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SigmaXL optimisation of oil spill removal from water using orange peels bio-adsorbent

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ABSTRACT

Traditional methods for oil spill cleanup, such as chemical dispersants and mechanical recovery, are often expensive and can harm marine ecosystems. If orange peels (OP) prove to be a cost-effective alternative, it could save money for companies and governments involved in oil spill response efforts. Response surface methodology (RSM) optimisation conducted in this study, with dosage ranging from 0.2 – 0.4 g and time from 41 – 50 min, identified OP particles of BSS 100 sieve size as an effective adsorbent for oil spill mop up. Using the basic SigmaXL features in Excel, a design of experiments (DOE) based on central composite design (CCD) indicates that the maximum adsorption capacity of OP is 34.17 g/g. This capacity is characterised by its limonene content, which enhances its sorption ability under optimal conditions of 0.2 g and 50 min. As such, a quadratic model, whose reliability is described by F, p-value, T and mean square (MS) model significance parameters, illustratively satisfy the predicted response variable at R^2 = 0.8988. As a result, the residual plots show a uniform distribution of residuals, while the 3D surface and contour plots indicate connection between the input and output variables. SigmaXL not only gives the optimal combinations but allows for further optimum variable predictions outside the boundaries chosen at 95% confidence and prediction intervals. This study also shed light on resource and time management with respect to OP utilisation for oil sorption, which is the sole aim of selecting the two factors analysed to minimise cost.

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1. INTRODUCTION

Abubakar & Alhassan (2021), Gote et al. (2023) and Veľková et al. (2023) discussed several oil spill clean-up strategies from water bodies, including chemical dispersant usage, burning, containment, recovery, sorbent application (adsorption or absorption), bioremediation, and phytoremediation. Since a large chunk of oil export (90%) is via water bodies (Saha & Majumdar, 2021) like the sea, river, and ocean, offshore spillage by accident from ships, tankers, or leaks in marine oil pipes is rampant (Wolok et al., 2021). Sorbent usage, cost, types, criteria for selection, storage and a detailed description of how it works in the clean-up of oil spillage from shorelines is found in ITOPF (2024). Examples of biosorbents already investigated for similar purpose are shown in Table 1. They are basically classified into animal/living organism waste or parts, plant biomass, and chemical compounds (e.g., plastics, rubber, polymers etc.).

Table 1. Different Sorbents Utilized for Oil Spill Clean Up in Previous Research

Category	Biosorbent Used	Study by
Animal Parts/Waste/Residue	Chitin	(Trang et al., 2023; Trang & Andreevna, 2020)
	Chitosan	(Barros et al., 2014; Khalifa et al., 2021; Oseke et al., 2018)
	Keratin	(Ifelebuegu & Johnson, 2017)
	Fish scale	(Lutfee et al., 2020)
	Human hair	(Kasundra et al., 2019; Kumar et al., 2019; Mehjabeen, 2022; Pagnucco & Phillips, 2018; Shah, 2020; Ukotije-Ikwut et al., 2016)
	Autochthonous consortium	(Taura et al., 2022)
Plant Biomass	Napier grass	(Obi et al., 2023)
	Lotus leaf	(Bhushan, 2019)
	Kapok fibres	(Abdullah et al., 2019; Azlin Shah et al., 2019; Wang et al., 2014)
	Eggshell	(Muhammad et al., 2012)
	Marine alga	(Tarbaoui et al., 2016)
	Banana stem	(Husin et al., 2011; Nazifa et al., 2018; Sathasivam & Mas Haris, 2010)
	Banana peels	(Abdullah et al., 2016; Asadu et al., 2022; Dawodu et al., 2021; El-Din et al., 2017; El-Nafaty et al., 2013)
	Plantain leaves	(Dagde, 2018; Eboibi et al., 2023)
	Ogbono shell	(Onwu et al., 2019)
	Groundnut husk	(Dagde, 2018; Oluwatoyin & Olalekan, 2021)
	Wool fibres	(Condurache et al., 2021)
	Sugarcane bagasse	(Díaz et al., 2022; Utomo et al., 2016)
	Pomelo peel	(Zamparas et al., 2020)
	Avocado peel	(Malhas & Amadi, 2023)
	Rice husk/straw	(Hoang & Pham, 2021; Kelle, 2018; Li et al., 2023; Pirestani et al., 2018; Ramakrishnan et al., 2021; Shi et al., 2022)
	Coconut husk	(Ifelebuegu & Momoh, 2015)
	Coconut coir	(Abel et al., 2020a, 2020b; Mukhair, 2016; Yusof et al., 2015)
	Cocoa pod	(Onwuka et al., 2018)
	African oil bean seed pod	(Obi & Ajiwe, 2022)
	Corn waste	(Asadpour et al., 2019; Maulion et al., 2015)
	Oil palm leaves	(Odunlami et al., 2022b)
	Pith bagasse	(Hussein et al., 2008; Hussein et al., 2009)
	Wheat husk	(Omar et al., 2023)
	Bambara nut husk	(Chukwujindu et al., 2020)
	Pineapple crown	(Etanuro et al., 2023)
	Rambutan peel	(Nguyen et al., 2024)
	Kenaf core fibres	(Salisu et al., 2019; Tan et al., 2021)

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	Mango shell	(Olufemi & Otolorin, 2017)
	Lemon peel	(Jopery et al., 2020; Tembhurkar & Deshpande, 2012)
	Jackfruit	(Wan Ibrahim et al., 2013)
	Solanum incanum leaves	(Abutaleb et al., 2021)
	Cissus populnea leave	(Honda et al., 2023)
	Aloe vera	(Meez & Hosseini-Bandegharaei, 2021)
	Phragmites australis	(Behnood et al., 2013)
	Calotropis gigantea	(Sukmawati, 2023)
	Azolla folliculoid	(Amin et al., 2015)
	Sterculis setigera	(Osemeahon & Dimas, 2020)
	Piliostigma reticulatum	(Dimas et al., 2021)
	Borassus aeothopum coir	(Arinze-Nwosu et al., 2019)
	Posidonia oceanica	(Jmaa & Kallel, 2019)
	Biopolymer	(Eweida et al., 2023; Omer et al., 2020; Omer et al., 2021)
	Sawdust	(Banerjee et al., 2006; Soliman et al., 2020)
	Bamboo fibre	(Adhithya et al., 2017)
	Water hyacinth	(Arquam et al., 2023; Khondoker et al., 2024)
	Lawson leaves	(Davey, 2022; Mahmoud et al., 2022)
	Papyrus plant	(Toamah & Fadhil, 2021)
Non-Agricultural or Chemical Adsorbent	Polyurethane foam	(Trang et al., 2023; Trang & Andreevna, 2020)
	Graphene	(Vocciante et al., 2019)
	Waste tyre powder	(Odeh & Okpaire, 2020)
	Solidifiers	(Federici & Mintz, 2014; NRT-RRT, 2007)
	Sponge	(Shittu et al., 2020)
	Polypropylene	(Bayat et al., 2005)
	Hafnium oxide ceramic	(Hussain et al., 2020)
	Coronavirus face masks	(Alatabe, 2024)
	Fabric	(Ku et al., 2021)
	Aerosol	(Nimy & Anitha, 2020)
	Aerogel	(Doshi et al., 2018; Zamparas et al., 2020)
	Peat	(Al-Ameri et al., 2019; Cojocaru et al., 2011; Rotar et al., 2014)
	Metal oxide	(Sayed et al., 2004)
	E-waste	(Ramakrishnan et al., 2021)
	Waste plastic	(Aboul-Gheit et al., 2006)
	Zeolite	(Danehpash et al., 2018; Kalbuadi et al., 2019)
	Nanomaterials	(Amar et al., 2019; Odunlami et al., 2022a; Siregar et al., 2019; Usman & Okoro, 2017)
	Composite	(Ibe, 2019; Izevbekhai et al., 2020; Mirzaei, 2021)

Source: Author's own data

A combination of some of these (Table 1) can be used (Saha & Majumdar, 2021; Tayeb et al., 2019), as well as some developed novel technologies/adsorbents such as the BIOBIND (Unbehaun et al., 2014), bionic oil adsorber (Barthlott et al., 2020), and water column (Barry et al., 2017). Current study sought to build on the existing literature that has explored numerous biosorbents for oil spill removal. However, some of these alternatives either lack the desired oil absorption capacity or are less readily available. Orange peel (OP) ability to decontaminate water and wastewater of toxic metals is well known (Hasan et al., 2021; Lima et al., 2020), but its performance in oil spill removal is still being researched (Abdullah et al., 2016; Okpanachi et al., 2019; Yao & Song, 2021) and is still at the experimental stage. OPs are byproduct of the citrus industry and are often discarded as waste. If they can be repurposed for oil spill cleanup, it could lead to more sustainable use of resources and reduce waste generation. Ideally, activated OP adsorbents could be more effective than non-activated ones due to their increased surface area and enhanced adsorption capacity. However, the natural OP used in this study can still serve as a viable adsorbent for certain applications, particularly when cost and environmental impact are the considerations.

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This study focuses on investigating the viability of utilizing OP waste as bio-adsorbent for oil spill cleanup from water. The specific goals include the physical alteration of the fruit peel to enhance its efficacy in oi spill remediation, analysis of the performance of the OP adsorbent by evaluating its sorption capabilities and other relevant properties like dosage and contact time using SigmaXL response surface methodology (RSM) software and characterize this bio-adsorbent based on this sorption capacity (q_e) using techniques such as Fourier Transform Infrared (FTIR) spectroscopy and Scanning Electron Microscopy (SEM). SigmaXL is an Excel add-in primarily designed for statistical analysis and Six Sigma tools. When it comes to RSM optimisation for processes such as oil spill decontamination of water, SigmaXL offers several advantages compared to other software. SigmaXL's combination of ease of use, comprehensive statistical analysis capabilities, graphical tools, Excel integration, cost-effectiveness, and customer support make it a compelling choice for individuals and organizations seeking to perform RSM optimisation and other statistical analyses within the familiar Excel environment. On a 3D surface plot, Omar et al. (2023) showed the effect of time and wheat straw dosage on oil removal capacity in STATISTICA. In addition, Asadu et al. (2022) juxtaposed ANFIS and Minitab towards optimising oil sorption with banana peel, Behnood et al. (2014) optimised the use of raw bagasse, Al-Ameri et al. (2019) used Box-Wilson RSM to find the optimal parameter combination on peat bagasse performance, Salisu et al. (2019) analysed the influence of monomer ratio and initiator concentration on grafting efficiency and oil sorption, Onwu et al. (2019) optimised 4 independent variables including dose and time, utilising ogbono shells, and Izevbekhai et al. (2020) revealed little interaction between polymer composite adsorbent dosage, contact time, and adsorption percentage. These studies were among the many adoptions of an RSM programme to optimise oil spill sorption using a specific biosorbent at the moment. Nevertheless, isotherm studies of oil spill mop up using OP or modified OP sorbent had been carried out (Okpanachi et al., 2019). Traditional methods of oil spill cleanup can be harmful to marine ecosystems (Dighiesh et al., 2019; Olajuyigbe et al., 2020), thus, finding a natural, biodegradable alternative like OP could help mitigate these negative effects. A report by U.S. Department of Agriculture (USDA, 2023) puts Brazil, followed by China, at the top of global orange production. United States being 6th in the list (2.54 million metric tons) in that year, perhaps could take advantage of OP capability, as the country is the one with the most recorded cases of oil spill in history. Hydrophobicity of an oil spill removal adsorbent refers to its ability to repel water and attract oil, facilitating the separation of oil from water. Adsorbents with higher hydrophobicity are more effective at removing oil from water surfaces (Peng et al., 2021). OP contains compounds like limonene, which are naturally hydrophobic. When OP is used as an adsorbent for oil spill cleanup, its hydrophobic properties enable it to selectively absorb oil while repelling water.

2. MATERIALS AND METHOD

2.1 Materials, analytical equipment and software

Main substances or materials used in this study are OP, distilled water and crude oil samples. Auxiliary apparatus for the hierarchical experimental runs involved are the flocculation apparatus, conical flask, oven, filter paper, test tube and beaker. FTIR 4100 series (Jasco Corp., Japan) and an appropriate high-performance DJ-SEM150 series SEM instrument (produced by Jiangsu Wuxi, China) used are some of the analytical equipment employed in the study. Laboratory outcome from this study was entered to SigmaXL RSM optimisation software installed in Windows 10 Laptop computer.

2.2. Peel sourcing and preparation

OPs were sourced from Maiduguri Monday Market area, Borno state, Nigeria. It was made to undergo thorough and repetitive cleaning process with distilled water, as carried out by Li et al. (2023) and Michael-Igolima et al. (2023), so as to detach the dust and small particles impurities from the biowaste. It was

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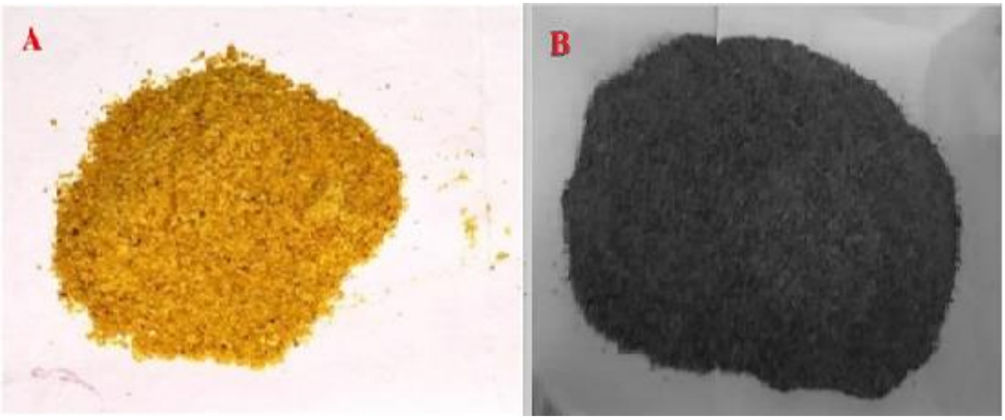

Fig. 1. (a) Grounded OPs, and (b) Peel surface after sorption

subsequently dried at 105℃ in an oven for 7 h to get rid of any moisture content, following Behnood et al. (2013) and Shittu et al. (2020) step. Post-drying, the OP sorbent was ground into smaller particles to ensure uniformity and stability in weight. To prepare the samples for batch adsorption tests, the grounded fruit waste was further processed by meshing and sieving using BSS 100 (150 nm) sieve (manufactured by Bionics Scientific, India), as used in Arquam et al. (2023). Doing that, uniformity and standardization of the bio-sorbent particles (Fig. 1a) for subsequent experimental procedures is ensured.

Literature findings show that sorption of different oil types (e.g., crude oil, diesel, kerosene, vegetable oil, lube oil, bilge oil, heating oil, waste oil, fuel oil and heavy oil) can be carried out using various sources of water, including distilled, tap, ocean and lake water (Jopery et al., 2020).

2.3. Biosorption experimentation

Stoke solution was prepared by adding 50 mL of crude oil into 450 mL of distilled water. Next, 50 mL was drawn from the stoke solution to carry out the biosorption studies based on the equivalent time and dosage. The biosorption experiments for oil spill removal were conducted using a flocculation unit comprising two main sections. In the mixing/stirring section consisting of 5 sets of electric stirring motors capable of variable speeds up to 250 rpm, each motor was equipped with a variable speed control regulator for precise adjustment. Additionally, a time control sensor was integrated with the electric motors to regulate the contact time between the adsorbate and biosorbent, similar to Olufemi & Otolorin (2017)'s approach. As for the biosorption experiments setup, it was carried out in a series of beakers, each containing 50 mL of crude oil solution at the desired adsorbent dosages, almost in accordance with Meez & Hosseini-Bandegharaei (2021). The mixtures were agitated at 250 rpm speed and 20 min duration using a shaker mixer. After agitation, the resulting mixtures were filtered and the adsorption data were recorded. The equilibrium adsorption capacity (q_t) was then determined using Equation (1) (Abdelwahab et al., 2021; Dagde, 2018; Kelle, 2018; Mehjabeen, 2022; Peng et al., 2021; Tabbakh & Barhoum, 2018);

$$q_t = \frac{M_{OP_w} - (M_{H_2O} + M_{OP_i})}{M_{OP_i}} \tag{1}$$

where, M_{OP_i} = initial mass of OP adsorbent (g), M_{OP_w} = mass of wetted adsorbent (g) and M_{H_2O} = mass of water adsorbed.

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2.4. FTIR and SEM characterisation

Surface functional groups and chemical bonds present on the surface of the OP biosorbent, playing a crucial role in the adsorption processes were identified via FTIR analysis. SEM was used to investigate and understand the surface texture (surface morphology – surface roughness, porosity and particle size distribution) of the sample at high magnification (Aboul-Gheit et al., 2006; Adhithya et al., 2017).

2.5. RSM optimisation by SigmaXL

To carry out RSM optimisation, SigmaXL V10 Excel add-in was downloaded. Under 'Response Surface' in the 'Design of Experiments' (DOE) drop down, 'Response Surface Designs' was entered. Under the next sub-window, number of factors (i.e., 2), 1 number of replicates, rotatable (alpha = 1.414) axial value and 1 number of responses was selected. Within the same window, low and high factor level and their names was set and 10 randomize run, central composite design (CCD) consisting of 2 centre points, were specified. CCD was favored over other design because it provides more axial design points (Rehman et al., 2022). Using fewer center points may result in less precision in estimating the curvature and lack of fit. However, it requires fewer experimental runs; hence, potentially lower cost and resource requirements (materials, time & labour). Yonguep & Chowdhury (2021) utilises more centre points (precisely, 5) in their study, generally enhancing the precision and reliability of the estimated response surface model – but characterised by increased cost and resource requirements.

A worksheet showcasing the predicted runs values for Factors A and B (dose and time) was observed and the respective response R (q_t) values were determined in the laboratory before filling the q_t empty cells in the worksheet. 'Analyze Response Surface Design' was clicked on under the 'Response Surface' dropdown, where alpha for Pareto Chart, available responses and model terms were selected. An option requesting the creation of the regular residual plots was picked. After this step, the RSM SigmaXL plugin is expected to give the regression model for the q_t response, model summary statistics, parameter estimates in terms of coded units, analysis of variance (ANOVA) for the model, Durbin-Watson Test for Autocorrelation in residuals, Pareto Chart and residuals with respect to the model terms. There also exists a 'Predicted Response Calculator', allowing users to determine the optimal values by changing the predictor variables simultaneously. This calculator was used to test for several other possibilities. 'Contour/Surface Plots' was then entered to display the 3D surface and contour plots emanating from the analysis. To precisely predict the optimal combination, 'Excel Solver' was used by defining the boundary values of A (0.2 – 0.4g) and that of B (41 – 50 min) as constraints, by changing A and B cell to maximise the target/response cell.

3. RESULTS AND DISCUSSION

3.1. Characteristics of the adsorbent

SEM analysis carried out, revealed the morphology and texture of the OP adsorbent before and after adsorption. Fig. 2a depicts the morphology of the virgin adsorbent before the crude oil adsorption. From it, numerous pores on the surface of the sample can be clearly deduced, indicating the potential for crude oil adsorption. In Fig. 2b, the SEM image clearly demonstrates that the pores observed on the virgin adsorbent in Fig. 2a have become filled due to the adsorption of crude oil on the surface.

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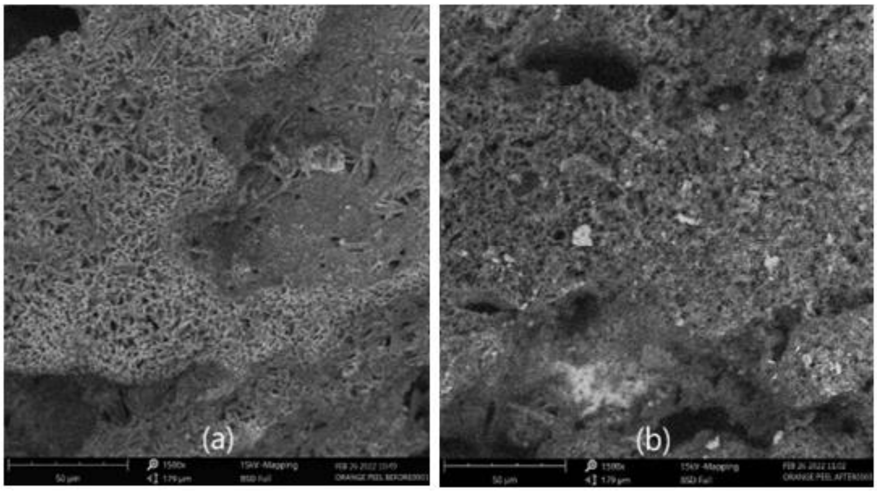

Fig. 2. SEM images of (a) fresh OPs adsorbent, and (b) spent (used) OPs adsorbent

Typically, the surface of the virgin adsorbent in Fig. 2a appears clean, with distinct features and a relatively smooth texture. Michael-Igolima et al. (2023) mentioned that smooth surfaces have minimal adsorption uptake due to the reduced number of active sites available for the adsorption and binding of contaminants. But after adsorption, the surface appears rougher or less uniform due to the presence of the adsorbed crude oil. Also, the features on the surface of the virgin adsorbent are more visible and easier to distinguish due to the absence of any adsorbed material. However, the visibility of surface features is reduced or altered after adsorption (Fig. 2b), as the adsorbed crude oil cover or fill in the pores, affecting the overall visibility of the surface morphology. Exactly the same way, the structure of lemon peel appeared to be coated prior to adsorption (Jopery et al., 2020). On the other hand, FTIR spectrum of the natural OP before biosorption (Fig. 3a) revealed several peaks corresponding to different organic functional groups described by Table 2.

Table 2. FTIR of OP sorbent pre- and post-adsorption of crude oil

No.	Peaks	Functional Group
Before Sorption		
1.	3865.48 & 3788.32 cm^{-1}	Vibrations of N-H and O-H
2.	3595.43, 3387.11 & 3317.67 cm^{-1}	Presence of alcohol with O-H stretch
3.	3240.52 & 2931.90 cm^{-1}	Presence of carboxylic acids with O-H stretch
4.	2137.20 & 1643.41 cm^{-1}	Presence of carboxylic acid with O-H stretch and secondary amine with N-H bend
5.	1543.10 & 1458.23 cm^{-1}	Presence of amines with N-H bend
6.	1381.08 & 1280.78 cm^{-1}	Presence of phenol and alcohol with O-H bend
7.	1033.88 cm^{-1}	Presence of alcohol and ether with C-O stretch
After Sorption		
1.	3973.49 & 3857.76 cm^{-1}	Vibrations of N-H and O-H functional groups
2.	3387.11 cm^{-1}	Alcohol with OH stretch and hydrogen bonding
3.	3263.66 & 2931.90 cm^{-1}	Presence of carboxylic acid with O-H stretch and methylene with C-H stretch
4.	2530.69 & 2137.20 cm^{-1}	Presence of carboxylic acid with O-H stretch and alkyne with C≡H stretch
5.	1643.41 & 1458.23 cm^{-1}	Presence of amide with C=O stretching and secondary amine with N-H bending
6.	1373.36 & 1288.49 cm^{-1}	Presence of phenol, alcohol with O-H bend and ether with C-O-H stretch
7.	1026.16 cm^{-1}	Presence of alcohol with C-O stretch

Source: Author’s own illustration

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However, in Fig. 3b, FTIR spectrum of the OP after biosorption exhibited several peaks indicating changes in the functional groups compared to the spectrum before biosorption, as evidenced in Table 2. These changes in the FTIR spectrum after bio-sorption indicate modifications in the surface functional groups of the OP, suggesting interactions between the biosorbent and the adsorbate (crude oil), which are crucial for the biosorption process. Some of these stretching vibrations are explained in the literature for other sorbents utilisation (Abutaleb et al., 2021; Meez & Hosseini-Bandegharaei, 2021; Mirzaei, 2021; Soliman et al., 2020).

A functional group that characterises biosorption taking place is the "vibrations of N-H and O-H" group (Abdullah et al., 2016; Al-Ameri et al., 2019). This functional group is associated with organic compounds containing nitrogen and oxygen atoms, which are commonly found in biomolecules such as proteins, amino acids, and carbohydrates. The presence of N-H and O-H vibrations indicates the involvement of these functional groups in the biosorption process, highlighting the interactions between the bio-adsorbent material (OP) and the adsorbate (crude oil) during the oil removal process from water. Respectively, they are 3865.48 cm^{-1} and 3788.32 cm^{-1} before adsorption (Fig. 3a) and 3973.49 cm^{-1} and 3857.76 cm^{-1} after adsorption (Fig. 3b).

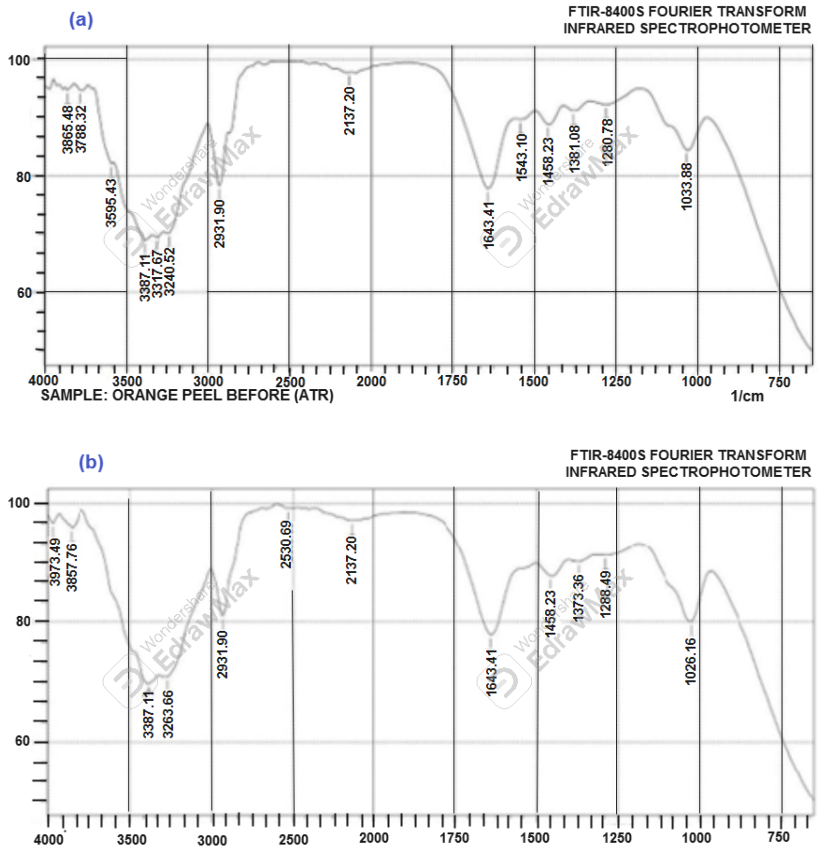

Fig. 3. FTIR spectrum of fresh OP (a) before biosorption, and (b) after biosorption

Source: Author’s own data

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3.2. Empirical optimum and RSM predicted model

At various dosage of the peel (Predictor A – specifically, 0.2 – 0.4 g) and contact time (Predictor B – i.e., 41 – 50 min) defined in SigmaXL, an experiment was conducted to determine the response (R) or q_t shown in Table 3. Similar to the predictors used in this work, Malhas & Amadi (2023) examined their influence on % removal of oil of different types using avocado peel sorbent. Obviously, the optimal parameters are 32.55 g/g q_t, 0.2 g adsorbent dosage and 49 min time. This is nearly equal to a predicted q_t of 34.17 g/g at the same values of A and B. Omar et al. (2023) obtained a maximum of 10.989 and 12.786 g/g from STATISCA programme based on 5g activated wheat straw dose taken to adsorb diesel, which obviously gives lesser capacity than OP used herein.

Table 3. Design of experiment for crude oil sorption using OP

Run	Std. Order	Center Points	Block	A: Dosage (g)	B: Time (min)	R: Adsorption Capacity (g/g)	Predicted (Fitted) R Values (g/g)
1.	6	1	1	0.2	41	32.4	31.673
2.	5	1	1	0.4	41	13.4	14.491
3.	3	1	1	0.2	49	32.5	32.889
4.	9	0	1	0.4	49	18.3	20.845
5.	1	1	1	0.2	49	32.55	32.889
6.	10	0	1	0.4	45	19.3	15.664
7.	2	1	1	0.3	45	14.9	11.076
8.	7	1	1	0.3	50	18.9	16.573
9.	8	1	1	0.3	45	11.6	11.076
10.	4	1	1	0.3	45	4.4	11.076

Std. Order stands for Standard Order and refers to the standardised order in which the experimental runs or data points are arranged in the RSM design matrix. Standardising the order helps in organising the experimental factors and responses systematically for analysis and interpretation. Center points are often replicated to estimate the experimental error and assess the model's predictive capability. Blocks can be used to account for external factors that may influence the experimental outcomes, such as batch effects or environmental conditions. The predicted q_t in Table 3 was based on Equation (2).

$$q_{t_{Prdct.}} = \begin{Bmatrix} 11.34358718 - 7.145740863A + 2.692805791B + 1.44523407AB \\ +11.89452077A^2 + 2.536502853B^2 \end{Bmatrix} \quad (2)$$

Only adsorbent dosage in this study is among the 5 parameter quadratic model predicted by Behnood et al. (2014). A plot of the predicted q_t response and the actual response is illustrated in Fig. 4.

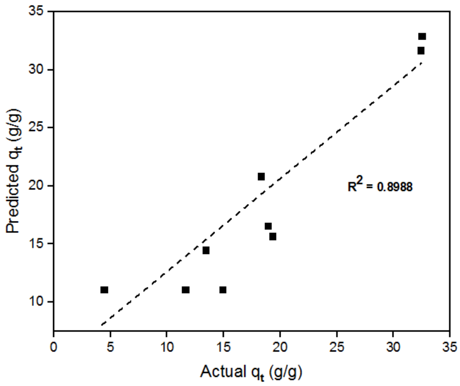

Fig. 4. Predicted versus actual adsorption capacity of oil by OP
Source: Author's own illustration

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There is apparent 10.12% non-fit in Fig. 4, which is insignificant to affect the model performance. SigmaXL, as a software tool for RSM optimisation, typically provides options for users to add and modify model terms in regression analyses. Adding an additional model term to Equation (2) may or may not necessarily improve the fit of the predicted versus actual plot to 100%. The decision to include additional model terms should be based on statistical criteria such as model significance, goodness of fit measures, and the theoretical relevance of the added terms. In the context of RSM and regression modelling, adding more terms to the model can lead to overfitting, where the model becomes too complex and captures noise in the data rather than the underlying relationships. This can result in a model that performs well on the existing data but fails to generalize to new data. Before adding more model terms, it is recommended to assess the model's adequacy using statistical diagnostics, such as the coefficient of determination (R^2), ANOVA, residual analysis, and other model evaluation techniques. This will help determine whether the current model adequately captures the relationships between the factors and responses, or if additional terms are needed to improve the model's predictive accuracy. Standard error of the coefficient (SE coefficient) measures the variability or uncertainty in the estimated coefficient. A lower SE coefficient (viz., that of 'A' ≡ 1.968) indicates a more precise estimate of the coefficient, as shown in Table 4.

Table 4. Parameter estimates (coded units)

Term	**Coefficient**	**SE Coefficient**	**T**	**P**	**VIF**	**Tolerance**
Constant	11.34358718	2.575533469	4.404	0.0117	-	-
A: Dose	-7.145740863	1.968033306	-3.631	0.0221	1.073830531	0.93124564
B: Time	2.692805791	2.183870185	1.233	0.2851	1.054561707	0.948261248
AB	1.44523407	2.446148197	0.590820	0.5864	1.036339108	0.964935118
A^2	11.89452077	3.414763981	3.483	0.0253	1.293159917	0.773299564
B^2	2.536502853	4.039771545	0.627883	0.5641	1.306429922	0.765444807

Source: Author's own data

T-value is the ratio of the estimated coefficient to its standard error. It is used to test the significance of the coefficient (Sawdi, 2021). A higher T-value (as in T = 3.483 & 4.404) indicates that the coefficient is more likely to be statistically significant. P-value indicates the probability of observing the estimated coefficient if the null hypothesis (that the coefficient is not significant) is true. A lower p-value (typically < 0.05) suggests that the coefficient is statistically significant (Eboibi et al., 2023). In Table 4, the Variance Inflation Factor (VIF) measures the multicollinearity between predictor variables in the regression model. A VIF value > 10 indicates high multicollinearity, which can affect the reliability of the coefficient estimates. Table 4 reports VIF < 2, demonstrating a reliable coefficient. Tolerance is the reciprocal of the VIF and indicates how much of the variance of a predictor variable is not explained by other predictor variables. A tolerance value ≅ 1 indicates low multicollinearity. Regarding the values in the 'P' column highlighted in red by SigmaXL, this typically indicates that the corresponding coefficients are statistically significant at a predetermined significance level (e.g., α = 0.05). When the P-value is < the significance level, the coefficient is considered statistically significant, and SigmaXL may highlight these values in red to draw attention to their importance in the regression model. In a Pareto Chart, the bars represent the magnitude of the coefficients of the model terms, and they are plotted in descending order of their absolute values. The x-axis typically represents the model terms, while the y-axis represents the magnitude of the coefficients, as illustrated in Fig. 5. Taller bars (A^2 & A) indicate model terms with larger coefficients, suggesting that these terms have a more significant impact on the response variable compared to model terms with shorter bars (B, B^2 & AB).

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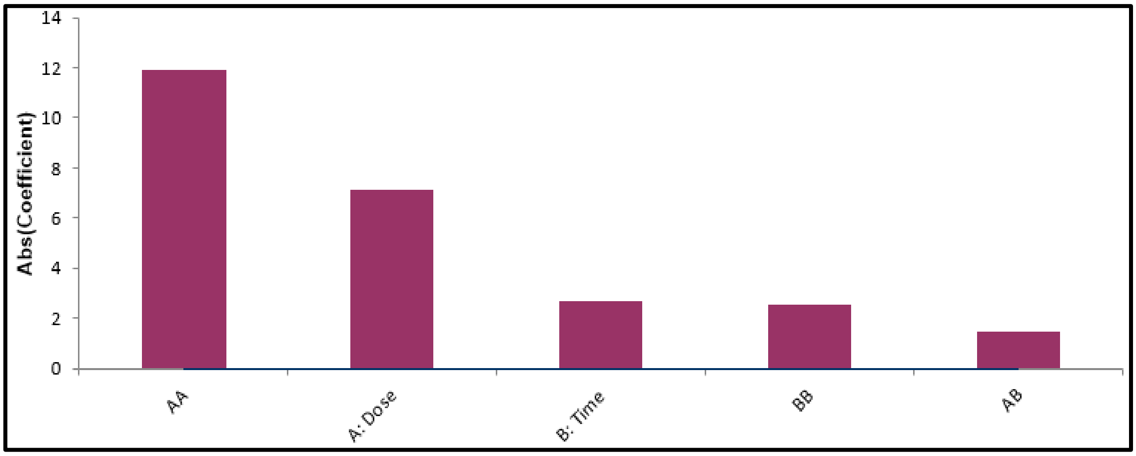

Fig. 5. Pareto Chart of Coefficients for Adsorption Capacity Quadratic Model

Source: Author's own data

3.3. ANOVA and residual reports

A significant F-value (F-statistic) and a low p-value < 0.05 (precisely 0.0404 in this study) indicate that the regression model as a whole is statistically significant and explains a significant amount of the variability in the response variable. Mean Square values (MS) provide information about the variance explained by the model (model MS) and the unexplained variance (error MS). A larger model MS (768.72) compared to error MS (equal to 86.565) suggests that the model is effective in capturing the relationships between the predictors and the response, as shown in Table 5.

Ideally, the F-statistic is the ratio of the MS for the model to the MS for the error. It is used to test the overall significance of the regression model (Salisu et al., 2019). A higher F-value indicates that the model is more likely to be statistically significant (Izevbekhai et al., 2020; Onwu et al., 2019). Sum of Squares (SS) model value = 768.72 indicates the total variability explained by the regression model whereas an SS error value of 86.565 represents the unexplained variability or residual error in the model. Degrees of freedom (DF) for the model is equal to the number of predictors (model terms) in the model (in this case, the DF Model = 5). The fewer the factors, the minimal the model terms given and vice versa, as observed in higher DF Model in Chukwujindu et al. (2020) who selected 4 input variables. The different DF values in Table 5 reflect the specific components of the ANOVA analysis, including the model, error, lack of fit, and pure error. Table 6 shows that a Durbin-Watson (DW) statistic close to 2 suggests no autocorrelation, while values significantly different from 2 indicate the presence of autocorrelation in the residuals.

Table 5. Analysis of variance for model

Source	**DF**	**SS**	**MS**	**F**	**P**
Model	5	768.72	153.74	7.104	0.0404
Error	4	86.565	21.641	-	-
Lack of Fit	3	31.440	10.480	0.190111	0.8944
Pure Error	1	55.125	55.125	-	-
Total (Model + Error)	9	855.29	95.032	-	-

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Table 6. DW test for autocorrelation in residuals and model summary statistics

Metric	Value
DW Statistic	0.999073
P-Value Positive Autocorrelation	0.0309
P-Value Negative Autocorrelation	0.9663
R^2	89.88%
R^2 Adjusted	77.23%
S (Root Mean Square Error)	4.652

Source: Author’s own data

In this investigation, the DW Statistic value is approximately 0.999073 (or ≅ 1), which by implication mean there is no autocorrelation present in the residuals. A low P-Value of 0.0309 < 0.05 points to the presence of positive autocorrelation, corresponding to P-Value > 0.05 for negative autocorrelation. An average magnitude of the residuals is suggested by the RMSE of 4.652 and an R^2 value of 89.88% show that the model explains approximately 0.8988 of the variance in the q_t response variable. Asadu et al. (2022) stated that the proximity between the R^2 and adjusted R^2 values (i.e., 0.7723) is an indication of goodness of fit of the data. In Fig. 6, which represents Frequency vs. Regular Residuals, the bars indicate the frequency or count of residuals falling within specific ranges or bins. A tall bar indicates a concentration of residuals around that value while a shorter bar implies a lower frequency of residuals within that specific range. A balanced distribution with bars of similar height across different ranges indicates a more uniform distribution of residuals.

It is observed that the plot of NSCORE vs. Regular Residuals in Fig. 7 shows a linear relationship where the points align closely along a diagonal line. This indicates that the residuals are normally distributed. In a regular residual vs fitted values plot, if the points are randomly scattered around the horizontal line at 0 on the Y-axis, it indicates homoscedasticity, suggesting that the variance of the residuals is constant across different levels of the predicted values. Any discernible patterns in the scatter plot, such as a funnel shape or systematic increase/decrease in residuals as fitted values change, may indicate issues like heteroscedasticity or non-linearity in the model. In Fig. 8, a consistent spread of points around the 0 line suggests that the model’s assumptions are met, and the residuals are unbiased and normally distributed (Behnood et al., 2014; Yonguep & Chowdhury, 2021). A residual consistently at zero across all observations, suggests that the model is accurately capturing the relationship between the predictor variables and the response variable. However, in Fig. 9, the residual was initially above 0 (implying underestimation) and fall below it (implying overestimation). The consistent shifts between overestimation and underestimation suggest the presence of systematic bias in the model. Having residuals consistently at zero does not necessarily mean the model is perfect. It could still be affected by issues like omitted variable bias, specification errors, or multicollinearity. However, the absence of any discernible pattern in the residuals suggests that the model is at least performing adequately in terms of capturing the overall relationship between the variables. Meanwhile, Fig. 10 and Fig. 11 are regular residual plots vs. model terms. When a plot of regular residuals against a specific model term was made, we observe a straight vertical dotted point, which implies that one of the predictor variables is a perfect linear function of another predictor variable or a combination of predictor variables in the model (a perfect multicollinearity issue).

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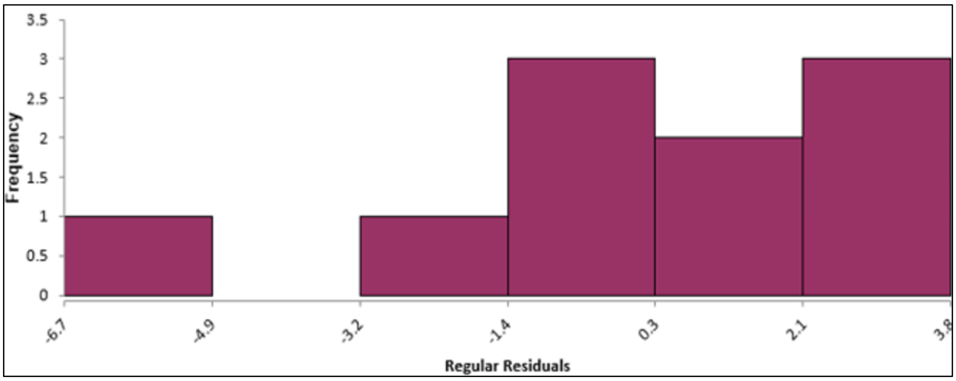

Fig. 6. Frequency vs regular residuals

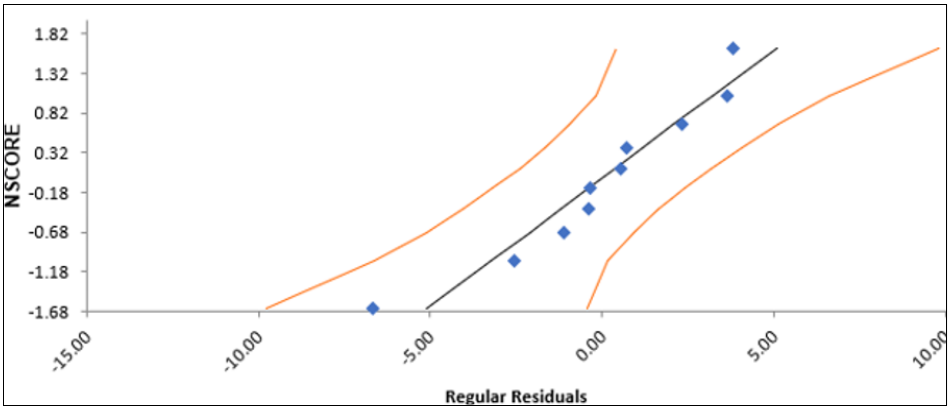

Fig. 7. Normal probability plot of regular residuals

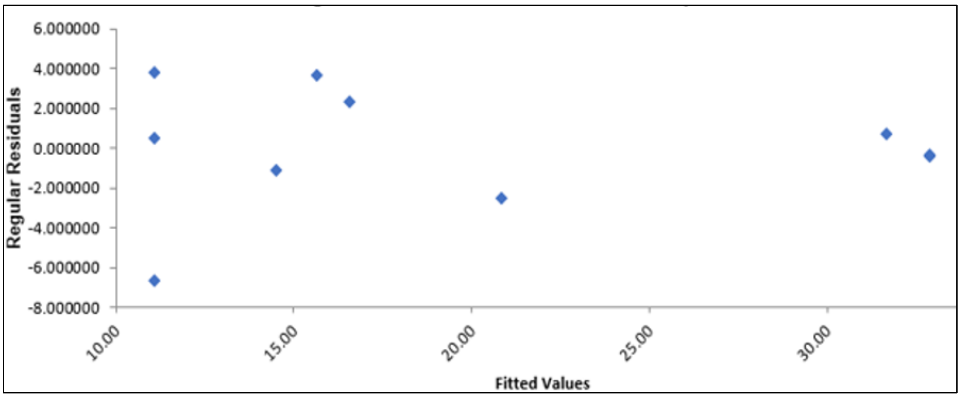

Fig. 8. Regular residuals vs predicted values

Source: Author's own data

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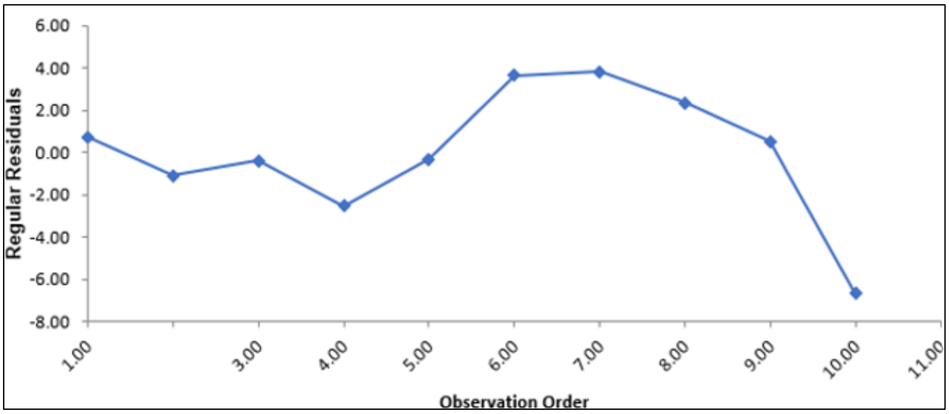

Fig. 9. Regular residuals vs data order

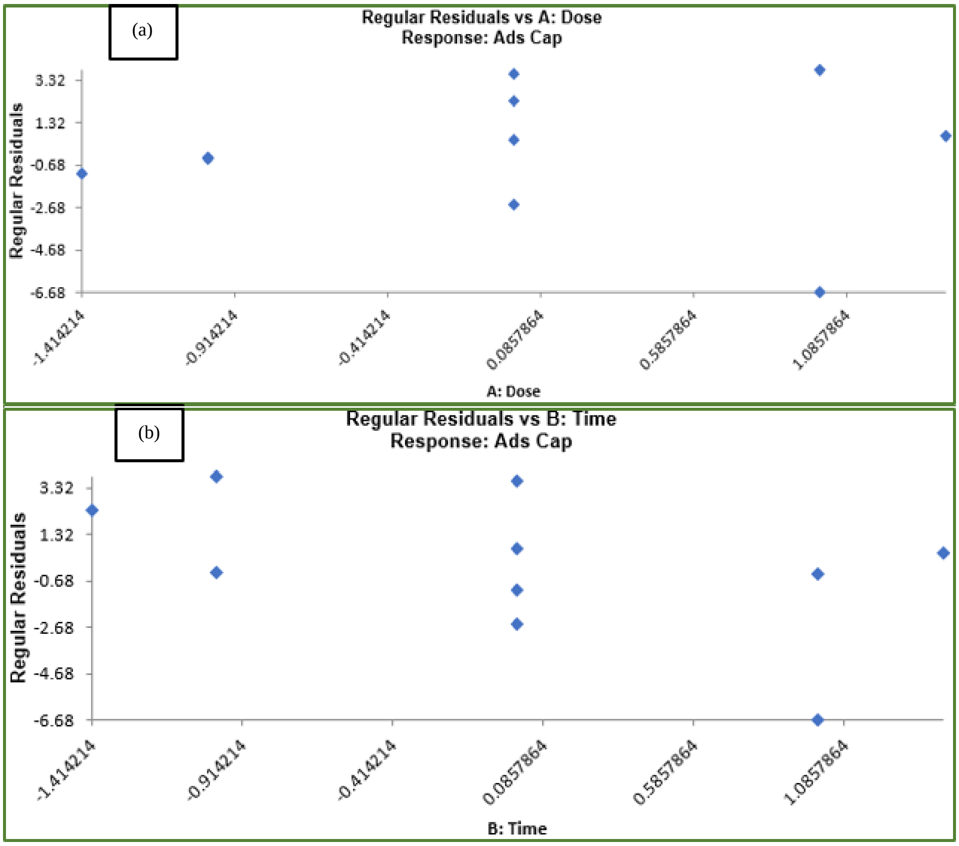

Fig. 10. Regular residuals vs (a) dose, and (b) time

Source: Author's own data

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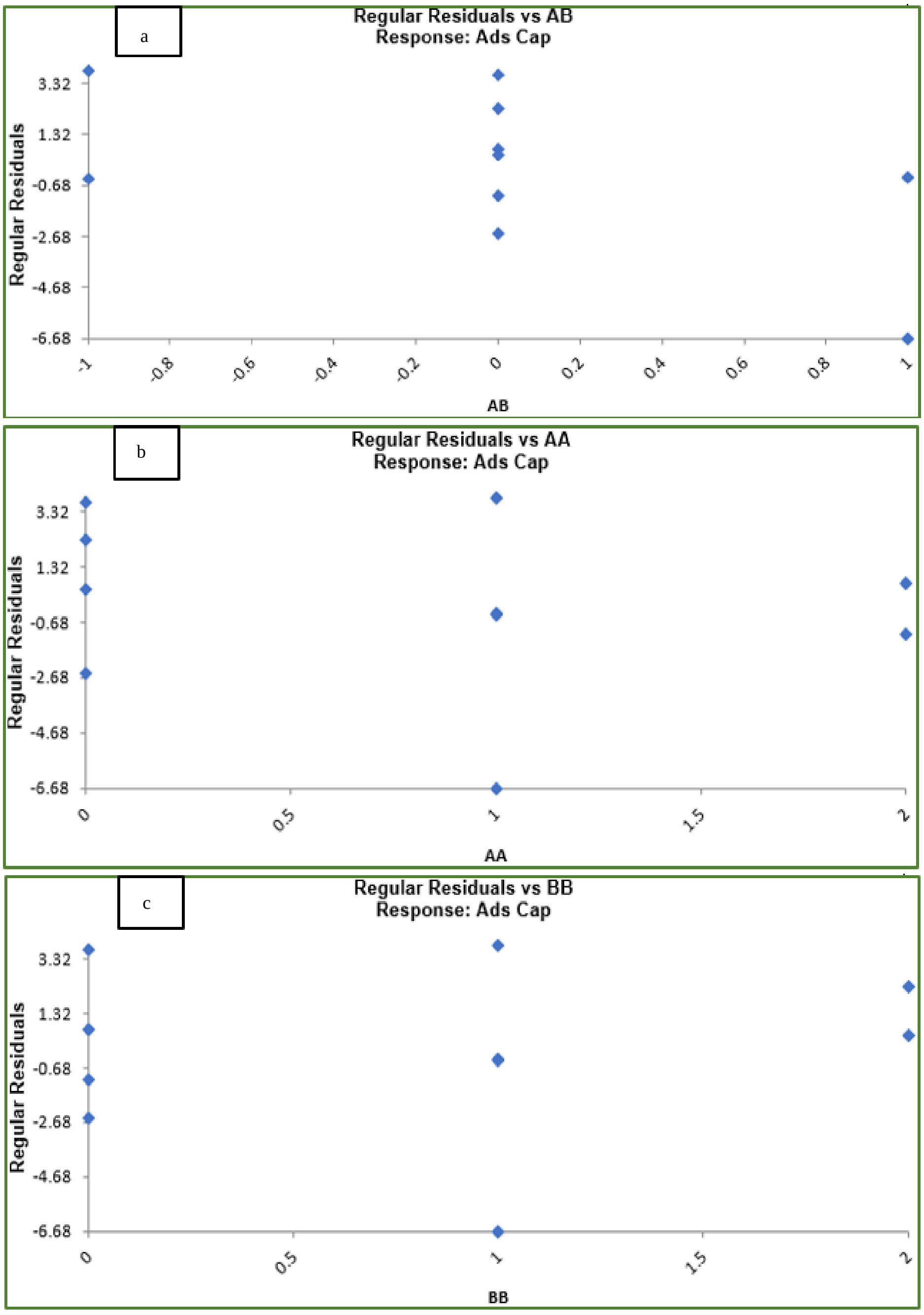

Fig. 11. Regular residuals vs (a) AB, (b), AA, and (c) BB

Source: Author’s own data

3.4. Predicted optimum

In contour and 3D surface plots of Fig. 12, a colour gradient is typically used to represent the range of values of the response variable. Lighter colours (e.g., white or yellow) often indicate higher values, while darker colours (e.g., blue or black) represent lower values. In the 3D surface plot, the colour shading on the surface represents the response variable's values at different combinations of predictor variables. The colour changes help in identifying regions of optimal or suboptimal response values.

The combination of A and B values corresponding to the peak or highest point on the 3D surface plot represents the optimal conditions for achieving the maximum adsorption capacity. Facing up orientation implies that the response variable (q_t) values are increasing as the predictor variables (A and B) increase. After careful observation, maximum q_t = 34.168 g/g is traced to 0.2 g OP dose and 50 min contact time. These optimal combinations can be compared with several possibilities shown in Table 7. Yao & Song (2021) reported a capacity of 59.7 g/g from 20 g dried OP ferrofluid utilisation, which is about twice the amount obtain in this study.

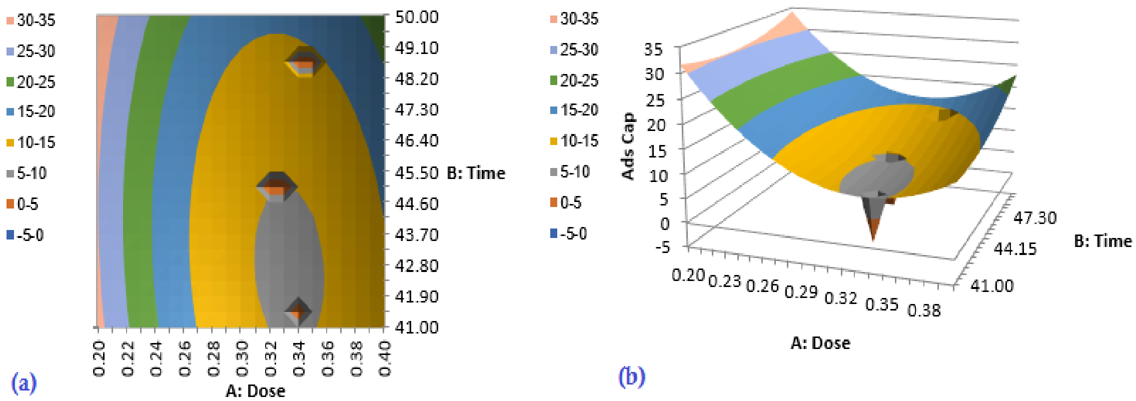

Fig. 12. RSM (a) contour, and (b) 3D surface plots

Source: Author's own data

Table 7. Predicted response calculator output based on random choice of A and B

A (g)	B (min)	Predicted Response (g/g)	Lower 95% CI	Upper 95% CI	Lower 95% PI	Upper 95% PI
0.1	45.5	73.2132	35.106824	111.319480	32.977393	113.448911
0.2	50	34.1679	35.106824	111.319480	32.977393	113.448911
0.4	50	22.7669	35.106824	111.319480	32.977393	113.448911
0.4	30	31.9327	35.106824	111.319480	32.977393	113.448911
0.4	20	74.0934	35.106824	111.319480	32.977393	113.448911
0.4	15	104.5682	35.106824	111.319480	32.977393	113.448911
0.2	70	112.3631	35.106824	111.319480	32.977393	113.448911
0.1	70	147.3239	35.106824	111.319480	32.977393	113.448911
0.1	20	155.7832	35.106824	111.319480	32.977393	113.448911

CI = Confidence Interval & PI = Prediction Interval

Source: Author's own data

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The 95% CI for the predicted response ranges from 35.10682378 g/g to 111.3194802 g/g, indicating the range within which the true mean response is likely to fall with 95% confidence, and the 95% PI for the predicted response ranges from 32.97739344 g/g to 113.4489105 g/g, portraying the range within which individual future observations are likely to fall with 95% confidence. Comparing predicted responses across different levels of adsorbent dosage and contact time can help identify alternative optimal conditions for maximizing adsorption capacity. Two situations are clear here. First, based on the provided data, the combination of A = 0.4 g and B = 15 min yields a predicted response of 104.5682 g/g, which is quite high. This combination suggests rapid adsorption with a relatively short contact time, making it a suitable choice for saving time and energy. The energy expanded would mainly be in the form of mechanical energy for mixing or agitation to ensure sufficient contact between the OP adsorbent and the solution containing the target substance (e.g., oil in the case of an oil spill), since it is a physical process (Nguyen et al., 2023). Moreover, Toamah & Fadhil (2021) mentioned that the available crude oil molecules are not proportionate to all the exchange site on the sorbent at high dosages of it. Obi et al. (2023) and Hussein et al. (2008) also affirmed that increasing the dosage reduces the adsorption capacity, which is evident in row 3 of Table 7 when the dose = 0.4 g (keeping B as 50 min). Secondly, to conserve or minimize the use of adsorbent and save on wastage and cost, a combination that achieves a reasonably high q_t while using the lowest possible dosage of OP sorbent should be employed. Thus, the combination of A = 0.1 g and B = 20 min yields $q_t =$ 155.7832 g/g, which is quite high compared to other combinations with the same dosage. But whether these choices are feasible must be tested in the laboratory. The optimum combination from the RSM software (Row 2, Table 7) may not align perfectly with either priority, but it represents a balanced approach with moderate adsorbent usage and contact time. Since the sorption of oil spill from water is made possible using OP, testing it before mass manufacturing is desired, based on protocols described by Cooper & Keller (1993).

4. CONCLUSION

The use of OPs for oil spill cleanup could create new economic opportunities, such as the development of OP collection and processing industries in regions where citrus farming is prevalent. To verify this claim, 0.2 – 0.4 g of 150 nm OP was added to 50 mL crude oil-water mixture to experimentally sorb it from water at varying contact time between 41 – 50 min, based on 10-run SigmaXL DOE. Earliest before RSM optimisation, an irregular OP surface after sorption, revealed by SEM and the N-H and O-H functional groups (3865.48 & 3788.32 cm^{-1} pre- and 3973.49 & 3857.76 cm^{-1} post- sorption peaks/wavenumbers) revealed by FTIR analysis, already signal the ability of OP to sorb oil from water. Later, an RSM-CCD optimisation returns 34.17 g/g as the OP adsorption capacity, corresponding to 0.2 g dosage and 50 min contact time. This fit is further supported by the PI, CI, DW statistics, p-value, F-value, RMSE, DF, SS, MS, T, VIF, SE coefficient and tolerance statistical and nonlinear regression model predictions estimates obtained, explaining the quadratic model, 3D surface and contour plots, and the predicted q_t response. Clearly, optimisation of crude oil removal from water using OP was successfully carried out using SigmaXL. But model precision improvement is needed by increasing the number of centre-points in the RSM software to increase R^2 and adjusted R^2 beyond their current value of 0.8898 and 0.7723, respectively. Other possible optimal combinations are respectively, A = 0.4 g, B = 15 min & 104.5682 g/g and A = 0.1 g, B = 20 min & 155.7832 g/g for time and resource management, based on the 'Predicted Response Calculator'.

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CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefits, commercial or financial conflicts and declare the absence of conflicting interests with the funders.

AUTHORS' CONTRIBUTIONS

A.M. Abubakar, H.D. Diriki, L.B. Umdagas: Conceptualisation; **H.D. Diriki, T. Saka**: Methodology; **A.M. Abubakar, K.C. Mukwana**: Data analysis, investigation, and writing-original draft; **W.C. Ulakpa, A.A. Bhutto**: Project administration, and formal analysis; **A. M. Abubakar, L.B. Umdagas, K. Khan**: Data Validation; supervision, and writing- review and editing.

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