Recycling of Waste Expanded Polystyrene as an Effective Adsorbent of Naphthalene from Aqueous Solution

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Abstract
Batch adsorption process factors [contact time (20–150 min), adsorbent dosage (0.5–1.5 g), adsorbate concentration (5–30 mg l\(^{-1}\)), and agitation rate (100–250 rpm)] were optimised based on D-optimal Design under the Response Surface Methodology (RSM) of the Design-Expert Software (7.6.8) for the removal of naphthalene from aqueous solution using adsorbent developed from Acetylated Waste Expanded Polystyrene (AWEPs). The maximum adsorption capacity (5.6608 mg g\(^{-1}\)) achieved was well fitted to Dubinin-Radushkevich Isotherm (\(R^2 = 0.9949\)). The SSE (< 0.05) and ARE (< 4.0 %) indicated pseudo-second-order as the most suitable model. This research has demonstrated the effectiveness of the WEPs for the removal of naphthalene from the aqueous solution.

Keywords
Adsorption, D-optimal, naphthalene, waste expanded polystyrene

1 Introduction
Polycyclic aromatic hydrocarbons (PAHs) are a class of organic compounds characterised by two or more fused benzene rings. They are carcinogenic, mutagenic, and toxic. There are sixteen listed PAHs as priority pollutants that have been linked to various health challenges in humans. PAHs are by-products of various anthropogenic and industrial activities, such as incomplete combustion of coal, fuel, garbage, oil, oil spillage, organic substances, polymers, refuse, tobacco smoke, and wood, among others. The negative effect of PAHs in the environment has been a great concern to researchers to mitigate their serious effects on the human body.

PAHs have a strong resistance to biological degradation, and some conventional physicochemical processes have not demonstrated the desired potency for their removal. However, adsorption processes, involving the use of activated carbon derived from synthetic, natural, and renewable sources have been deployed for the successful removal of PAHs. This may not be unconnected to the advantages of the ease in operation, cost-effectiveness, and insensitivity to toxic substances compared to other separation techniques. Activated carbon, commonly used in the adsorption process, has high adsorption capacities for a wide range of pollutants because of its porous microstructures and large surface areas. However, the purchase cost and the cost of regeneration of AC are expensive, besides there is 10–15 % loss during regeneration. Adsorbents such as carbon nanotube, zeolite, diatomite, and organoclay have been used for the adsorption of PAHs from aqueous solutions.

Naphthalene is an important PAH that has a molecule containing two benzene rings with molecular formula \(C_{10}H_{8}\) obtainable from petroleum refining and coal tar distillation. Its presence in the environment is more pronounced, relative to the other types of PAHs. Some authors have used various types of adsorbent originated from clay, coal, and agricultural biomass for the removal of naphthalene from wastewater. Expanded polystyrene (EPS) are agglomerated small and expandable plastic “beads” used to produce food packages, because of their calendaring surface, which prevents absorption of water, oil, beverages, and other processed food products. Used EPS has relatively low scrap value and is discarded after use. Its non-biodegradable nature makes it persist in the environment and its build-up reduces the holding capacity of landfills or dumpsites. Reuse of EPS for environmental remediation is reported in the works of and Ajlil, and Reuse of EPS waste into an effective adsorbent for the removal of naphthalene from the aqueous solution.

2. Experimental
2.1 Materials and sample preparation
WEPs, sourced from dumpsites were washed to remove oil and dirt from their surfaces. The collected WEPs were soaked in detergent solution and stirred until the strength...
of the detergent became weak. They were then rinsed thoroughly with a copious amount of distilled water before being sun-dried and oven-dried at 105 °C to constant weight. They were reduced to relatively uniform sizes to reduce the impact of calendaring and expose the pores. All the reagents used in this research were of analytical grade and used without further purification.

[Acetic acid (γ: 60.052 g mol⁻¹, T0: 118 °C, ρ: 1.05 g cm⁻³, Tm: 16.6 °C, CAS 64-19-7), NaOH (γ: 39.997 g mol⁻¹, ρ: 2.13 g cm⁻³, T0: 1.388 °C, CAS 1310-73-2), Naphthalene (Tm: 80.26 °C, γ: 128.1705 g mol⁻¹, T0: 218 °C, ρ: 1.14 g cm⁻³, CAS 91-20-3), Ethanol (γ: 33.37 °C, ρ: 46.07 g mol⁻¹, Tm: -114.1 °C, Density: 789 kg m⁻³, CAS 64-17-5)].

2.2 Activation of WEPs
The WEPs were mixed in 100 ml of acetic acid (c = 4.15 mol dm⁻³) at a ratio of 1.5:1 (mass of activant/mass of precursor) and microwaved in the oven at 600 Hz for 90 min. The excess acid was boiled off. The pH of the acetylated WEPs (AWEPs) developed was neutralized with NaOH and oven-dried to constant weight.

2.3 Characterisation of AWEPs
The ash and moisture contents were determined by the ASTM analytical method and the method adopted by Ekpete and Horsfall. The ash and moisture contents were calculated according to Eqs. (1) and (2). Fourier Transform Infrared Spectroscopy (FTIR) was used to determine the functional group on the surface of AWEPs before and after adsorption. The samples were prepared with potassium bromide at a 1:10 ratio and pressed into the pelletized disc. The FTIR spectrum of WEPs, AWEPs, and spent WEPs (SWEPs) adsorbents were recorded within the range of 400–4000 cm⁻¹.

\[
\text{ash content} \% = \frac{m_1 - m_2}{m_2} \times 100 \quad (1)
\]

\[
\% \text{ moisture content} = \frac{m_3 - m_2}{m_3 - m_1} \times 100 \quad (2)
\]

where \(m_1\) is the mass of ash, \(m_2\) is the mass of the dried sample, \(m_3\) is the mass of crucible, and \(m_3\) is the mass of crucible with the wet sample.

2.4 Batch adsorption
2.4.1 Adsorption studies
A stock solution of naphthalene of 200 mg l⁻¹ was prepared by dissolving 200 mg of naphthalene in 100 ml of ethanol. Distilled water was added to make 1 l. The stock solution was further diluted with distilled water accordingly to produce the desired concentration. The study type used for this experimental design for the adsorption study was the Response Surface Methodology (RSM). The initial design suggested by the Design-Expert software (7.6.8) was D-optimal. Zero (0) centre point was chosen for the design with no blocks selected, and a build time of 875 min was used for the design model. The factors considered were activant concentration (A), impregnation ratio (B), microwave time (C), and microwave frequency (D) while the response is adsorption capacity. Determined amount (0.5–1.5 g) of AWEPs was mixed with 100 ml naphthalene solution of specific concentration (5–30 mg l⁻¹) and shaken on a rotary shaker at a specific agitation rate (100–250 rpm), and at room temperature (28±2 °C), according to the D-optimal Design (Table 1). The mixture was centrifuged and the supernatant was analysed using UV-Spectrometer (UV-6100A, manufacturer: METASH A-MATRIX) at a wavelength of 275 nm. Different concentrations (5–50 mg l⁻¹) of naphthalene were prepared earlier, and UV-Spectrometer (UV-6100A) at a wavelength of 275 nm was used to develop the calibration curve from which the equation for evaluating the naphthalene concentration from absorption was determined. The adsorption capacity and removal efficiency were evaluated by Eqs. (3) and (4).

\[
\text{adsorption capacity} = \frac{(Y_o - Y_t)V}{m} \quad (3)
\]

\[
\text{removal (}%) = \frac{Y_o - Y_t}{Y_o} \times 100 \quad (4)
\]

where \(Y_o\) (mg l⁻¹) is the initial concentration of naphthalene solution in contact with adsorbent, \(Y_t\) (mg l⁻¹) is the final concentration of naphthalene solution after the batch adsorption procedure at any time \(t\), \(m\) (g) is the mass of adsorbent, and \(V\) (l) is the volume of the naphthalene in solution.

2.4.2 Effect of adsorption factors
The optimum conditions for the adsorption of naphthalene using AWEPs was subjected to OFAT procedures during which the effects of contact time and concentration were investigated when one of the factors was varied at a time.

2.5 Investigation of suitable adsorption isotherms
The adsorption data obtained were fitted to selected isotherm models. Their constants were evaluated, and the
correlation coefficient ($R^2$) was used to express the extent of correlation between the experimental data and the model predicted values.\(^\text{41}\)

**2.5.1 Langmuir isotherm model**

The Langmuir isotherm model is expressed by Eq. (5) and the linear form Eq. (6) was used to generate the plot of \(\frac{Y_e}{q_e}\) against \(Y_e\), which gave a straight-line graph with a slope \(\frac{1}{q_mK_L}\) and intercept of \(\frac{1}{q_m}\):

\[
q_e = \frac{q_mK_LY_e}{1 + K_LY_e}
\]  \hspace{1cm} (5)

\[
\frac{Y_e}{q_e} = \frac{1}{q_mK_L} + \frac{Y_e}{q_m}
\]  \hspace{1cm} (6)

where \(q_e\) is the adsorption capacity at equilibrium (mg g\(^{-1}\)), \(Y_e\) is the equilibrium concentration of the adsorbate solution (mg l\(^{-1}\)), \(K_L\) is the constant related to the free energy of adsorption (l mg\(^{-1}\)), and \(q_m\) is the maximum adsorption capacity at monolayer coverage (mg g\(^{-1}\)).

**2.5.2 Freundlich isotherm model**

The Freundlich isotherm model equation, Eq. (7), assumes a heterogeneous adsorbent surface with its adsorption sites at varying energy levels. Its linear form Eq. (8) was used to generate the plot of ln\(q_e\) against ln\(Y_e\), that is needed to determine the Freundlich constants (\(K_f\) and 1/n).\(^\text{42}\)

\[
q_e = k_fY_e^n
\]  \hspace{1cm} (7)

\[
q_e = \frac{1}{n}\ln Y_e + \ln k_f
\]  \hspace{1cm} (8)

where \(k_f\) is the Freundlich constant, and \(q_e\) is the adsorption capacity at equilibrium (mg g\(^{-1}\)).

**2.5.3 Temkin isotherm model**

Temkin isotherm model explicates that the adsorbate-adsorbent interactions and the related change in heat and/or energy of adsorption are assumed to be linear -+-+characterised by a uniform distribution of binding energy and up to some maximum binding energy.\(^\text{42}\) Such an assumption cannot hold for a logarithmic relationship. The Temkin isotherm model is expressed by Eq. (9), while its linear form is expressed by Eq. (10), and was further simplified to Eq. (11).

\[
q_e = \frac{RT}{b}\ln(AY_e)
\]  \hspace{1cm} (9)

\[
q_e = \frac{RT}{b}\ln A + \frac{RT}{b}\log Y_e
\]  \hspace{1cm} (10)

\[
q_e = B\ln A + B\ln Y_e
\]  \hspace{1cm} (11)

where \(B = RT/b\), \(B\) is the molecular interaction parameter related to the heat of adsorption, \(A\) and \(B\) are the Temkin isotherm constants, \(T\) (K) is the absolute temperature, and \(R\) is the ideal gas constant (8314 J mol\(^{-1}\) K\(^{-1}\)).

**2.5.4 Dubinin-Radushkevich isotherm**

Dubinin-Radushkevich (D-R) isotherm Eq. (12) assumes that pore filling influenced the adsorption mechanism in micropores and not a layer-by-layer formation of a film in the walls of the adsorbent pores.\(^\text{43}\) The linear form of the D-R isotherm equation is expressed in Eq. (13) and was used to plot ln\(q_e\) against \(\varepsilon\), needed to determine the \(q_m\) and \(\beta\) from the intercept and slope.

\[
q_e = q_{me}e^{-\beta\varepsilon^2}
\]  \hspace{1cm} (12)

\[
\ln q_e = \ln q_{me} - \beta\varepsilon
\]  \hspace{1cm} (13)

where \(\beta\) (K\(^2\) mol\(^2\)) is the free energy of sorption per mole of the naphthalene as it migrates to the surface of WEPs from an infinite distance in the solution, \(q_m\) is the maximum adsorption capacity, and \(\varepsilon\) is the Polanyi potential (l mol\(^{-1}\)), which is expressed by Eq. (14):

\[
\varepsilon = RT\ln \left(1 + \frac{1}{Y_e}\right)
\]  \hspace{1cm} (14)

where \(R\) is the universal gas constant (8.314 J mol\(^{-1}\) K\(^{-1}\)), \(T\) is the absolute temperature (K), and \(Y_e\) is the equilibrium concentration of naphthalene.

**2.6 Adsorption kinetics studies**

The pseudo-first-order model, pseudo-second-order model, and intraparticle diffusion model were employed to evaluate the experimental data generated in this study.

**2.6.1 Pseudo-first-order model**

This model (Eq. (15)) is based on a solid capacity and its plot of ln\((q_e - q_t)\) vs \(t\) that gives a straight line from which \(K_1\) and \(q\) were evaluated based on the slope and intercept.

\[
\ln(q_e - q_t) = \ln q_e - K_1t
\]  \hspace{1cm} (15)

where \(q_e\) is the equilibrium adsorption capacity (mg g\(^{-1}\)), \(q_t\) is the adsorption capacity at time (mg g\(^{-1}\)), \(K_1\) is the pseudo-first-order rate constant (l min\(^{-1}\)), and \(t\) is the time taken.

**2.6.2 Pseudo-second-order model**

This model (Eq. (16)) was used to plot \(t/q_e\) vs \(t\), which gave a straight line from which \(q_e\) and \(K_2\) were evaluated.
\[ \frac{t}{q_t} = \frac{1}{q_K} + \frac{t}{q_e} \]  

(16)

where \( K_2 \) is the rate constant of pseudo-second-order adsorption (g mg\(^{-1}\) min\(^{-1}\)).

6.3 Intraparticle diffusion model

This model indicates that the rate-limiting step is the transport of the solute from the bulk of the solution to the adsorbent pores through the intraparticle process. It was expressed according to Eq. (17):

\[ q_t = k_{\text{diff}} t^2 + C \]  

(17)

where \( k_{\text{diff}} \) is the intraparticle diffusion rate constant (mg g\(^{-1}\) min\(^{-0.5}\))., \( t \) is time, and \( C \) is constant.

2.7 Test of the kinetics model

The impact of various error functions on the predicted isotherm parameters was analysed to determine the order of suitability of the selected isotherm models. Error functions such as Average Relative Error (ARE) and Sum of Error Square (SSE) were calculated according to the Eqs. (16) and (17):

\[ \text{ARE} = \frac{100}{n} \sum_{i=1}^{n} \left| \frac{q_{e,\exp} - q_{e,\text{cal}}}{q_{e,\exp}} \right| \]  

(16)

\[ \text{SSE} = \sum_{i=1}^{n} \left( \frac{q_{e,\text{cal}} - q_{e,\exp}}{n} \right)^2 \]  

(17)

where \( q_{e,\exp} \) is the adsorption capacity at equilibrium experimental (mg g\(^{-1}\)), \( q_{e,\text{cal}} \) is the adsorption capacity at equilibrium calculated (mg g\(^{-1}\)), and \( n \) is the number of data points.

3 Results and discussion

3.1 Physicochemical analysis of the adsorbent

There was a significant difference in the ash content between the WEPs (0.10 %) and AWEPs (0.39 %) (Table 2). This may be due to the impact of the activant on the composition of the untreated WEPs, and this further suggested that the activation process was evident. The moisture content of AWEPs (14.82 %) was higher than that of WEPs (0.90 %), and this may be due to the soaking step during the activation process.

3.2 FTIR characterisation of WEPs and AWEPs

The IR peaks observed in the WEPs ranged from 623.1 cm\(^{-1}\) to 3933.4 cm\(^{-1}\) and the peak height ranged from 21.8 cm\(^{-1}\) to 36.9 cm\(^{-1}\) (Fig. 1a). AWEPs had IR peaks that ranged from 613.7 cm\(^{-1}\) to 3892.3 cm\(^{-1}\) and peak height ranged from 3.5 cm\(^{-1}\) to 21.8 cm\(^{-1}\). The IR peak for the spent SAWEPs ranged from 618.5 cm\(^{-1}\) to 3930.4, while the peak height ranged from 10 cm\(^{-1}\) to 27.4 cm\(^{-1}\). The IR peaks observed in the WEPs shifted from (623.1–3933.4 cm\(^{-1}\)) to (613.7–3892.3 cm\(^{-1}\)) on AWEPs. All these changes suggested the impact of acetic acid activation on the surface improvement of the WEPs. The aromatic C–H band present in both WEPs and AWEPs is attributed to the presence of an aromatic benzene ring that is not split by the acidic modification on the WEPs. C–C–O at 1332 cm\(^{-1}\) was found in WEPs but disappeared in AWEPs due to the modification by acetic acid. These bands indicate the possible involvement of these functional groups on the surface of AWEPs in the naphthalene adsorption process.

WEPs and AWEPs surface chemistry were found to be different as some of the functional groups such as O–H and C–C–O disappeared due to the activation process.

3.3 Design summary for the adsorption capacity of AWEPs for naphthalene

Run 4 (60 min, 100 rpm, 5 mg l\(^{-1}\), and 1.5 g) gave the lowest adsorption capacity (0.1623 mg g\(^{-1}\)), while Run 3 (30 min, 100 rpm, 30 mg l\(^{-1}\), and 0.5 g) gave the highest adsorption capacity (5.6608 mg g\(^{-1}\)) (Table 3), which

| Table 2 – Proximate analysis of WEPS and AWEPS |
| Sample | Ash content / % | Moisture content / % |
|--------|----------------|---------------------|
| WEPs   | 0.10           | 0.90                |
| AWEPs  | 0.39           | 14.82               |

WEPs – Waste Expanded Polystyrene, AWEPs – Acetylated Waste Expanded Polystyrene
is higher than adsorption capacity of 1.44, 1.595, 1.527, and 4.39 mg g\(^{-1}\), reported by Ania et al.,\(^{49}\) Murilo et al.,\(^{10}\) and Alade et al.,\(^{8}\) for activated carbons, activated clay, and flamboyant pod activated carbon studied for the removal of naphthalene. A quadratic model was selected for this study because of its least standard deviation (10.33) and high \(R^2\) (0.9835). The \(R^2\) (0.9548) was very close to the adjusted \(R^2\) of 0.9548, with less than 0.2 differences, as normally expected.\(^{44}\) This indicated no large block effect nor any possible problem with the model and data obtained.\(^{30}\) Adequate Precision, which measures the signal-to-noise ratio of the data, was 28.793, which was greater than the desired value (4.0), thus making the developed model very suitable to navigate the design space.\(^{31}\)

### Table 3 – Results of responses from adsorption experimental data

| Run | A: time/min | B: agitation rate/rpm | C: concentration/mg l\(^{-1}\) | D: dosage/g | adsorption capacity/mg g\(^{-1}\) |
|-----|-------------|-----------------------|-------------------------------|-------------|-------------------------------|
| 1   | 30.00       | 100.00                | 17.50                         | 1.50        | 0.6188                        |
| 2   | 45.00       | 250.00                | 30.00                         | 1.50        | 0.7565                        |
| 3   | 30.00       | 100.00                | 30.00                         | 0.50        | 5.6608                        |
| 4   | 60.00       | 100.00                | 5.00                          | 1.50        | 0.1623                        |
| 5   | 30.00       | 100.00                | 5.00                          | 0.50        | 0.4869                        |
| 6   | 60.00       | 100.00                | 30.00                         | 1.50        | 1.4231                        |
| 7   | 30.00       | 250.00                | 5.00                          | 1.50        | 0.3072                        |
| 8   | 30.00       | 250.00                | 17.50                         | 0.50        | 2.8130                        |
| 9   | 60.00       | 100.00                | 17.50                         | 0.50        | 2.9000                        |
| 10  | 30.00       | 175.00                | 30.00                         | 1.50        | 1.3072                        |
| 11  | 60.00       | 250.00                | 5.00                          | 0.50        | 0.9217                        |
| 12  | 60.00       | 100.00                | 30.00                         | 1.50        | 1.3072                        |
| 13  | 30.00       | 100.00                | 30.00                         | 0.50        | 3.0521                        |
| 14  | 60.00       | 250.00                | 30.00                         | 1.50        | 0.5898                        |
| 15  | 60.00       | 250.00                | 17.50                         | 0.50        | 4.4434                        |
| 16  | 60.00       | 175.00                | 17.50                         | 1.00        | 0.4065                        |
| 17  | 30.00       | 250.00                | 5.00                          | 1.50        | 1.9608                        |
| 18  | 45.00       | 250.00                | 17.50                         | 1.00        | 0.9717                        |
| 19  | 60.00       | 250.00                | 30.00                         | 0.50        | 4.7043                        |
| 20  | 30.00       | 100.00                | 5.00                          | 0.50        | 0.9838                        |
| 21  | 30.00       | 175.00                | 17.50                         | 1.00        | 0.4500                        |
| 22  | 45.00       | 100.00                | 17.50                         | 1.00        | 1.3630                        |
| 23  | 45.00       | 175.00                | 23.75                         | 1.00        | 2.2489                        |
| 24  | 45.00       | 175.00                | 5.00                          | 1.00        | 0.3304                        |
| 25  | 30.00       | 250.00                | 30.00                         | 1.00        | 2.2217                        |

### 3.4 Analysis of variance (ANOVA) for adsorption capacity of AWEPs

Prob > F of any term or model, less than 0.05, at a 95% confidence interval is taken as significant.\(^{31}\) The model F-value of 34.16 implies the model is significant and it has about 0.01% chance of occurrence due to noise. Thus, C, AB, AC, AD, BC, BD, and CD are significant model terms (Table 4). The “Lack of fit F-value” of 0.63 implies the Lack of fit is not significant relative to the pure error, which makes the model fit\(^{31}\) and there is a 62.61% chance that a “Lack of fit F-value” this large could occur due to noise. This is further illustrated by Fig. 2, showing the effects of the model terms concerning Normal % probability. The points are distributed on the normal line starting from approximately 2 to 97% on normal percentage distribution, Y-axis, and −1.5 to 2.5 on internally studentized residuals, X-axis though there is a stacking of the points.\(^{31}\)
The model equations, (Eqs. (18) and (19)), show the relationship between the adsorption capacity and the selected factors that can be used to predict the naphthalene adsorption. Model terms B, D, AD, D^2, A^2, and C^2 have positive coefficients, which indicate an increase in the adsorption capacity of AWEPs in the factors. The negative coefficients as observed in A, C, AB, AC, BD, CD, and B^2 indicate an antagonistic influence of these factors on the adsorption capacity of naphthalene. The empirical model equations in terms of coded factors are given in Eq. (18), for the significant and non-significant terms.

\[
(\text{adsorption capacity})^{-1} = +3.01 - 1.83A + 2.46B - 25.23C + 6.27D - 30.38AB - 31.98AC + 24.36AD + 33.65BC - 22.59BD - 24.79CD + 8.26A^2 - 0.6B^2 + 7.35C^2 + 19.45D^2
\]  

A, B, C, and D are the coded variables for activant concentration, IMR, microwave time, and frequency, respectively.

3.6 Model graph for the selected factors on adsorption capacity for naphthalene

The plot of agitation rate with time (Fig. 3) shows a gradual decrease in adsorption capacity before a steep slope moving upward, and this indicates that the two factors cause a decrease, and then finally increase the adsorption rate. There is a slight decrease in the slope of concentration against time before (Fig. 4) and thus, means that the ...
Fig. 3  – (a) Contour plot, and (b) 3D plot showing the relationship between time and agitation rate.

Fig. 4  – (a) Contour plot, and (b) 3D plot showing the relationship between time and concentration.

Fig. 5  – (a) Contour plot, and (b) 3D plot showing the relationship between dosage and time.
Fig. 6 – (a) Contour plot, and (b) 3D plot showing the relationship between concentration and agitation rate

Fig. 7 – (a) Contour plot, and (b) 3D plot showing the relationship between dosage and agitation rate

Fig. 8 – (a) Contour plot, and (b) 3D plot showing the relationship between dosage and concentration
lower value of these factors decreases the adsorption rate. Contrary to Figs. 3 and 4, there is a parabolic curve, an increase in adsorption capacity in the plot of dosage and time (Fig. 5). Further increase in dosage value with time caused a decrease in adsorption capacity (Fig. 6). Concentration against the agitation rate plot shows an increase in adsorption capacity (Fig. 4). Other factors that increased the adsorption rate were the dosage value and agitation rate (Fig. 7), but the contrary result was experienced in Fig. 8, in which the dosage and concentration decreased with the adsorption rate.

3.7 Numerical optimisation studies on adsorption capacity

Numerical optimisation was obtained from the software. The four factors (time, agitation rate, concentration, and dosage) were all set to “is in range” (Table 4), while the adsorption capacity was set to “maximise” with its upper and lower limit, respectively. The desirability values were 0.994, and the optimum values suggested by the software were 60 min, 100 rpm, 5 mg l\(^{-1}\), and 1.5 g for time, agitation rate, concentration, and dosage, respectively (Fig. 9).

Table 5 – Selected factors used for optimisation showing their respective ranges

| Name                  | Goal            | Lower Limit | Upper Limit |
|-----------------------|-----------------|-------------|-------------|
| time                  | is in range     | 30          | 60          |
| agitation rate        | is in range     | 100         | 250         |
| concentration         | is in range     | 5           | 30          |
| dosage                | is in range     | 0.5         | 1.5         |
| (adsorption capacity) | ^−3 maximise    | 0.00551244  | 233.908     |

3.8 Effect of concentration on adsorption of naphthalene

An increase in the adsorbate initial concentration (10–30 mg l\(^{-1}\)) leads to an increase in the adsorption capacity (1.921–5.9217 mg g\(^{-1}\)) of the adsorbent due to the increase in the driving force of the concentration gradient (Fig. 10a). The adsorption of naphthalene was rapid at the initial stage of the contact time (30 min) for all the concentrations. This was because, in the beginning, all active sites on the adsorbent were vacant, hence, adsorption proceeded at a faster rate. After this, the rates of adsorption and desorption tended to be equal, and the extent of adsorption reduced and eventually became almost constant at equilibrium. The percentage of removal also increased with increasing time for all the concentrations (Fig. 10b).

3.9 Effect of dosage on the adsorption of naphthalene

An increase in adsorbent dosage (0.5–2.5 g) led to a reduction (5.5739–1.1930 mg g\(^{-1}\)) in adsorption capacity (Fig. 11a), because of the effect of partial aggregation of naphthalene on the adsorbent surface, resulting in a decrease in total surface area available for naphthalene molecules. An increase in the adsorbent dosage increased the removal efficiency of naphthalene from 92.9 % to 99.4 %.
at 180 min, due to the availability of more adsorption sites on the adsorbent (Fig. 11b).\textsuperscript{52,53}

3.10 Adsorption isotherm study

3.10.1 Langmuir isotherm model

The values of $q_m$ and $K_L$ for AWEPs (Fig. 12) were $-8.7108 \text{ mg g}^{-1}$ and $-0.1106$, respectively. The low value of $R^2$ (0.4238) indicated that the experimental equilibrium data were not well described by the Langmuir model. The maximum adsorption capacity ($q_m$) obtained from this research ($-8.7108 \text{ mg g}^{-1}$) (Table 5), was lower for the adsorption of naphthalene onto mesoporous molecular sieves, zeolite, Mesoporous organosilica, and banana peel activated carbon, respectively.\textsuperscript{10,54}

3.10.2 Freundlich isotherm model

The Freundlich model to estimate $K_f$ and $1/n$ are $-0.0511 \text{ l mg}^{-1}$ and $0.8059$ from its intercepts and the slope, respectively (Fig. 13). The values of $1/n$ ranging from 0 to 1, indicates the model’s favourability for the adsorption process.\textsuperscript{55} and this value is lower than the previous research, except 0.7986 derived by Gupta and Gupta.\textsuperscript{57} The negative $K_f$ value obtained from this study was lower than...
the $K_f$ value derived by Carla et al., Chang et al., Gupta and Gupta, respectively. The $R^2$ (0.9777) was relatively high, thus making the Freundlich isotherm a better model compared to the Langmuir (0.4238) (Table 5).

### 3.10.3 Temkin isotherm model

Estimated Temkin isotherm parameters $A$ and $B$ were 6.0670 l g$^{-1}$ and 1.2408 J mol$^{-1}$, respectively, (Fig. 14), with an $R^2$ value of 0.9883, which is higher than the $R^2$ values of 0.4238 and 0.9777 obtained for Freundlich and Langmuir, suggesting that the data better fitted Temkin isotherm than the other two.

### 3.10.4 Dubinin-Radushkevich isotherm for the effect of concentration

The Dubinin-Radushkevich isotherm model is described by the plot of $\ln(q_m - q_e)$ against $E^2$ (Fig. 15) and the estimated parameters, $q_m$ and $\beta$ are 25.1851 mg g$^{-1}$ and $1 \cdot 10^6$ KJ$^2$ mol$^2$, respectively.

The mean free energy of biosorption $\beta$ determines the biosorption mechanism as either a physical or chemical process. The biosorption process is chemically driven if the value of $\beta$ is greater than 8 KJ$^2$ mol$^2$, but involves physical mechanism if less. The value obtained in this study indicated that the adsorption of naphthalene onto AWEPs was driven by a chemical process. The $R^2$ value was 0.9949, which was the highest of all the isotherm models investigated.

### Table 6 – Comparison of Langmuir and Freundlich Isotherm Parameter to other studies

| Adsorbent                      | Langmuir parameter | Freundlich parameters | Author                        |
|--------------------------------|--------------------|-----------------------|-------------------------------|
| Mesoporous Molecular Sieves   | 0.10624            | NR                    | Murilo et al. (2004)$^{10}$   |
| Zeolite                        | 0.769              | 4.215                 | Chang et al. (2004)$^{56}$    |
| Mesoporous Organosilica       | 0.0466             | 0.227                 | Carla et al. (2011)$^{54}$    |
| Banana Peel Activated Carbon  | NR$^*$             | 21.54                 | Gupta and Gupta (2015)$^{57}$ |
| AWEPs                          | −8.7108            | −0.0511               | This study                    |

$^*$NR – not reported

Fig. 16 – Plot of $\ln(q_m - q_e)$ against time for the effect of concentration

Fig. 17 – Plot of $t/q_e$ against time for the effect of concentration

Fig. 18 – Plot of $q_e$ against $\ln t$ for the effect of concentration

Fig. 19 – Plot of $q_e$ against $T^{0.5}$ for the effect of concentration
3.11 Investigation of adsorption kinetics

3.11.1 Pseudo-first-order kinetics model

The estimated $k_1$ values of 0.023, 0.020, and 0.026 had no visible trend (Fig. 16, Table 7). There is a wide disparity between the calculated equilibrium adsorption capacity $q_{e,cal}$ and the experimental equilibrium adsorption capacity $q_{e,exp}$ values, contrary to a good correlation expected. This suggests that the adsorption of naphthalene onto AWEPs does not fit the first-order kinetics. The $R^2$ for 10, 20, and 30 mg l$^{-1}$ are 0.9609, 0.7838, and 0.8034, respectively and are relatively high.

### Table 8 – Kinetic parameters obtained for the kinetics models

| Kinetics model          | Parameters | 10 mg l$^{-1}$ | 20 mg l$^{-1}$ | 30 mg l$^{-1}$ |
|-------------------------|------------|----------------|----------------|----------------|
| Pseudo-first order      | $q_{e,cal}$/mg g$^{-1}$ | 1.648          | 1.389          | 1.933          |
|                         | $q_{e,exp}$/mg g$^{-1}$ | 1.922          | 3.878          | 5.922          |
|                         | $K_1$/min$^{-1}$ | 0.023          | 0.020          | 0.026          |
|                         | $R^2$      | 0.9609         | 0.7838         | 0.8034         |
| Pseudo-second order     | $q_{e,cal}$/mg g$^{-1}$ | 2.349          | 4.297          | 6.545          |
|                         | $q_{e,exp}$/mg g$^{-1}$ | 1.922          | 3.878          | 5.922          |
|                         | $K_2$/g mg$^{-1}$ min$^{-1}$ | 0.011          | 0.013          | 0.010          |
|                         | $R^2$      | 0.9750         | 0.9804         | 0.9785         |
| Elovich                 | A          | 0.140          | 0.872          | 1.816          |
|                         | B          | 1.951          | 1.298          | 0.900          |
|                         | $R^2$      | 0.8905         | 0.6349         | 0.5503         |
| Intraparticle diffusion  | $K_{id}$  | 0.120          | 0.172          | 0.246          |
|                         | C          | 0.434          | 1.821          | 3.060          |
|                         | $R^2$      | 0.8194         | 0.5322         | 0.4504         |

3.11.2 Pseudo-second-order kinetics model for the effect of concentration

The $q_{e,cal}$ obtained from the plot of $t/q$ vs $t$ (Fig. 17) were 2.349, 4.297, and 6.545 mg l$^{-1}$, showing a direct relationship with the increase in concentration. The values of $q_{e,cal}$ were relatively closer to the values of $q_{e,exp}$. The values of $K_2$ (0.011, 0.013, and 0.010 g mg$^{-1}$ min$^{-1}$) had no particular pattern with increasing concentration. The $R^2$, (0.9750, 0.9804, and 0.978) were higher than the $R^2$, obtained for the first-order model (Table 7).

3.11.3 Elovich kinetic model

The plot of $q_e$ against $\ln t$ for Elovich kinetic model (Fig. 18) gave the values of initial adsorption rate, $\alpha$ as 0.14, 0.872, and 0.814, while the rate of surface coverage, $\beta$, were 1.951, 1.298, and 1.900 for 10, 20 and 30 mg l$^{-1}$, respectively. The $R^2$ (0.8905, 0.6349, and 0.5503) displayed an inverse relation with the initial concentration. The increasing order of suitability of the kinetic models, based on $R^2$ obtained, was Elovich < pseudo-first-order < pseudo-second-order kinetic model. Therefore, the adsorption experiment of naphthalene onto AWEPs is best described by the pseudo-second-order kinetic model.

3.11.4 Intraparticle diffusion model

The kinetic parameter (C) obtained from the plot of $q_e$ against $t^2$ (Fig. 19) was 0.434, 1.821, and 3.06 for 10, 20, and 30 mg l$^{-1}$, respectively, implying that values of $C$ were directly proportional to the surface adsorption of naphthalene in the rate-controlling step. The intraparticle diffusion rate constant, $K_{id}$ values were 0.12, 0.172, and 0.246, showing that $K_{id}$ increased with concentration. Intraparticle diffusion becomes the sole rate-limiting step if the plot is linear and passes through the origin. This study deviated from this condition, thus, intraparticle diffusion is not the sole rate-limiting step. The $R^2$ value obtained reduced with increasing concentration.

3.12 Error analysis for the kinetic models

It was found that the SSE$_{\text{pseudo-first-order}}$ value for the effect of concentration ranged between 0.017 and 0.89, while the value obtained for the pseudo-second-order ranged between 0.260 and 0.054. ARE$_{\text{pseudo-first-order}}$ value for the effect of concentration was greater than 9 %, while ARE$_{\text{pseudo-second-order}}$ value for the same factor was less than 4 % (Table 8). Therefore, the pseudo-second order model better predicts the adsorption of naphthalene on AWEPs than the pseudo-first order model.

### Table 9 – SSE and ARE values for kinetic models

| Kinetic model     | Concentration/mg l$^{-1}$ | SSE     | ARE/%  |
|-------------------|---------------------------|---------|--------|
| pseudo-first order| 10                        | 0.0107  | 2.0366 |
|                   | 15                        | 0.8850  | 9.1689 |
|                   | 20                        | 0.5698  | 9.6227 |
| pseudo-second order| 10                        | 0.0260  | 3.1737 |
|                   | 15                        | 0.0250  | 1.5435 |
|                   | 20                        | 0.0554  | 1.5028 |

4 Conclusion

This research successfully demonstrated the suitability of expanded polystyrene waste (WEPs) products as an effective adsorbent. The activant (acetylene) used improved the
surface characteristics of the WEPS, and this influenced its adsorptive properties, particularly for the removal of naphthalene from aqueous solution. The adsorption process was chemically driven, as suggested by the fitness of the data generated to Dubinin–Radushkevich isotherm and pseudo-second-order kinetic models. This, therefore, opens more opportunities to explore the WEPS for the adsorption of organic pollutants from aqueous solution and real-life wastewater.

List of abbreviations and symbols

| Abbreviation | Description |
|--------------|-------------|
| a_k          | Khan model exponent |
| b_k          | Khan constant |
| A            | Temkin isotherm constant |
| ANOVA        | analysis of variance |
| ARE          | average relative error at monolayer coverage |
| AWEPS        | acetylated waste expanded polystyrene |
| b            | heat of adsorption constant |
| B            | Temkin isotherm constant |
| BET          | Brannen–Emmet–Teller method |
| d            | interlayer spacing, m |
| DOE          | design of experiment |
| EPS          | expanded polystyrene |
| FTIR         | Fourier transform infrared spectroscopy |
| H            | standard enthalpy, J mol$^{-1}$ |
| h            | initial adsorption rate as $t \rightarrow 0$, mg g$^{-1}$ min$^{-1}$ |
| IMR          | impregnation ratio |
| $K_1$        | rate constant of pseudo-first order adsorption, 1/min |
| $K_2$        | rate constant of the pseudo-second, g mg$^{-1}$ min$^{-1}$ |
| $k_{diff}$   | rate constant for intraparticle diffusion, m g$^{-1}$ min$^{-1}$ |
| $k_f$        | Freundlich constant |
| $K_e$        | free energy of adsorption constant |
| MBN          | methylene blue number |
| MW           | microwave |
| n            | Hill coefficient of binding interaction of the adsorbate |
| OFAT         | one factor at a time |
| PAHs         | polycyclic aromatic hydrocarbons |
| $q_e$        | adsorption capacity at equilibrium, mg g$^{-1}$ |
| $q_m$        | maximum adsorption capacity, mg g$^{-1}$ |
| $q_s$        | theoretical isotherm saturation capacity, mg g$^{-1}$ |
| $q_1$        | adsorption capacity at time $t$, mg g$^{-1}$ |
| r            | inverse power of distance from the surface, m$^{-1}$ |
| R            | universal gas constant, J mol$^{-1}$K$^{-1}$ |
| RE           | removal efficiency |
| RSM          | response surface methodology |
| SSE          | sum of square error |
| T            | absolute temperature, K |
| V            | volume, dm$^3$, cm$^3$ |
| WEPS         | waste expanded polystyrene |
| $\Delta G$  | change in Gibbs free energy, J mol$^{-1}$ |
| $\Delta S$  | standard entropy change, J K$^{-1}$mol$^{-1}$ |
| $\beta$      | free energy of adsorption per mole, KJ mol$^{-2}$ |
| $\epsilon$  | Polanyi potential, J mol$^{-1}$ |
| $\gamma_e$   | equilibrium concentration, mg l$^{-1}$ |
| $\gamma_0$   | adsorbate initial concentration, mg l$^{-1}$ |
| $\theta$     | degree of surface coverage |

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Recikliranje ekspandiranog polistirena kao učinkovitog adsorbenta naftalena iz vodene otopine

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Šaržni faktori procesa adsorpcije (vrijeme kontakta 20 – 150 min), doziranje adsorbenta (0,5 – 1,5 g), koncentracija adsorbata (5–30 mg l⁻¹) i brzina miješanja (100–250 min⁻¹) optimizirani su na temelju D-optimalnog dizajna primjenom metodologije odzivne površine (RSM) programa Design-Expert (7.6.8) za uklanjanje naftalena iz vodene otopine pomoću adsorbenta razvijenog iz acetiliranog otpadnog ekspandiranog polistirena (AWEP). Ostvareni maksimalni adsorpcijski kapacitet (5,6608 mg g⁻¹) dobro je prilagođen izotermi Dubinin-Radushkevich (R² = 0,9949). SSE (< 0,05) i ARE (< 4,0 %) označili su pseudo-drugi red kao najprikladniji model. Ovo istraživanje pokazalo je učinkovitost WEP-a za uklanjanje naftalena iz vodene otopine.

Ključne riječi
Adsorpcija, D-optimalnost, naftalen, otpadni ekspandirani polistiren

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