

Optimization of Soaking Time and HCl Activation on the Properties of Adsorbent Synthesized from Robusta Coffee Husk Waste

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Abstract The increasing severity of water pollution caused by industrial, agricultural, and domestic activities requires the development of efficient and sustainable adsorbents. This study investigates the optimization of soaking duration and hydrochloric acid (HCl) activation on the physicochemical properties of activated carbon synthesized from Robusta coffee husk waste. The activation process used HCl at concentrations of 5%, 10%, and 15%, with immersion durations of 1, 2, and 3 days. Characterization of the resulting activated carbon included moisture content, ash content, and iodine adsorption capacity, analyzed in accordance with the Indonesian National Standard (SNI) No. 06-3730-1995. Experimental results revealed that all samples met the standard limits for moisture (<15%) and ash content (<10%), with the lowest values recorded at 0.0023% and 1.0056%, respectively. However, none of the samples achieved the minimum iodine adsorption threshold of 750 mg/g, with the highest capacity measured at 660.33 mg/g under 2 day immersion with 5% HCl. Statistical analysis using two-way ANOVA demonstrated that both HCl concentration and soaking duration, as well as their interaction, had a statistically significant effect ($p < 0.05$) on all measured properties. The optimum condition for producing high quality activated carbon was identified as 5% HCl concentration with 2 days of soaking. This finding highlights the potential of coffee husk biomass as a cost effective and environmentally friendly precursor for activated carbon production.

INTRODUCTION

The degradation of water quality resulting from extensive activities such as industrialization, intensive agricultural practices, and domestic waste has reached an alarming level. Hazardous contaminants including heavy metals, organic compounds, and excessive nutrients not only disrupt the ecological balance of aquatic biota but also contribute to toxicity chains that threaten human health through bioaccumulation. Amid the complexity of these challenges, adsorption technology offers an innovative solution by employing sorbent materials to selectively capture pollutants at the solid liquid interface.

Among the cost effective and efficient approaches to mitigating the adverse impacts of water pollution, adsorption stands out as particularly promising. This method has been demonstrated to be effective in removing color, odor, and harmful contaminants due to its high sorptive capacity. Although activated carbon remains one of the most effective adsorbents, its relatively high cost has led researchers to explore biomaterials as viable alternatives. These biomass based materials are not only more accessible but also generally require lower production costs (Septiani et al., 2023).

One of the promising raw materials for producing activated carbon is the coffee plant, whose primary waste product is the coffee bean husk (Nurmalita et al., 2022). Coffee husks contain a substantial amount of cellulose, ranging from 15% to 43%. The high cellulose content, along with other organic compounds rich in carbon elements, indicates the considerable potential of coffee husks as a precursor for activated carbon production (Ratman et al., 2024). In several coffee producing regions, unmanaged waste particularly Robusta coffee bean husks remains a persistent environmental issue, as it poses a risk of contamination if not properly treated.

Previous studies have demonstrated that activated carbon derived from spent coffee grounds can effectively remove pollutants such as ammonia from liquid effluents in urea fertilizer industries (Desniorita et al., 2022). These findings highlight the potential of coffee bean husks as a viable raw material for developing natural adsorbents. Through appropriate physical or chemical activation processes, coffee husk waste can be converted into activated carbon, enhancing its economic value while simultaneously reducing its environmental impact.

METHODS

The research design used in this study is an experimental laboratory method. The independent variables in this study are the hydrochloric acid (HCl) concentration and soaking duration, while the dependent variables is the adsorption effectiveness of the resulting activated carbon. The relationship among these variables will be statistically analyzed using a two-factor analysis of variance (ANOVA) with replication. This research design was chosen to make sure the study answers the research questions clearly and accurately.

The equipment used in this study includes a volumetric flask, beakers, grinder, glass funnel, porcelain crucibles, aluminum foil, glass stirrer, tray, desiccator, 80-mesh sieve, drying oven, combustion furnace for activated carbon production, and a titration apparatus for iodine adsorption capacity testing. The materials utilized consist of simplisia (dried raw material) of Robusta coffee bean husk waste, hydrochloric acid (HCl) at concentrations of 5%, 10%, and 15%, sodium thiosulfate (Na₂S₂O₃), potassium iodide (KI), Iodin (I₂) and starch indicator.

Research Stage

The experimental procedures were conducted through several phases, as illustrated schematically in **FIGURE 1**.

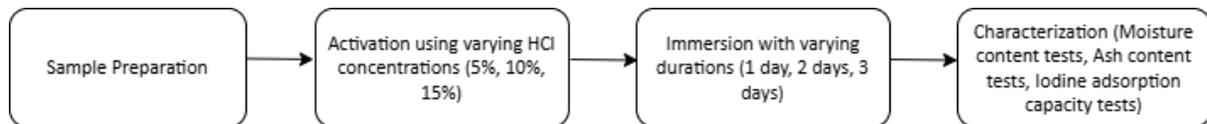


FIGURE 1. Schematic diagram of the research stages.

Sample Collection And Preparation

Robusta coffee husk samples were obtained from the coffee milling process in Kalibanger Village, Semarang Regency. The samples were subsequently cleaned to remove impurities such as stones and residual coffee beans. The robusta coffee husks were then subjected to combustion in a furnace using a metal tray to produce charcoal. The resulting charcoal was ground using a grinder to obtain a fine powder, which was subsequently sieved through an 80-mesh screen.

Activated Carbon Production and Characterization

The resulting charcoal was ground into a fine powder using a grinder and subsequently sieved through an 80-mesh screen. A total of 25 grams of the sieved charcoal was then chemically activated using hydrochloric acid (HCl) at varying concentrations of 5%, 10%, and 15%. The samples were subjected to different soaking durations of 1, 2, and 3 days. Following the soaking process, the activated carbon was thoroughly rinsed with aquadest and dried in an oven at 120°C for 1 hour until completely dry.

The produced activated carbon was characterized through moisture content analysis, ash content analysis, iodine adsorption capacity analysis.

Moisture Content Test

One gram of activated carbon was placed in an oven at 150°C for 30 minutes, then cooled in a desiccator for 5 minutes. The moisture content was calculated with formula [1] below.

$$\text{Moisture Content Test (\%)} = \frac{w_1 - w_2}{w_1} \times 100\% \tag{1}$$

Where

W1 = Weight before heating (g)

W2 = Weight after heating (g)

Ash Content Test

One gram of activated carbon was placed in a furnace at 500°C for 1 hour, then cooled in a desiccator for 30 minutes. Ash content was calculated using the following formula [2] below.

$$\text{Ash Content Test (\%)} = \frac{w_1}{w_2} \times 100\% \quad (2)$$

Where

W1 = Weight of total ash (g)

W2 = Weight of the sample (g)

Iodine Adsorption Capacity Test

A 0.5 g sample of activated carbon was treated with 100 mL of 0.1 N iodine solution and KI⁻, stirred using a magnetic stirrer for 15 minutes. Then, 25 mL of the filtrate was titrated with 0.1 N sodium thiosulfate (Na₂S₂O₃) solution until a yellow color appeared. When the yellow color began to fade, 1 mL of 1% starch solution was added as an indicator. Titration continued until the solution turned from dark brown to colorless. Iodine adsorption capacity was calculated using the following formula [3] below.

$$\text{Iodine Absorbed } \left(\frac{mg}{g}\right) = \frac{6 - \left(\frac{V \times N}{0.1}\right) \times 12.69 \times 10}{w} \quad (3)$$

Where

V = Volume of Na₂S₂O₃ used (mL)

N = Normality of Na₂S₂O₃

W = Mass of the sample (g)

RESULTS AND DISCUSSION

The activated carbon obtained in this study was evaluated in accordance with the quality requirements specified in the Indonesian National Standard (SNI) No. 06-3730-1995 for technical-grade activated carbon. The parameters examined include moisture content, ash content, and iodine adsorption capacity. The experimental work was conducted at the Chemical Engineering Laboratory, Universitas Muhammadiyah Surakarta.

Moisture Content Analysis

Moisture content analysis was performed on activated carbon samples subjected to different HCl concentrations and immersion durations. As depicted in Table 1 and Figure 2, all samples exhibited moisture content levels below the SNI maximum threshold of 15%. The lowest moisture content was recorded at 0.0050% for the 1 day immersion in 10% HCl. For the 2 days immersion, the lowest value was 0.0037% at 5% HCl, and for the 3 days immersion, it was 0.0060% at 15% HCl. A general trend of decreasing moisture content with increasing HCl concentration was observed for 2 and 3 days immersions, whereas the 1 day immersion results were more fluctuating across concentrations.

TABLE 1. Moisture content results (in percentage)

Soaking Time	HCL Concentration		
	5%	10%	15%
	%	%	%
1	0,0067	0,0050	0,0055
2	0,0037	0,0048	0,0063
3	0,0153	0,0072	0,0060

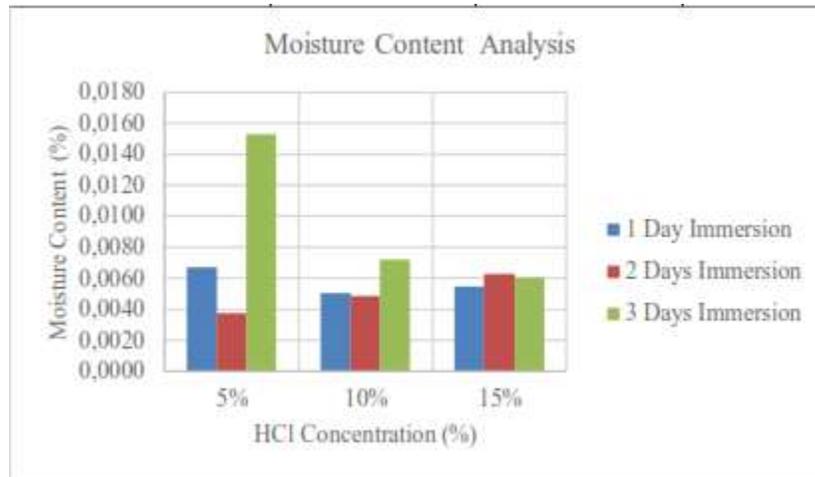


FIGURE 2. Graph of Moisture Content Results

These low moisture values indicate that the activated carbon samples successfully met the SNI standard, which is critical as excessive moisture can reduce adsorption efficiency and accelerate material degradation. Furthermore, low moisture levels imply that the available active sites on the adsorbent surface are not significantly blocked by water (Naftaly & Takwanto, 2024). The data suggest that varying HCl concentrations during activation did not lead to significant increases in moisture content, demonstrating that HCl activation at 5–15% remains a safe and effective treatment.

ANOVA two-factor with replication analysis confirmed that both immersion time and HCl concentration had statistically significant effects on moisture content. The F values for immersion time and HCl concentration were 746.2114 and 357.7915, respectively, both substantially exceeding the critical F value, with p-values of 1E-10 and 2.65E-09 ($\alpha = 0.05$). Additionally, their interaction effect was also significant, with an F value of 411.1159 and a p-value of 3.55E-10. These results emphasize the importance of optimizing the combination of immersion time and HCl concentration to achieve ideal moisture content in activated carbon.

Ash Content Analysis

The presence of metal oxides in activated charcoal is indicated by its ash content, which consists of residual minerals. During the carbonization process, these minerals cannot be volatilized (Maylani et al., 2023). According to **Table 2** and **Figure 3**, the ash content test results for the activated carbon show that all samples exhibited very low ash levels, ranging from 1.0040% to 1.0170%, which is significantly below the maximum ash content allowed by the Indonesian National Standard (SNI), set at 10%. This demonstrates that activation using HCl solution was effective in removing most of the unwanted inorganic compounds. The lowest ash content, 1.0040%, was recorded in the sample treated with 15% HCl for 3 days, indicating the effectiveness of this treatment in reducing ash content.

TABLE 2. Ash content results (in percentage)

Soaking Time	HCL Concentration		
	5%	10%	15%
	%	%	%
1	1,0116	1,0116	1,0116
2	1,0114	1,0114	1,0114
3	1,0107	1,0107	1,0107

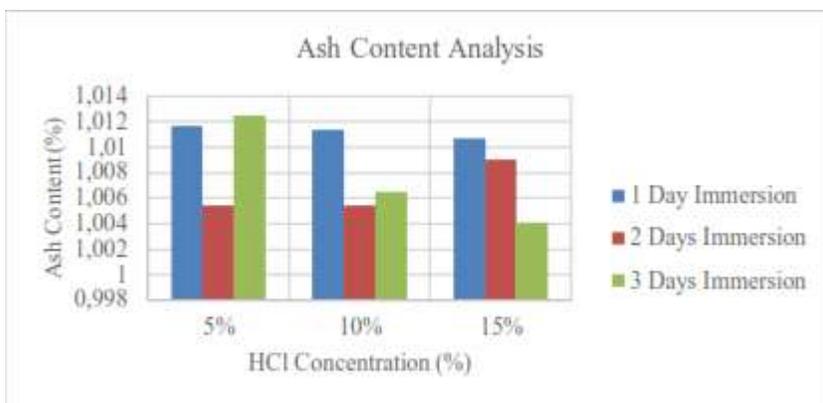


FIGURE 3. Graph of Ash Content Results

The data show that higher HCl concentrations and longer immersion durations tend to reduce ash content, although slight fluctuations are still observed. This can be explained by the fact that chemical activation with HCl helps dissolve inorganic compounds from the carbon material, and longer immersion time allows the chemical reactions to proceed more optimally. Higher HCl concentrations also contribute to accelerating the dissolution of ash-forming components. Therefore, a combination of 15% HCl concentration and 3-day immersion can be considered the most effective and optimal condition for producing activated carbon with the lowest ash content.

Based on the results of the two-factor ANOVA with replication for the ash content data, both immersion time and HCl concentration were found to have a statistically significant effect on the ash content of the activated carbon. This is indicated by the F-values for immersion time (437.5213) and HCl concentration (113.8471), both of which are far above the critical F-value of 4.256495. Moreover, the extremely low p-values (1.08E-09 for immersion time and 4.08E-07 for HCl concentration) compared to the alpha level of 0.05 confirm the statistical significance of these effects. This means that both variables exert a real influence on ash content outcomes.

The interaction between immersion time and HCl concentration also showed a significant effect, with an F-value of 257.2563, exceeding the critical value of 3.633089, and a p-value of 2.87E-09, which is well below the significance threshold. This indicates that the two factors interactively influence the final ash content. Therefore, to obtain activated carbon with low ash content that meets quality standards, an appropriate combination of immersion duration and HCl concentration must be applied during the activation process.

Iodine Adsorption Capacity

The iodine adsorption capacity is a key indicator of the microporous structure of activated carbon and its ability to adsorb small molecules. As illustrated in Table 3 and Figure 4, none of the tested samples met the minimum SNI requirement of 750 mg/g. The highest adsorption capacity recorded was 566.4816 mg/g in the sample treated with 15% HCl for 3 days, while the lowest was 8.6292 mg/g in the 1-day immersion with 15% HCl.

TABLE 3. Ash content results (in percentage)

Soaking Time	HCL Concentration		
	5%	10%	15%
	%	%	%
1	128,1690	128,1690	128,1690
2	48,4758	48,4758	48,4758
3	8,6292	8,6292	8,6292

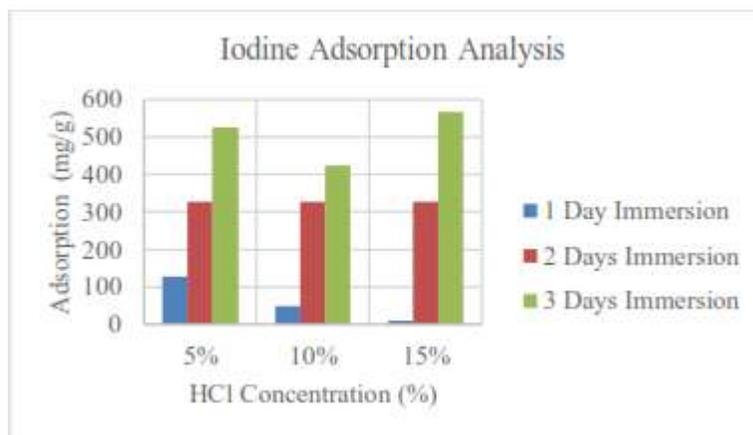


FIGURE 4. Graph of Ash Content Results

The adsorbent’s capacity to uptake small molecules was evaluated using an iodine solution. This approach also indicates the presence of a microporous structure within the adsorbent, as a higher iodine uptake generally corresponds to a more optimal adsorbent performance (Manurung et al., 2022). Based on the graph in Figure 4, all tested activated carbon samples exhibited iodine adsorption capacities significantly below the minimum standard set by the Indonesian National Standard (SNI), which is 750 mg/g. The highest observed value was 566.4816 mg/g, obtained under treatment with 15% HCl for an immersion period of three days. Conversely, the lowest value was recorded at 8.6292 mg/g under a one-day immersion using the same HCl concentration. These findings suggest that the activation conditions employed in this study were insufficient to produce activated carbon with iodine adsorption performance that meets SNI quality standards.

In terms of trends, a general increase in iodine uptake was observed with prolonged immersion time, particularly at HCl concentrations of 5% and 15%. At 5% HCl, iodine adsorption increased significantly from 128.1690 mg/g (1-day immersion) to 526.6350 mg/g (3-day immersion). Similarly, at 15% HCl, the adsorption capacity improved markedly from 8.6292 mg/g to 566.4816 mg/g after three days of immersion. These results indicate that both higher HCl concentration and longer immersion time play crucial roles in enhancing the porosity of activated carbon. Nevertheless, the resulting iodine values remain suboptimal, warranting a reevaluation of the activation parameters, such as the type of activating agent, carbon washing procedures, or stirring duration. Optimization is therefore essential to achieve iodine adsorption levels compliant with the SNI standard.

According to the results of a two-factor ANOVA with replication, both immersion time and HCl concentration exerted statistically significant effects on iodine adsorption. This is evidenced by the F-values: F(immersion time) = 555.6512 and F(HCl concentration) = 10.76744, both of which exceed the critical F-value of 4.256495. Additionally, the p-values of 3.73×10^{-10} and 0.004097, respectively, are well below the alpha level of 0.05, confirming the significance of both factors. These findings suggest that variations in immersion duration and acid concentration significantly influence the adsorption capacity of the resulting activated carbon.

Moreover, the interaction between immersion time and HCl concentration also showed a significant effect on iodine uptake, with an F-value of 12.74419 exceeding the critical F-value of 3.633089 and a p-value of 0.000948. This result highlights that the combination of these two variables has a synergistic effect on the adsorption performance. Therefore, to achieve iodine numbers that meet or exceed the minimum standard of 750 mg/g, it is necessary to simultaneously optimize both immersion conditions and acid concentration rather than evaluating them in isolation.

CONCLUSION

Based on the experimental results, all activated carbon samples exhibited moisture and ash contents well below the maximum thresholds specified by the Indonesian National Standard No. 06-3730-1995 (15% and 10%, respectively). The lowest moisture content (0,0023%) was achieved at 15% HCl concentration with 1 day of soaking, while the lowest ash content (1,0056%) was obtained with 3 days of soaking under the same acid concentration. Despite these favorable results, none of the samples met the minimum iodine adsorption capacity of 750 mg/g, with the highest recorded value being 660,33 mg/g. Statistical analysis (ANOVA) indicated that both HCl concentration

and soaking duration had significant effects on all measured properties ($p < 0,05$). Among the tested conditions, the sample treated with 5% HCl and soaked for 2 days demonstrated the best overall quality and is recommended as the optimal condition for producing activated carbon from this precursor.

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