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Adsorption of Some Heavy Metals and Dyes in Aquatic Environment by Using Sludge Biochars Obtained at Different Pyrolysis Temperatures

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ABSTRACT
The complex mixture of organic matter, metals, and potentially harmful substances found in sewage sludge, a wastewater treatment byproduct, presents risks to human health and can foster antibiotic resistance. Numerous studies have investigated the influence of pyrolysis temperature on biochar characteristics and performance. Investigating the effects of pyrolysis temperature on sludge-derived biochar's properties and the fate of heavy metals and azodyes throughout the process is the specific aim of this study. This reduction primarily stems from the volatilization of carbon, the major constituent, at temperatures around 600°C. In experiments evaluating contaminant removal, biochar produced at 500°C (Biochar 500) was used at a concentration of 5g/ L, with a contact time of 30 minutes, an average solution temperature of 23.4°C, and an average pH of 7.94. Under these conditions, the average removal percentage of trace metals ranged from 80.0% for selenium (Se) and arsenic (As) to 99.79% for cobalt (Co). The removal efficiency for methylene blue (MB) ranged from 99.69 to 99.96%. Optimal removal of the harmful substances was achieved with a specific adsorbent dosage (5mg/ L), a controlled solution pH, and a 30-minute contact time. The study demonstrates that even small amounts of the adsorbent are highly effective in removing MB from water. The Langmuir adsorption model predicted a maximum MB adsorption capacity of 965mg/ g, which aligns with experimental results obtained from using MB to treat wastewater contaminated with methylene blue.

INTRODUCTION

Wastewater treatment generates significant volumes of sewage sludge, a complex mixture of organic matter, nutrients, heavy metals, pathogens, and antibiotics. This composition results in considerable environmental and health challenges. The ongoing trends of urbanization and population growth are intensifying the risks associated with this waste stream, necessitating effective management. A critical issue is the potential for antibiotic-resistant microorganisms in the sludge to spread disease and worsen the global antibiotic resistance crisis, especially when the sludge is incorporated into agricultural soils. Furthermore, the presence of heavy metals and other toxins poses a risk of contaminating soil and water, impeding safe disposal and beneficial reuse **(Akdag, *et al.*, 2018; Ateshan, *et al.*, 2019)**. Pyrolysis offers a promising solution by converting sewage sludge into biochar, a stable carbon form that can store carbon in soil, promoting carbon sequestration and reducing greenhouse gas emissions. Higher pyrolysis temperatures intensify dehydration, decarboxylation, and decarbonylation reactions, lowering the hydrogen-to-carbon (H/C) and oxygen-to-carbon (O/C) ratios in the resulting biochar, which are critical indicators of its chemical composition and reactivity **(Ateshan, *et al.*, 2020; Akpasi, *et al.*, 2023**).

Biochar with lower hydrogen-to-carbon (H/C) and oxygen-to-carbon (O/C) ratios exhibits greater charring and stability, which in turn makes it more beneficial for soil improvement and carbon sequestration. By controlling pyrolysis conditions and selecting specific feedstocks, these H/C and O/C ratios can be manipulated to tailor biochar properties for diverse applications, including soil amendment, water treatment, and carbon sequestration. Notably, studies suggest that biochar with an H/C ratio under 0.3 is effective in reducing N_2O emissions (**Dinc *et al.*, 2020; Ateshan & Misnan, 2025a)**, and biochar with a high proportion of aromatic carbon and low O/C and H/C ratios proves most effective for sequestering carbon in soil (**Goktepeli *et al.*, 2023**).

The pyrolysis temperature exerts a substantial influence on the physical, chemical, and functional attributes of biochar, thereby enabling its customization for specific intended uses. As an illustration, an escalation in pyrolysis temperature results in an expansion of biochar's specific surface area and micropore volume. For instance, biosolids-derived biochar exhibited a progression in specific surface area from 48.25 $m^2 \cdot g^{-1}$ to 65.74 $m^2 \cdot g^{-1}$ and in micropore volume from 0.0313 $cm^3 \cdot g^{-1}$ to 0.0369 $cm^3 \cdot g^{-1}$ upon increasing the temperature from 700 to 900 °C (**He *et al.*, 2018; Goktepeli, 2023**).

As pyrolysis temperatures increase from 400 to 800°C, biochar derived from waste biomass experiences an increase in both BET surface area and zeta potential. This enhancement in surface properties and thermal stability is a direct consequence of higher temperatures. The chemical characteristics of biochar are also temperature-dependent. Higher pyrolysis temperatures tend to lower yields and decrease nitrogen content, while simultaneously increasing carbon concentration and aromaticity. This shift occurs because nitrogen compounds volatilize, and the intensified charring process promotes carbon retention and reduces water-soluble carbon. Temperature also impacts biochar's acid-base properties; higher temperatures reduce acidic functional groups and increase basic functional groups, thereby influencing its adsorption capacity. The optimal pyrolysis temperature is not universal but depends on the biochar's intended use. For instance, biochar produced at lower temperatures (around 400°C) is more suitable as a microbial carrier, beneficial for agricultural applications like plant growth and nitrogen fixation. Pyrolysis above 400°C effectively inactivates pathogens and antibiotic-resistant bacteria, making the biochar safer. A key challenge is therefore to match the pyrolysis temperature to the specific biochar application, which often requires monitoring biochar properties to achieve the desired characteristics (**Kane *et al.*, 2022; Ateshan & Misnan, 2025b**).

Pyrolysis offers a promising and viable method for sludge recycling. This process effectively reduces sludge volume, eliminates pathogens, and converts organic components into valuable bio-oil, pyrolysis gas, and biochar. Sludge-derived biochar, a nutrient-rich, porous material containing nitrogen, phosphorus, and potassium, shows potential as a soil amendment. When applied, it can enhance soil properties by lowering bulk density, increasing porosity and water retention, improving nutrient retention, and adsorbing pollutants, all of which contribute to better plant growth. The effectiveness of sludge-derived biochar in revitalizing degraded soils is significantly impacted by its elemental makeup, physicochemical properties, and pore structure, necessitating further detailed investigation into these aspects. Moreover, the presence of heavy metals in sludge-derived biochar requires research to understand and mitigate environmental release risks **(Ateshan, 2025)**. Ultimately, the pyrolysis temperature employed is a key factor in shaping the final characteristics of the biochar produced (**Li *et al.*, 2022**).

Methylene blue (MB), a widely used textile dye, also serves medical purposes as a stain, antiseptic, and diagnostic agent. However, its extensive discharge into water bodies, estimated at 85,000 tons annually along with other dyes and pigments, poses a significant environmental challenge and a threat to human and animal health, causing symptoms like irritation and gastrointestinal distress. This necessitates its removal from wastewater, making it a critical area for environmental and public health efforts. MB is often used in research to evaluate adsorption and photocatalysis processes. A key challenge in these studies is the aggregation of MB molecules in solution, a process influenced by concentration and temperature. This aggregation can affect process efficiency, as adsorbents optimized for MB monomers may prove ineffective against larger aggregates due to pore size limitations (**Al-Wabel *et al.*, 2023**). Spectroscopic methods for determining methylene blue (MB) concentration are significantly hindered by its aggregation, as various MB species display differing optical spectra. The self-aggregation of MB in water, leading to dimer formation, has been thoroughly investigated. These studies typically aim to establish the visible light absorption spectra of monomers and dimers, as well as the thermodynamic parameters governing their equilibrium (predominantly the equilibrium constant at room temperature, and less often, enthalpy and entropy). However, our prior findings indicate considerable uncertainty within the published molar attenuation coefficients and thermodynamic parameters. This uncertainty is attributed to the common but flawed assumption that the monomer's spectroscopic behavior is temperature-independent. This assumption is incompatible with the observed phenomenon where the monomer spectrum's maximum attenuation coefficient, obtained through extrapolation at extremely low MB concentrations (6.3×10^{-7} M), diminishes slightly with increasing temperature (**Amin *et al.*, 2024**). This study investigates the influence of pyrolysis temperature on sludge-derived biochar characteristics (**Haider, 2025)**. It further analyzes how pyrolysis temperature affects the behavior and transformation of heavy metals and azodyes during the pyrolysis process."

MATERIALS AND METHODS

- **Sampling**

From a wastewater treatment plant in west Baghdad, 2000g of fresh sewage sludge was collected. The samples were then dried at 85°C for 2 hours and cooled to room temperature.

- **Pyrolysis trials**

The sewage sludge samples were pyrolyzed at different temperatures. The first sample was pyrolyzed at 300^0C; the second was conducted at 400 ^{0}C, the third at 500 ^{0}C, finally the fourth sample pyrolysis was conducted at 600^0C. The pyrolysis time was 3.0 hours, and the heating rate was 25^0C/ min; the pyrolysis process was conducted using electric furnace (JSMF-120HT, Korea) in nitrogen gas environment to inhibit the oxygen.

- **Chemical characteristics of sludge and biochar (APHA, 2024)**

- The Thermo Scientific FLASH 2000 CHNS/O Analyzers (USA) were employed to determine the elemental analysis (CHNS) of the sludge and biochar.
- pH values were determined for the prepared samples (sample/water, 1:20, w/v), and the analysis were conducted using pH meter (PHS-25 Bench Top PH Tester).
- The BET (Brunauer-Emmett-Teller) surface area, pore size of the samples was analyzed with Analyzer (AMI Micro-300, UK).
- Trace metals (Ni, Cr, Cd, Pb, Zn, Se, As, Fe, Mn, Co, Cu) were determined using ICP/MS (ICP-MS Agilent 7500ce Inductively Coupled Plasma Mass Spectrometer, USA); the analysis process was conducted according to **EPA (2007)**.

- **Water analysis (APHA, 2024)**

- Trace metals (Ni, Cr, Cd, Pb, Zn, Se, As, Fe, Mn, Co, Cu) were determined using ICP/MS (ICP-MS Agilent 7500ce Inductively Coupled Plasma Mass Spectrometer, USA); the analysis process was conducted according to **EPA (2007)**.
- Methylene blue dye (MB) was determined using $\lambda = 665$ nm a Jenway 6300 UV/VIS spectrophotometer Spectrophotometer (Jenway 6300, USA).

- **Statistica analysis**

SPSS 19.0 was used for data analysis in the present study.

RESULTS AND DISCUSSION

Suldge and biochar

The results obtained from the elemental analysis of sludge and biomass CHNS for sludge and biomass were monitored. The percentages of elements decreased with increasing pyrolysis temperature, as the percentages of carbon decreased from 27.0 to 18.4% in the case of sludge and biomass at 600°C, as is the case with the elements hydrogen, nitrogen and sulfur, as shown in Table (1) and Fig. (1).

While similar observations have been made in other studies, they often focused on shorter residence times. A wide array of factors, including pyrolysis temperature, gas residence time, feed rate, particle size, reaction atmosphere, sludge composition, and catalysts, all impact pyrolysis yield and product distribution. The inherent variability in sewage sludge composition contributes to differing pyrolysis yields and product characteristics. Activated sludge mainly consists of proteins, carbohydrates, and lipids, which decompose at temperatures of 200– 635°C, 209– 800°C, and 164– 497°C, respectively, falling within the temperature range used in this study. The sludge also contains celluloses, hemicelluloses, and lignin, which decompose thermally between 450–600°C. Other studies have reported comparable gas yields, around 34 wt%, when subjected to high temperatures (850°C) and residence times ranging from 1 to 2 hours. The resulting syngas, primarily a mixture of CO_2, CO, H_2, and CH_4 (constituting up to 90 vol%), also contains hydrocarbons, nitrogen, and other minor constituents. Higher temperatures generally lead to a reduction in CO_2 content and an increase in the volumetric percentage of H_2, indicating that temperature significantly influences the syngas composition (**Ali, *et al.*, 2013; Al-Addous, *et al.*, 2017**).

Table 1. Characteristics of sludge and biochar

No	Parameter	Elemental analysis (%)			
	Sludge & Biochar	C	H	N	S
1	Sludge	27.2	3.66	4.8	0.28
2	Biochar (300 ^{0}C)	23.8	2.14	3.9	0.19
3	Biochar (400 ^{0}C)	21.6	1.89	3.3	0.16
4	Biochar (500 ^{0}C)	19.6	1.64	2.8	0.12
5	Biochar (600 ^{0}C)	18.4	1.1	2.24	0.09

The results for the biocar samples show that the pH increases with increasing pyrolsis temperature from 300 to 600 degrees Celsius, where it increases from 7.46 to 8.6 and also the ash percentages increases in biochar samples with increasing pyrolysis temperature from 300 to 600 ^{0}C, as shown in Table (2) and Fig. (2).

Table 2. Characteristics of sludge and biochar

No	Parameter / Sludge & Biochar	pH (-)	Ash (%)	H/C (-)
1	Sludge	7.05	47.2	0.13
2	Biochar (300 ^{0}C)	7.46	58.1	0.09
3	Biochar (400 ^{0}C)	7.9	68.2	0.09
4	Biochar (500 ^{0}C)	8.3	71.6	0.08
5	Biochar (600 ^{0}C)	8.6	76.3	0.06

The results for the biocar samples show that the surface area increases with increasing pyrolsis temperature from 300 to 600 degrees Celsius, where it increases from 7.31 to 11.23m^2/ g and also the pore size and pore width increase in biochar samples with increasing pyrolysis temperature from 300 to 600^{0}C, as shown in Table (3) and Fig. (3).

Table 3. Characteristics of sludge and biochar

No	Parameter / Sludge & Biochar	Surface area (m^2/g)	Pore size (cm^3/g)	Pore width (nm)
1	Sludge	4.12	0.011	58.4
2	Biochar (300 ^{0}C)	7.31	0.038	24.7
3	Biochar (400 ^{0}C)	8.86	0.047	21.2
4	Biochar (500 ^{0}C)	9.82	0.051	17.6
5	Biochar (600 ^{0}C)	11.23	0.069	14.1

Table (4) shows the levels of trace metals, including Ni, Cr, Cd, Pb, Zn, Se, As, Fe, Mn, Co, and Cu, in both sludge and biochar samples. The concentration of these metals decreased with increasing pyrolysis temperature. An increase in pyrolysis temperature led to a loss of organic mass, which in turn resulted in the enrichment of heavy metals in the biochar. The concentrations of various heavy metals in the biochar derived from sewage sludge (SSB) were higher than those found in the original sewage sludge.

Theoretically, metals such as Cu, Pb, Cr, Ni, and Zn are not expected to be released at temperatures up to 750°C, given the vapor pressures of their typical compounds (sulfides, carbonates, hydroxides, and organically bound forms). However, the high gas velocities produced during pyrolysis can entrain metal-containing particles, necessitating the use of effective particle collection systems. In contrast, cadmium (Cd) is known to volatilize at a pyrolysis temperature of 625°C. Studies have demonstrated that extending the residence time from 1 to 4 hours at this temperature reduced the Cd content in carbonized sludge from 52 to 18%. Furthermore, a temperature of 750°C with a 1-hour residence time led to 95% Cd evaporation. Other research has indicated that higher pyrolysis temperatures reduce the concentration of heavy metals in biochar while simultaneously increasing their concentration in the evolved gas **(Alyun *et al.*, 2018; Chen *et al.*, 2021)**.

Table 4. Trace metals of sludge and biochar

No	Sludge & Biochar / Parameter	Unit	Sludge	Biochar (300 ^{0}C)	Biochar (400 ^{0}C)	Biochar (500 ^{0}C)	Biochar (600 ^{0}C)
1	Ni	mg/kg	1.38	0.48	0.33	0.24	0.21
2	Cr	mg/kg	3.41	0.52	0.26	0.19	0.11
3	Cd	mg/kg	0.17	0.14	0.12	0.093	0.058
4	Pb	mg/kg	0.46	0.24	0.13	0.085	0.047
5	Zn	mg/kg	38.6	24.8	16.2	11.3	7.2
6	Se	mg/kg	0.06	0.02	0.01	0.01	0.01
7	As	mg/kg	0.074	0.03	0.01	0.01	0.01
8	Fe	mg/kg	112.2	71.4	33.1	24.3	19.6
9	Mn	mg/kg	14.2	6.6	3.4	2.1	1.3
10	Co	mg/kg	0.042	0.03	0.01	0.01	0.01
11	Cu	mg/kg	23.5	17.4	13.5	9.4	5.5

Industrial wastewater treatment

In the present study, the biochar samples were evaluated for using in trace metals removal from industrial textile wastewater. The biochar samples used in the experiments was the biochar 500^{0}C (5 g/l dose), and the contact time was 30min and the solution temperature in average was 23.4^{0}C, and the pH was 7.94 in average. The removal percentage of trace metals in average ranged from 80.0% in case of Se, As to 99.79% in case of Co, as shown in Table (5) and Fig. (4).

Table 5. Average values of trace metals of industrial wastewater & treated wastewater that treated by biochar (500^0C)

No	Samples / Parameter	Unit	Raw industrial wastewater (textile industries)	Treated wastewater	Removal (%)
1	Ni	mg/l	0.031	0.003	90.32
2	Cr	mg/l	0.075	0.013	82.67
3	Cd	mg/l	0.006	0.001	83.33
4	Pb	mg/l	0.15	0.006	96.00
5	Zn	mg/l	0.23	0.03	86.96
6	Se	mg/l	0.005	0.001	80.00
7	As	mg/l	0.005	0.001	80.00
8	Fe	mg/l	0.43	0.062	85.58
9	Mn	mg/l	0.02	0.001	95.00
10	Co	mg/l	1.89	0.004	99.79
11	Cu	mg/l	0.096	0.003	96.88

In the present study, the biochar samples were evaluated for using in methylene blue dye (MB) removal from industrial textile wastewater. The biochar samples used in the experiments was biochar 300^0C (5g/ l dose), biochar 400^0C (5g/ l dose), biochar 500^0C (5g/ l dose), and biochar 600^0C (5g/ l dose), and the contact time was 30min and the solution temperature in average was 23.4^0C, and the pH was 7.94 in average. The removal percentage of MB in average ranged from 99.91 to 99.96%, as shown in Table (6) and Fig. (5).

Biochar has garnered significant interest as a methylene blue (MB) adsorbent due to its lower cost and abundant raw material sources compared to activated carbon. Studies have demonstrated the effectiveness of HNO_3-modified biochar derived from reed biomass (ABC) for MB adsorption. The adsorption mechanisms of biochar derived from diverse feedstocks, including pine wood, pig manure, and cardboard, have been the subject of research. Additionally, the efficacy of hickory biochar, pyrolyzed at varying temperatures, in MB removal has been assessed. The adsorption mechanism of biochar originating from the co-pyrolysis of solid waste has also been investigated. While these investigations underscore the viability of biochar from solid waste biomass, a critical limitation often observed is extremely low maximum adsorption capacities, thereby posing a significant challenge in developing high-capacity biochar from novel raw materials **(Rafiq *et al*., 2016; Tomczyk *et al*., 2020)**.

Table 6. Average values of MB of industrial wastewater & treated wastewater

No	Samples / Treated by:	Unit	Raw industrial wastewater (textile industries)	Treated wastewater	Removal (%)
1	Biochar (300 ^{0}C)	mg/l	29.8	0.093	99.69
2	Biochar (400 ^{0}C)	mg/l		0.071	99.76
3	Biochar (500 ^{0}C)	mg/l		0.026	99.91
4	Biochar (600 ^{0}C)	mg/l		0.013	99.96

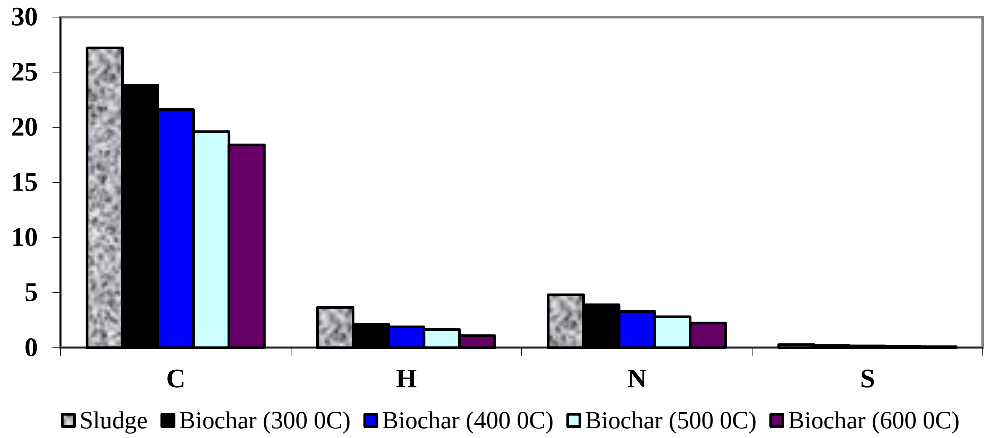


Fig. 1. Characteristics of sludge and biochar (C, H, N, S %)

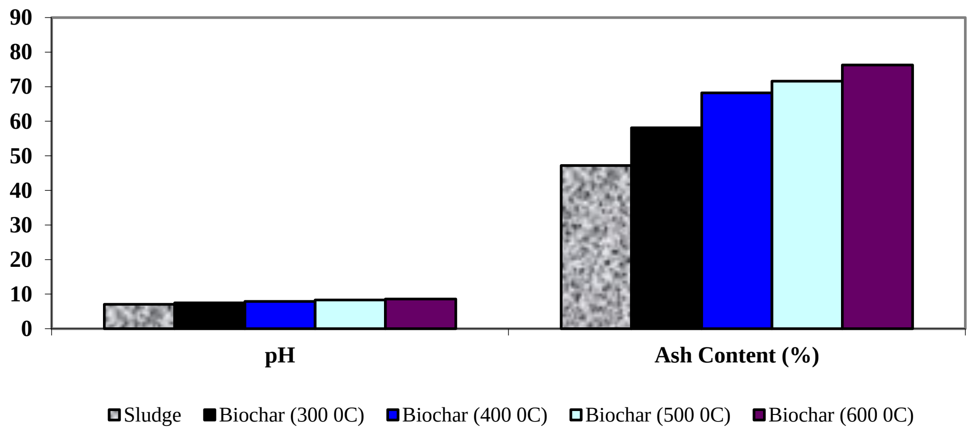


Fig. 2. Characteristics of sludge and biochar (pH, Ash Content %)

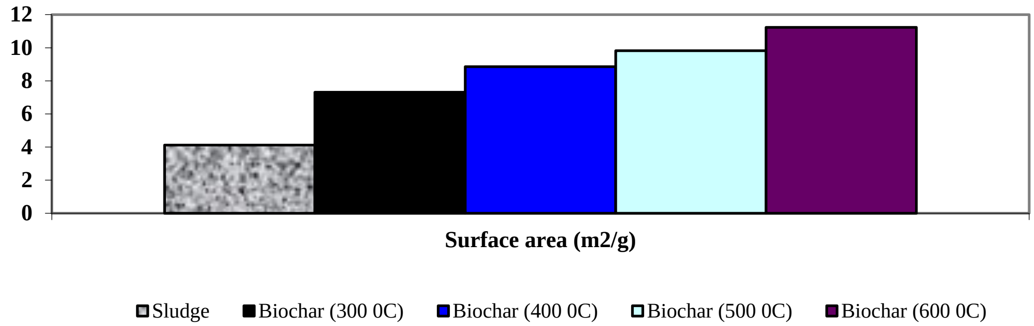


Fig. 3. Characteristics of sludge and biochar (Surface area m^2/g)

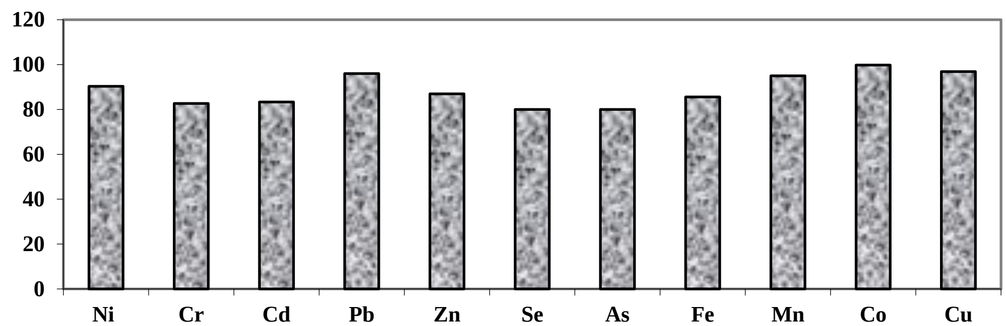


Fig. 4. Average values of trace metals in mg/l of industrial wastewater & treated wastewater that treated by biochar (500^0C)

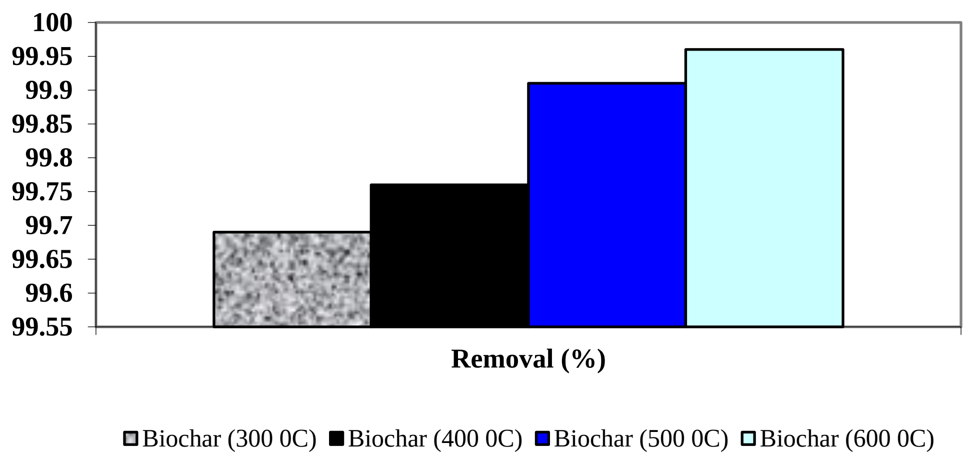


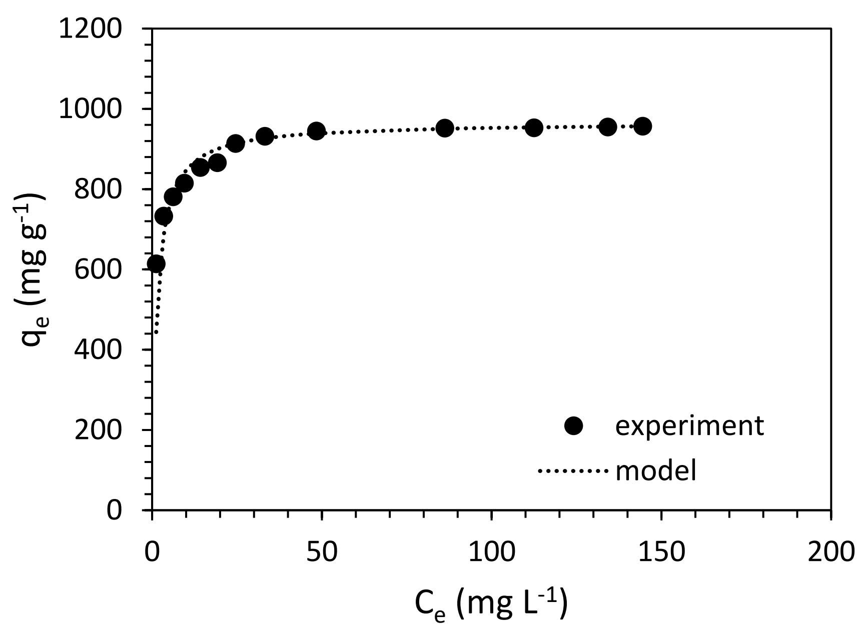
Fig. 5. Average values of MB of industrial wastewater & treated wastewater

Linear regression of isotherm models

Linear regression, a widely used isotherm fitting method, employs the linear least-squares approach. This technique, leveraging the coefficient of determination, creates structurally adaptable isotherm equations, facilitating easy fitting to experimental data. Specifically, the Langmuir isotherm model can be linearized into six distinct forms of simple linear regression, each tailored to different parameters. A core theoretical assumption is that the independent variable, representing the experimental input, is free from error. However, in practice, rearranging the regression equation (numerator and denominator) can affect the correlation results. Consequently, variations in the fitted equation's parameters can be used to identify the most appropriate error distribution through comparative analysis **(Al-Husseini & Al-Salman, 2020; Xia *et al.*, 2020; Azemi *et al.*, 2021; Zhao, *et al.*, 2021)**.

MB Removal Efficiency:

Observed removal efficiencies of methylene blue (MB) ranged from 99.69% to 99.96%. The highest removal efficiency, 99.96%, was achieved under the optimized conditions of pH 7.94, a contact time of 30 minutes, an initial MB concentration of 5.8mg/ L, and an adsorbent dosage of 5mg/ L (**Abdullah *et al.*, 2020; Al Sailawi *et al.*, 2020; Abdullah *et al.*, 2022)**. The maximum adsorption capacity was 965mg/ g, which indicates that a small amount of the adsorbent was sufficient to effectively remove MB from the aqueous solution, as illustrated in Fig. (6).



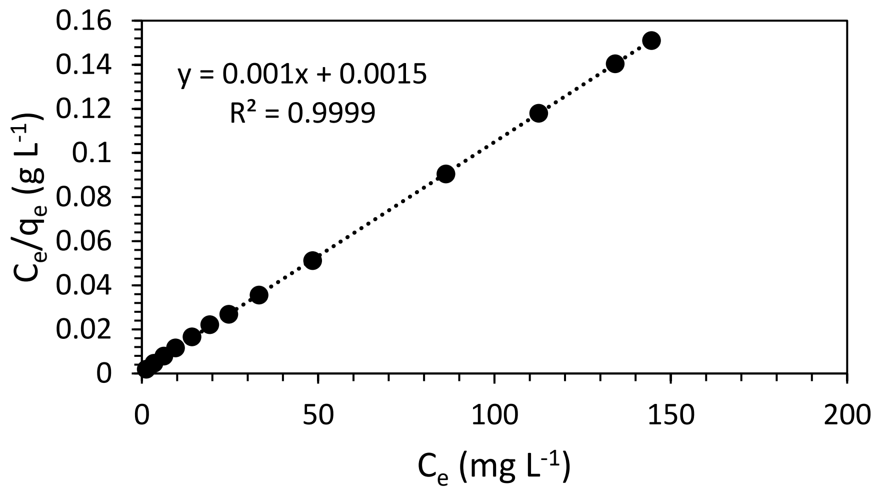


Fig. 6. Langmiur adsorption isotherm of MB from wastewater using biochar (Biochar 600^{0}C) (Linear model)

CONCLUSION

MB removal efficiency varied from 99.69% to 99.96%. The highest removal efficiency, 99.96%, was observed under optimal conditions: pH 7.94, a contact time of 30 minutes, an initial MB concentration of 5.8 mg/L, and an adsorbent dosage of 5 mg/L. Furthermore, the maximum adsorption capacity of 965mg/ g demonstrates that even a small quantity of the adsorbent can effectively remove a substantial amount of MB from aqueous solutions. These findings highlight the efficacy of the biochar preparation method, the stability of the resulting particles, and their potential for industrial wastewater purification and environmental remediation. Analysis of sludge and biochar samples revealed that the concentrations of trace metals (Ni, Cr, Cd, Pb, Zn, Se, As, Fe, Mn, Co, Cu) generally decreased with increasing pyrolysis temperature. Biochar samples produced at higher pyrolysis temperatures exhibited a superior capacity for metal removal.

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