

Tea Waste-Activated Carbon Utilized for Methylene Blue dye and iodine value Adsorption

Shubhank Machhopuriya^{1*}, Dr. J.K. Shrivastava²

¹M.Tech. Scholar, Department of Chemical Engineering, Ujjain Engineering College, Ujjain

²Professor, Department of Chemical Engineering, Ujjain Engineering College, Ujjain

Abstract - The study investigates the possibility of utilizing waste tea to generate biochar and activated carbon to remove methylene blue number (MBN) and iodine value (IV) from wastewater. As a by-product of tea production, tea waste was transformed into activated carbon, and then the effectiveness of the activated carbon in removing dye was examined through batch studies. As a result of its porous structure and large surface area, the results indicated that tea waste-activated carbon has a high adsorption capacity for methylene blue. It was found that the contact time affected the adsorption process. Without oxygen, tea waste samples were treated at a range of temperatures (400, 500, 600, and activated carbon), and the resulting changes in adsorption characteristics were investigated later. Consequently, dye removal systems that take advantage of adsorption are designed to be cost-effective. During the experiment, biochar and activated charcoal were utilized for the purpose of adsorbing methylene blue dye and determining the iodine value. The effluent and wastewater significantly reduced methylene blue dye and iodine value. Within the scope of our experimental inquiry, it was demonstrated that activated charcoal could remove significantly higher amounts of methylene blue dye and iodine value. There is a correlation between the duration of the response (60, 120, and 180 minutes) and the amount of methylene blue that is absorbed. After 180 minutes, a tea waste-activated sample was found to have achieved the highest possible percentage of methylene blue dye removal, which was 98.22%. The measurement of iodine reveals that the highest amount of adsorption that may be achieved using ACTW is 1275.68 mg/g.

Key Words: Tea waste, Activated carbon, Methylene blue, Iodine value, Wastewater treatment, Adsorption capacity, Sustainable adsorbent

1. INTRODUCTION

In recent decades, the increasing industrialization and globalization of manufacturing processes have led to a substantial rise in the production and utilization of synthetic dyes and pigments across various industries worldwide. While these dyes contribute significantly to product aesthetics and functionality, their improper disposal and release into natural water bodies have become a growing environmental concern. Many synthetic dyes are persistent organic pollutants, resistant to conventional wastewater treatment methods. They can cause adverse impacts on

aquatic ecosystems and human health due to their toxic and carcinogenic properties (Aboua K. N. et al., 2015), (Abuelnoor N. et al., 2021).

Exploring sustainable and alternative sources of activated carbon, like agricultural waste products, for environmental remediation has gained attention in recent years. Tea waste, a by-product of tea production, is abundant and rich in organic constituents, making it a potential candidate for producing activated carbon with favorable adsorption properties (Shrestha, L. K. et al., (2019). Various industries, such as paper, food, plastics, and textiles, extensively use dyes, resulting in wastewater with distinctive characteristics. Organic substances in sewage, when discharged into natural water bodies, can be harmful to aquatic life, necessitating additional chemical and biological oxygen. Certain dyes can produce intense hues even at minimal concentrations. Techniques such as ozonation, oxidation, filtering, and coagulation with metal compounds eliminate color from wastewater. However, these technologies often lead to high sludge production, harming the environment. Therefore, activated carbon adsorption is recognized as a promising technique for effectively removing color from wastewater with minimal waste generation (Gowtham, B. 2021), (Hussain, S., et al., 2018).

The activated carbon, generated through hydrothermal carbonization and KOH activation, demonstrated a substantial surface area of 862.2 m²/g and efficiently eliminated methylene blue of 415.8 mg/g (Tran, T. H., et al., 2020). The specific surface area of the synthesized adsorbents varied from 89 to 345 m²/g, and the adsorption capabilities of the charcoal adsorbents for methyl red in an aqueous solution ranged from 26 to 135 mg/g (Paluch D. et al., 2023). Mesoporous PALB, with 816 m²/g surface area and 1 nm radius, efficiently removes >98% MB dye from aqueous solution at 303 K, pH 8, 50 mg/L initial dye concentration, 0.2 g/L adsorption dose, and 30 min contact time. PALB has a high monolayer adsorption capacity (Q_{max}) of 263.95 mg/g, outperforming other adsorbents for MB dye removal in aqueous solutions (Jabar, J. M. et al., 2022). The highest removal of 246.5 mg/g was attained at an initial RBBR concentration, achieving a rate of 95.33% (Gembo, R. O. et al., 2024). Mesoporous magnetic biochar (MBC) with a surface area of 94.2 m²/g, achieving a maximum adsorption capacity of 353.4 mg/g (Mortada, W. I., et al., (2024).

Granular activated carbon generated from sugarcane bagasse (SCBAC) was synthesized using steam activation, resulting in a 489 m²/g surface area, and exhibited adsorption capacities ranging from 290 to 403 mg/g, with efficiencies over 90% following seven regeneration cycles utilizing ethanol (Sutthasupa, S. et al., (2023). Co-pyrolysis generated biochar, exhibiting adsorption capacities of 30.98 (20°C), 29.74 (30°C), and 31.45 (40°C) mg/g, respectively (Mei, M., et al., 2022). Activated carbon sourced from durian shell debris, with a surface area of 348 m²/g and an iodine adsorption value of 634 mg/g, and the most excellent adsorption capacity was 57.47 mg/g (Tran, Q. T. et al., 2022). Activated carbon (WT-AC) produced from waste tea, with an iodine value of 593.41 mg/g, eliminates 89.2% of MB and completely removes Cd²⁺ (Mariah, M. A. A., Rovina, et al., 2023). Peanut hulls exhibited the highest adsorption effectiveness (>95%) among natural adsorbents, although activated carbon derived from sunflower shells demonstrated superior performance (Sawalha, H. et al., 2022). Biochar obtained from orange (*Citrus sinensis*) peels (OPBC) activated with the adsorption, exhibiting a maximum capacity of 208.3 mg/g (Jawad, A. L. I., et al., 2019). Activated carbon was synthesized utilizing ZnCl₂, H₃PO₄, H₂SO₄, NaOH, and KOH, with the ZnCl₂-derived activated carbon exhibiting the highest iodine number (1519 mg/g) and specific surface area (1029 m²/g), efficiently eliminated heavy metals (99.9% Hg, 74.7% Cu (Bai, X. et al., 2023). The Langmuir isotherm accurately characterized the adsorption process, with mason pine-derived hydrochar (MPHC) demonstrating the maximum methylene blue removal capacity (155.1 mg/g) (Zhang, X., et al., 2024). Green algae *Ulva lactuca* (GABS) biochar, the Langmuir isotherm model most accurately characterizes adsorption, exhibiting a maximum capacity of 303.78 mg/g (Shoailb, A. G., et al., 2024). *Eucheuma cottonii* seaweed biochar, resulting surface area of 640 m²/g, yielding a maximum adsorption capacity of 133.33 mg/g (Saeed, A. A. H., et al., 2020). Luffa vine (LVAC) porous biochar, demonstrated a substantial specific surface area (1075.17 m²/g) and elevated MB adsorption capacity (559.11 mg/g). LVAC exhibited 83% efficiency following eight cycles, indicating superior recyclability (Xu, R., et al., 2024). Mugwort (*Artemisia vulgaris*) biochar and activated biocarbon demonstrated a substantial surface area (974.4 m²/g) and maximal adsorption capacities methylene blue (164.14 mg/g) and iodine (948 mg/g) (Wiśniewska, M. et al., 2022). Activated carbon from pistachio with a maximum surface area attained of 1468 m²/g, with methylene blue (341 mg/g) and iodine (1276 mg/g) adsorption capabilities, respectively (Baytar, O. et al., 2018).

This study seeks to examine the viability of employing activated carbon obtained from tea waste for the adsorption of methylene blue dye from aqueous solutions. The explicit aims are to transform tea waste biomass into biochar and activated carbon for the removal of methylene blue dye and the assessment of iodine value. This project aims to utilize

tea waste as a precursor for activated carbon manufacture and evaluate its effectiveness in dye removal, thereby contributing to sustainable and eco-friendly wastewater treatment solutions and environmental protection.

2 Material and Methods

2.1 Feedstock

The tea waste was collected from the tea-making stall in Ujjain. Then, tea waste was washed many times till all the impurities were removed and sun-dried, and then hot air-oven dry for 22 hours to completely dry. Tea waste biomass was thermally processed by placing it in a zip lock bag for further use.

2.2 Semi-batch Pyrolysis Setup

The stainless-steel sample container contained nitrogen gas and pyrolysis product inlets and outlets. A hole for the thermocouple was on the sample container's side. Pyrolysis of tea waste for biochar. It's semi-batch. In each trial, Tea waste weighed 50 grams. In each experiment, the reactor was purged with nitrogen (N₂) gas at 100 mL/min for 30 minutes to ensure an inert environment; then, nitrogen flow was adjusted to the goal flow rate. The reactor's internal pressure was 0.1 MPa throughout the experiment. In each experiment, the reactor was heated at 20°C/min from ambient temperature (30 °C) to the target temperature of 400, 500, and 600°C. After that, the reactor was kept at the target temperature (the residence time). If this occurs, the system should be allowed to cool before removing the biochar sample from the reactor. The biochar samples are designated as TW400, TW500, and TW600.

2.3 Chemicals used

Methylene blue dye, 0.1N Iodine solution (12.700 g of iodine and 19.100 g of potassium iodide (KI) in 1 Liter of Distilled Water), 0.05N Sodium Thiosulphate solution (12.5 gm Na₂S₂O₃·5H₂O in 1 Liter Distilled water), 1% Starch solution, Activated carbon.

2.4 Preparation of Activated Carbon -

TW600 has been taken for further activation treatment. The TW600 was blended with KOH in a 1:1 ratio. The resulting solution was oven-dried for 18 hours. Subsequently, the dried sample was placed in a ceramic boat and within a tubular muffle furnace for two hours of heating under an argon (Ar) inert atmosphere at 800°C. Subsequently, vacuum filtration was employed to rinse the acquired sample with a 0.1M HCl solution, followed by (DI) deionized water. Subsequently, oven-dry the acquired sample for 24 hours. Subsequently, it was placed in a desiccator for the subsequent adsorption analysis. Methylene blue and iodine adsorption are utilized to evaluate the heat-treated tea waste chars and teawaste-activated carbon. The activated carbon

sample derived from tea waste biochar at 600°C is designated TWAC.

2.5 Iodine Value or Iodine Number -

The iodine solution was standardized by adding two drops of starch solution to 10 ml of 0.1 N iodine solution in a conical flask. The iodine solution changed from a light yellow to a dark blue color. The resultant solution was titrated with 0.05N sodium-thiosulfate until it turned colorless. The buretreading corresponds to a blank reading (B). Activated carbon was accurately measured at 0.2 grams. It was added to the iodine flask, which needed to be dry. Then, 40 cc of 0.1N iodine solutions were added. After a proper four-minute shaking, the flask was filtered. The filtrate was collected in a dry flask, and ten cc of it was titrated against a standard sodium-thiosulphate-solution using starch as an indicator (Jeyakumar, R. S., et al., 2014).

$$\text{Iodine Value} = C \times \text{conversion factor,}$$

$$\text{Conversion factor} = (127 \times \text{Normalcy of iodine} \times 40) / (\text{Wt. of Sample blank reading})$$

$$C = (B-A)$$

2.6 Methylene Blue Number / Methylene Value (MNV) -

0.05 grams of activated carbon was agitated for 3 hours at 200 rpm with 100ml of MB solution to measure the methylene blue number of the carbons. The residual methylene blue concentration was then measured by measuring the absorbance at 664 nm using a UV/visiblespectrophotometer after filtering the solution with the Whatman 41 filterpaper (Wu, Z., et al., 2014).

Calculations-

M-mass of the adsorbent in grams, V-volume of the solution in liters, and MBN-Methylene Blue Number, Co and Ce-initial and equilibrium concentrations of MB (mg/l), respectively.

3. Literature Survey

Table -1: Comparison of methylene blue adsorption using activated carbon

S. No.	Sample Name	Iodine Value (mg/g)	Methylene Blue Number (mg/g)
1	Coffee husk waste	-	415.8
2	African almond (Terminalia catappa L) leaves	-	263.95
3	Mesoporous magnetic biochar	-	353.4
4	Sugarcane bagasse-derived	-	403

S. No.	Sample Name	Iodine Value (mg/g)	Methylene Blue Number (mg/g)
5	Durian shell-activated carbon	634	57.47
6	Orange (Citrus sinensis) peels biochar	-	208.3
7	Green algae Ulva lactuca biochar sulfur	-	303.78
8	Eucheuma cottonii seaweed biochar	-	133.33
9	Luffa vine (LVAC) porous biochar	-	559.11
10	Mugwort (Artemisia vulgaris) biochar	948	164.14
11	Pistachio shells activated carbon	1276	341

4. Result and Discussion

The iodine adsorption capacity per gram of carbon increased in pyrolyzed tea waste biomass, correlating with the observed changes in biomass.

Table -2: Iodine value at different temperatures treated biomass, and activated carbon-

S. No.	Sample Name	Iodine Value (mg/g)
1.	TWR	390.25
2.	400	591.05
3.	500	649.75
4.	600	705.57
5.	TWAC	1275.68

According to the calibration curve, the relation between the concentration versus absorbance and the initial absorbance and final absorbance: -

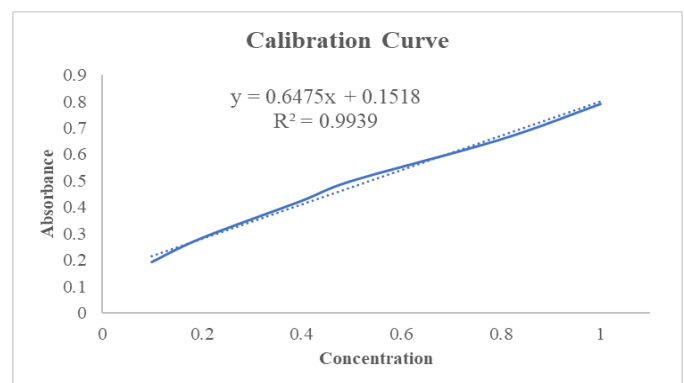


Chart -1: Calibration Curve for MBN

Table -3: Methyleneblue number for biomass, biochar and activated carbon

S. No.	Sample Name	MBV (mg/g) 60 min.	MBV (mg/g) 120 min.	MBV (mg/g) 180 min.
1.	TWR	13.34	13.45	14.94
2.	TW400	32.85	34.85	40.05
3.	TW500	45.44	45.51	50.80
4.	TW600	54.08	54.79	59.49
5.	TWAC	164.36	179.23	191.24

After the pyrolysis process, as the temperature rises surface area of the biochar also increases, and the micropores of the treated biochar boost the adsorption-capacity of iodine and methyleneblue dye following increasing order, including the following:

$$TWAC > 600 > 500 > 400$$

Methylene blue adsorption increases with increasing reaction durations (60, 120, and 180 minutes). A tea waste-activated sample showed a maximum percentage elimination of 98.22% of the methylene blue dye after 180 minutes.

3. CONCLUSIONS

Using tea waste biochar and tea waste activated carbon (TWAC) for methylene blue dye adsorption has been extensively studied and proven to be a viable and effective method for wastewater treatment. TWAC exhibits a high adsorption capacity for methyleneblue dye due to its porous structure and large surface area. This makes TWAC an efficient adsorbent for removing dyes from aqueous solutions. The contact time parameter significantly influences the adsorption efficiency. Optimizing these parameters is crucial to maximize the dye removal performance of TWAC. The adsorption of methylene blue rises as the reaction durations (60, 120, and 180 minutes) increase. A tea waste-activated sample demonstrated a maximal removal rate of 98.22% of methylene blue dye after 180 minutes. The iodine evaluation indicates that the highest adsorption capacity of ACTW is 1275.68 mg/g.

Utilizing TWAC for dye adsorption offers ecological benefits by repurposing tea waste, reducing waste generation, and minimizing synthetic adsorbents. It also provides economic advantages by utilizing a low-cost and renewable biomass resource. TWAC has diverse application potential in textiles, paper, leather, and dye manufacturing industries, where dye-contaminated wastewater is a concern. Its effectiveness and affordability make it a practical alternative to conventional adsorbents.

REFERENCES

- [1] Aboua, K. N., Yobouet, Y. A., Yao, K. B., Goné, D. L., & Trokourey, A. (2015). Investigation of dye adsorption onto activated carbon from the shells of Macoré fruit. *Journal of Environmental Management*, 156, 10-14.
- [2] Abuelnoor, N., AlHajaj, A., Khaleel, M., Vega, L. F., & Abu-Zahra, M. R. (2021). Activated carbons from biomass-based sources for CO₂ capture applications. *Chemosphere*, 282, 131111.
- [3] Bai, X., Quan, B., Kang, C., Zhang, X., Zheng, Y., Song, J., ... & Wang, M. (2023). Activated carbon from tea residue as an efficient adsorbent for environmental pollutant removal from wastewater. *Biomass Conversion and Biorefinery*, 13(15), 13433-13442.
- [4] Baytar, O., Şahin, Ö., Saka, C., & Ağrak, S. (2018). Characterization of microwave and conventional heating on the pyrolysis of pistachio shells for the adsorption of methylene blue and iodine. *Analytical Letters*, 51(14), 2205-2220.
- [5] Gembo, R. O., Odisitse, S., Msagati, T. A., & King'onde, C. K. (2024). Unlocking the valorization potential of Rooibos tea waste-derived activated carbon in the adsorptive removal of remazol brilliant blue R dye. *Next Sustainability*, 4, 100054.
- [6] Gowtham, B. (2021). Experimental study on performance assessment of Fenton and photo-Fenton oxidation process for methylene blue. *Proceedings of the International Academy of Ecology and Environmental Sciences*, 11(2), 43.
- [7] Hussain, S., Anjali, K. P., Hassan, S. T., & Dwivedi, P. B. (2018). Waste tea as a novel adsorbent: a review. *Applied Water Science*, 8, 1-16.
- [8] Jabar, J. M., Odusote, Y. A., Ayinde, Y. T., & Yilmaz, M. (2022). African almond (*Terminalia catappa* L) leaves biochar prepared through pyrolysis using H₃PO₄ as chemical activator for sequestration of methylene blue dye. *Results in engineering*, 14, 100385.
- [9] Jawad, A. L. I., Al-Heetm, D. T., & Abd Rashid, R. (2019). Biochar from orange (*Citrus sinensis*) peels by acid activation for methylene blue adsorption. *Iranian Journal of Chemistry and Chemical Engineering*, 38(2), 91-105.
- [10] Jeyakumar, R. S., & Chandrasekaran, V. (2014). Preparation and Characterization of Activated Carbons Derived from Marine Green Algae *Ulva fasciata* sp. *Asian Journal of Chemistry*, 26(9).
- [11] Mariah, M. A. A., Rovina, K., Vonnice, J. M., & Erna, K. H. (2023). Characterization of activated carbon from waste

- tea (*Camellia sinensis*) using chemical activation for removal of methylene blue and cadmium ions. *South African Journal of Chemical Engineering*, 44, 113-122.
- [12] Mei, M., Liu, J., Wang, T., Chen, S., Liu, D., & Li, J. (2022). Co-pyrolysis of sewage sludge and grape dreg to produce efficient and low-cost biochar for methylene blue removal: adsorption performance and characteristics. *Desalination and Water Treatment*, 248, 277-287.
- [13] Mortada, W. I., Ghaith, M. M., Khedr, N. E., Ellethy, M. I., Mohsen, A. W., & Shafik, A. L. (2024). Mesoporous magnetic biochar derived from common reed (*Phragmites australis*) for rapid and efficient removal of methylene blue from aqueous media. *Environmental Science and Pollution Research*, 31(29), 42330-42341.
- [14] Paluch, D., Bazan-Wozniak, A., Wolski, R., Nosal-Wiercińska, A., & Pietrzak, R. (2023). Removal of methyl red from aqueous solution using biochar derived from fennel seeds. *Molecules*, 28(23), 7786.
- [15] Saeed, A. A. H., Harun, N. Y., Sufian, S., Siyal, A. A., Zulfiqar, M., Bilad, M. R., ... & Almabhashi, N. (2020). *Eucheuma cottonii* seaweed-based biochar for adsorption of methylene blue dye. *Sustainability*, 12(24), 10318.
- [16] Sawalha, H., Bader, A., Sarsour, J., Al-Jabari, M., & Rene, E. R. (2022). Removal of dye (methylene blue) from wastewater using bio-char derived from agricultural residues in palestine: Performance and isotherm analysis. *Processes*, 10(10), 2039.
- [17] Shoaib, A. G., Van, H. T., Tran, D. T., El Sikaily, A., Hassaan, M. A., & El Nemr, A. (2024). Green algae *Ulva lactuca*-derived biochar-sulfur improves the adsorption of methylene blue from water. *Scientific Reports*, 14(1), 11583.
- [18] Shrestha, L. K., Thapa, M., Shrestha, R. G., Maji, S., Pradhananga, R. R., & Ariga, K. (2019). Rice husk-derived high surface area nanoporous carbon materials with excellent iodine and methylene blue adsorption properties. *C*, 5(1), 10.
- [19] Sutthasupa, S., Koo-Amornpattana, W., Worasuwannarak, N., Prachakittikul, P., Teachawachirasiri, P., Wanthong, W., ... & Chaiwat, W. (2023). Sugarcane bagasse-derived granular activated carbon hybridized with ash in bio-based alginate/gelatin polymer matrix for methylene blue adsorption. *International Journal of Biological Macromolecules*, 253, 127464.
- [20] Tran, Q. T., Đo, T. H., Ha, X. L., Duong, T. T. A., Chu, M. N., Vu, V. N., ... & Song, P. (2022). Experimental design, equilibrium modeling and kinetic studies on the adsorption of methylene blue by adsorbent: activated carbon from durian shell waste. *Materials*, 15(23), 8566.
- [21] Tran, T. H., Le, A. H., Pham, T. H., Nguyen, D. T., Chang, S. W., Chung, W. J., & Nguyen, D. D. (2020). Adsorption isotherms and kinetic modeling of methylene blue dye onto a carbonaceous hydrochar adsorbent derived from coffee husk waste. *Science of the Total Environment*, 725, 138325.
- [22] Wiśniewska, M., Rejer, K., Pietrzak, R., & Nowicki, P. (2022). Biochars and activated biocarbons prepared via conventional pyrolysis and chemical or physical activation of mugwort herb as potential adsorbents and renewable fuels. *Molecules*, 27(23), 8597.
- [23] Wu, Z., Zhong, H., Yuan, X., Wang, H., Wang, L., Chen, X., ... & Wu, Y. (2014). Adsorptive removal of methylene blue by rhamnolipid-functionalized graphene oxide from wastewater. *Water research*, 67, 330-344.
- [24] Xu, R., Wei, J., Cheng, D., Wang, W., Hong, L., Chen, Y., & Guo, Y. (2024). Abundant porous biochar derived from luffa vine for removal of methylene blue: Selective adsorption and mechanistic studies. *Industrial Crops and Products*, 219, 119114.
- [25] Zhang, X., Chen, F., Liu, S., Lou, J., Liu, W., Rousseau, D. P., & Van Hulle, S. (2024). Synthesis, characterization, and methylene blue adsorption isotherms of hydrochars derived from forestry waste and agro-residues. *Biomass Conversion and Biorefinery*, 14(2), 1809-1824.