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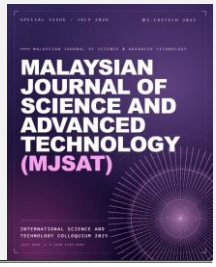


# Comparative Performance of NaOH and KOH-Activated Rice Husk Carbon for Heavy Metal Removal from Agricultural Runoff

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**ARTICLE**

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**ABSTRACT**

Removal of heavy metal from agricultural runoff by adsorption of activated carbon (AC) derived from rice husk (RH) were investigated. In this work, rice husk-derived activated carbon (RHAC) was chemically impregnated with sodium hydroxide (NaOH) and potassium hydroxide (KOH) separately and its difference in absorption efficiency and physicochemical properties were characterized using Fourier Transform Infrared Spectroscopy (FTIR) and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP OES). The removal efficiencies of heavy metal were strongly dependent on types of activation agent used. Adsorption performance analysis from KOH-activated RHAC revealed a significant removal of lead (Pb) at 65.7% and cadmium (Cd) at 85.0% respectively, attributed to the presence of stronger carbonyl and hydroxyl functional groups. Conversely, NaOH-activated RHAC performed better in achieving 60.0% for copper (Cu) and 15.8% for chromium (Cr), likely due to possible desorption or competitive adsorption. The results highlight the critical role of activation agents in influencing the adsorption behavior of heavy metals in real agricultural runoff. Overall, rice husk was successfully valorized into low-cost adsorbent thus, highlighting its potential in agricultural wastewater treatment.



## 1. INTRODUCTION

*Oryza sativa*, rice serves as a staple food component for almost fifty percent of the global population [1]. Annual rice production is anticipated to surpass 550 million tons by 2030, with rice husk (RH) constituting 25% of the harvested rice paddy [2]. The RH residue is typically discarded through on-site burning causing disposal and environmental problems. Meanwhile, the accumulated of heavy metal in water system can cause fatal diseases even at very low concentrations. Increasing concern for these problems, have been motivating researchers to transform RH into AC due to its low cost, availability, and eco-friendly [3]. Many studies have shown a significant rate of heavy metal removal by AC through this simple technology with economic feasibility.AC is commonly activated using NaOH and KOH, which play a definite role in induce the porosity thus, enhancing the adsorption efficiencies [4].

In this study, both alkaline agents are extensively used to improve pore formation and surface properties of AC. Different activation agents may vary in etching intensity, resulting in unique pore structures and surface chemistries that influence adsorption efficacy. Therefore, the comparison of NaOH and KOH activation is scientifically vital to clarify how activation chemistry influences the physicochemical features of RH-derived activated carbon. While prior research investigates either NaOH or KOH in isolation, frequently employing synthetic solutions that lack in real organic complexity of real agricultural runoff, thereby hinders direct comparison between these two activation agents. In the present study, attempts have been made to assess systematically how activation chemistry governs the physicochemical properties of RH-derived AC and evaluate their efficacy in removing heavy metals from actual agricultural runoff.

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## 2. METHODOLOGY

### 2.1 Chemical and Materials

RH was procured from a local rice mill while agricultural runoff samples were collected from Kampung Hutan Arau. Activating agents, sodium hydroxide (NaOH), Potassium hydroxide (KOH), nitric acid ($HNO_3$, 65%), and deionized water were purchased from Merck.

### 2.2 Sample Preparation

RH was cleaned and washed repeatedly in deionized water to remove dust and other impurities, then dried in oven at 105°C for 48 hours. Agricultural runoff was filtered through a 0.45µm membrane filter, acidified to pH of 2 with 65% $HNO_3$ and stored at 4°C until further use.

### 2.3 Preparation of Activated Carbon

RHAC were produced by two-stages carbonization process at temperature 500°C and 700°C for 2 hours in an N2 gas atmosphere. The char was ground, sieved (200µm), and chemically activated. To activate RH, 10g of char was mixed with 10g NaOH or KOH, dissolved in 50mL deionized water, stirred into a slurry, then dried at 105°C. The samples were rinsed several times with deionized water to get pH 6 to 7. The RH activated samples were designated as NaOH-RHAC and KOH-RHAC.

### 2.4 Fourier Transform Infrared Spectroscopy (FTIR)

Analysis of both chemically activated RH was carried out using Perkin Elmer System 2000 FTIR spectrometer (Waltham, MA, USA) with the KBr pellet method. Samples were prepared by mixing activated carbon with KBr in a ratio of 1:120 mg of samples. The sample was then pressed into pellets. The spectrum resolution used was 4 $cm^{-1}$ in a spectral range of 4000 to 400 $cm^{-1}$ with 32 scan times.

### 2.5 Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

ICP-OES were employed for the quantification of trace metals (Cu, Pb, Cr, Cd). The samples undergo baseline concentrations through 0.45µm, and acidified runoff prior analysis to prevent clogging of the instrument.

### 2.6 Adsorption Experiments

0.5g of RHAC was equilibrated with 100mL of acidified runoff. The pH was adjusted to 5-5.5 using diluted NaOH [5]. Then, the mixtures were shaken at 150 rpm for 24 hours at room temperature. Adsorption performance was assessed based on adsorption capacity (qe) and the removal efficiency (%) of the adsorbent was calculated using the following equations:

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (1)$$

$$Percentage\ Removal = \left(\frac{C_0 - C_e}{C_0}\right) \times 100 \quad (2)$$

Where:

- $C_0$ = Initial concentration of heavy metals (mg/L)
- Ce = Initial concentration after adsorption (mg/L)
- V = Volume of solution (L)
- m = Mass of activated carbon used (g)

## 3. RESULTS AND DISCUSSION

### 3.1 Physical Characteristics of Sample Preparation

The physical changes observed during sample preparation are summarized in Table 1.

**Table 1**. Summary of operational conditions, product yields, and physical characteristics at each stage of RH carbon production and activation

| Operation | Conditions | Outcome/ Observation |
|---|---|---|
| Initial washing | Deionized water rinse | Removed visible soil; colour changed from pale brown to straw-yellow |
| Oven-drying | 105°C, 24 hours | RH became friable and free flowing |
| Carbonization | Muffle furnace, 500°C, 2 hours | 65g char obtained from 200g raw husk (Yield = 32.5%); black, porous, and powdery |
| Ash determination | Proximate analysis, 750°C | 19.5g ash from 65g char; 30wt% ash; grey residue |
| Chemical activation | NaOH/ KOH, 1:1 (w/w), 700°C, 2 hours | 60g each product NaOH-RHAC cohesive, slightly plastic, KOH-RHAC hard and brittle |

Upon completion of carbonization at 500°C, about 32.5% of black and porous char was produced from 200g raw RH. High ash content (~30%) was obtained attributed to the inherent silica in RH. After subsequent chemical activation with NaOH and KOH (1:1 w/w) at 700°C for 2 hours resulted in distinct physical differences as shown in Figure 1.

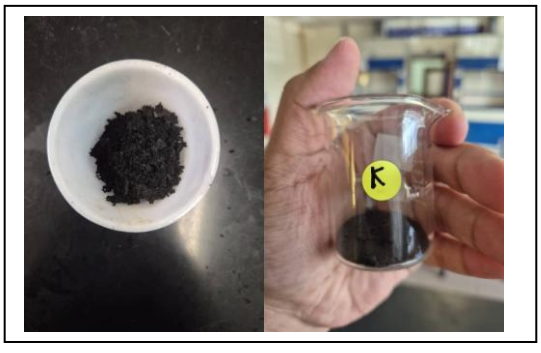


**Fig.1.** Physical appearance of NaOH-RHAC (left) & KOH-RHAC (right)

NaOH-RHAC appeared in reduced friability, while KOH-RHAC was brittle and powdery. This indicates KOH facilitated better pore development contributed to improved structural properties of AC compared to NaOH-RHAC.

*Fourier Transform Infrared Spectroscopy (FTIR) Analysis*

FTIR spectra of NaOH and KOH – RHAC are shown in (Figure 2, and Table 2).

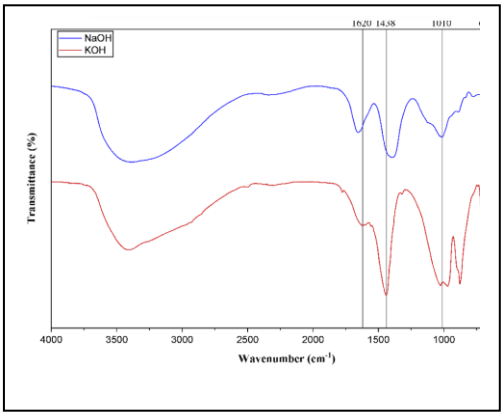


**Fig.2**. FTIR spectra of NaOH-RHAC and KOH-RHAC

**Table 2:** FTIR peak assignments and corresponding functional groups of NaOH-RHAC and KOH-RHAC

| Wavenumber ($cm^{-1}$) | Functional Group | NaOH-RHAC | KOH-RHAC |
|---|---|---|---|
| ~3430 | O-H Stretching (hydroxyl groups) | Broad | Broad |
| ~1620 | C-O Stretching (carbonyl or carboxylic groups) | Medium | Strong |
| ~1438 | Aromatic C=C or $CH_2$ bending | Weak | Moderate |
| ~1010 | C-O stretching (ether, phenol, alcohol groups) | Moderate | Strong |
| ~669,508 | Si-O / metal-O vibrations (silica content) | Present | Present |

The broad peak around ~3430$cm^{-1}$ was attributed to the bending vibration of hydroxyl(O-H) may be due to the presence of hydroxyl and alcohol. These functional groups can facilitate hydrogen bonding and act as active sites for cation exchange [6] particularly under slightly acidic conditions [7]. The adsorption peaks around 1620 $cm^{-1}$ were assigned to C=C and are more prominent in KOH-RHAC which contributes to metal ion chelation [8]. The presence of C-O stretching at ~1010 $cm^{-1}$ is also more intense in KOH-RHAC compared to NaOH-RHAC, signifying the phenolic and ether functionalities which are particularly effective in ion exchange and ligand interaction. Meanwhile, peaks at 669 and 508 $cm^{-1}$ correspond to Si-O and metal-O stretching, indicating the presence of silica-derived moieties in RH. which affect surface polarity and contribute to secondary adsorption mechanism [9].

*3.2 ICP-OES Analysis of Heavy Metal Removal*

ICP-OES is an analytical technique used for the detection of chemical elements. This technique enables precise quantification of metal ions present in the water sample before and after adsorption treatment. The results of NaOH and KOH derived AC in terms of removal efficiency and adsorption capacity, are summarized in Table 3.

**Table 3:** Initial metal concentrations and adsorption performance of NaOH-RHAC and KOH-RHAC, expressed as final concentration (mg/L), removal efficiency (%), and adsorption capacity, qe (mg/g)

| Metal | (Initial) | NaOH-RHAC (Final / % / qe) | KOH-RHAC (Final / % / qe) |
|---|---|---|---|
| Pb | 0.067 | 0.026/61.2/0.008 | 0.023/65.7/0.009 |
| Cr | 0.019 | 0.016/15.8/0.001 | 0.019/0.0/0.000 |
| Cd | 0.020 | 0.018/10.0/0.0002 | 0.003/85.0/0.003 |
| Cu | 0.050 | 0.020/60.0/0.006 | 0.028/44.0/0.004 |

Throughout the adsorption of heavy metal, KOH- RHAC showed significant performance for Pb (65.7%) and Cd (85.0%) respectively. This result is consistent with FTIR result, indicating enriched C=O and C-O functionalities that promote metal chelation. Previous work by Xia *et.al* [10] similarly reported that $Cd^{2+}$ readily forms stable inner-sphere complexes with oxygenated groups enhancing its immobilization on functional surfaces. The adsorption capacities (qe) recorded for Pb (0.009 mg $g^{-1}$) and (0.003 mg $g^{-1}$) for Cd.This result align with the range reported for chemically activated rice husk-derived carbons (typically 0.002–0.015 mg $g^{-1}$), indicating comparable performance with similar materials reported in the literature [11]. In contrast, NaOH-RHAC exhibited higher $Cu^{2+}$ removal (60.0%) compared to KOH RHAC (44.0%). This difference possibly is due to less competition or a stronger affinity of $Cu^{2+}$ to the specific functional groups present in NaOH-RHAC. About 15.8% of Cr removal was obtained by NaOH-RHAC whilst KOH-RHAC showed no net uptake. This finding may be attributed to chromium speciation in aqueous solution, where Cr exists as both Cr (III) and Cr(VI), which exhibit markedly different adsorption mechanisms. Cr (III) is predominantly cationic and can be readily adsorbed through electrostatic attraction, whereas Cr(VI) commonly forms oxyanions that display weaker affinity toward carbonaceous surfaces [12]. Adsorption capacities ($q_e$) further support these findings. For Pb and Cd KOH-RHAC achieved the highest $q_e$ values (0.009 and 0.003 mg/g) respectively, confirming its superior affinity. In contrast, Cr exhibited a negligible of 0 $q_e$, due to its poor binding affinity and possible interferences in the adsorption system.

## 4. CONCLUSION

This study shows the potential of RH for the removal of heavy metal in agricultural runoff. The adsorption efficiencies using different activating agents on RHAC has been investigated. The findings confirmed that the activation agent plays an important role in surface porosity and physicochemical properties of AC. Impregnated with NaOH and KOH results in a porous, with oxygenated functional groups (C=O and C-O) as stipulated by FTIR. The highest adsorption of Pb (65.7%) and Cd (85.0%) were obtained from RH activated with KOH. Conversely, NaOH-RHAC produced high removal capacity for Cu adsorption, resulting in a 60.0% removal rate than KOH-

RHAC which managed only 44% removal. However, Cr removal was minimal in KOH-RHAC due to variations in Cr speciation and the competitive desorption process suggesting a lack of affinity for Cr under these specific activation conditions. Despite being confined to laboratory-scale evaluation, future research should incorporate comprehensive pore structure analysis (e.g., BET and pore distribution) to further clarify structure-function correlations for the assessment of scalability and real-world performance. These developments will enable the practical application of RHAC tailored to the target pollutants in agricultural runoff.

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