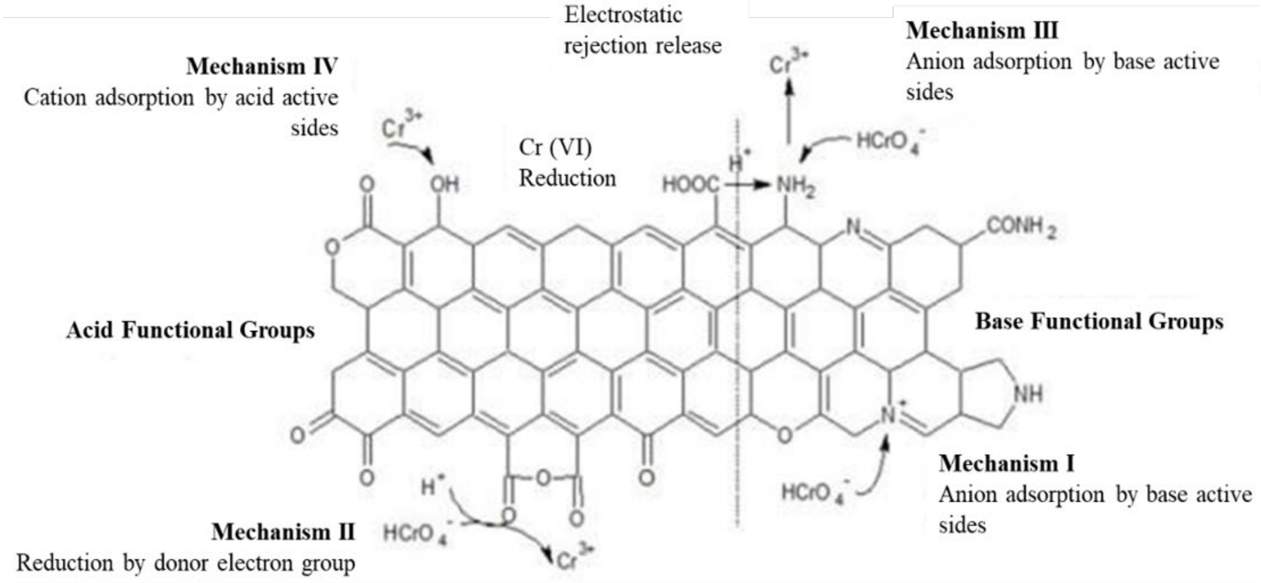

Fig. 5. Adsorption mechanism of Cr(VI) ions in activated carbon surface.

Effect of Contact Time. The effect of contact time was conducted by varying the duration of stirring between the adsorbent and adsorbate in the batch adsorption process. Various contact times of 5, 30, 60, 120, 180, 240, 300, 360, 480, 600, and 720 minutes were used. The results of removal percentage by contact time variations is presented in Fig. 6. The graph indicates that the removal percentage of Cr(VI) ions at a contact time of 5 minutes is 21.06%, followed by a increasing the removal percentage at a contact time of 30 minutes. This is then followed by a gradual increase until equilibrium is reached at a contact time of 600 minutes, with an adsorption percentage of 66.90%. A similar trend was observed in the research conducted by [17]. This phenomenon is attributed to the high availability of active sites on the H_3PO_4-activated rice straw adsorbent at the beginning of the adsorption process, allowing Cr(VI) to be readily bound to the adsorbent surface. One of the proposed mechanisms is the adsorption of Cr(VI) by functional groups containing oxygen, either through ion exchange or complexation [17]. This mechanism may underlie the adsorption process in this study, as evidenced by the high presence of oxygen-containing functional groups in the FT-IR spectra obtained. The reduction of Cr(VI) ions can be visually observed because Cr(VI) ion solutions are yellow in color and will turn orange as the concentration of Cr(VI) ions increases [30]. Fig. 7 illustrates the color change of the solution before and after the adsorption process.

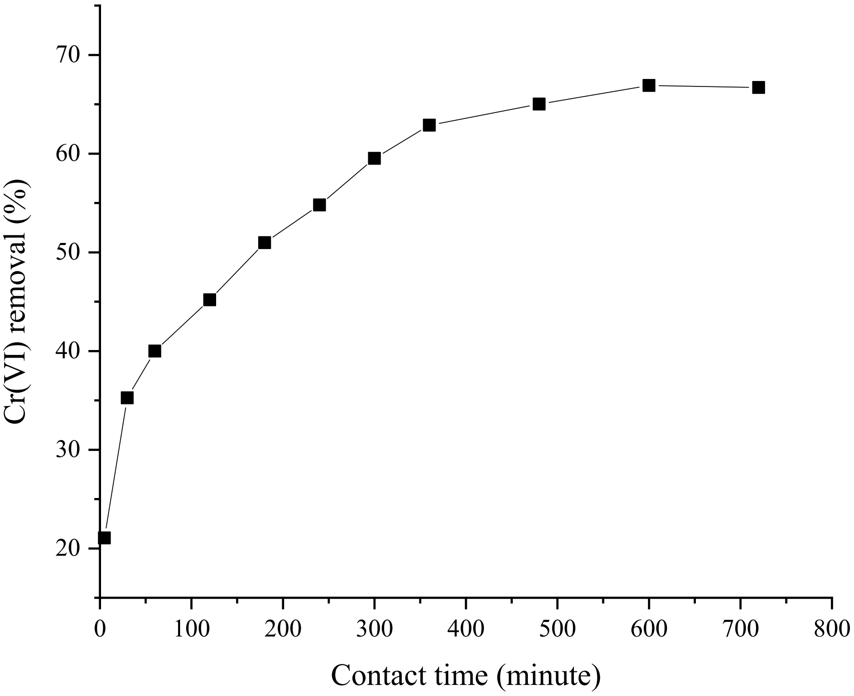

Fig. 6. Graph of the effect of varying contact time on the removal percent of Cr(VI) ions.

Fig. 7. Comparison of working solution before (working solution) and after adsorption with contact time of 60 and 360 minutes.

Conclusions

An adsorbent based on carbon derived from rice straw was successfully prepared, heated to 450°C for 2 hours, and activated with H_3PO_4 for the reduction of Cr(VI) in aqueous solutions. The obtained results revealed an optimum pH at pH 2 and equilibrium at a contact time of 600 minutes, with a Cr(VI) reduction percentage of 66.90%.

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