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Experimental Performance Study of Activated Carbon Loaded with Nickel Oxide Nanoparticles from Pomegranate Peels in Diesel Desulfurization

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Abstract: This study develops a green methodology for adsorptive desulfurization of diesel fuel with NiO-NP-doped AC prepared using Iraqi pomegranate peel extract. The NiO-NPs were generated greenly by reduction of nickel nitrate via phytochemicals generated from pomegranate peel, then these NiO-NPs were calcined and loaded on AC (10 wt.%). Characterization by XRD, FTIR, SEM, and EDS supported the formation of a mesoporous AC-NiO composite with a surface area of 775 $m^2 g^{-1}$ and NiO particles of ~30-50nm sizes having both crystalline and amorphous forms. The optimization studies showed that the catalyst loading of 0,75 g gave the highest sulfur removal efficiency, i.e., 90,76% with a reaction temperature of 90°C and a reaction time of 90 minutes, respectively. These are mild conditions that provide a good compromise between performance and energy economy. The degrees of removal achieved herein compare favorably with those obtained by other adsorbents and thus spotlight the potential of this green-engineered catalyst for industrial-scale sustainable settings.

Keywords: Activated carbon, Nickel oxide nanoparticles, Pomegranate peel extract, Green synthesis, Diesel fuel, Sulfur removal

Introduction

Energy production is perhaps one of the most important contemporary dilemmas, as it is intrinsically related to economic growth and economic activities. This generation of goods, service provision, and the entire economic activity ultimately rest on how energy consumption occurs Between 1980and 2023, the annual global energy usage increased significantly, with total consumption reaching approximately 404quadrillion BTUs in 2023(Raimi, Zhu, Newell, & Prest, 2024). Although the share of fossil fuels is declining after the eighty s to the year 2013, these fuels still provide almost 82 percent of the world's energy, with close to half of that coming from petroleum (Abas, Kalair, & Khan, 2015). Petroleum is, therefore, the main pillar of global energy supply but contributes significantly to environmental problems through sulfur compounds such as free sulfur, sulfides, disulfides, mercaptans, thiophene, benzothiophene, and dibenzothiophene. When burned, these turn into harmful SOx and result in air pollution, global warming, and acid rain (Shi & Wu, 2021; Obaid et al., 2021). Over the last few years, sulfur restrictions have become draconian and efforts made to remove it from fuels such as diesel and gasoline (Haruna, Merican, Musa, & Abubakar, 2022). The most advanced and adopted industrial standard for sulfur removal is catalytic hydrodesulfurization (HDS) under which many of the sulfur compounds, including sulfides and thiophenes, can be removed completely (Tanimu & Alhooshani, 2019). There are, however, some compounds, dibenzothiophenes being one of them, which show lower reactivity to HDS treatments, thus requiring harsher conditions for the reaction to proceed to achieve the necessary sulfur removal, thereby making the procedures energy demanding and costlier (Sikarwar, Gosu, & Subbaramaiah, 2019). The catalysts for HDS will be expensive and sometimes they will also induce undesirable side reactions, which will lower the octane rating of gasoline (Tanimu & Alhooshani, 2019).

Activated carbon (AC) is an imperative support material for adsorptive desulfurization because of its high surface area, highly developed porosity, and significant surface functional groups which assist in the dispersion and

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prevent agglomeration of NiO nanoparticles as well as provide large surface areas for adsorption with sulfur compounds. The combination of the physical adsorption characteristics of AC and the chemical adsorption characteristics of NiO improve overall sulfur removal efficiencies. The mesoporous characteristics of the AC allow for mass transfer and availability of active sites, facilitating the kinetics and capacity of desulfurization. In this work, AC helps to stabilize the NiO nanoparticles but also contributes to a significant portion of the composite's total BET surface area (775 m^2/g) that will have a primary implication on the adsorptive performance.

To counteract the effects of these limitations, other ways of ultra-deep desulfurization have been given considerable attention. These include biodesulfurization, oxidative desulfurization (ODS) with state-of-the-art catalysts, and extractive desulfurization with ionic liquids(Tanimu et al., 2022). Among these methods, adsorptive desulfurization (ADS) presents a viable option as it is considered to be economically favorable and energy-efficient. It operates under atmospheric pressure and temperature without requiring costly hydrogen or catalysts. Certain adsorbents have very high adsorption capacities to remove refractory aromatic sulfur compounds(Boshagh, Rahmani, & Zhu, 2022; Mikhail, Zaki, & Khalil, 2002).

The adsorptive mechanism for desulfurization of organic sulfur compounds on activated carbon impregnated with NiO involves many interacting processes and could be described on the level of basic equations. It is actually a two-step sorption process: initial physical adsorption onto the activated carbon support of high surface area and the second step being the reactive or adsorption desulfurization, wherein NiO nanoparticles interact with organic sulfur compounds through a chemisorptive method. As for the reactive adsorption desulfurization path, NiO nanoparticles interact with thiophene in this way:
$NiO+C4H4S \rightarrow NiSx+C4H4$

Here, thiophene (C4H4SC4H4S) undergoes C-S bond cleavage on NiO active sites, from which formation of nickel sulfide species (NiSxNiSx) occurs, with the hydrocarbon portion being released back to the fuel streams. This mechanism is assisted by activated carbon's high surface area (775 m2/g in this study), which provides spectacular dispersed conditions for the NiO nanoparticles and accessibility to active sites so that desulfurization occurs effectively through the synergy of physical and chemical adsorption processes.

In recent studies, enhanced adsorbents have been developed with much better adsorption capacity, their regeneration capacity, and selectivity(Lata, Singh, & Samadder, 2015). Sulfur compounds degrade the quality of fuels as well as damage the environment, thus creating a need for cheap and effective removal methods. For example, the removal of dibenzothiophene (DBT) from the model oil using acid-activated carbon-based adsorbents loaded with magnetite nanoparticles has been shown(Jha et al., 2019; Sadare & Daramola, 2019). Other researcher synthesized a new ZnO-nanobentonite (ZnO/NB) nanocomposite using hexadecyltrimethylammonium bromide (HDTMA) as a surfactant, which show outstanding efficiency in removing xylenol orange (XO) dye from wastewater(Obaid, Tameemi, & Hameed, 2021).

This study concentrates on the adsorptive desulfurization of model diesel fuel with porous activated carbon support, packed with Ni oxide nanoparticles made from local Iraqi source of pomegranate. It emphasizes the much-needed innovative technologies of desulfurization based on sustainable economic viability against an environmentally sustainable saviour of cleaner fuel desired for a better earth. The fix is most viable because it replaces unabated imports (with local ingredients) with sustainable practices and improved environmental friendliness. Therefore, the adsorptive capacity of activated carbon in activated carbon-nanocomposite in this method is enhanced with the addition of Ni oxide-nanoparticles increased by incorporation of additional resources such as pomegranates, thus enabling the removal of Sulphur components from diesel fuels. As a global society, making strides to reduce fossil fuel burning impacts of the environment can only assist in improving air pollution and other environmental concerns and upgrade competition by reducing their effects in the energy sector. Continued investigation of emerging technologies in sustainable, environmentally friendly solutions can also promote local economies and promote sustainability through custodianship for the environment and promote precedence for future solutions and green energy practices.

Preparation of Nickel Oxide Nanoparticles Using Pomegranate Peel Extract and Their Application in Adsorptive Desulfurization

Experimental Setup

The experimental setup for the adsorptive desulfurization study comprised several key stages: synthesis of NiO nanoparticles via green chemistry, preparation of NiO-loaded activated carbon composite, model diesel fuel formulation, and batch adsorption experiments. Preparation of NiO nanoparticles (NiO-NPs) using pomegranate (Punica granatum) peel extract occurs by some eco-friendly, multistep method that associates waste valorization with a few principles of green chemistry.

Preparation of Pomegranate Peel Extract

Freshly collected pomegranate peels were carefully washed to remove all dirt and oven-dried at 60°C for 16 hours so as to preserve the bioactive compounds important for nanoparticle synthesis (Vinay, 2019). The dried peels were then ground in an electric mill into fine powder to increase surface area for better extraction efficiency (Ben-Ali, Akermi, Mabrouk, & Ouederni, 2018). In extraction, 10 g of powder was blended with 100 mL of distilled water, known for its high purity and absence of interfering ions, and kept under magnetic stirring at 60°C and 200 rpm for 1 hour to facilitate adequate polyphenol and flavonoid extraction. Both act as natural reducers and stabilizers for green nanosynthesis(Shanmugavadivu, Kuppusamy, & Ranjithkumar, 2014). The mixture was centrifuged, and the supernatant was filtered through a Whatman No. 1 paper to obtain a clear extract free from any particulate matter. This is then used immediately for synthesis if required. Else, it is stored in dark bottles at 4°C to retain its stability (Sandhya, Khamrui, Prasad, & Kumar, 2018).

Preparation of Nickel Nitrate Solution

The nickel(II) nitrate hexahydrate [$Ni(NO_3)_2 \cdot 6H_2O$] solution at 1 M concentration was prepared applying the laboratory-defined protocols stated by Kumar et al.(Sandhya et al., 2018). Correct weighing of 290.8 g $Ni(NO_3)_2 \cdot 6H_2O$ (molar mass = 290.8 g/mol) was done on an analytical balance. The salt was transferred to a 1000 mL volumetric flask half-filled with 500 mL deionized water so as to avoid early crystallization. A magnetic stirrer working at 200 rpm stirred the mixture until dissolution was complete(Ravikumar et al., 2019). The solution was then made up to the mark with deionized water while stirring was continued to avoid concentration fluctuations. Such precautions are crucial to help ensure success and reproducibility in further downstream projects(Akram et al., 2022).

The solution was stored inside a dark, sealed glass bottle that had been labeled with details of the concentration and preparation date, as well as with the hazard warnings. It was then kept in a cold, dry location away from light for the prevention of degradation. Handling procedures required strict safety measures that necessitated wearing nitrile gloves and goggles, with working done under a fume hood, this being a feature of known toxics $Ni(NO_3)_2$, oxidative in nature; risk involved includes inhalation or skin contact (Dichala, Therios, Koukourikou-Petridou, & Papadopoulos, 2018).

This method is highly cited in nanomaterial synthesis and analytical chemistry (Sandhya et al., 2018), with further use ensuring the exact ion availability of nickel for nanoparticle fabrication requiring pure and consistent solutions. The hexahydrate form is highly soluble in aqueous media, making it a convenient reagent for performing controlled reactions involved in green chemistry applications.

Green Synthesis of NiO Nanoparticles Using Pomegranate Peel Extract

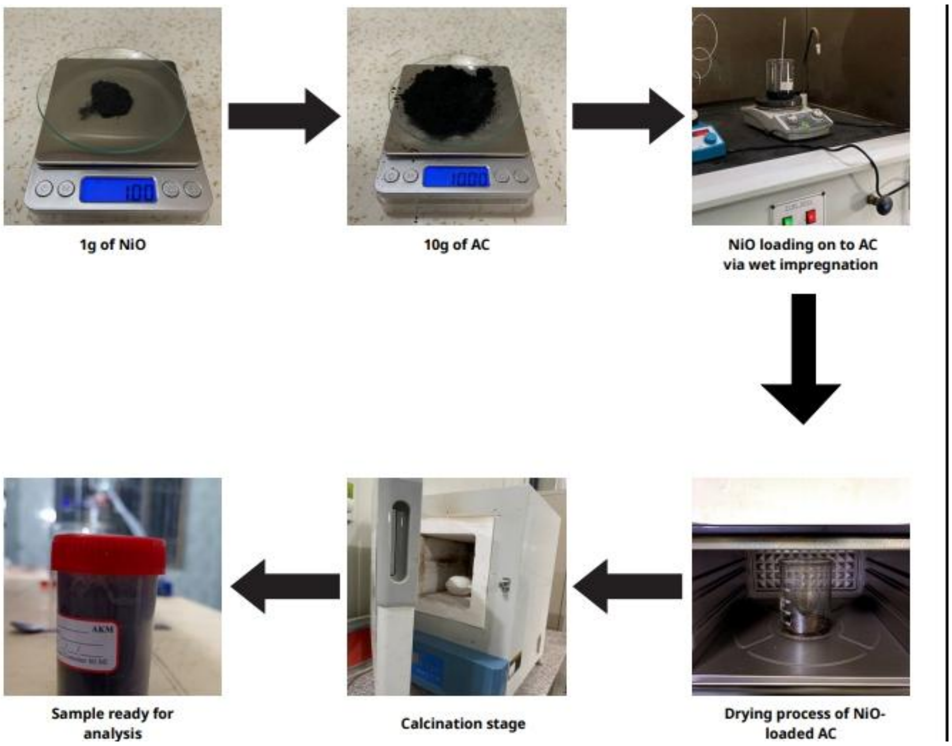
NiO nanoparticles were prepared by a non-conventional green method, using the peel extract of P. granatum as a source of phytochemicals which helped in reduction as well as a stabilizing agent. A typical method involved mixing 100 mL of 1 M nickel nitrate [$Ni(NO_3)_2 \cdot 6H_2O$] solution with 100 mL of aqueous pomegranate peel extract. Afterward, the mixture was maintained at 60°C with stirring at 200 rpm for an hour to accomplish reduction of Ni^{2+} ions by polyphenols and flavonoids in the extract. These active compound possess the ability to reduce ions and provide steric stabilization to the nanoparticles, thus preventing their aggregation (Narayanankutty, Sharma, & Singh, 2023; Saleh, Hussein, Ray, & Elzaref, 2024).

The initial pH of the mixture was approximately 5.5, which was then adjusted to ~10 using 1M NaOH, a necessary step to improve nanoparticle nucleation and growth (Besenhard et al., 2020). A color change from green to dark brown/black, which can be associated with available surface plasmons in the nanoscale particles ((Jawale et al., 2022)), confirmed that NiO NPs were now formed. After leaving to cool to room temperature, the colloidal suspension was distilled, and nanoparticles were extracted using vacuum filtration. The precipitate was washed multiple times with deionized water to eliminate any chemicals used in the process. The precipitate dried at 80°C for 12 hours. The nickel hydroxide intermediates were then transformed into crystalline NiO nanoparticles by calcining at 420°C for 3 hours in a muffle furnace, which was confirmed by XRD (Narayanankutty et al., 2023). In this case, we applied the principles of sustainable chemistry by utilizing agricultural waste (the pomegranate peel), and did not use any toxic reductants, and collected high value nanoparticles that have utility in catalytic, electronic and biomedical applications.

Incorporating 10% NiO Nanoparticles onto Activated Carbon

The activated carbon employed in this study was sourced from commercial-grade AC from Sigma-Aldrich Washing with deionized water and drying at 120°C for 12 hours preceded its use to eliminate impurities and adsorbed moisture. Physical characterization of the AC surface was conducted by Brunauer–Emmett–Teller (BET) analysis; the value of 775 m²/g indicated that the AC surface was largely mesoporous. Scanning electron micrographs showed evenly dispersed NiO nanoparticles over the AC surface, whereas energy-dispersive X-ray spectroscopy checked their elemental composition. The AC's huge surface area and porosity were operative in effectively dispersing the NiO nanoparticles and opening up maximum adsorption sites for thiophene removal.

A 10 wt% nickel oxide nanoparticle (NiO NP)-activated carbon composite was created by loading the nanoparticles onto the carbon support following a procedural protocol to adequately disperse the nanoparticles across the carbon support(Dinh & Gomes, 2020). Based on preliminary dispersion studies, 1 g of NiO NPs was dispersed into 100 mL of deionized water via magnetic stirring (800 rpm, 1 h) to provide optimal colloidal stability and minimal agglomeration of the nanoparticle suspension. Next, 9 g of activated carbon was slowly added into the suspension while stirring (700 rpm, 1.5 h) till the activated carbon infiltrated the NiO NP suspension heterogeneously covering the surface of the carbon support and penetrating the porous matrix of the activated carbon(Arabzadeh, Ghaedi, Ansari, Taghizadeh, & Rajabi, 2015). Then, the composite was dried at 120 °C for 12 h until no moisture was detected before calcination at 450 °C for 3 h in static air. The calcination temperature of 450 °C was selected based on optimization studies that demonstrated this temperature effectively promotes the transformation of nickel hydroxide intermediates into crystalline NiO nanoparticles while preserving the structural integrity and porous nature of the activated carbon, ensuring optimal composite properties (Singh et al., 2025).



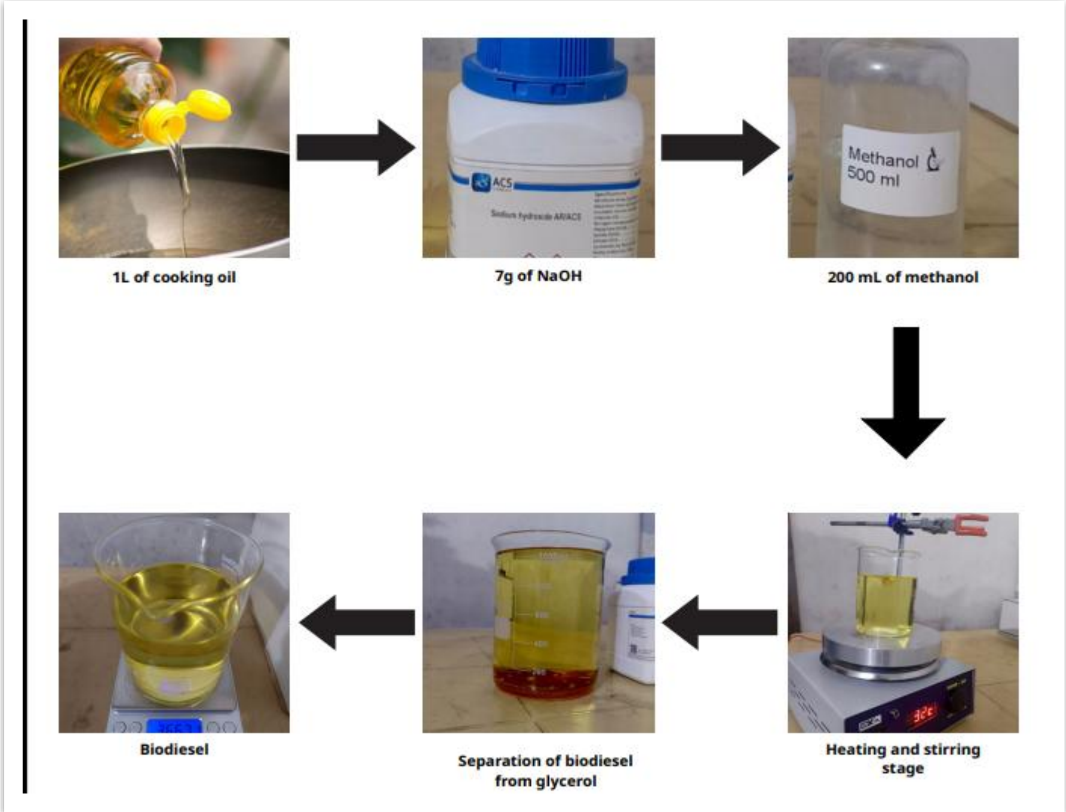
Scheme1. Incorporating 10% NiO nanoparticles onto activated carbon

This approach highlights the importance of agitation parameters (speed, time), temperature and thermal conditions, and interfacial interactions in achieving high loading efficiency and spatial uniformity. Accurate, adaptive, and directed loading will directly affect composite performance in environmental applications such as pollutant adsorption, with both accessibility to loadable nanoparticles, and distribution of nanoparticles being the main factors determining effectiveness in catalysis applications as illustrated in Scheme 1.

Biodiesel from Waste Cooking Oil and Its Preparation

Biodiesel was prepared from waste cooking oil (WCO) via a base-catalyzed transesterification process, following established protocols]. The resulting biodiesel was characterized and confirmed to comply with ASTM D6751 standards, ensuring its suitability as a solvent for the model diesel fuel in this study (Foo et al., 2022). To begin, 1 L of WCO was filtered to eliminate solids materials and residual water content, as residual water content is important for reducing saponification side reactions that decrease yield in the presence of alkaline conditions. The filtered WCO was then preheated to 60 °C to reduce viscosity and improvements on reaction rate and therefore kinetics (Jain, Sharma, & Rajvanshi, 2011) as illustrated in Scheme 2 . A sodium methoxide catalyst was synthesized by dissolving 7 g of NaOH in 200 mL of anhydrous methanol while stirring continuously. The sodium methoxide catalyst solution was then added to the heated WCO and stirred mechanically for 1 hour at 60 °C at a stirring speed of 600 rpm. After the reaction was complete the contents were placed into a separatory funnel and allowed to settle for 24 hours, after the 24 hours there were two distinct layers, one that contained biodiesel and the other that contained glycerol(Cetinkaya & Karaosmanoğlu, 2004).

After it was clarified, the crude biodiesel was separated and washed with warm deionized water (50°C) twice to remove residual catalyst and glycerol. The washed biodiesel was dried over anhydrous sodium sulfate. Finally, all characterizations confirmed compliance with ASTM D6751 standards. This demonstrates the method's ability to synthetize biodiesel from waste cooking oil as a suitable alternative fuel. This implementation method supports the principles of a circular economy by finding new use for waste product, while diverting the potential reliance on fossil-derived diesel as alternative fuel in the transportation sector with possible ability to expand for industrial projects(Abd Rabu, Janajreh, & Honnery, 2013).



Scheme 2. Preparation of biodiesel from waste cooking oil and its preparation

Preparation of a Model Diesel Fuel Containing Thiophene

A model diesel fuel containing 500 ppm thiophene was prepared to assess desulfurization efficiency, following the established protocol for simulating sulfur containing fuels in adsorption and catalytic studies(Dai et al., 2008). The theoretical calculation of thiophene required to make the desired solution utilized the density of thiophene

(C_4H_4S, density = 1.05 g/mL). Thus, to obtain the concentrations of thiophene required for the 500 ppm solution for 366.71 g of biodiesel, 183.36 mg of thiophene was required and calculated as follows(Haji & Erkey, 2003):

$$ppm = \frac{mass\ of\ solute\ (mg)}{mass\ of\ solutiob\ (kg)}$$

The corresponding volume was determined using the compound's density:
Volume (µL) = mass (g) / density (g/mL)
Volume (µL)=0.18336 g / 1.05 g/mL=174 µLVolume (µL)=1.05g/mL0.18336g=174µL

A measured volume of thiophene (174 µL) was placed in 366.71 g of biodiesel in a sealed glass flask. The contents were vortexed for 5 minutes, which dispersed thiophene uniformly in the biodiesel. This mixture had to be thoroughly mixed due to the objective of picturing real-world sulfur distributions in fuel matrices(Seredych & Bandosz, 2011).

This method is common (eg adsorptive, oxidative, or extractive desulfurization) in the literature, and produced a reproducible model system for evaluating the sulfur-removal efficiencies of innovative materials or catalysts. The fuel was kept under inert conditions to prevent the thiophene from degrading prior to subsequent experimental studies, as illustrated in Scheme 3.

Scheme 3. Preparation of a model diesel fuel containing thiophene

Characterization of the Catalyst

The synthesized catalyst undergoes rigorous characterization using advanced techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR). These analyses provide insights into the structural, morphological, and chemical properties of the material. Additionally, graphical representations and images are utilized to interpret the findings comprehensively.

Experimental Procedure: Desulfurization of Diesel Samples

In this work, desulfurization experiments were conducted on the prepared model biodiesel with thiophene additions using a simple batch reactor system consisting of a water bath to control the temperature, a three-hole flask of suitable capacity, and a magnetic stirrer and heater to ensure the homogeneity of the reaction medium and precise control of operating conditions. This system is one of the simplest and most widely used in laboratory studies, as it allows temperature control (via the water bath), adjustable stirring speed, and the possibility of taking samples during the reaction through the flask's side ports. It also ensures a homogeneous distribution of the adsorbent within the biodiesel medium. as illustrated in Figure 1.

The adsorptive desulfurization process on synthesized Biodiesel. A total of 13 experiments are conducted to evaluate the efficiency of sulfur removal. a set amount of biodiesel (50 mL) was used in each experiment, and particular operating parameters were applied. The sulfur content post-treatment is measured to determine the effectiveness of the adsorbent in reducing sulfur levels. The catalyst efficiency was measured as a function of the amount of the catalyst (nanofilament NiO+ activated carbon), temperature and adsorption time. This step is critical for optimizing the process parameters and achieving desirable desulfurization outcomes. All the steps of biodiesel preparation and catalysis synthesis is summarized in scheme 5 for clarification.

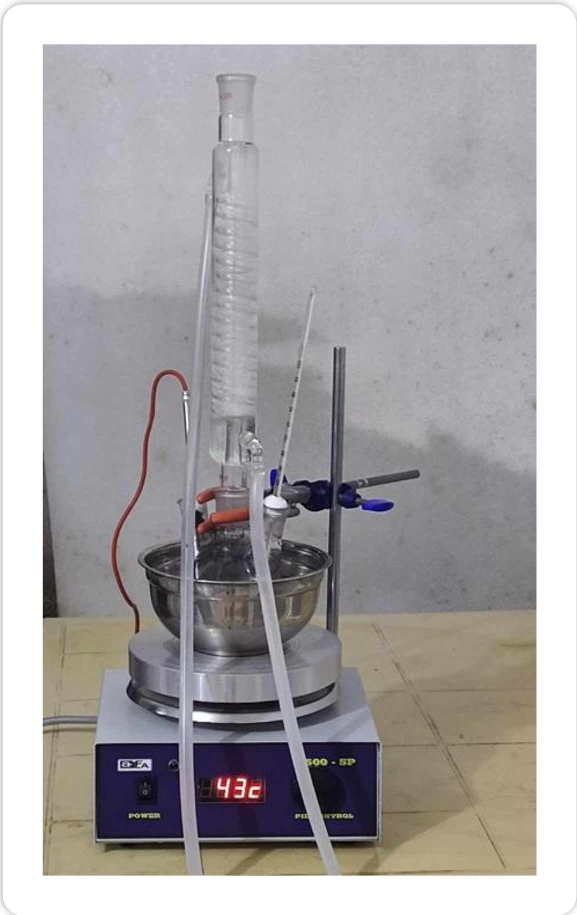
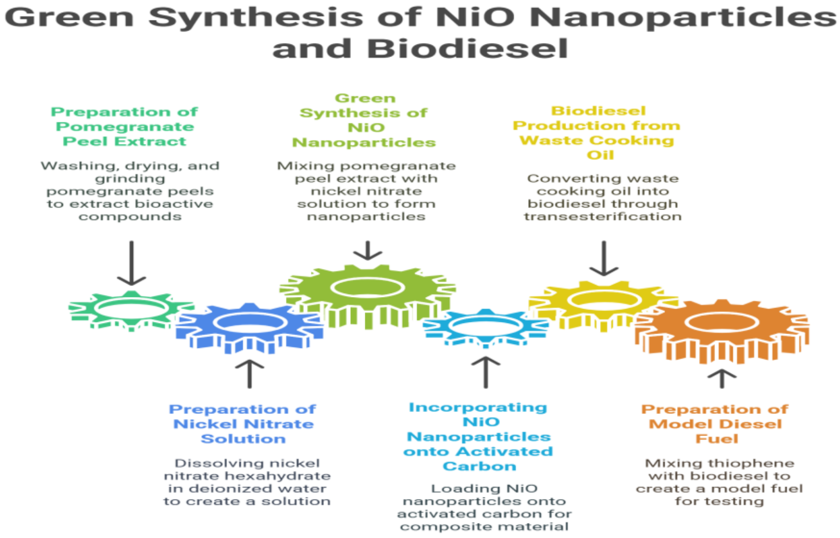


Figure 1. Photograph of the simple batch reactor apparatus



Scheme 5. Schematic representation of the experimental procedure.

Results and Discussion

X-Ray Diffraction (XRD)

The X-ray diffraction (XRD) (figure 2) pattern is indicative of a composite material together with activated charcoal and NiO. The pattern displayed sharp intense peaks in the region of 2θ about 30° to 45° associated with the crystalline NiO phases(Hu et al., 2008). These peaks can be indexed to the following planes of face-centered cubic NiO: (111), (200) and (220). These peaks indicate the presence of well-crystallized NiO material in the sample(Wubie, 2019). Along with these strong reflections, the pattern showed a weak broad hump or slope at lower 2θ values (about 10°-30°), characteristic of the amorphous nature of activated charcoal(Khalil, Elhamdy, & Elsamahy, 2022). The broad feature arises from the amorphous nature of carbon structure where there is not long range order that is presence in the sharp reflections of the crystalline NiO phase.

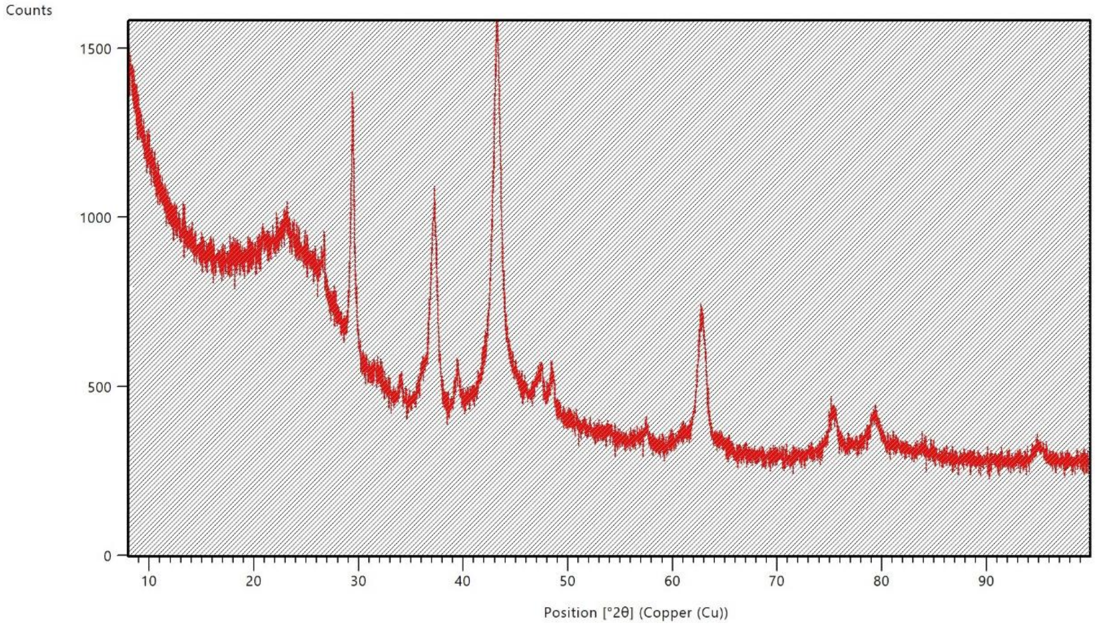


Figure 2. XRD pattern of NiO nanoparticles incorporated with activated carbon.

The presence of both sharp crystalline peaks and a broad amorphous background in the XRD pattern shows that the activated carbon was successfully added to NiO. The position and intensity of the NiO peaks indicate that the crystalline structure of NiO is preserved upon composite formation, and, similarly, the amorphous phase of carbon was preserved. The preservation of both phase structures could be advantageous for applications in catalysis, energy storage, or for adsorption where a blend of the useful properties of crystalline metal oxides, and porous high-surface-area carbon is desired. Collectively, XRD shows the formation of a NiO–AC composite with both crystalline and amorphous domains while respecting the structures of both components within the hybrid material.

FTIR

Figure 3 shows the FTIR spectrum of NiO nanoparticles incorporated with activated carbon. This specialized characterization technique reveals critical information about the chemical bonding, functional groups, and structural properties of the composite material. The FTIR spectrum shows several characteristic absorption bands that attest to the presence of NiO nanoparticles. Particularly, the low wavenumber region at about 520-550 cm^{-1} shows a very strong absorption peak that corresponds to the vibrations of the Ni-O bond(Hong et al., 2021). This particular and primary peak stands to identify NiO materials; it appears quite sharp in the spectrum and suggests the formation of NiO nanostructures(Bharathy & Raji, 2014).

The spectrum also presents bands between 660-730 cm^{-1}, indicating Ni-O-H vibrations (Tanimu & Alhooshani, 2019) . Hydroxylated nickel oxide species form on NiO nanoparticle surfaces upon moisture exposure or under certain synthesis conditions. These bands indicate partial surface hydroxylation over NiO nanoparticles, which could greatly alter their catalytic and adsorptive properties.

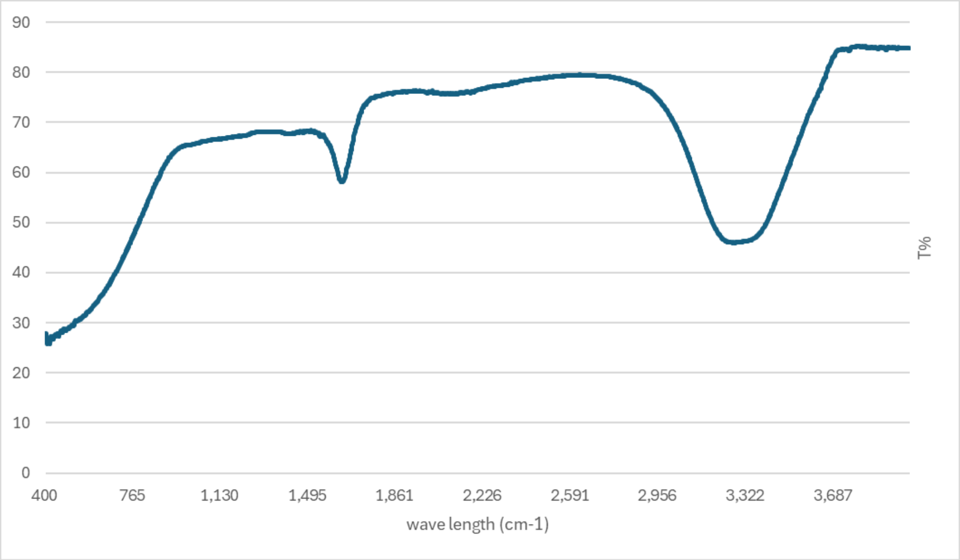


Figure 3. FTIR spectra of NiO nanoparticles incorporated with activated carbon.

A broad absorption band is observed around 3400-3500 cm^{-1} in the spectrum. This absorption corresponds to the stretching vibration of hydroxyl (-OH) groups (Abbasa et al., 2024; Han et al., 2013), indicating adsorption of water molecules or the presence of surface hydroxyl groups on the NiO nanoparticles and the activated carbon support. Of course, an absorption band occurring around 1630-1650 cm^{-1} might be assigned to the bending vibrations of these hydroxyl groups, thus confirming their presence within the system.

The spectrum has special features of activated carbon-type materials in the region 1000-1500 cm^{-1}. Stretching of C-O bonds may be responsible for the band observed at 1030 cm^{-1}[3], whereas bands appearing in the region 1380-1384 cm^{-1} can represent carboxylate groups on the surface of the activated carbon or nitrate species that exist as impurities of the synthesis(Khodair et al., 2022). The absorption band at 1384 cm^{-1} is interesting as it may indicate the presence of carboxylate groups on NiO[3], nitrate species (NO_3^-) formed during synthesis or adsorption, or vibrations of NiO_3^- in the material. A surface modification or adsorption process could have occurred at the interface of NiO and the carbon substrate, as evidenced by this spectral feature.(Li et al., 2014).

The spectrum also suggests possible adsorption phenomena. In the literature for similar compounds, peaks for adsorbed NO_2 species that exhibit an asymmetric stretching vibration of NO_3 on the surface of activated carbon were indicated by peaks in the 1380-1385 cm^{-1} range (Li et al., 2014). This is indicative of potential exposure to nitrogen oxides in the composite or possibly that nitrate groups were incorporated at synthesis during production for this composite.

FTIR spectroscopy does not directly measure particle size but features within the spectrum can serve as evidence potentially about the morphology of the NiO nanoparticles. For example, the sharpness and intensity of the Ni-O vibration band in the low wavenumber region relates to crystallinity/particle size. Comparing to similar research, the spectral features both suggest that the NiO nanoparticles are in the possible size range of 5-15 nm corresponding to ultrafine spherical particles that are generally produced at moderate temperatures (close to 300°C) (Hong et al., 2021).

There are no significant peaks unrelated to the spectum that would suggest undesirable cross-contamination/species, and all bands matched characteristic NiO bands and bands of activated carbon; thus we feel confident to state that the purity in the composite for the NiO portion is high. Overall, the spectral features characteristic of NiO nanoparticles (and activated carbon) characterized the spectrum with few unassigned bands. This either suggests that only minimal by products formed in the synthesis (Biju & Khadar, 2003).

The FTIR spectral characteristics of the NiO-activated carbon composite lend to possible good catalytic applications, where the Ni-O bond sites and surface hydroxyl groups are ideal for catalytic reactions, as these sites can enhance electron transfers and absorb the reactants which are reducible via OH-. The activated carbon provides the large surface area, while the presence of NiO nanoparticles provides catalytic active sites for many reaction mechanisms including oxidation mechanisms. The spectrum shows indications that adsorbing material may dispose of optimize gaseous pollutants. Research has shown which materials showing spectral features such as this can show effective removal of nitrogen oxides or some other type of pollutants. The spectrum indicates that the hydroxyl groups and oxygenated functionality contributes(Shanthakumari & Latha).

Energy-Dispersive X-Ray Spectroscopy (EDS)

An energy-dispersive X-ray spectroscopy (EDS) analysis in Figure 4 was conducted to determine the elemental content of the NiO nanoparticles supported onto activated carbon. The EDS spectrum showed that the primary elements are carbon (C), oxygen (O) and nickel (Ni) containing weight percentages of 68.1%, 14.0% and 8.3% respectively (C > O > Ni) which is attributed to the activated carbon matrix. The other background peaks of oxygen and nickel indicate through elemental analysis that NiO nanoparticles contained in the composite material. Minor elements (potassium (K), calcium (Ca), sulfur (S), silicon (Si), magnesium (Mg), phosphorus (P), manganese (Mn), and chlorine (Cl)) were detected in trace amounts and may have originated from precursor salts, synthetic reagents used or impurities. In regard to elemental distribution, these findings are consistent with FTIR results and indicate that the NiO phase on the activated carbon support is fairly well dispersed, and that the NiO, Actived Carbon composite itself forms part of the composition. Further, the absence of considerable contamination, and clear presence of target elements can be seen and supports the success of the synthesis process and ability of the material to be used for future catalytic or adsorption applications**.**

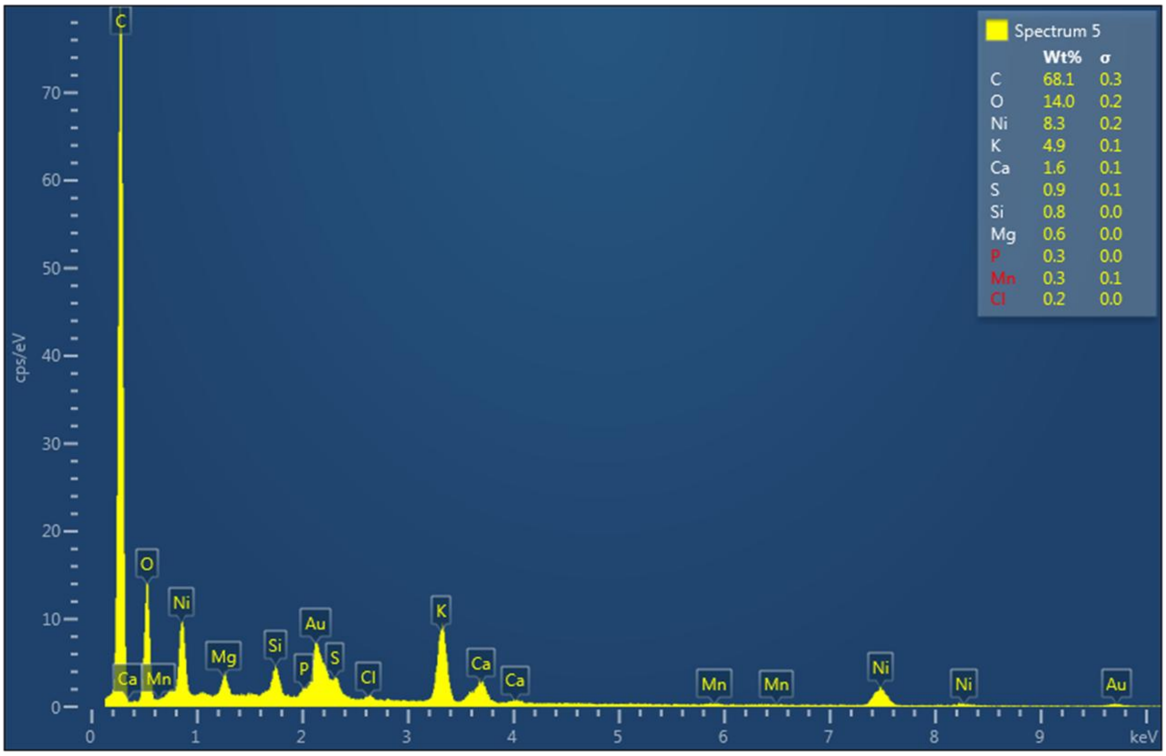


Figure 4. EDS spectra of NiO nanoparticles incorporated with activated carbon

Field Emission Scanning Electron Microscopy (FESEM)

Field Emission Scanning Electron Microscopy (FESEM) analysis was used to evaluate the surface morphology and particle size distributions for the NiO nanoparticles supported on activated carbon. Figure 5 displays the SEM image at a magnification of 135,000× which shows a rough heterogeneous surface with a high density of nearly spherical nanoparticles dispersed across the activated carbon surface. The measured particle size shown by the marker (D1 = 30.76 nm) indicates the NiO particles are of nanoscale dimension size. The nano-sized particles tend to form agglomerates or clusters on the porous carbon substrate. The observed morphology indicates sufficient tree-like dispersion of the NiO nanoparticles in turn likely led to an increase in surface area and accessible active sites. The porous nature of the activated carbon support is also visually seen to provide a strong hold on the anchoring of the nanoparticles. The morphological characteristics are corroborated by the EDS and FTIR results indicating the synthesis of a nanostructured NiO/activated carbon has been successful and could be used in future studies with potential for high-performance catalytic or adsorptive properties(Sekar, Kim, & Lee, 2020).

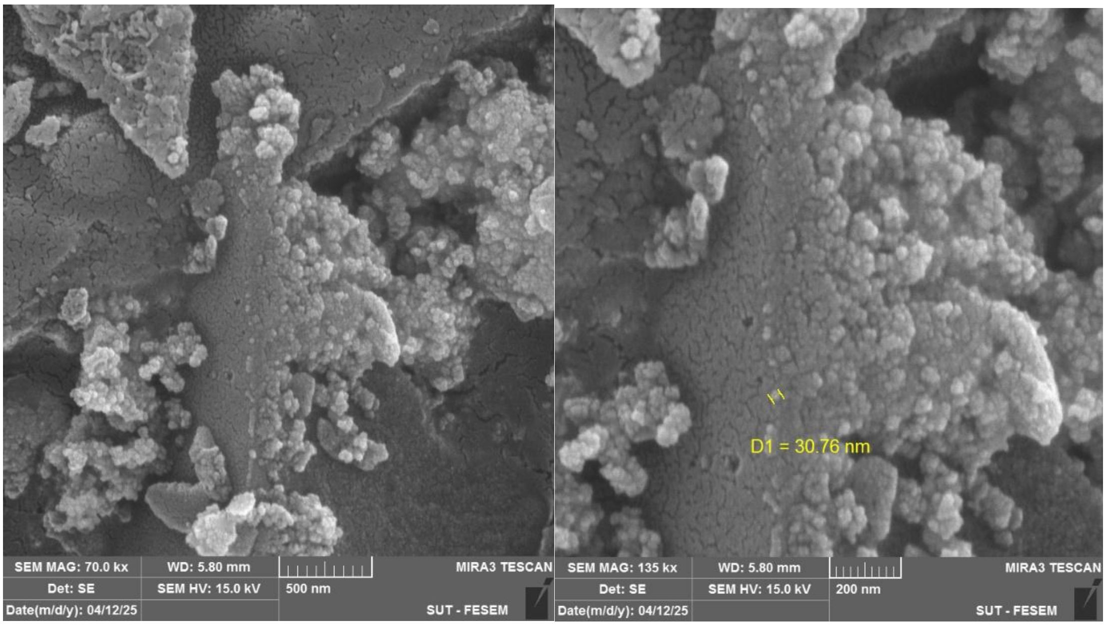


Figure 5 .(a) Low- and (b) high-magnification FE-SEM images of NiO nanoparticles with activated carbon.

Nitrogen Adsorption–Desorption

Nitrogen adsorption–desorption measurements were conducted to assist in understanding the textural properties of the NiO nanoparticles supported by activated carbon. The resulting isotherm (Figure 6) displays a typical type IV isotherm type with a clear hysteresis loop, confirming its mesoporous nature. The BET surface area of the material was 775.01 m² g⁻¹, which is a significant value for NiO/activated carbon composites.

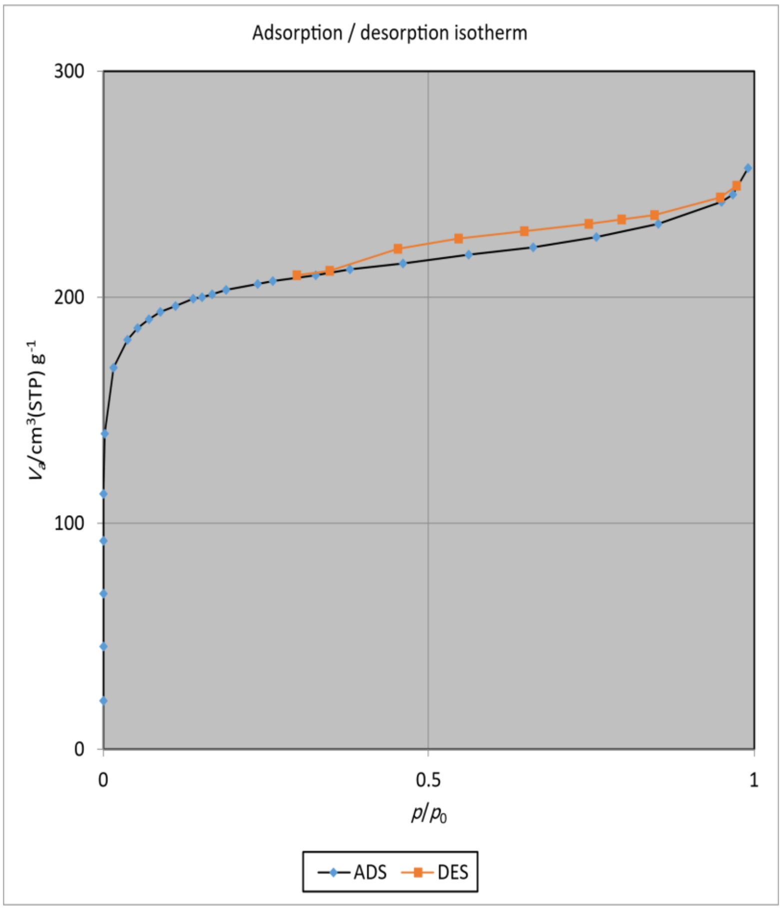


Figure 6. N2 adsorption-desorption isotherms of the NiO in activated carbon

The high surface area can be attributed to the synergistic effects of highly dispersed NiO nanoparticles along with the porous nature of activated carbon, as evidenced by SEM analysis. In terms of literature reports of similar materials, the BET surface areas of NiO/activated carbon composites range from 400 to 650 $m^2\ g^{-1}$, while the sample in this investigation shows very high surface area and it appears that the conditions of this synthetic strategy provided for a well-dispersed nanoparticle distribution avoiding pore blockage by NiO. This high surface area values of the present material is expected to enhance its performance in applications such as catalysis, adsorption, and for energy storage, since an increased accessible surface is highly advantageous(Khaleed et al., 2017; Yadav & Tripathi, 2017).

Adsorptive Desulfurization Performance

Determine optimal loading for sulfur removal from model diesel with an initial sulfur concentration of 500 ppm. The removal efficiency is calculated using the equation:

$$\text{Removal efficiency}=\frac{Intial\ concnetration-final\ concentration}{intial\ concentration}\times 100$$

The experimental data analysis suggests that intermediate catalyst dosages perform better than extreme values. Dosage 0.75 g NiO+AC showed improved sulfur removal for a series of temperature and time conditions, with a maximum removal efficiency of 89.14% obtained under mild conditions (30°C, 90 min), while the maximum sulfur content after treatment was 46.2 ppm remaining at elevated conditions (90°C, 90 min), thus resulting in a sulfur removal efficiency of 90.76%.

Higher catalyst loads (1.0 g) were able to compete in performance, especially at high temperatures, with 48 ppm remaining sulfur content (90.4% removal) at 90°C for 60 minutes. Yet, considering the slight improvement from the 0.75 g dosage, it might be agglomeration of the catalyst that limited accessibility to active sites, consistent with what has been observed in polyoxometalate catalyst studies, where a catalyst dosage higher than 70-75 mg decreased the desulfurization rate. 0.5 g dosages, on the other hand, fell short of impressive performances in all cases, with sulfur removal efficiencies ranging from 79.4-87.2 %, indicating that the density or distribution of active sites for the adsorption and burning of sulfur compounds may be less optimal (Mousavi et al., 2023; Pedram et al., 2022).

Reaction time optimization indicates that the duration of 60 to 90 minutes offers the most practical sulfur removal in a balanced relation with energy consumption. Using the best-performing catalyst dosage at 90-gc-NiO+AC (0.75 g), sulfur removal increased marginally from 90.7 to 90.76% by extending the reaction time from 30 to 90 min, such that longer contact times may enable further adsorption and oxidation of sulfur compounds. Nonetheless, the negligible uplift in performance after 60 minutes and from 60 to 90 minutes, wherein removal was roughly ~89.14% to 90.76%, denoted diminishing returns for long reaction time.

This time dependency pattern favors 30-60 minutes industrial reaction time reported in the literature to balance complete desulfurization under energy efficiency considerations. The 60-min optimum time from this study is supported by reports on amphiphilic catalysts where the desulfurization reaction stabilized within 10 min, although prolonged reaction times might have been required for complete conversion of sulfur compounds in complex diesel matrices.

The optimum range for catalyst dosages obtained from this study was between 0.75-1.0 g, which is fairly in agreement with what has been reported in literature regarding nickel-based desulfurization catalysts. NiO-MoO3 catalyst supported on activated carbon showed an optimum dosage of 0.5 g in oxidation of dibenzothiophene, whereby 71% sulfur removal was achieved. Amphiphilic catalysts showed an optimal dosage of 0.63 g (1.5 wt% of model oil) that gave a 96.1% desulfurization rate. The value obtained in this study, of 0.75 g, is a reasonable intermediate that indicates a good utilization of active sites without too much formation of aggregates by the catalysts.

Better than many conventional approaches, this work revealed an amazing sulfur removal rate of 90.76% at mild conditions (90°C, normal pressure). For example, whereas NiO/Fe_2O_3 systems need 160°C for 93.4% efficiency, polyoxometalate catalysts need 70°C to achieve 92.17% sulfur removal. Made in an environmentally friendly manner (using agricultural waste and nickel nitrate), requires less energy than high-temperature methods, and has a competitive surface area (775.01 m^2/g) that is comparable to commercial catalysts, thus the great performance of the pomegranate-derived NiO/AC catalyst is especially remarkable. This effectiveness shows the potential of green-engineered catalysts for industrial desulfurization, so balancing practical performance with sustainability.

The temperature optimizing results show that the pomegranate-based NiO/AC catalyst was effective at a moderate temperature of 90°C--in contrast to some studies which have operated at around 150°C and higher temperatures (i.e., Temperature is almost equal to 150°C) to achieve proper results. The studies on NiO/ZnO-TiO2 adsorbents (i.e. natural zeolites and titanias where very little details were provided) reported breakthrough sulfur capacities of 6.71% at unreported temperatures. The studies using NiO/Fe2O3 catalysts required 160°C (i.e. Temperature is approximately equal to 160°C) in order to reveal promising conversions. Achieving reasonable sulfur removal at 90°C is advantageous in terms of energy consumption and process economics.

The optimal operating times of 60-90 minutes for the reaction times shown in this study are consistent with typical industrial practice and literature values. Response surface methodology studies on NiO-MoO3 catalysts reported similar maximum desulfurization conditions at moderate successes at lower temperatures and catalyst additions(Abdulhadi & Alwan, 2023). The time that has occured for the reaction times have reasonable times relative to the rapid desulfurization in 3-10 minutes (i.e. The times occurred in the time scales of convenience) as is commonly employed in industry but remembering that these systems were not referred to real diesel fuel matrices where there would be significant complexity, as well as the moderate activity of the catalyst. Table 1. Effect of catalyst amount, temperature, and reaction time on sulfur removal efficiency.

The drop in desulfurization effectiveness when process variables are changed beyond their optimal values is due to a number of interrelated variables. In the case of the catalyst dosage, using more than the optimal 0.75 g of NiO-AC composite, doesn't lead to a linear increase in sulfur removal, and can lead to agglomeration of the particles in the reactor and overlapping of the active sites that thus leads to a decreased effective surface area available for the adsorption, and in turn less overall effective performance. This phenomenon is often described as a "site saturation effect", where additional adsorbent does not add significantly to removal because there are less thiophene molecules in solution than the number of active sites available. In a mathematical sense, this occurred with the plateau appearing in the Langmuir isotherm at a high adsorbent concentration.

Similarly, using temperatures or reaction times which are higher than their respective optima (90 °C and 90 min in this study) does not lead to proportionately increased efficiency, but rather an increase in energy use which increases exuberantly higher. High temperatures may potentially also even induce desorption of sulfur species that had already been adsorbed, as well as structural changes to the adsorbent itself, therefore reducing its adsorption capacity. Long contact times likely also end up reaching equilibrium, at which no further contact with the adsorbent would assist, and may in fact even allow additional desorption or structural degradation of the adsorbent.

Table 1. Sulfur removal of samples as a function of amount of catalyst, temperature and time.

Amount (g) (NiO+AC)	Temperature (c)	Time (min)	Sulfur Content ppm	After Sulfur Content ppm	Removal (%)
0.75	30	90	500	54.3	89.14
0.75	60	60	500	100	80
0.75	90	90	500	46.2	90.76
0.5	30	60	500	87	82.6
1	90	60	500	48	90.4
1	60	90	500	95	81
0.5	90	60	500	76	84.8
0.5	60	90	500	103	79.4
1	30	60	500	57	88.6
0.5	60	30	500	64	87.2
0.75	90	30	500	46.5	90.7
0.75	30	30	500	52.3	89.54
1	60	30	500	53	89.4

Conclusion

This study highlights the potential of utilizing locally sourced materials like pomegranate peel extract for nanoparticle synthesis, offering a cost-effective and environmentally sustainable solution for fuel purification. Further exploration into this method could pave the way for scalable applications in industrial desulfurization processes.

Based on the XRD, FTIR FE-SEM and BET results of NiO nanoparticles with activated carbon, we have substantial evidence that a composite material that retains the characteristic chemical bonding properties of both materials was successfully synthesized. The demonstration of vibration bands associated with Ni-O, the presence of hydroxyl functional groups, and the carbon functionality all distinguish the nature of this material and suggest possible application in catalysis, adsorption and environmental remediation.

The spectral analysis shows a material with significant surface functionality, allowing for high reactivity and adsorption capabilities. These features indicate that NiO-activated carbon composite may be applicable in our case studies from water purification, gas separation, energy storage systems to heterogeneous catalysis. It would be worthwhile to use additional characterization methods to understand the more comprehensive morphological and performance characteristics of this composite material.

Studies using 0.75 grams of NiO-loaded activated carbon (AC) at 90 degrees Celsius for 90 minutes show the best sulfur removal rate of 90.76%. Under reasonable running conditions, these conditions offer a good mix between performance and energy economy. Given the materials and mild conditions applied to prepare the catalyst, the general removal efficiencies achieved are competitive. Future development efforts could target even bettering the catalyst material as well as modifying other parameters to increase general emission control efficiency while preserving the environmental benefits of the catalyst system.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest.

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