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EVALUATION OF THE EFFICIENCY OF *AZADIRACHTA INDICA* (NEEM) LEAF POWDER AND ITS ACTIVATED CARBON IN THE ADSORPTION OF BENZENE AND TOLUENE FROM SIMULATED REFINERY WASTE WATER

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ABSTRACT

This study assessed the efficiency of Azadirachta indica (Neem) leaf powder (NLP) and its activated carbon (NLAC) as potential adsorbents for the extraction of benzene and toluene from simulated refinery wastewater. The objective is to evaluate the capacity of these materials to enhance the quality of industrial effluents and assist developing nations in attaining the Sustainable Development Goals (SDGs), specifically Goal 6 (Clean water and Sanitation), Goal 12 (Responsible Consumption and Production), and Goal 15 (Life on Land). Azadirachta indica leaf powder and its activated carbon were prepared by air-drying Azadirachta indica leaf in the shade at room temperature (25 °C) for 72 hr, followed by oven drying at 105 °C for 30 min until they were crisp, and chemical activation with phosphoric acid. The adsorbents were assessed by Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and Brunauer-Emmett-Teller (BET) analysis. The FTIR analysis identified the functional groups in the adsorbent that are accountable for the adsorption property. The surface presents a fibrous composition including irregular macropores and enlarged cavities that may facilitate the diffusion of adsorbate molecules through the macropores. The batch adsorption process was studied under numerous conditions, encompassing varying concentrations, pH, contact time, adsorbent amount, and temperature. The findings indicated that activated carbon derived from Neem leaf, possessing a surface area of 427.154 m²/g, exhibited substantial adsorption of benzene and toluene, attaining removal efficiency of 74 % and 81 % respectively, under optimal conditions of (pH 10, contact time 75 min, adsorbent dosage 1 g, temperature 60 °C, and an initial concentration of 100 mg/L). Neem leaf powder was determined to be ineffective for adsorption, due to its limited surface area (294.381 m²/g) and insufficient pore structure. The findings underscore the efficacy of NLAC as an economical and sustainable material for the clean-up of toxic substances from industrial effluents.

Keywords: Adsorption, Activated Carbon, Adsorbent, Adsorbate, Azadirachta indica Leaf, Benzene, Toluene

INTRODUCTION

Water is a ubiquitous solvent essential for various human activities, with the primary concerns being its quantity and purity (Sadiq *et al.,* 2022). Globally, the population continues to expand, already exceeding 8 billion individuals, while wastewater generation is also escalating, anticipated to rise by 51 % by 2030 from the 2020 aggregate of 380 billion m³ (Qadir *et al.,* 2020). Growing development coupled with technology has resulted in the production and discharge of vast quantities of pollutants in the ecosystem (Marathe, 2022). Oil-based companies, like oil refineries, constitute significant economic sectors that produce substantial quantities of hazardous waste (Otitolaiye, 2022). Such dangerous and cancer-causing contaminants may result in several health issues, including asthma, allergies, dermal irritation, respiratory difficulties, nausea, diaphoresis, emesis, cognitive impairment, hypertension, migraines, and potential abnormalities in chromosomes. Consequently, it is a significant impediment to the conservation of water resources (Al-Ghouti and Sweleh, 2019). The petroleum sector and oil extraction are the primary contributors of polluted wastewater (Abuhasel *et al.,* 2021). The occurrence of hydrocarbons, which include benzene, toluene, ethyl benzene, xylenes (BTEX), and polycyclic aromatic hydrocarbons (PAHs), in petroleum-based waste water warrants significant concern.

Oil companies and petrochemical enterprises produce substantial amounts of contaminants, including petroleum hydrocarbons, originating from various roots such as oil storing facilities, cleaning areas, and container sanitizing operations. If this effluent is not properly released and managed, it can significantly affect the ecosystem. Consequently, it is imperative to perform a thorough evaluation and assessment of oily effluent characteristics before commencing any sort of clean-up procedures (Sanghamitra *et al.,* 2021).

Effective control and disposal of effluent are crucial for safeguarding human health, protecting the ecosystem, and maintaining the long-term conservation of hydrological resources (Singh *et al.,* 2023).The purification of effluent entails the elimination or mitigation of impurities and toxins via diverse chemical, physical (Arslan *et al.,* 2024), and biological techniques (Daverey *et al.,* 2019). Management techniques may encompass screening, sedimentation, filtration, adsorption (Fiyadh *et al.,* 2023), biological oxidation, disinfection, and advanced treatment techniques (Da Silva et al., 2023) such as membrane filtration (Al-Asheh *et al.,* 2021) and advanced oxidation (Dewi *et al.,* 2021; Irshad *et al.,* 2022).

This study presents the evaluation of the efficiency of Neem leaf powder and its activated carbon as a green adsorbent for the cleanup of organic pollutants, thereby supporting sustainable development goals (SDGs), which are critical in several nations.

MATERIALS AND METHODS

Equipment/Instrument

Oven, weighing balance, muffle furnace, pH meter, water bath, mechanical shaker (STUART SF1, Bibby Scientific UK), UV-Vis Spectrophotometer (THERMO SCIENTIFIC GENESYS180, China), Fourier Transform Infrared

Spectrophotometer (CARY630-FTIR, India), Scanning Electron Microscope (THERMO FISHER PRISMA E, USA), Brunauer-Emmett-Teller Analyzer (QUANTACHROME, Germany).

Sample Collection and Adsorbent Preparation

Fresh Neem (Azadirachta indica) leaves were collected from the twigs of a mature tree sourced locally from Hayin Malam Bello Rigasa, Kaduna, identified at Herbarium unit of the department of Biological Science, Nigerian Defence Academy, Kaduna, Nigeria (voucher no. NDA/BIOH/2024/36), cleaned, air-dried in the shade at room temperature (25 °C) for 72 hr, followed by oven drying at 105 °C for 30 min until they were crisp. Ground into a fine powder and sifted through a 220 µm mesh (Husaini *et al.* 2023). For activated carbon, 100 g of leaves powder was carbonized in furnace at 500 °C for two hr, then chemically activated with 85 % phosphoric acid (H_3PO_4) and heated in an oven for 2 hr at 150 °C. The sample was washed with distilled water, dried at 60 °C in an oven for 2 hr, and stored in an airtight container (Husaini *et al.,* 2023).

Adsorbate Preparation

Stock solutions of benzene and toluene were prepared at 500 mg/L each in a 1000 cm^3 volumetric flask, with working solutions of 20, 40, 60, 80, and 100 mg/L, each made in a 250 cm^3 volumetric flask, generated via serial dilutions (Khader *et al.,* 2024).

Characterization of The Adsorbent (Ftir, Sem, Bet)

FTIR was conducted using ATR mode with spectra recorded over a range of 4000-650 cm^{-1}. The ATR-FTIR spectra were acquired utilizing a spectral resolution of 4 cm^{-1} and a total of 8 scans (Joel *et al.,* 2024). SEM analysis was performed to assess surface morphology, a working distance of 12.9-13.5 mm and an acceleration voltage of 30.00 kV were employed at various magnifications, utilizing the secondary electron image (SEI) as the detector. (Husaini and Ibrahim, 2019). And BET analysis determined surface area, pore volume, and diameter using nitrogen adsorption at 77 K utilizing a QUANTACHROME NOVA 4200e equipment.

Adsorption Optimization Studies

Batch experiments were conducted to evaluate the effects of dosage (0.2–1 g), pH (2–10), contact time (15–75 min), temperature (20–60 °C), and initial concentration (20–100 mg/L) to determine the optimal parameters for the removal of Benzene and Toluene. Adsorbate-adsorbent mixtures were agitated at 300 rpm, and filtrates were analyzed with a UV-Vis spectrophotometer. The concentration of the contaminant in the solution was determined by measuring the absorbance at the maximum wavelengths of ʎ max = 254 nm for Benzene and ʎ max = 261 nm for Toluene (Khader *et al.,* 2024).

Pollutant removal efficiencies, and equilibrium adsorption capacities, were computed. The data were analyzed to evaluate the efficacy of neem leaves powder and its activated carbon as adsorbents for pollutant removal.

The pollutant removal % and adsorption capacity were determined for each experiment using Eqn 1 (Mahdi *et al.,* 2023) and Eqn 2 (Sadare *et al.,* 2022), respectively.

$$\%\ \text{Removal of Benzene or Toluene} = (Co - Ce)/Co \times 100 \ldots \quad (1)$$

Where:

C_o = Initial concentration of Benzene or Toluene before the adsorption process (mg/L)

C_e = Equilibrium concentration of Benzene or Toluene in the filtrate after adsorption process (mg/L)

$$qe = (Co - Ce)/M \times V \quad (2)$$

Where:

qe = Adsorption capacity (mg/g)

C_o = Initial concentration of Benzene or Toluene (mg/L)

C_e = Equilibrium concentration of Benzene or Toluene after adsorption (mg/L)

m = Mass of Adsorbent (g)

V = Volume of Adsorbate (L)

The initial and post-adsorption data were analyzed to assess the efficacy of Neem leaves powder and its activated carbon in pollutant removal.

RESULTS AND DISCUSSION

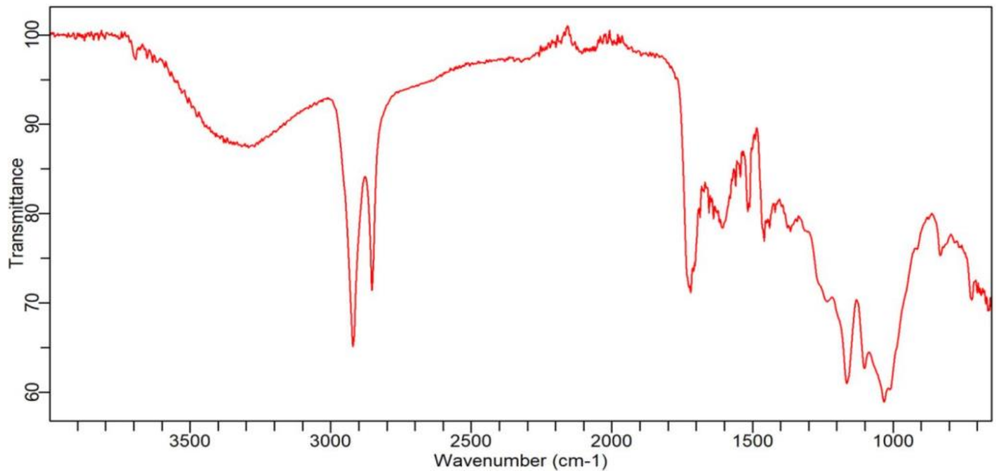

Figure 1: FTIR Spectra for NLP

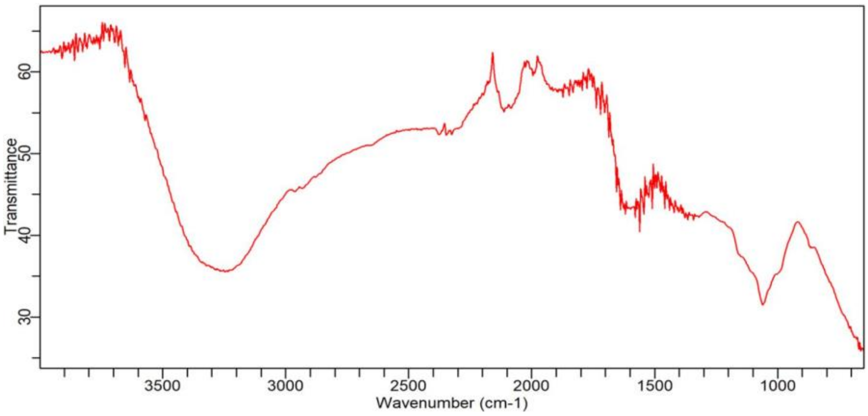

Figure 2: FTIR Spectra for NLAC

The functional groups in Neem leaf powder and Neem leaf activated carbon were studied using FT-IR spectroscopy. Figure 1 and 2 showed the distinct FT-IR peaks for Neem leaves powder and Neem leaves activated carbon. The stretching vibration of the O-H bond results in the broad absorption band at 3373 cm^{-1} for NLP and 3233 cm^{-1} for NLAC (Mordhiya *et al.,* 2023). The peaks at 2920-2853 cm^{-1} for NLP indicate the presence of aliphatic C-H bonds (alkanes) and C-H stretching groups (Mordhiya *et al.,* 2023). The peak at about 2111-1996 cm^{-1} for NLAC indicates the presence of C≡C stretching bonds characteristic of the alkyne group (Abel *et al.,* 2020). The adsorbent may possess aromatic rings, indicated by a distinct band at 1664 cm^{-1} for NLP and at 1599 cm^{-1} for NLAC, which is indicative of the C=C stretching vibration of the aromatic ring (Pathania *et al.,* 2017). The peak between 1233-1030 cm^{-1} in NLP indicates the existence of C-O stretching bonds, while the peak at 1060 cm^{-1} in NLAC signifies the presence of carboxylic, alcoholic, phenolic, ether, and ester groups (Naga *et al.,* 2019). The C=O stretch in NLP occurs at 1720-1654 cm^{-1}, but in NLAC it is observed at 1873-1720 cm^{-1}, indicating the presence of carboxylic, ketonic, phenolic, and ester groups (Mordhiya *et al.,* 2023). The signal at 1543-1517 cm^{-1} for NLP indicates the presence of N-H bending bonds in amines. The -C-H bending in NLP is identified at 1459-1364 cm^{-1}, indicating the presence of alkane groups, while aromatic bonds are observed at 833-723 cm^{-1} (Mordhiya *et al.,* 2023). Consequently, these represent the interpretations derived from the FT-IR spectra.

Plate 1a: SEM image of NLP at 100x

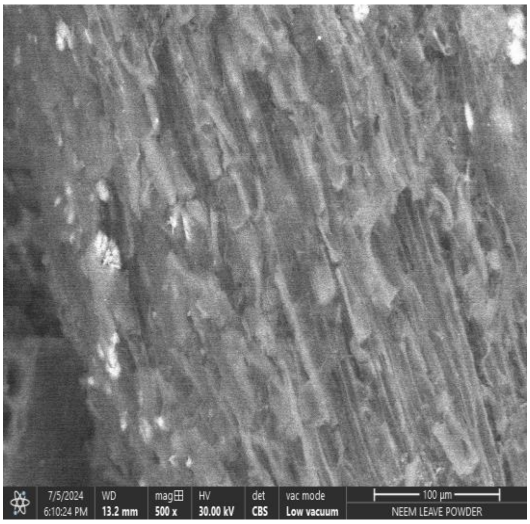

Plate 1b: SEM image of NLP at 500x

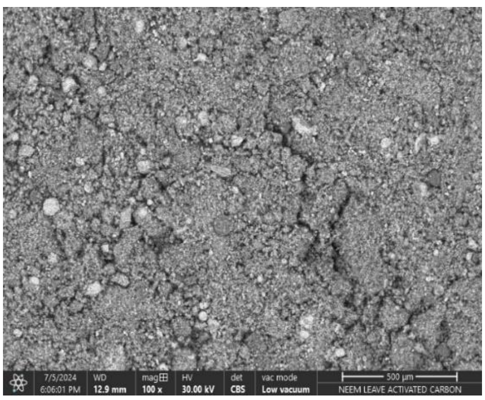

Plate 1c: SEM image of NLAC at 100x

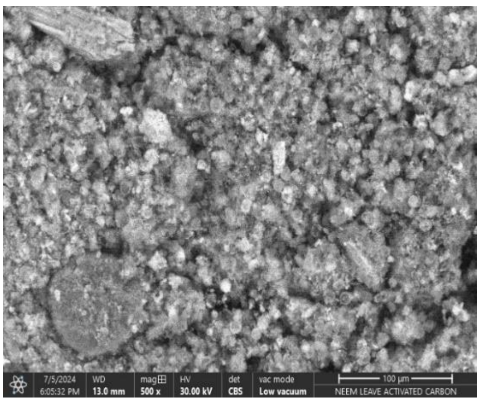

Plate 1d: SEM image of NLAC at 500x

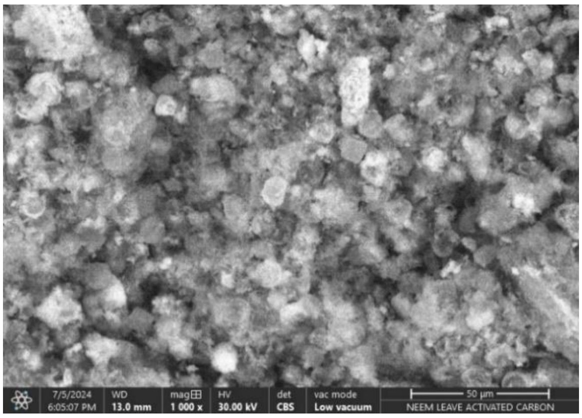

Plate 1e: SEM image of NLAC at 1000x

Scanning Electron Microscopy Analysis (SEM)
Scanning Electron Microscopy (SEM) is employed for the morphological investigation of all samples. NLP exhibited fibrous, irregular structures with fewer pores, while NLAC showed granular, fragmented morphology with highly porous surfaces. This structural difference supports the enhanced adsorption performance of the activated form (Sadare *et al.,* 2022).

Brunauer-Emmett-Teller Analysis (BET)
Table 1: BET Result of Neem leaves powder and its activated carbon

S/N	Adsorbent	BET Surface Area (m^2/g)	Pore Size (nm)	Pore Volume (cm^3/g)
1	Neem leaves powder	294.381	2.128	0.165
2	Neem leaves activated carbon	427.154	2.105	0.218

NLAC had a higher surface area (427.154 m^2/g) and pore volume (0.218 cm^3/g) compared to NLP (294.381 m^2/g and 0.165 cm^3/g, respectively) The increase in surface area after activation indicates successful development of additional micropores and mesopores, likely due to chemical activation (H_3PO_4 treatment) (Foo and Hameed, 2010). Both fell within mesoporous ranges (2–50 nm), suitable for adsorption of organic molecules. Activation improved pore density rather than width (Rouquerol *et al.,* 2013). The increase in pore volume confirms successful pore development during activation. Higher pore volume enhances adsorption capacity (Yagmur *et al.,* 2021).

Adsorption Optimization Studies
Effect of Concentration on Adsorption of Benzene and Toluene Using Neem Leaves Activated Carbon

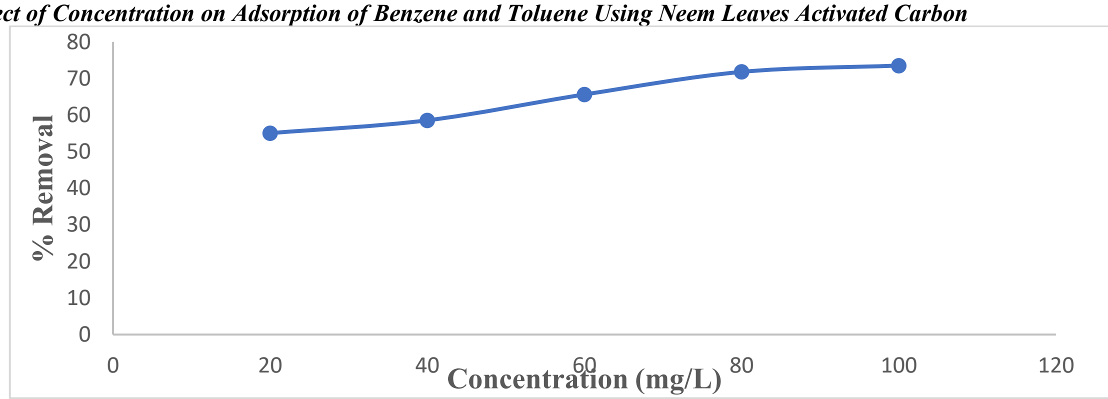

Figure 3: Effect of Concentration on Removal of Benzene

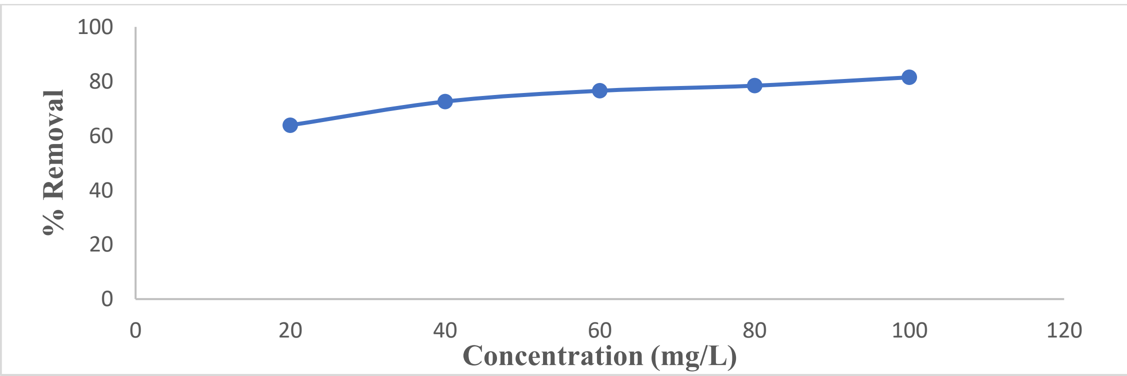

Figure 4: Effect of Concentration on Removal of Toluene

Figures 3 and 4 illustrate the impact of concentration variations on the uptake of Benzene and Toluene by the activated carbon derived from Neem leaves. The observed trend indicated that the removal efficiencies increased with initial concentration due to a higher driving force and greater interaction with available sites. Benzene removal rose from 55 % to 74 %, and toluene from 64 % to 81 % (Sadare *et al.,* 2022), with slight plateaus at higher concentrations due to site saturation. Once these sites are occupied, new molecules in the solution have fewer binding opportunities, leading to a decelerated enhancement in adsorption efficiency (Melaphi *et al.,* 2023). This trend aligns with findings from analogous studies, including Melaphi *et al.* (2023), which examined the adsorptive removal of BTEX compounds utilizing activated carbon sourced from macadamia nut shells, revealing a plateau in adsorption efficiency at elevated concentrations due to site saturation.

Effect of Dose on Adsorption of Benzene and Toluene Using Neem Leaves Activated Carbon

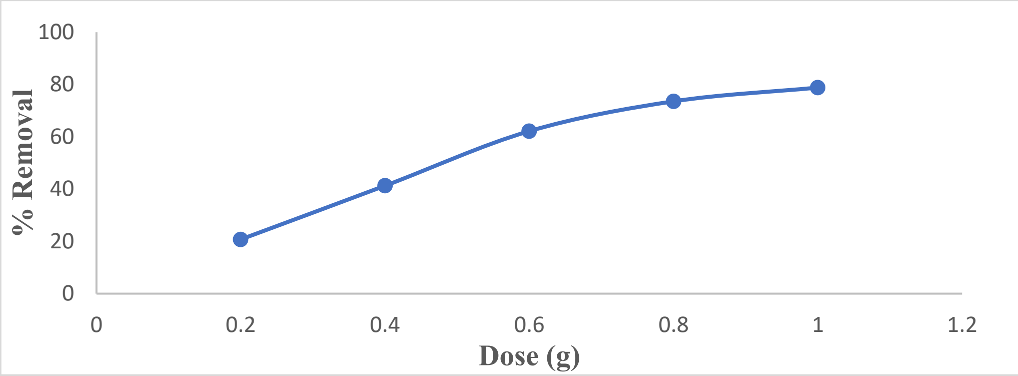

Figure 5: Effect of Dose on Removal of Benzene

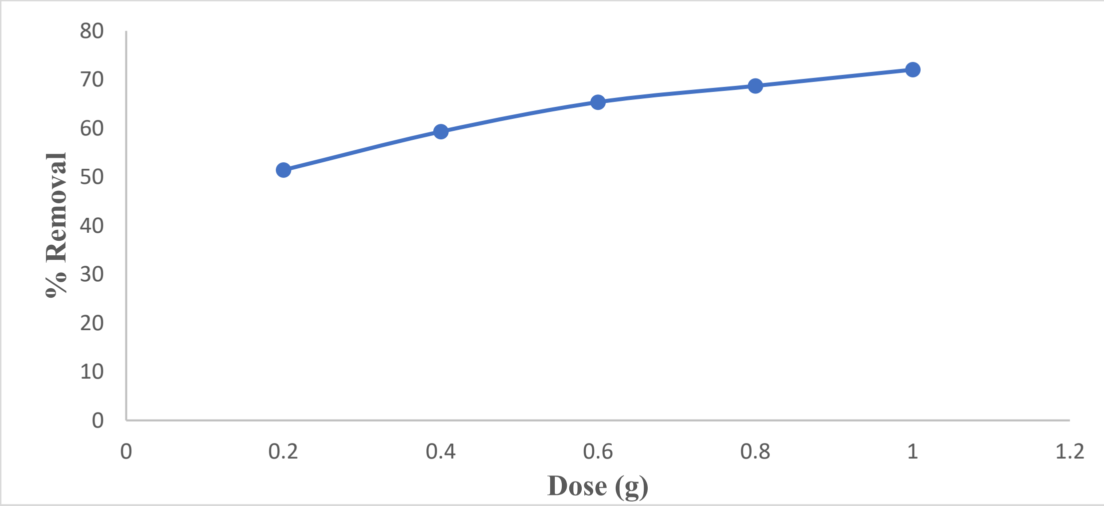

Figure 6: Effect of Dose on Removal of Toluene

A distinct pattern was noted indicating that the percentage removal of contaminants increased with an increase in the adsorbent dosage (Figures 5 and 6). Increased dosage led to higher removal due to more available active sites (Khader *et al.,* 2024). Benzene removal improved from 21 % to 79 %, and toluene from 51 % to 72 %. The removal rate plateaued at higher dosages due to saturation (Melaphi *et al.,* 2023). Comparable trends have been documented in additional research, including the study by Melaphi *et al.* (2023) regarding the adsorptive elimination of BTEX compounds utilizing activated carbon sourced from macadamia nut shells,

Effect of Contact Time on Adsorption of Benzene and Toluene Using Neem Leaves Activated Carbon

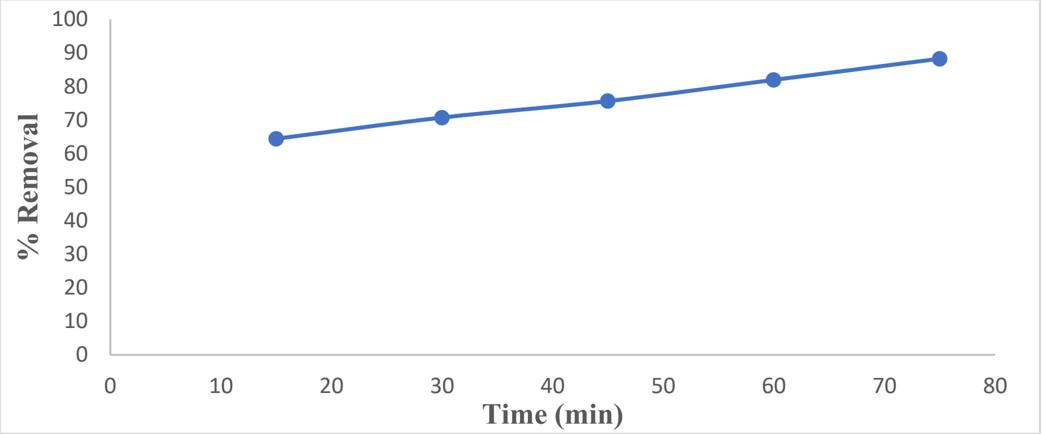

Figure 7: Effect of Contact Time on Removal of Benzene

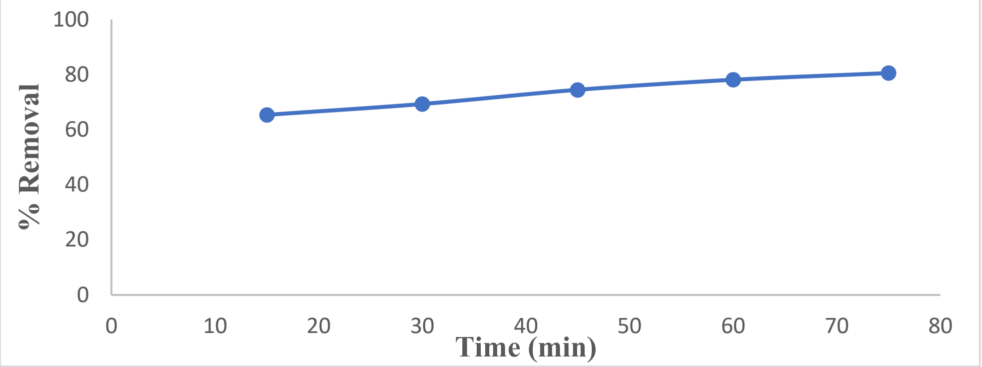

Figure 8: Effect of Contact Time on Removal of Toluene

Figures 7 and 8 illustrate the impact of contact time on the adsorptive removal of Benzene and Toluene chemicals from simulated refinery wastewater. A consistent trend was noted in which Adsorption increased with time, reaching equilibrium after 75 min. Benzene and toluene removals rose from 64 % and 65 % to 88 % and 81 %, respectively. Early rapid adsorption was followed by slower uptake, indicating saturation of sites (Sadare *et al.,* 2022).

This behavior aligns with findings from analogous studies, including those by Melaphi *et al.* (2023) on the adsorptive elimination of BTEX compounds utilizing activated carbon from macadamia nut shells.

Effect of pH on Adsorption of Benzene and Toluene Using Neem Leaves Activated Carbon

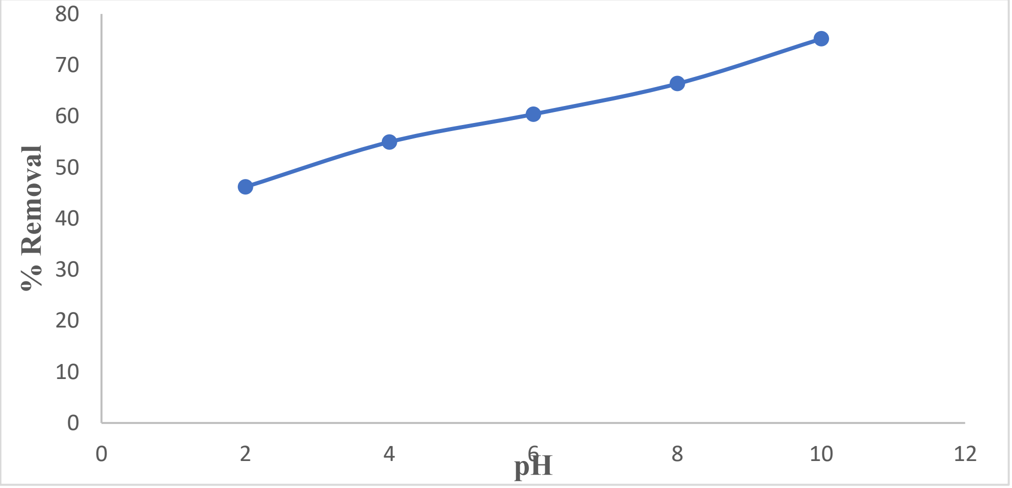

Figure 9: Effect of pH on Removal of Benzene

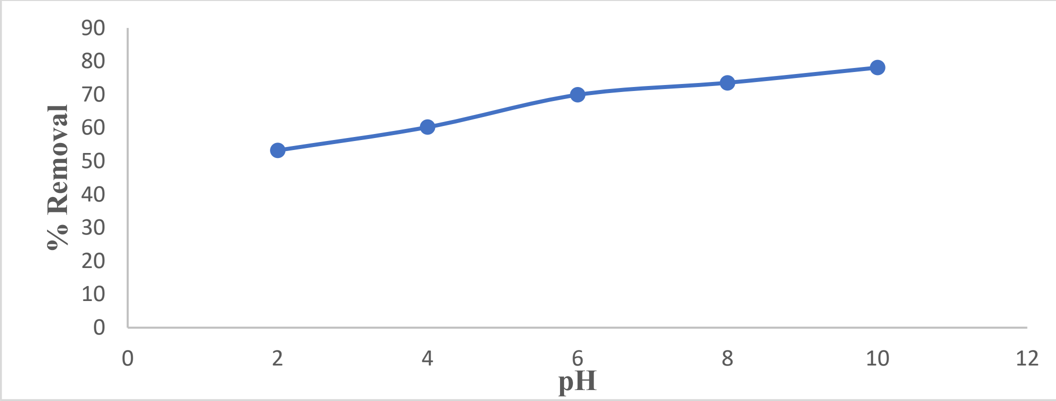

Figure 10: Effect of pH on Removal of Toluene

The influence of pH on the adsorption removal of Benzene and Toluene utilizing Neem leaves activated carbon is illustrated in Figures 9 and 10, respectively. The observed pattern indicated that the rising pH enhanced removal due to reduced competition with H^+ ions and favorable surface charge. Benzene removal increased from 46 % to 75 %, and toluene from 53 % to 78 %, with peak efficiency at pH 10. Adsorption plateaued as saturation approached (Mordhiya *et al.,* 2023). These findings align with analogous studies, including those by Das *et al.* (2020) regarding the adsorption properties of Indian Neem leaves for the removal of methyl violet dye.

Effect of Temperature on Adsorption of Benzene and Toluene Using Neem Leaves Activated Carbon

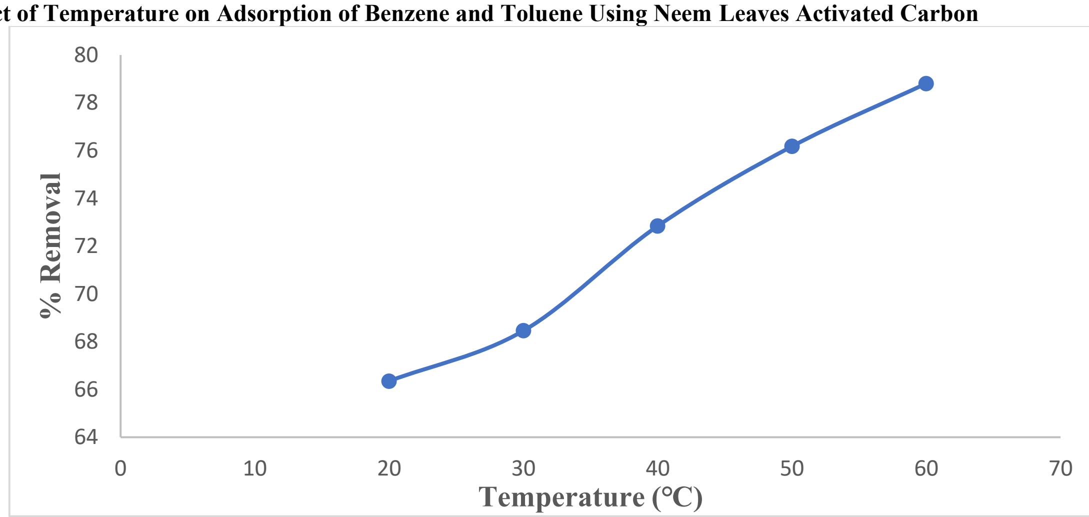

Figure 11: Effect of Temperature on Removal of Benzene

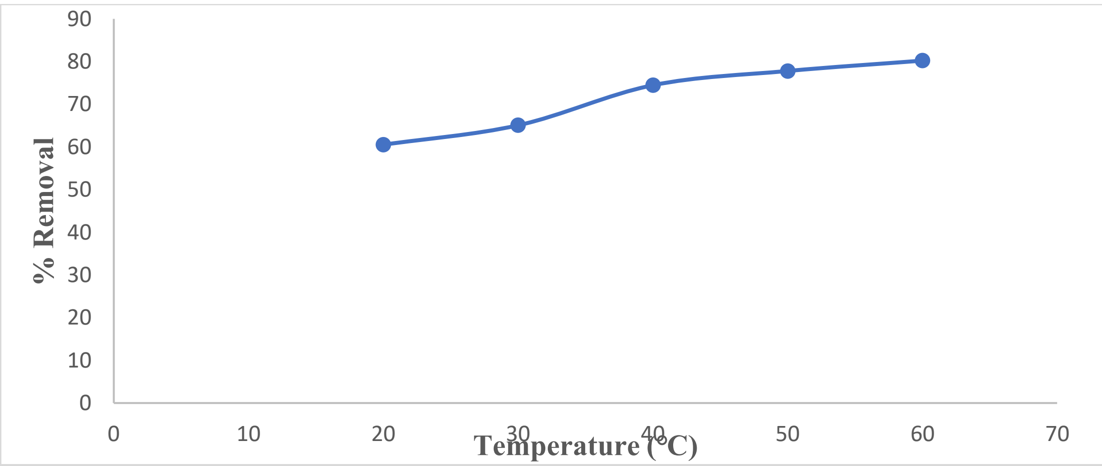

Figure 12: Effect of Temperature on Removal of Toluene

The influence of temperature on the removal of Benzene and Toluene utilizing Neem leaves activated carbon has been examined and is illustrated in Figures 11 and 12, respectively. A discernible trend was noted indicating that the process was endothermic. Higher temperatures (20–60 °C) improved molecular mobility and diffusion into pores. Benzene and toluene removal rose from 66 % to 79 % and from 61 % to 80 %, respectively. Slight plateauing near 60 °C indicated proximity to optimal capacity (Mordhiya *et al.,* 2023). The results align with prior research, including that of Das *et al.* (2020), which examined the adsorption properties of Indian Neem leaves for the removal of methyl violet dye, revealing a comparable endothermic adsorption mechanism.

Adsorption of Benzene and Toluene Using Neem Leaves Powder

Neem leaf powder was ineffective, with absorbance remaining high (4.9) under varying conditions. Its lower surface area and pore volume, coupled with polar surface chemistry, hindered interaction with nonpolar benzene and toluene (Khosrowshahi *et al.,* 2022). NLP lacked the structural and chemical features essential for effective adsorption, unlike NLAC, which benefited from activation-enhanced porosity and surface reactivity (Abdul Rasheed *et al.,* 2024).

CONCLUSION

This study conducted batch experiments on the adsorption of Benzene and Toluene from simulated industrial wastewater utilizing neem leaf and its activated carbon. The adsorbents were evaluated utilizing Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and Brunauer-Emmett-Teller (BET) methods. The absorption of benzene and toluene (mg/g) was observed to rise with increasing concentration, contact duration, adsorbent dosage, pH, and temperature. Activated carbon derived from neem leaf, possessing a surface area of 427.154 m²/g, achieved an optimal pH of 10, a contact period of 75 min, an adsorbent dosage of 1 g, a temperature of 60 °C, and an initial concentration of 100 ml/L. The percentage elimination of Benzene and Toluene was determined to be 74 % and 81 %, respectively, utilizing NLAC.

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