Parametric Modeling for Activated Carbon Synthesis

Muhammad Usman[1*], Azhar Hussain[1], Muhammad Najam UL Hassan[1], Ali Nasir[1], Ramzan Abdul Karim[2], Muhammad Yasir[3], Aneela Wakeel[1], Abdul Moiz Rao[1], Muhammad Ahmed[1], Muhib Ullah[1], Khurram Imran[2]

[1]University of Engineering and Technology (UET), Taxila, Department of Mechanical Engineering,
***chusman67728@gmail.com**

[2]Ghulam Ishaq Khan Institute of Engineering Sciences and Technology (GIKI), Faculty of Materials and Chemical Engineering, Pakistan, azharhussain@uettaxila.edu.pk

[3]Institute of space technology, Department of materials science and engineering, Pakistan, najamulhassan2001@gmail.com

Abstract

This study investigates the synthesis of activated carbon from waste tires, focusing on how heating rates influence activation energy and product quality. Through controlled experiments, the research evaluates the impact of varying thermal treatments on the activation energy and the resultant activated carbon's surface area, pore size, and volume. The objective is to establish the optimal conditions that enhance adsorptive properties while maximizing energy efficiency in the production process. Findings indicate that slower heating rates are conducive to producing activated carbon with higher surface areas and smaller, more uniform pore sizes, traits desirable for effective adsorption. Specifically, activated carbon produced under slow heating exhibited a surface area of 621 m^2/g and a pore size of 292 Å, compared to 570 m^2/g and 308 Å under faster heating conditions. This demonstrates a direct correlation between the heating rate and the material's structural characteristics that affect its adsorption capacity.

Incorporated within this study parametric model that forecasts the qualities of activated carbon for various heating regimens, facilitating the precise adjustment of the synthesis process. The study reveals a nuanced understanding of energy consumption in the synthesis process. Lower heating rates, while beneficial for product quality, necessitate a reevaluation of energy expenditure to ensure economic viability. Conversely, the slight reduction in quality observed with rapid heating suggests a potential for time and energy savings, offering a trade-off between efficiency and product performance. The research bridges the gap between process parameters and activated carbon quality, providing valuable insights for the development of more sustainable, cost-effective production methods. By identifying ideal heating rates, the study paves the way for advancements in activated carbon manufacturing, promising significant improvements in environmental sustainability and economic efficiency. This contributes to a broader application of activated carbon in pollution control and resource recovery, underscoring the importance of optimized manufacturing processes.

Keywords: Activated Carbon, Synthesis, Waste Tires, Heating Rates, Activation Energy, Thermal Treatment, Energy Efficiency, Sustainable Manufacturing, Pollution Control.

1. Introduction

Activated carbon, a material known for its ability to adsorb pollutants, has a history rooted in ancient practices yet remains crucial in modern environmental and industrial applications[1]. Its earliest recorded use traces back to civilizations such as the Sumerians and Egyptians, who utilized charred wood for metal purification and as a fuel that doesn't produce smoke. The material's journey through history showcases its evolving role, from improving air quality in the 18th century to decolorizing sugar in the 19th century, highlighting its diverse utility.

The core attribute of activated carbon lies in its microporous structure, which grants it a high surface area for adsorption. This characteristic is pivotal for addressing contemporary challenges like water pollution, where activated carbon serves as a key method for removing a wide range of contaminants from water sources. The increasing concern over water pollutants due to industrial discharges has amplified the demand for effective treatment solutions, positioning activated carbon as an essential tool in water and wastewater management[2].

With the parametric model established, predictive capabilities are utilized based on quality of final product and activation energy values to forecast the properties of activated carbon under different synthesis scenarios. This enables the fine-tuning of the production process to achieve desired specifications efficiently. Moreover, the model supports decision-making by ranking the relative importance of different variables in achieving the optimal performance of the activated carbon for specific applications, such as pollutant adsorption or dye removal.

Recent advancements have focused on sustainable and efficient production methods for activated carbon, particularly from biomass-derived materials. This shift aims to overcome the drawbacks of conventional production techniques, which can be energy-intensive and involve harmful chemicals[3]. Research has explored using waste materials, such as sawdust from specific trees like Subabul, for producing activated carbon[4]. This approach not only leverages waste but also presents a greener alternative for carbon capture technologies.

2. Methodology

2.1 Analytical Approach

The analytical approach of this research hinges on the synthesis of activated carbon from waste tires, optimizing energy consumption through the Arrhenius equation, which models temperature's effect on reaction rate constants. The equation:

$$k(T) = A \cdot e^{-RTE} \qquad \ldots(1)$$

$$dt/d\alpha = k(T) \cdot (1-\alpha) \qquad \ldots(2)$$

$$a = -\frac{E}{R} \qquad \ldots(3)$$

$$E = -aR \qquad \ldots(4)$$

provides a framework to relate the rate constant $k(T)$ and the activation energy E with the heating process. By applying linear regression to the experimental data, denoted as (xi, yi), with xi=$T1$ and $yi = ln(k(T))$, both activation energy and pre-exponential factor are derived, informing the synthesis optimization.

This method was employed in the study to enhance the surface area of activated carbon for efficient removal of methylene blue dye (MB)[5]. Findings show that demineralization, particularly using NaOH and H2SO4, substantially increased the surface area from 53 m^2/g to 257 m^2/g and enabled a significant reduction in metal content, Zn^{2}+, Al^{3}+, Ca^{2}+, and Mg^{2}+, facilitating maximum MB removal efficiencies and optimizing the adsorption process[6]. The study indicates that adsorption is site-specific and chemically driven, as the most effective dye removal reached 90.13% with a 4 M KOH solution and the best conditions for MB removal were identified at a 1.2 g adsorbent dosage, 120-minute contact time, pH 8, a temperature of 20°C, and an initial dye concentration of 10 mg/L. These insights are critical for determining the optimal conditions for activated carbon production and its application in dye adsorption.

2.2 Experimental Approach

In this study, the production of activated carbon was derived from the thermal decomposition and chemical activation of waste tire pieces. Initially, these pieces were cleansed with a lukewarm water and acetone mixture, followed by drying at 75°C to eliminate moisture. The dry tire material was then pyrolyzed in a tubular furnace under an inert argon atmosphere, heating up to 700°C at specific rates to form char. Subsequently, this char was chemically activated using a concoction of hydrochloric acid (HCl) and potassium hydroxide (KOH) to etch a microporous structure onto its surface, a crucial attribute for enhancing its adsorptive capacity.

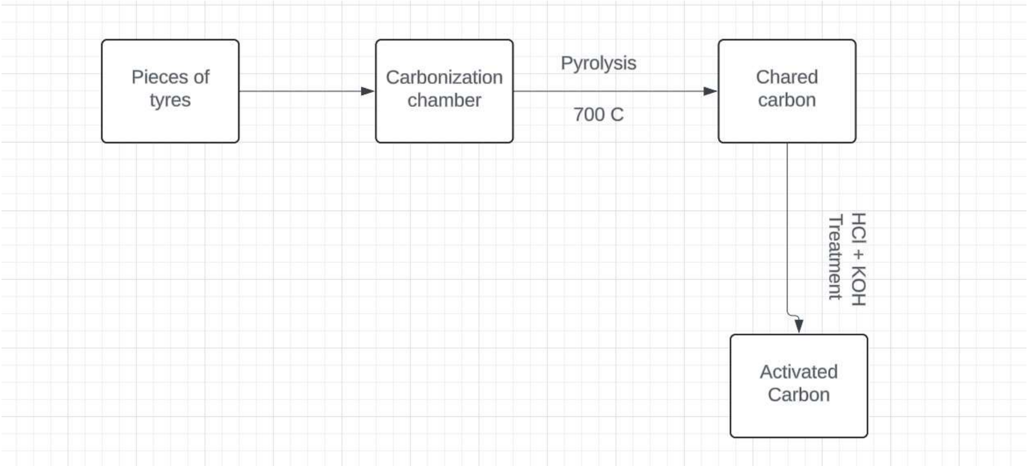

Figure 1: Schematic Diagram of Experimental Work

The final activated carbon's structural and chemical attributes were meticulously examined to determine its quality and effectiveness for adsorption applications. Scanning Electron Microscopy (SEM) was utilized for analyzing the surface morphology, while the Brunauer-Emmett-Teller (BET) method provided insights into the surface area and pore volume. This approach allowed for a detailed investigation into the impact of varying heating rates on the synthesis efficiency, optimizing both the energy consumption and the activated carbon's adsorptive properties, thereby contributing to the development of sustainable waste tire management and carbon capture solutions.

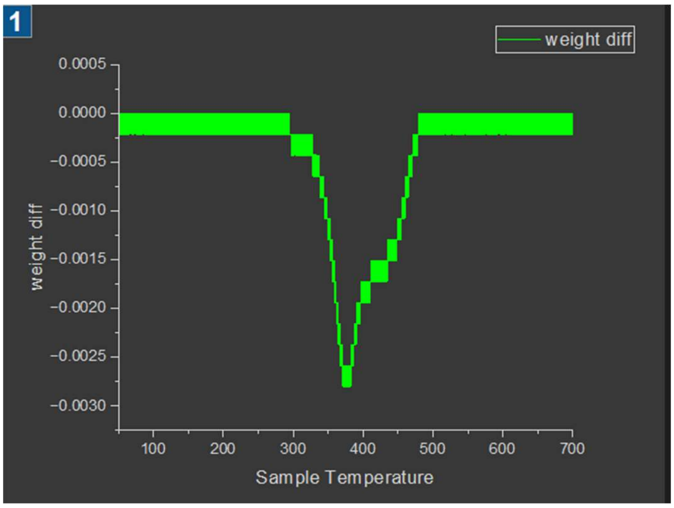

Figure 2: Unsubstracted weight difference observed against sample Temperature.

The graph presents a pronounced weight loss in the sample as temperature increases, indicative of the primary thermal decomposition or pyrolysis phase, likely corresponding to the volatilization of volatile components. This behavior aligns with the findings of the previous studies which investigated the effect of heating rates on the synthesis of activated carbon from tires, suggesting that the most significant weight change occurs at elevated temperatures, essential for optimizing the activation process[7]. The stabilization of weight at higher temperatures suggests the end of major reactions and the formation of a carbonaceous char, a precursor to activated carbon.

In the first test, samples underwent a controlled heating process at a constant rate of 10°C/min up to a maximum temperature of 700°C. This methodical increase in temperature facilitates a gradual pyrolysis, allowing for detailed observation of the thermal degradation and weight loss corresponding to different temperature stages, which is crucial for understanding the reaction kinetics and the development of pore structure in the activated carbon.

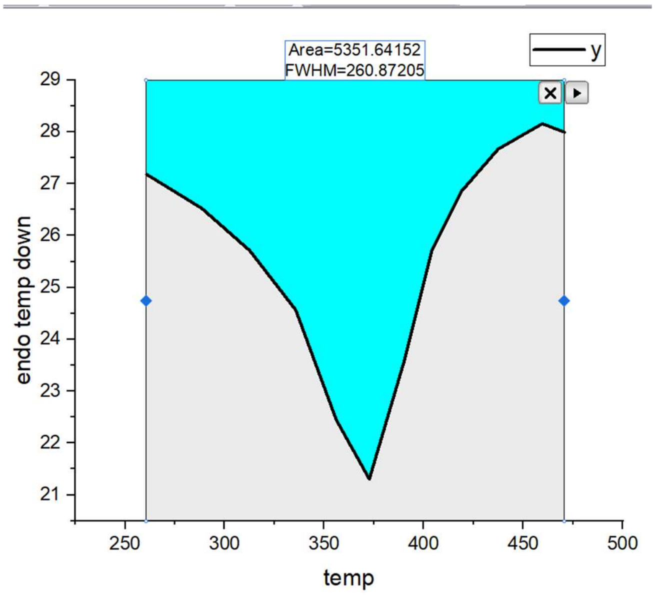

Figure 3: AUC for Test-1

For the second test, the heating rate was initially set at 20°C/min up to 500°C, after which it was accelerated to 50°C/min until reaching 700°C. This two-phase heating approach was employed to examine the effects of a rapid temperature increase on the activation process and final product characteristics. The faster rate influences the size and distribution of the pores by altering the thermal stress and reaction pathways within the carbonizing material.

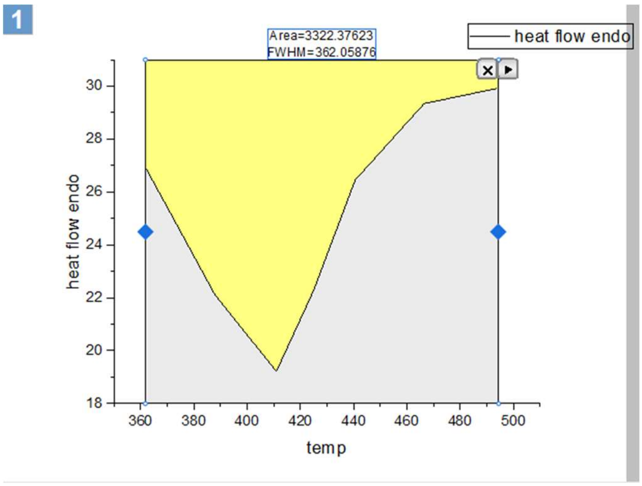

Figure 4: AUC for Test-2

In the analysis of the two tests, the first showed a significant exothermic peak with an AUC of 5351.64, implying substantial energy release at a constant heating rate of 10°C/min up to 700°C, which may denote exothermic reactions such as the combustion of volatile materials or structural reorganization within the material. The second test revealed an exothermic response with a lower AUC of 3322.37, across a broader temperature range, as evidenced by the FWHM of 362.06, due to a more dynamic heating regime that commenced at 20°C/min to 500°C and then escalated to 50°C/min up to 700°C. This suggests that the faster heating rate and subsequent ramp may lead to different reaction kinetics, potentially affecting the structure and quality of the synthesized activated carbon.

Table 1: Activation Energy for tests performed.

Test	**Activation Energy (kJ/mol)**	**Slope**
1st Test - 1st Activation Energy	28.7	-1.5
1st Test - 2nd Activation Energy	23	-1.2
2nd Test - 1st Activation Energy	18.7	-0.97
2nd Test - 2nd Activation Energy	25.5	-1.4

Table 2: Power computed for both tests.

Test	**Power (kW/mol)**	**Power (kW/g)**
First Test	0.014	0.00117
Second Test	0.012	0.001

2.3 Quality Analysis

The quality of activated carbon produced under different heating conditions reveals distinct characteristics in terms of surface area, pore size, and pore volume, which are critical for its adsorption capacity. For the slow heating process, the activated carbon exhibited a surface area of 621 m²/g, a pore size of 292 Å, and a pore volume of 0.34 cm³/g. These properties indicate a highly porous material capable of facilitating adsorption through an extensive network of micropores, suitable for removing small molecules and impurities from liquids or gases.

In contrast, the activated carbon produced through fast heating with a ramp presented a slightly lower surface area of 570 m²/g and a larger pore size of 308 Å, alongside a marginally higher pore volume of 0.35 cm³/g. This suggests that while the material remains effective for adsorption purposes, the rapid temperature changes may lead to a less optimal pore structure for certain applications, potentially affecting its selectivity and capacity for specific contaminants.

Table 3: Comparison of Quality Parameters for both Tests.

	Surface Area (m²/g)	**Pore Size (Å)**	**Pore Volume (cm³/g)**
Slow Heating	621	292	0.34
Fast Heating with Ramp	570	308	0.35

Standard Activated Carbon	1012	229	0.38

Comparing these results to standard activated carbon, with a surface area of 1012 m^2/g, a pore size of 229 Å, and a pore volume of 0.38 cm^3/g, it's evident that the laboratory-synthesized products, especially from slow heating, approach the qualities of commercially available activated carbon but with room for improvement in maximizing surface area and optimizing pore size for enhanced adsorption performance. These findings underscore the importance of controlled thermal processing in tuning the adsorptive properties of activated carbon derived from waste tires, presenting a sustainable approach to material recovery and environmental remediation.

2.4 Correlation between Activation Energy and Quality

Relationship between activation energy values and the quality parameters of activated carbon:

- Activated carbon produced through slow heating, which involves higher activation energies, tends to exhibit larger surface areas and smaller pore sizes. This indicates that a gradual increase in temperature, allowing for more energy to be absorbed over time, is conducive to forming a dense microporous structure that enhances adsorption capabilities[8].
- Conversely, activated carbon synthesized with fast heating and a ramp, characterized by lower activation energies, generally shows slightly reduced surface areas and larger pore sizes. This suggests that rapid temperature increases lead to less controlled pore development, potentially affecting the uniformity and efficiency of the adsorption process.
- The pore volume of activated carbon remains relatively consistent across different heating strategies, indicating that the total volume accessible for adsorption is less affected by the rate of temperature increase and more by the overall thermal treatment process.

3. DOE and Decision Making

The synthesis of activated carbon from waste tires is significantly enhanced when integrating Design of Experiment (DOE) and decision-making frameworks, which refine the optimization of critical process variables, including temperature, heating rate, and gas flow. These factors are integral to determining the activation energy and the resultant carbon quality[9]. Utilizing factorial designs or response surface methodology within DOE enables a systematic investigation of the variable interactions. This strategy assists in pinpointing the most advantageous conditions that amplify the activated carbon's surface area, pore size, and volume. The DOE approach not only facilitates statistical analysis of experimental data but also aids in developing predictive models that correlate process parameters with quality outcomes.

Moreover, multi-criteria decision analysis techniques, embedded within these decision-making frameworks, allow for the prioritization of quality parameters aligned with targeted application needs, thus steering the synthesis process toward the most efficacious conditions. This integrated approach promises to refine the analytical methods used to determine activation energy, employing DSC and DTG curves to advance the understanding of thermal and kinetic behaviors in the production of activated carbon. Future research endeavors can build upon these methodologies to advance precision in establishing activation energy values and enhancing the performance of activated carbon for specific environmental and industrial applications.

4. Conclusion

In conclusion, this research has demonstrated that the heating rates significantly affect both the activation energy required and the quality of activated carbon produced from waste tires. Slow heating rates were found to favor the creation of activated carbon with higher surface areas and more desirable pore structures, enhancing its adsorption efficiency. In contrast, fast heating rates led to slight reductions in these quality parameters. These insights not only highlight a sustainable method of converting waste tires into valuable activated carbon but also stress the importance of process optimization for improving material properties. Moving forward, further optimization of the synthesis parameters, informed by this research, could lead to advancements in activated carbon production that are both environmentally and economically beneficial.

References

[1] E. Kassahun, S. Mekuria, and S. M. Beyan, “Specific Surface Area Enhancement of Waste Tire-Based Activated Carbon by Demineralization Technique and Adsorption of Methylene Blue,” *International Journal of Chemical Engineering*, vol. 2022, 2022, doi: 10.1155/2022/8198551.

[2] S. Al-Mutlaq and E. Mahal, “Synthesis of activated carbon from scrap tires with petroleum kerosene as a decomposition auxiliary agent,” *Eurasian Chemical Communications*, vol. 4, no. 7, 2022, doi: 10.22034/ecc.2022.316296.1268.

[3] E. Kassahun, S. Tibebu, Y. Tadesse, and N. Awish, “Synthesis Optimization of Activated Carbon Driven from Scrap Tire for Adsorbent Yield and Methylene Blue Removal under Response Surface Methodology,” *Advances in Materials Science and Engineering*, vol. 2022, 2022, doi: 10.1155/2022/2325213.

[4] S. A. Borghei *et al.*, “Synthesis of multi-application activated carbon from oak seeds by KOH activation for methylene blue adsorption and electrochemical supercapacitor electrode,” *Arabian Journal of Chemistry*, vol. 14, no. 2, 2021, doi: 10.1016/j.arabjc.2020.102958.

[5] R. B. González-González *et al.*, "Characterization of chemically activated pyrolytic carbon black derived from waste tires as a candidate for nanomaterial precursor," *Nanomaterials*, vol. 10, no. 11, 2020, doi: 10.3390/nano10112213.

[6] A. E. Ogungbenro, D. V. Quang, K. A. Al-Ali, L. F. Vega, and M. R. M. Abu-Zahra, "Synthesis and characterization of activated carbon from biomass date seeds for carbon dioxide adsorption," *J Environ Chem Eng*, vol. 8, no. 5, 2020, doi: 10.1016/j.jece.2020.104257.

[7] M. 'Anwar, A. 'Hussain, M. ' 'Abu Sufyan, M. 'Usman, and M. ' Najam ul Hassan, "Effect of heating rate on synthesis of Activated carbon from Tyre," Konya, Turkey, 2024, pp. 430–436.

[8] F. Taufany *et al.*, "The Synthesis of Activated Carbon from Waste Tyre as Fuel Cell Catalyst Support," *Evergreen*, vol. 9, no. 2, 2022, doi: 10.5109/4794166.

[9] N. Genli, Şahin, O. Baytar, and S. Horoz, "Synthesis of activated carbon in the presence of hydrochar from chickpea stalk and its characterization," *Journal of Ovonic Research*, vol. 17, no. 2, 2021, doi: 10.15251/jor.2021.172.117.