RASĀYAN *J. Chem.*
Vol. 19 | No. 1 | 331-339 | January-March | 2026
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

AN ACTIVATED CARBON ADSORBENT FOR IRON METAL: A SYNTHESIS AND CHARACTERIZATION OF THE HUSK AND STALK OF CORN

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ABSTRACT

This study was conducted to synthesize and characterize activated carbon from corn stalk and husk waste using an acid activator. The synthesis was carried out through carbonization at 400 °C for 3 hours, followed by activation using HCl with five variations of activated carbon weight combinations: BJ_{100}, KJ_{100}, $BJ_{30}KJ_{70}$, $BJ_{70}KJ_{30}$, and $BJ_{50}KJ_{50}$. Characterization was performed using FTIR and SEM-EDS. The results of the FTIR spectra revealed differences in functional groups between the various combinations, which identified the diversity of chemical composition on the activated carbon surface. Morphological analysis using SEM revealed that each combination had successfully formed pores, albeit with varying numbers and sizes. Optimization tests were conducted by comparing the reduction of iron (Fe) level under varying pH conditions, concentrations, and contact times. The concentration of 10 mg/L is the optimum concentration to reduce the Fe metal concentration. The optimum pH is at pH 3 for BJ_{100} and $BJ_{30}KJ_{70}$, and at pH 4 for KJ_{100}. The ideal contact duration for activated carbon to adsorb Fe is 30 minutes. BJ_{100} and KJ_{100} followed the Freundlich model, while the activated carbon combination $BJ_{30}KJ_{70}$ has a maximal adsorption capacity of 0.5996 mg/g and adhered to the Langmuir model. These results demonstrate that activated carbon derived from corn stalks and corn husks can effectively reduce the concentration of Fe metal.

Keywords: Carbonization, Freundlich, HCl, Langmuir, Reduction

RASĀYAN *J. Chem.*, Vol. 19, No.1, 2026

INTRODUCTION

Corn is a source of carbohydrates that is widely consumed by Indonesians. Apart from being a primary food ingredient, it also plays a crucial role in Indonesia's agricultural sector. According to data from the Statistics Indonesia, the corn harvest area in Indonesia in 2023 was 2.48 million hectares, and in 2024, it increased to 2.55 million hectares.[1] This abundant corn production can lead to an increase in agricultural waste, highlighting the importance of addressing challenges in agricultural waste management, particularly in creating environmentally friendly and economically valuable solutions. Among corn plant parts, stovers, such as leaves, roots, and stalks, are the most abundant waste, despite their low digestibility.[2] The composition of corn plant waste, based on its proportion, consists of stems (50%), leaves (20%), cobs (20%), and husk (10%) of the total corn plant by-products.[3] Chemically, corn husk contains 38.2 % cellulose, 6.6 % lignin, 2,8 % ash, and 44.5 % hemicellulose.[4] Corn stalks contain cellulose, hemicellulose, lignin, and ash, comprising approximately 42.6%, 21.3%, 8.2%, and 29.4% of the total mass respectively. Based on the cellulose content of corn stalks and corn husks, these two parts have the potential to be processed into activated carbon manufacturing materials. Previous research

indicates that activated carbon from natural materials can potentially remove metals, such as Cl, Cd, and Fe, with adsorption efficiencies of 96.08% and 88.31%.[5] This research aims to determine the optimal combination of activated carbon derived from corn stalk and husk in reducing the concentration of iron metal (Fe). The biomass waste is used for metal reduction support on sustainable development purposes (in line with SDG-6: Clean water and sanitation); therefore, this research has a significant impact on environmental aspects.

EXPERIMENTAL

Materials

The raw materials used were corn stalk and husk, distilled water, HCl (Merck), H_2SO_4 (Merck), HNO_3 (Merck), $FeSO_4.7H_2O$, and methylene blue (Merck).

Equipment

Equipment includes a Memmert Oven, furnace, beaker glass (100 mL), OHAUS, Erlenmeyer flask, desiccator, funnel, volumetric flask (1000 mL), drop pipette, hot plate, measuring cylinder, magnetic stirrer, graduated pipette, watch glass, and particle size distribution (100 mesh).

Methods

Preparation of Activated Carbon

A total of 2,000 g of corn stalks and husks were washed, cut into small pieces, and dried until completely dry, then weighed and combined into the following mixtures: BJ_{100}, KJ_{100}, $BJ_{30}KJ_{70}$, $BJ_{70}KJ_{30}$, and $BJ_{50}KJ_{50}$. The materials were carbonised at 400°C for 3 hours, ground, and then sieved using a 100-mesh sieve. Activation was carried out using 3M HCl for 7 hours with a carbon-to-activator ratio of 1:10 [6]. After soaking, the samples were filtered, rinsed with distilled water until a neutral pH (6–7) was reached, as confirmed by pH paper, and then dried in an oven at 110 °C.[7]

Quality Test of Activated Carbon

Moisture Content

One gram of activated carbon was heated at 105°C for 3 hours, cooled in a desiccator, and then weighed.

Ash

Two grams of activated carbon are measured, then heated in a furnace at 550 °C for three hours until the entire sample is converted to ash. The sample is cooled in a desiccator and then weighed again.

I_2 Adsorption

The activated carbon (0.2 gram) is mixed with 25 mL of 0.1 N I_2 solution, shaken for 15 minutes, then filtered and titrated with 0.1 N sodium thiosulfate. If the colour is pale yellow, add 1% amylum indicator. Titration is stopped when the blue colour disappears. The same procedure is performed on the blank solution.

Methylene Blue

Half a gram of activated carbon is mixed with 50 mL of 50 ppm methylene blue solution, then stirred using a magnetic stirrer for 15 minutes. The concentration of filtered and remaining methylene blue is measured using UV-Vis at 665 nm[8], and its adsorption is calculated using a specific formula[9]:

$$MB_N = \frac{C_O - C_e}{M} \times V$$

Characterisation of Activated Carbon

Characterisation of activated carbon was conducted on BJ_{100}, KJ_{100}, and the optimal combination of activated carbon, using FTIR to identify functional groups in the test samples and SEM-EDS to analyze the morphological structure.

Optimisation of Activated Carbon

For every 100 mL of a 10 mg/L Fe solution was mixed with 1.25 g of adsorbent was mixed, stirred for 30 minutes, and then filtered. The resulting solution was stored for Fe (II) analysis using an Atomic Absorption Spectrophotometer (AAS). This was done for each combination.[10]

Optimisation of Activated Carbon Conditions

pH

1.25 g of activated carbon BJ_{100}, KJ_{100}, and the optimum combination were added to 100 mL of adsorbate solution with pH variations of 3, 4, 5, and 6, stirred for 30 minutes, filtered, and analysed for Fe (II) content, with a fixed concentration parameter of 10 mg/L [10].

Concentration

1.25 g of activated carbon BJ_{100}, KJ_{100}, and the optimum combination were added to 100 mL of adsorbate solution with varying concentrations of 5 mg/L, 8 mg/L, 10 mg/L, and 12 mg/L, stirred for 30 minutes, filtered, and analysed for Fe (II) content and optimum pH parameters. [10].

Contact Times

1.25 g of activated carbon BJ_{100}, KJ_{100}, and the optimum combination were added to 100 mL of adsorbate solution with optimum pH and concentration parameters, and the mixtures were contacted for 30, 45, 60, and 75 minutes. The mixtures were then filtered and analysed for Fe (II) content. [10.]

Adsorption Isotherm

Based on the results of the optimisation of activated carbon concentration, which has been measured using AAS with a concentration variation of 5 ppm, 8 ppm, and 10 ppm. Testing the appropriate adsorption isotherm model for the absorption process of iron metal ions by BJ_{100}, KJ_{100}, and their optimal combinations was carried out by calculating the Langmuir and the Freundlich equations:

Freundlich [9]: $Log\ q_e = \log K_F + (\frac{1}{n}) \log C_e$ Langmuir [9]: $\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \left(\frac{1}{q_m}\right) C_e$

Where, q_m is the theoretical maximum adsorption capacity when the carbon surface is completely covered with metal ions (mg·g^{-1}), C_e is the equilibrium concentration of metal ions in the solution (mg L^{-1}), q_e is the amount of metal ion adsorbed at the specified equilibrium (mg g^{-1}), K_L (L mg^{-1}), K_F (L mg^{-1}), 1/n (L mg^{-1}) are the constants for Langmuir and Freundlich, respectively.

RESULTS AND DISCUSSION

Preparation of Activated Carbon

An increased corn harvest can become waste that pollutes the environment if not handled properly. Utilizing corn plant waste as a base material for producing activated carbon is expected to reduce the amount of agricultural waste, particularly from corn plants. Table-1 presents the results of the activated carbon activation process following the carbonization process.

Table-1: Activation Process Result

Activated Carbon	Sample (g)	Activated Carbon (g)	Yield
BJ_{100}	100	28.0304	28%
KJ_{100}	100	23.1413	23%
$BJ_{70}KJ_{30}$	100	27.9220	28%
$BJ_{50}KJ_{50}$	100	23.0854	23%
$BJ_{30}KJ_{70}$	100	25.0095	25%

The activated carbon yield ranged from 23 to 28%. Activated carbon from BJ_{100} produces a higher yield than KJ_{100}, because BJ_{100} has a lower water content (Table-2). The combination of $BJ_{70}KJ_{30}$ gives the highest yield (28%) and has the highest water content. This is due to the hygroscopic nature of activated carbon, which causes it to absorb water vapour from the air, given its high affinity for water.[11,12]

Quality Test of Activated Carbon

The moisture content calculation aims to assess the hygroscopic characteristics of the produced activated carbon (Table-2). Elevated and diminished water content reflect the volume of water occupying the

activated carbon. Relatively high moisture increases water adsorption capacity, competing with metal adsorption.[12] The activated carbon produced in this study meets the standard, as its moisture is below 15%, except for the $BJ_{70}KJ_{30}$ activated carbon. The low moisture indicates that, at the time of carbonisation, the moisture associated with the raw material is released before activation. The reduction in moisture content is closely linked to the hygroscopic properties of the activator.[13] As the pore size increases, the surface area expands correspondingly, increasing adsorption ability. Conversely, the higher the moisture content, the activated carbon is filled with[14] ash content testing aims to determine the percentage of mineral matter that remains after evaporation during the ignition process. All activated carbon samples meet the quality standards. The higher ash content can reduce the adsorption capacity of activated carbon due to the presence of minerals such as potassium, calcium, magnesium, and sodium in the pores.[15]

Table-2: Quality Standard Test Results

Activated Carbon	Physical Quality of Charcoal (%)		Adsorption Capacity (mg/g)		Surface Area*(m^2/g)
	Moisture Content	Ash	I_2	Methylene Blue	
SNI 06-3730-1995	< 15	<10	> 750	>120	-
BJ_{100}	12.8	2.0	548.382	0.2618	969
KJ_{100}	13.0	2.0	456.716	0.2471	914
$BJ_{70}KJ_{30}$	15.9	3.1	306.453	0.1971	729
$BJ_{50}KJ_{50}$	14.2	2.9	450.186	0.2023	749
$BJ_{30}KJ_{70}$	12.5	2.4	526.600	0.2351	870

Remarks: *surface area measured from the methylene blue adsorption

The I_2 adsorption test can be a good indicator of the microporosity of activated carbon; high results indicate that more micropores are formed in the carbon produced.[16] The iodine values in this study were relatively low, thus not meeting the Indonesian National Standard (SNI). These low values may be due to the contaminants on the surface of the activated carbon or inadequate pore opening, which may be blocked by residual activator substances that have not been thoroughly cleaned. [17,18] The activated carbon produced has a methylene blue adsorption result that is still below standard.

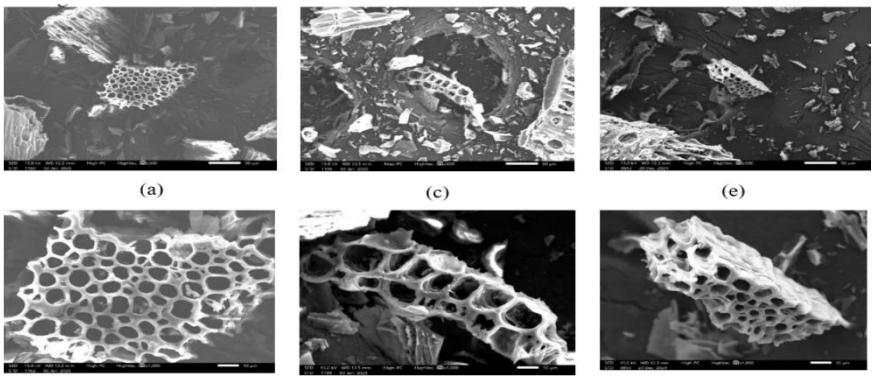


Fig.-1: (a) 300x, (b)1000x $BJ_{100,}$ (c) 300x, (d) 1000x KJ_{100}, (e) 300x, (f) 1000x $BJ_{30}KJ_{70}$

The reduction in methylene blue colour after adsorption reflects the pores formed and indicates the ability of activated carbon to adsorb high-molecular substances, such as dye molecules.[19] BJ_{100} shows a higher adsorption capacity than KJ_{100}, and the $BJ_{30}KJ_{70}$ combination shows a higher than the other combinations. These results align with the larger surface area of BJ_{100} compared to KJ_{100}, and $BJ_{30}KJ_{70}$ has better quality test results. Activated carbon with a smaller surface area is likely to be affected by micropores that inhibit the absorption of methylene blue.[20] In general, commercial activated carbon that is often used has a surface area of around 800-1500 m^2/g [19], and the results of this study fall within that range.

BJ_{100} has many pores of varying sizes. In contrast, KJ_{100} activated carbon appears to have fewer pores, with larger sizes. $BJ_{30}KJ_{70}$ activated carbon also shows considerable pore development. The composition

of the elements on the surface of activated carbons BJ_{100}, KJ_{100}, and $BJ_{30}KJ_{70}$ was determined through EDS analysis. The EDS results in Table-3 indicate that activated carbon possesses the highest percentage of the element C. The element is generated by the decomposition of organic substances and the oxidation of metals.[21]

Table-3: SEM-EDS Spectrum Interpretation

Element	Mass (%)			Atom (%)		
	BJ_{100}	KJ_{100}	$BJ_{30}KJ_{70}$	BJ_{100}	KJ_{100}	$BJ_{30}KJ_{70}$
C	75.80	81.78	82.05	88.82	86.39	88.76
O	2.87	16.21	10.27	2.52	12.85	8.34
Mg	0.27	-	0.37	0.16	-	0.27
Al	0.55	0.18	0.11	0.35	0.08	0.05
Si	2.79	0.16	0.38	0.27	0.07	0.18
Cl	19.84	1.67	6.22	7.88	0.60	2.28
K	-	-	0.15	-	-	0.05
Ca	-	-	0.45	-	-	0.15

The carbonization process at temperatures < 900 °C will not yield pure carbon content. Conversely, at temperatures greater than 900 °C and carried out in a vacuum, carbon with higher purity is achieved.[22]

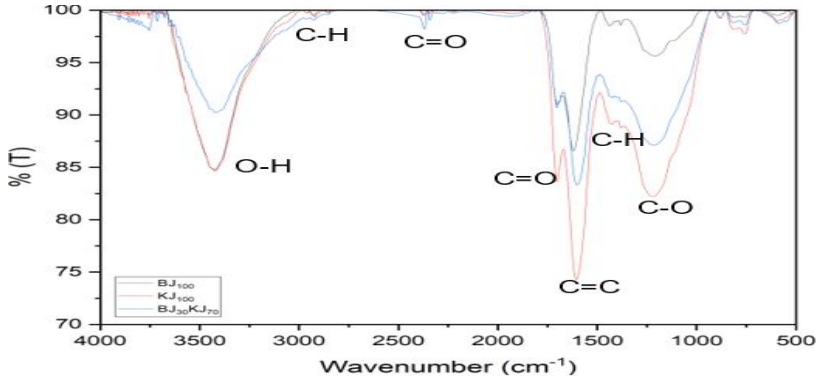


Fig.-2: FTIR Spectrum of the Sample

There are functional groups that appear at wave numbers 3423.13 cm^{-1} (BJ_{100}), 3422.77 cm^{-1} (KJ_{100}), and 3421.14 cm^{-1} ($BJ_{30}KJ_{70}$), reflecting the O-H vibrations of the hydroxyl group.[9] The presence of O-H groups indicates the presence of cellulose structures that are known to have great potential as adsorbents, due to the bound OH-groups' ability to interact with adsorbate components.[23] Other peaks at wave numbers 2924.07 cm^{-1}(BJ_{100}) and 2922.54 cm^{-1} (KJ_{100}) correspond to C-H vibrations from CH_3 and CH_2 groups.[9] In addition, wavenumbers 1702.45 cm^{-1} (BJ_{100}), 1702.88 cm^{-1} (KJ_{100}), and 1703.05 cm^{-1} ($BJ_{30}KJ_{70}$) identify the presence of C=O bonds from acetyl groups in lignin and ester groups in hemicellulose. Other wave numbers are 1619.80 cm^{-1} (BJ_{100}), 1604.77 cm^{-1} (KJ_{100}), and 1600.60 cm^{-1} ($BJ_{30}KJ_{70}$), which are aromatic C=C functional groups, a characteristic feature of carbon materials. The next peaks at wavenumbers 1208.94 cm^{-1} (BJ100), 1221.48 cm^{-1} (KJ100), and 1214.20 cm^{-1} ($BJ_{30}KJ_{70}$) originate from the stretching of C-O bonds within lignin.[9] The functional groups of activated carbon, which obtained O–H, C=O, and C–O stretching vibration, play an essential role as an absorbent of pollutants.[24]

Optimisation of Fe Removal using Activated Carbon

Activated carbon produced from a mixture of corn stalks and corn husks, namely $BJ_{70}KJ_{30}$, $BJ_{50}KJ_{50}$, and $BJ_{30}KJ_{70}$, showed similar variations in results. Among the three combinations, the $BJ_{30}KJ_{70}$ combination, which consists of 30 g of corn stalk waste and 70 g of corn husk waste, showed better result compared to the other combinations (Table-2). This was supported by the greater surface area of $BJ_{30}KJ_{70}$, which allowed for better adsorption capabilities than the other combinations. However, the I_2 and methylene blue adsorption values still did not meet the quality standards. Overall, neither the combined activated carbon nor the one consisting of one main ingredient showed significant differences in results. Figure-3

shows that the adsorption of BJ_{100} activated carbon decreases from pH 3 to 5 and then rises again at pH 6, although it remains lower than at pH 3. Conversely, KJ_{100} shows an increase from pH 3 to 4, followed by a decrease up to pH 6. . As for $BJ_{30}KJ_{70}$, the adsorption process continued to experience a decrease in the percentage of Fe (II) metal adsorption from pH 3 to pH 6. Optimal adsorption occurs at pH 3 for BJ_{100} (0.563 mg/g) and $BJ_{30}KJ_{70}$ (0.667 mg/g), and at pH 4 for KJ_{100} (0.597 mg/g). Optimal adsorption occurs at pH 3 for BJ_{100} (0.667 mg/g) and $BJ_{30}KJ_{70}$ (0.563 mg/g), and at pH 4 for KJ_{100} (0.585 mg/g). The decrease in adsorption is caused by competition between metal ions and H^+ ions for active sites, and repulsive forces between the positively charged adsorbent surface and metal ions. At concentrations of 5–10 mg/L, the adsorption capacity increases; however, it decreases significantly at 12 mg/L, as the saturation point is reached due to the limited adsorption space. Excessively high adsorbate concentrations can also reduce adsorption efficiency.

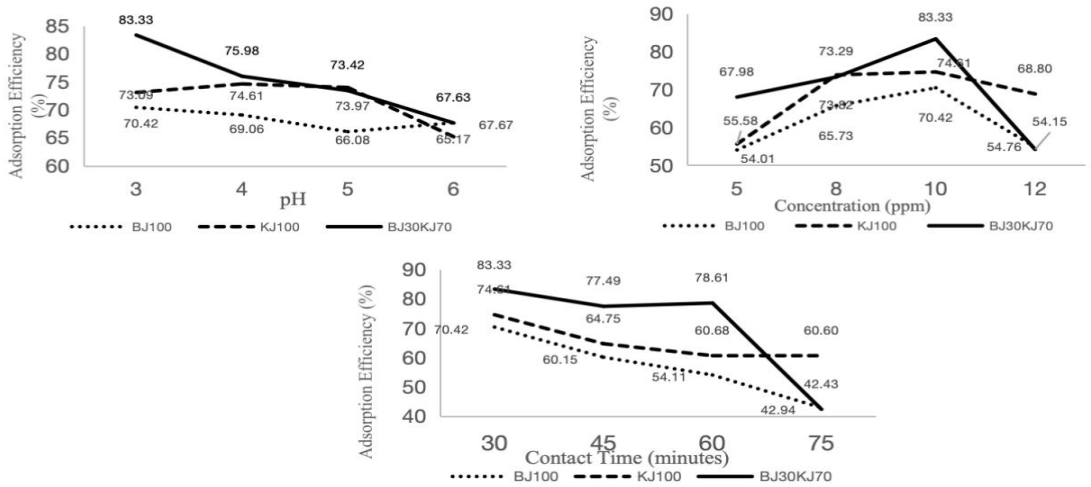
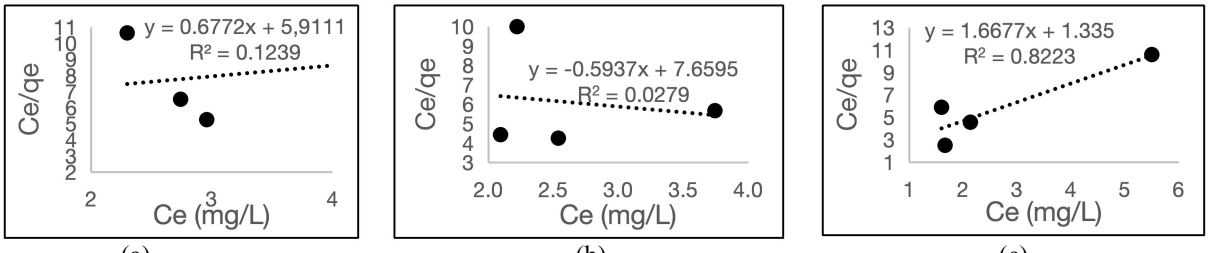


Fig.-3: Graph of pH, Concentration, and Contact Time; Effect on Adsorption Efficiency

The three activated carbons (BJ_{100}, KJ_{100}, $BJ_{30}KJ_{70}$) showed a rapid increase in the first 30 minutes because the adsorbent surface was still clean. [25] After reaching a contact time of 30 minutes, the adsorption ability began to decrease, as indicated by the reduced weight of Fe^{2+} adsorbed. This occurs because the concentration of Fe^{2+} ions is insufficient to interact with the adsorbent. This is attributed to the limited number of active sites on the activated carbon.[26] The adsorption process will cease when the interaction between metal ions and the adsorbent reaches saturation. In this condition, there is a rapid and continuous collision between the adsorbent and adsorbate particles. In addition, during the adsorption process, the pores of the adsorbent can shrink, allowing the adsorbate to be released again.[27] Based on the results of this study, the optimum contact time for the three activated carbons was 30 minutes.

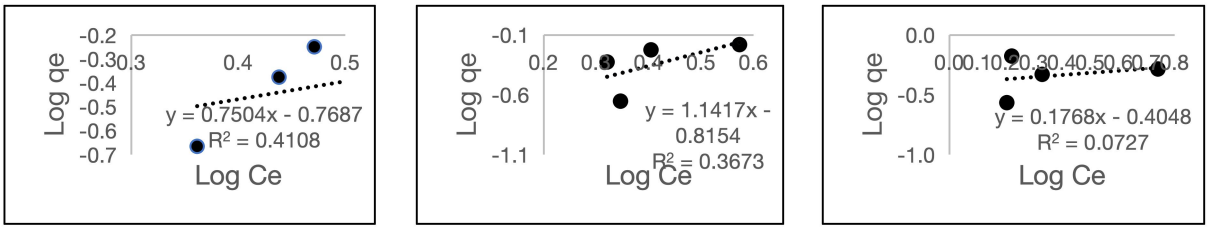
Adsorption Isotherm

The adsorption isotherm was determined by converting the Langmuir and the Freundlich equations into linear form, followed by calculating the adsorption capacity of activated carbon. The equilibrium model is determined based on a high correlation coefficient (r) value.[28]



(a) (b) (c)

Fig.-4: Linearization Curves of Langmuir (a) BJ_{100}, (b) KJ_{100}, (c) $BJ_{30}KJ_{70}$



(b) (b) (c)

Fig.-5: Linearization Curves of Freundlich (a) BJ_{100}, (b) KJ_{100}, (c) $BJ_{30}KJ_{70}$

Based on the R^2 values in Fig.-4 and 5, the Freundlich isotherm equation is more suitable for describing the adsorption of Fe^{2+} by BJ_{100} and KJ_{100} activated carbon derived from corn cobs and husks. The Freundlich model assumes that adsorption occurs on a heterogeneous adsorbent surface, with each molecule having a different adsorption potential. Relatively weak Van Der Waals forces occurred between the adsorbate and adsorbent. [28]

Table-4: Langmuir and Freundlich Constant Values

Activated Carbon	Freundlich	Langmuir
BJ_{100}	K_F = 0.1703, 1/n = 0.7504, n = 1,3326	K_L = 0.5421, q_m = 1.4766
KJ_{100}	K_F = 0.1529, 1/n = 1.1417, n = 0,8759	K_L = 1.2492, q_m = 0.1684
$BJ_{30}KJ_{70}$	K_F = 0.3937, 1/n = 0.1768, n = 5,656	K_L = 1.2492, q_m = 0.5996

The Freundlich and Langmuir constant values are described in Table-4. The Freundlich isotherm has K_F values of 0.1703 (BJ_{100}) and 0.1529 (KJ_{100}) and 1/n values of 0.7504 (BJ_{100}) and 1.1417 (KJ_{100}). The k value indicates the relative Freundlich adsorption capacity, and 1/n describes the adsorption intensity. The adsorption mechanism is categorised as favourable if the 1/n value is smaller than 1 or close to 0, indicating a strong interaction between the adsorbate and adsorbent. From the data processing, a 1/n value greater than 1 was obtained, namely 1.1417 (KJ_{100}). This value indicates a weak interaction between the KJ_{100} adsorbent and Fe^{2+} ions in the adsorbate, which can be classified as an unfavourable adsorption mechanism. . The maximum capacity value (q_m) is 1.4766 for BJ_{100} and 0.1684 for KJ_{100}. Meanwhile, $BJ_{30}KJ_{70}$ activated carbon is in line with the Langmuir isotherm equation, which assumes that adsorption occurs on a specific and homogeneous adsorbent surface, allowing only a monolayer of adsorbate to form on the adsorbent surface.[29] The use of activated carbon from agricultural waste, such as corn stalks and husks, has the potential to be a sustainable precursor that can support sustainable development, SDG number 6 regarding sanitation and clean water, so it is hoped that it will be able to have an impact on improving human health and the environment.

CONCLUSION

Activated carbon derived from corn stalks and corn husks can reduce the concentration of iron metal (Fe). The optimum combination of activated carbon is $BJ_{30}KJ_{70}$, with an efficiency of reducing the concentration of Fe (II) ions by 67.65%, from an initial concentration of 10 mg/L Fe metal to 3.235 mg/L. The optimization process yielded optimal pH values of 3 for BJ_{100} and $BJ_{30}KJ_{70}$, and 4 for KJ_{100}, along with an optimal concentration of 10 mg/L and a contact time of 30 minutes. The adsorption isotherm model of $BJ_{30}KJ_{70}$ activated carbon follows the Langmuir model, which refers to monolayer adsorption with a maximum adsorption capacity of 0.5996 mg/g. In comparison, BJ_{100} and KJ_{100} follow the Freundlich model, which refers to multilayer adsorption. This study supports SDG 6 for water and sanitation.

ACKNOWLEDGEMENTS

The authors are grateful for the support provided by all parties, including the Chemistry Laboratory of the Faculty of Mathematics and Natural Sciences at Pakuan University, Bogor, and the National Research and Innovation Agency (BRIN).

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. The authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID IDs, given below:

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Cite this article as: Ani Iryani *et al.*, *Rasayan J. Chem.*, 19(1), 331-339(2026), https://doi.org/10.31788/RJC.2026.1919513.

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