

Carbon-Oxalate (C-Ox) Practicum Waste as an Adsorbent Material Candidate in the Chemistry Laboratory

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ABSTRACT. This study aims to treat carbon and oxalate practicum waste as an adsorbent material candidate in the Chemistry Laboratory. Practicum waste was used to synthesise carbon-oxalate (C-Ox) by using wet (C-OxW) and semi-wet (C-OxDs) impregnation methods. The products were tested by SEM and FTIR. The adsorption was tested by contacting each 1 gram of C-Ox with various concentrations of chromium ions waste for 24 hours while shaking. The filtrate was measured by AAS. The data obtained were compared with standard activated carbon (CC) as control and analyzed with Fre\undlich and Langmuir isotherm equations. The results showed that C-Ox can be used as an adsorbent in the chemistry laboratory. Based on the SEM test, the pores of C-OxW were seen to be most than C-OxDs and CC. Adsorption of chromium ions of C-OxW and C-OxDs were more effective than CC. C-Ox test by FTIR showed very sharp wavenumbers appeared at 3674.5 and 3705.41 cm⁻¹ for the hydroxyl groups; 1697.4 cm⁻¹ for C=O; and 1141.8 cm⁻¹ for C-O. The determination coefficient (R²) of Freundlich isotherm of CC and C-Ox were higher than Langmuir isotherm, and on the other hand, R² of C-OxDs was shown oppositely.

Keywords: adsorbent, candidate, carbon-oxalate, waste

INTRODUCTION

Practicum was very important in chemistry learning. Practicum can develop process skills, motor skills, and the formation of scientific attitudes. The formation of this scientific attitude includes: conducting investigations, research, and studying nature in more depth. Practicum can also increase interaction between groups so that they can appreciate the work of the others. Thus emotional, social, and spiritual intelligence can develop (Akmalia & Silaban, 2018; Merdekawati, 2017). The important role of practicum in chemistry learning was very dependent on laboratory facilities and infrastructure. One of them was material (Harefa & Purba, 2019).

Chemical laboratory materials consist of general materials and special materials. Special materials must be treated specifically because they have a high level of purity, and have unstable properties. The cost of procuring special materials was very expensive and difficult to obtain (Harta et al., 2020). One of the special ingredients was activated carbon, oxalic acid, and chromium compounds.

Activated carbon, oxalic acid, and chromium compounds were used in the Analytical and Physical Chemistry Laboratory of the Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Pendidikan Ganesha (Undiksha). Activated carbon was used 560 grams every year and each researcher were used 400-500 grams. Meanwhile, oxalic acid was used at 1460 grams per year and some chromium compound was used at 50 grams or 15 g/L per year. The impact of using special materials was produced in waste (Putra et al., 2017). It became an environmental problem that must be solved immediately.

The problems with the use of activated carbon, oxalic acid, and chromium ions were storage, processing, and health effects. Carbon-oxalic waste was dominated by physics chemistry practicum, namely Freundlich isotherm. Meanwhile, saturated oxalic acid waste was produced from the practicum of the effect of temperature on solubility. Oxalic acid waste has been used for some qualitative chemistry practicum, virulence factor for diseases (Uloth et al., 2015), precipitated as ferrous oxalate (FeC₂O₄·2H₂O) (Liu et al., 2019). However, the number of waste was still abundant, and in the long term, the waste becomes a problem in the place and it was dangerous for the practitioner.

Health impacts of saturated oxalic acid waste according to Regulation (EU) No. 1907/2006 were coughing, shortness of breath, agitation, spasms, nausea, vomiting, collapse, and circulatory disorders. The systemic effects were decreased blood calcium levels, toxic effects on the kidneys, cardiovascular disorders, and the risk of serious injury to the eye. Besides that, according to Regulation number 04 / BIM / PER / 1/2014, oxalic acid can cause explosions with air on continuous heating. The critical range for oxalic acid was 15 K below the flashpoint. There was also a risk of explosion when oxalic acid reacted with chloric, sodium hypochlorite, strong oxidizing agents, and silver. Saturated oxalic acid reacted with alkaline, ammonia, or mercury to be an explosion because its reaction was exothermic. Meanwhile, the impact of laboratory chromium waste was very dangerous for the environment. The presence of chromium waste at oxidation numbers of +3 (Cr (III) and +6 (Cr(VI)). Cr(VI) was highly soluble in water and has higher toxicity in the human body than Cr(III) (Fenti et al., 2020). In addition, Cr(VI) also causes carcinogenic, mutagenic, and teratogenic effects (Prajapati et al., 2022). Cr(VI) was speciated as dichromate, hydrogen chromate, and chromate (Ajoke, 2021). The speciation of Cr(VI) was strongly influenced by pH. When pH is 2-6.5, chromium was in the main species $HCrO_4^-$ and when pH > 6.5-8 with the main species CrO₄²⁻ (Zheng & Duan, 2022).

Various techniques have been applied to remove Cr(VI), such as chemical precipitation, ion exchange, and redox, (Mahmoud et al. 2021; Pan et al. 2022), electrolysis, phytoremediation, and adsorption (Qi et al., 2021). Among these techniques, the adsorption technique was considered the most effective, inexpensive, easy to operate, accessible, available, and profitable method (Dim et al., 2021).

The choice of adsorbent was an important matter in the adsorption technique. Several adsorbents have been applied to remove hexavalent chromium, such as activated carbon (Hoang et al., 2021), natural oxides, and mineral composites (Punia et al., 2021). All of these adsorbents have given good results, but they are expensive and difficult to use on an industrial scale (Picazo-Rodríguez et al., 2022).

In recent years, applications for low-cost adsorbent materials such as natural materials, minerals, agricultural wastes, forest wastes, and industrial wastes have been developed (Picazo-Rodríguez et al., 2022). All these wastes have not shown maximum results in absorbing chromium ions waste. To increase the adsorption power, it is necessary to add epoxy, carboxyl, and hydroxyl groups. The presence of these groups on graphene oxide has been shown to increase arsenic adsorption (Singh et al., 2022). Thus, a selective, biodegradable, biocompatible, low-cost, environmentally friendly, efficient, and high adsorption adsorbent was created (Mahmoud et al., 2021).

Carbon-oxalate laboratory waste contained epoxy, carboxyl, and hydroxyl groups. The waste was studied as an adsorbent of chromium ions waste in the laboratory. Thus, an effective, inexpensive, easy to operate, accessible, available, and profitable adsorbent was obtained to treat chrome ions waste. The sections studied in this paper were morphology, FTIR spectrum, adsorption of chromium waste, and adsorption patterns of carbon-oxalate waste to chrome waste.

EXPERIMENTAL SECTION

Material and Methods

The materials were used K₂Cr₂O₇ pa Merck, demineralized water, Whatman filter paper with 125 mm diameter, charcoal pa, and laboratory waste of Analytical Chemistry and Physical Chemistry, Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Pendidikan Ganesha. The laboratory waste was carbon and oxalic acid. While the tools were used glassware, hot stirrer, ZHWY-304 shaker, UF160 oven, Shimadzu's IR Affinity-1 FT, SEM type JEOL JSM 6510-LA, and AAS Shimadzu AA-63.

Carbon-oxalate (C-Ox) Synthesis

Synthesis of C-Ox was carried out using 2 impregnation methods, namely the wet method and the semi-wet method. Wet impregnation method, 10 grams of carbon waste was immersed in saturated oxalic acid waste for 24 hours. Then the two mixtures were shaken for 24 hours using a shaker. The mixture was filtered, washed with demineralized water until pH 7, and dried in an oven at 100°C, then carbon-oxalate (C-OxW) was formed. C-OxW was tested by FTIR and SEM.

In the semi-wet impregnation method, 10 grams of carbon waste was immersed in saturated oxalic acid waste. The mixture was heated and stirred until dry. The mixture was heated at 100°C, and carbon-oxalate (C-OxDs) was formed. C-OxDs was tested by FTIR and SEM.

Implementation of C-OxW and C-OxDs as an Adsorbent

The implementation of C-OxW and C-OxDs as an adsorbent was carried out on chromium (VI) ion waste. The maximum adsorption pattern was determined by contacting the C-OxW and C-OxDs into the chromium ions waste solution at pH 6. One gram of C-OxW and C-OxDs were contacted with chromium ions waste with concentrations of 130, 200, 300, and 400 ppm for 24 hours. Filtering was done after the contact time. The filtrate was measured by AAS. The amount of chromium ions adsorbed per gram of adsorbent (mg/g) was calculated by the equation (Kebede et al. 2022).

$$qt = \frac{(Co - Ct)xV}{m}$$

In this case, V was the volume of solution (mL), m was the mass of C-OxW and C-OxDs, and Co, Ct was the concentration of chromium ions (ppm) at 0 and t seconds. The data was compared with standard carbon (CC) as a control, then it was analyzed by Langmuir and Freundlich equations.

RESULTS AND DISCUSSION

Morphology CC, C-OxW and C-OxDs

The morphology of CC, C-OxW, and C-OxDs were measured. SEM can determine the porosity of a sample (Min & Jung, 2017). Based on the SEM test, the pores of C-OxW were seen to be more than that of C-OxDs and CC. The results of these measurements were presented in **Figure 1**.

FTIR Spectrum

The FTIR spectra of CC, C-OxW, and C-OxDs were presented in **Figure 2**. The wavenumbers of oxalates bound to carbon show the strain vibrations of the hydroxyl groups (O-H) at 36200-3700 cm⁻¹. The hydroxyl groups were characterized by strong and sharp adsorption at 3674.5 and 3705.41 cm⁻¹. The standard oxalic acid C=O group strain vibration was found at 1697.4 cm⁻¹, while the standard oxalic acid C-O group strain vibration was found at 1141.8 cm⁻¹. These results were in line with the research of (Febriaty & Alimuddin, 2016). The wavenumbers were very sharp on C-OxW, less sharp on C-OxDs, and not visible on CC. These data indicate that the impregnation technique was a significantly different result in C-OxW and C-OxDs.

The FTIR spectrum of C-OxW shows a bond between carbon and oxalate very clearly. The binding energy between activated carbon functioned with oxalic acids was -34.79 kcal mol⁻¹. This bond was very strong accompanied by a large change in the structure of carbon compared to carbon without oxalates. This change was particularly evident for the bond angle and torsion, and not for the bond length. Nonfunctional carbon has very weak bonds and the presence of oxalate functional groups on the surface of carbon was very important for adsorption (Henry, 2013). The minimum energy structure of carbonoxalate was depicted in **Figure 3**.



Figure 1. The morphology of CC (a), C-OxW (b), and C-OxDs (c)



Figure 3. The minimum energy structure of carbon-oxalate (Henry, 2013)

Figure 3 shows the optimal geometry of the carbon-oxalate structure when activated carbon has functioned with a carboxylate group. The distance between the centre of mass of oxalate and carboxylic acids H1, O1, C1, and O2 was 2.31; 3.25; 4.24, and 5.36 Å respectively. It was clear that the double-charged oxalate was attracted to the OH portion of the carboxylic acid group, and it was not to O2 (H1 has a +0.41 charge and O1 has a 0.45 charge) (Henry, 2013).

Sorption of CC, C-OxW, and C-OxDs on Chromium lons Waste

The sorption of CC, C-OxW, and C-OxDs was increased with the increasing concentration of chromium ions waste. The amount of chromium ions adsorbed by CC, C-OxW, and C-OxDs was presented in **Figure 4**. The amount of chromium ions waste adsorbed by C-OxW and C-OxDs was higher than CC at each concentration because C -OxW and C-OxDs contain carboxyl groups (-COO-). Carboxyl groups (-COO-) can be used as a host and bonded chromium ions due to electrostatic forces (Jiang et al., 2020). The impact of electrostatic forces also occurs on metalbinding organic ligands (-COOH) (Hosseinzadeh, 2019).

The adsorption efficiency of every 1 gram of CC, C-OxW, and C-OxDs with every chromium ion concentration (400 ppm, 300 ppm, 200 ppm, and 130 ppm) showed a difference. C-OxW has the relative same efficiency as every chromium ion concentration waste. The adsorption of C-OxDs was increased at 130-300 ppm and it was stable at 300-400 ppm. Meanwhile, CC showed a decrease in the adsorption efficiency of chromium ions waste. The adsorption efficiency of chromium ions on C-OxW and C-OxDs was well than CC. It was effect by the modification of the oxalate group on the carbon (Scheufele et al., 2016). This has shown that carbon impregnation with oxalate is an appropriate step to increase the carbon adsorption capacity to chromium ions. Impregnation with suitable chemicals boosted the adsorptive capacity of activated carbon to remove hazardous substances (Sultana et al., 2022).



Figure 4. Relationship between initial concentrations of chromium ions and adsorbed chromium ions



Figure 5. Relationship between initial concentration of chromium ions waste and adsorption efficiency

Adsorption Patterns of CC, C-OxW, and C-OxDs

The adsorption isotherm was used to determine the adsorption pattern of CC, C-OxW, and C-OxDs. The adsorption isotherm shows the number of target species was distributed between the liquid and solid phases when the adsorption process reaches equilibrium. Adsorption isotherm was used to determine the maximum metal adsorption capacity of the adsorbent which was expressed in terms of the amount of metal adsorbed per unit mass of the adsorbent used (mg/g or mmol/g). Langmuir and Freundlich's equations were used to describe equilibrium adsorption to remove chromium ions (Attia et al., 2010; Khawaja et al., 2015). The linear form of Langmuir and Freundlich isotherms was represented by the following equation.

Langmuir (Vunain et al., 2021).

Ce/qe = 1/KLQo + Ce/Qo

In this case: KL = Langmuir equilibrium constant for adsorption (L/mg), Qo = maximum adsorption capacity (mg/g), qe = amount adsorbed at

equilibrium (mg/g), Ce = equilibrium concentration (mg/L).

Freundlich (Dada et al., 2012).

 $\log qe = \log KF + 1/n \log Ce$

In this case, KF and n are the isotherm constants of the Freundlich equation.

The determination coefficient (R^2) of the Langmuir isotherm of CC, C-OxW, and C-OxDs were 0.935, 0.008, and 0,889 respectively, whereas R^2 of Freundlich isotherm of CC, C-OxW, and C-OxDs were 0.998, 0.914, and 0.623 respectively. Based on the correlation, the Freundlich isotherm of CC and C-OxW were higher than the Langmuir isotherms. CC and C-OxW were described as heterogeneous adsorption on the surface, reversible molecules interaction, and a nonideal adsorption process (Mustapha et al., 2019). Meanwhile, the correlation coefficient for C-OxDs showed that the Langmuir isotherm was higher than the Freundlich isotherm. C-OxDs was shown that adsorption homogeneously on the surface and adsorption on the monolayer (Scheufele et al., 2016).



Figure 6. Freundlich isotherm adsorption pattern of CC (a), C-OxW (b), and C-OxDs (c)



Figure 7. Langmuir isotherm pattern of CC (a), C-OxW (b), and C-OxDs (c)

CONCLUSIONS

Based on the results of the study, it can be concluded that C-Ox can be used as an adsorbent in the chemistry laboratory. Based on the SEM test, the pores of C-OxW were seen to be most than C-OxDs and CC. Adsorption of chromium ions of C-OxW and C-OxDs were more effective than CC. C-Ox test by FTIR showed very sharp wavenumbers appeared at 3674.5 and 3705.41 cm⁻¹ for the hydroxyl groups; 1697.4 cm⁻¹ for C=O; and 1141.8 cm⁻¹ for C-O. The determination coefficient (R^2) of Freundlich isotherm of CC and C-Ox were higher than Langmuir isotherm, and on the other hand, R^2 of C-OxDs was shown oppositely.

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