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Activated Carbon Materials as Ideal Adsorbents for the Desulfurization of Fossil Fuels

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Abstract

Removal of organosulfur compounds from diesel is of interest from scientific, social, economic, and environmental viewpoints. Production of clean fuel remains a primary goal of the petroleum refining industry. The reduction of sulfur content below stringent ppm levels in diesel fuels becomes increasingly challenging due to the presence of sterically hindered sulfur compounds, such as 4,6-dialkyldibenzothiophenes, which are resistant to removal over conventional supported mixed sulfide hydrodesulfurization catalysts. Consequently, alternative and complementary technologies based on novel approaches, including adsorption, oxidation, and chelation, are being actively explored to target these refractory sulfur species. Any significant breakthrough in desulfurization technology has the potential to positively impact human well-being by improving air quality, reducing sulfur oxide emissions, and contributing to a cleaner environment, thereby supporting efforts to mitigate climate-change-related risks. In this context, a group of activated carbon materials with high specific surface area and pronounced microporosity was systematically characterized using X-ray diffraction (XRD) for their potential application as adsorbents in the desulfurization of straight-run diesel obtained from the Cauvery Basin Refinery (CBR). In addition, the adsorption-based approach offers several advantages over conventional hydroprocessing routes, including operation under milder conditions, lower hydrogen consumption, and reduced capital and operating costs. The structural and textural properties of activated carbons, such as pore size distribution, surface heterogeneity, and crystallinity, play a crucial role in determining their affinity toward sulfur-containing aromatic compounds. Understanding the relationship between adsorbent structure and desulfurization performance is therefore essential for the rational design of efficient sorbents. The present investigation contributes to this objective by providing insights into the suitability of activated carbon materials as promising candidates for deep desulfurization of diesel fuels, particularly for the removal of refractory sulfur compounds that limit the effectiveness of conventional refining technologies.

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INTRODUCTION

A Web of Science search conducted with specific keywords, namely, “adsorptive desulfurization,” yielded hardly 47 results as of 28th January 2026 [1–2]. An order of increase in the results to 298 was observed when the keywords were rephrased as “desulfurization and adsorption.” This number shrinks to only 40 results when publications in the past five years (2025–2021) were scanned through [1–30]. In addition to the transportation sector, the chemical industry also has its own share of

environmental contamination with S-containing compounds [4]. Adsorption-based techniques have been widely adopted for both liquid and gaseous streams contaminated with sulfur. Theoretical methods based on density functional theory (DFT) facilitate the establishment of the structure–property–function relationships of various carbon-based adsorbents and screen out ideal candidates for desulfurization applications based on an understanding of the reaction mechanism [5, 12, 31]. Biobased materials such as nanocellulose have been explored as adsorbents for desulfurization aimed at attaining sustainability development goals such as good health and well-being (SDG 3), clean energy (SDG 13), and climate action (SDG 17) [3–6].

Among the various adsorbents developed for desulfurization of gasoline and diesel, activated carbon remains a low-cost and highly innovative option. The objective of this study was to develop a regenerable activated carbon-based adsorbent to reduce sulfur in straight-run (SR) Cauvery Basin Refinery diesel from approximately 737 ppm to less than 200 ppm. The feed employed was the straight-run feed from Narimanam, Tamil Nadu. Thus, the problem of the exploitation of activated carbon materials as adsorbents for the desulfurization of a medium S-containing straight-run diesel fraction with a sulfur content of 737 ppm, from the Cauvery Basin Refinery, India, has been elucidated [7–11].

MATERIALS AND METHODS

Calgon carbon was purchased from Calgon Carbon (Tianjin) Co. Ltd., China; Adsorbent carbon was purchased from Adsorbent Carbons Pvt. Ltd., Tuticorin, India; IG 18 (18 × 40 mesh), IG 12 Indo (12 × 40 mesh), and IG 8 (8 × 30 mesh) were purchased from German Carbon Limited, India; AC 4 (4 × 8 mesh, coconut-shell-based), AC 6 (6 × 12 mesh), and AC 12 (12 × 30 mesh) were purchased from Active Carbon India Pvt. Ltd., India; Activated carbon materials 60 CTC 12 × 30 and 60 CTC 4 × 8 were purchased from Sud Chemie India Pvt. Ltd.

Tailoring the Surface Properties of Carbon Samples (Adsorbent Carbon (A) and Calgon Carbon (B))

Activation with Concentrated HNO_3

The treatment with HNO_3 changed the surface chemistry of the carbon materials. Such an oxidative treatment results in the formation of oxygen-containing surface functional groups (carbonyl and carboxyl groups). The presence of such surface functional groups, in most cases, enhances the adsorption capacity of carbon materials [12–19]. Two commercial activated carbon materials, adsorbent carbon (A) and calgon carbon (B), were treated with concentrated HNO_3. The wt.%/wt.% ratio of activated carbon to concentrated HNO_3 was 1:5. The oxidative treatment of activated carbon with concentrated HNO_3 was carried out at 60°C for 2 h under reflux conditions in a 2-liter RB flask. The contents were cooled to room temperature, washed with water, and dried at 110°C for 2 h.

Activation Under an Ar Atmosphere

Ar activation involved the thermal activation of nitric-acid-treated carbon materials A and B at 800°C under an Ar atmosphere for 2 h in a quartz tube. After activation, the carbon samples were termed nitric-acid-treated Ar-activated carbon materials [20–27].

Characterization

A Rigaku Miniflex II desktop X-ray diffractometer operating at 30 kV and 15 mA with Cu Kα radiation (λ = 1.5418 Å) at a scan rate of 1°/min was used to record the X-ray diffraction (XRD) patterns of the different materials. A scan range (2θ) of 5–90° was used to produce the diffraction profiles.

The Bragg equation is used to determine the interlayer spacing, as demonstrated below:

$$d = n\lambda/2\mathrm{Sin}\theta$$

where, λ is the wavelength of the radiation (CuKα) used = 0.15405 nm

where θ is the diffraction angle of the peak position.

Table 1. Properties of straight-run diesel from the Cauvery Basin Refinery distillation unit were used in the studies.

Property	Value
Total sulfur content (in ppm)	737
Flash point (°C)	93
Aniline point (°C)	81
Viscosity (at 40°C in cSt)	4.04
Pour point (°C)	+6
Density (g/cc)	0.8553
Diesel index	60
Cetane index	53

The average crystallite sizes along the c-axis (stacking axis), L_c, and along the a-axis, L_a, were calculated using the Scherrer equation.

$$L = K\lambda/B\cos\theta$$

Where, $L = L_c$ or L_a, B is the half-width of the peak in radians, and K is the form factor. The lattice dimension affects the form factor K. L_c and L_a values were calculated using K values of 0.9 and 1.84, respectively.

RESULTS AND DISCUSSION

The physicochemical properties of the Cauvery Basin Refinery diesel are summarized in Table 1.

The essential criteria for an ideal adsorbent are that the adsorbent should be capable of desulfurizing the feed under mild operating conditions, for example, low pressure (5–10 bar) and low temperatures (less than 250°C). In addition, the adsorbent should also be easily regenerated under modest conditions of temperature and pressure using either solvent, hydrogen, or air.

Several commercially available activated carbon materials (IG 18 × 40, IG 12 × 10, IG 8 × 30, AC 4 × 8, AC 6 × 12, AC 12 × 30, Calgon carbon as received, and adsorbent carbon as received) of varying physical and chemical properties were tested as adsorbents for the removal of organosulfur compounds from straight-run Cauvery Basin Refinery diesel [32]. Prior to the actual testing of the activated carbon material for the said application, a variety of activated carbon materials were systematically characterized by XRD to obtain knowledge on the structural features of the material, which will establishment of a structure–property–function relationship.

Structural (Crystallographic) Properties of Carbon Materials

XRD Analysis

XRD patterns of adsorbent carbon as received, adsorbent carbon treated with concentrated HNO_3, and adsorbent carbon treated with concentrated HNO_3 and subsequently activated in an Ar environment are shown in Figure 1.

The aforementioned peaks could be indexed to the (002) and (10) planes of activated carbon, respectively.

The phase structure of the carbon adsorbent remained unaltered upon nitric acid treatment (Figure 1(a) and (b)). However, in the case of adsorbent carbon treated with nitric acid followed by activation in an Ar atmosphere, an additional intense and narrow diffraction peak is observed at $2\theta = 26.7°$ (Figure 1(c)), attributable to the (002) reflection from highly crystalline graphitic carbon. Nitric-acid-treated and Ar-activated adsorbent carbon (Figure 1(c)) is more crystalline than either the as received adsorbent carbon or adsorbent carbon treated with nitric acid alone. Thus, Ar activation at 1073 K improved the crystallinity of the nitric-acid-treated carbon adsorbent.

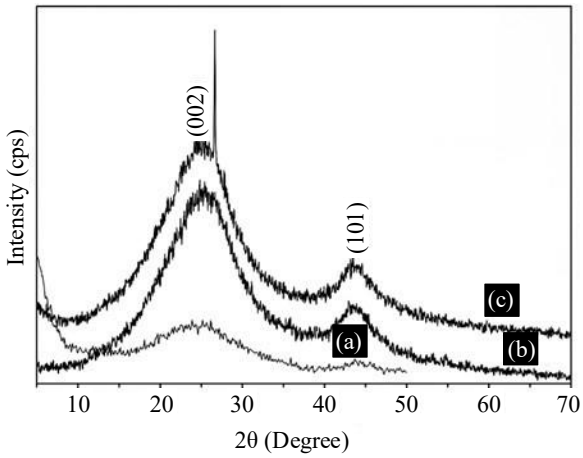

Figure 1. XRD patterns of (a) adsorbent carbon as received, (b) adsorbent carbon treated with HNO_3, and (c) adsorbent carbon treated with HNO_3 and activated with Ar.

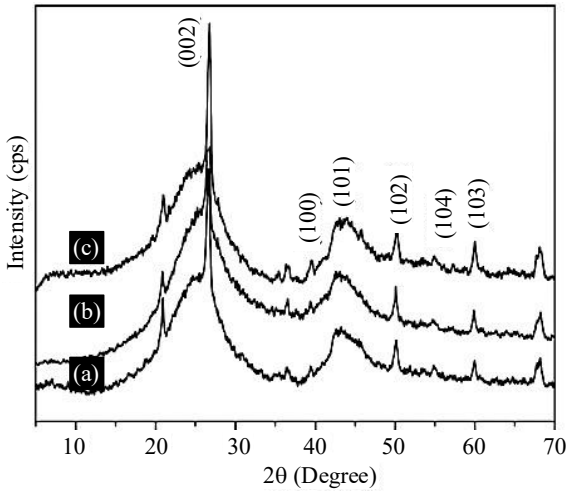

Figure 2. XRD patterns of (a) Calgon carbon as received, (b) Calgon carbon treated with HNO_3, and (c) Calgon carbon treated with HNO_3 followed by Ar activation.

Figure 2 shows the XRD patterns of the as received Calgon carbon, Calgon carbon treated with HNO_3, and Calgon carbon treated with HNO_3 followed by Ar activation in Figure 2(a), (b), and (c).

The diffraction peaks arising from each of these carbon samples were indexed and are typical of a graphitic carbon structure. Neither HNO_3 treatment (Figure 1(b)) nor HNO_3 treatment with subsequent Ar activation (Figure 2(c)) significantly altered the structure of the original Calgon carbon sample (Figure 2(a). Thus, neither HNO_3 treatment nor Ar activation has a significant influence on the phase structure of Calgon carbon.

There is a marked difference in the structural order of adsorbent carbon and Calgon carbon. No diffraction peaks were observed for the adsorbent carbon or its modified forms beyond $2\theta = 50°$ (Figure 1), in sharp contrast to the characteristic diffraction peaks resulting from Calgon and modified Calgon carbon above $2\theta = 50°$ (Figure 2). Thus, Calgon carbon appears to be structurally more ordered than adsorbent carbon [28–32].

As the latest research trend is toward a paradigm shift from fossil fuels to biobased fuels, current research efforts have been devoted to the desulfurization of biogas and other biofuels through the adsorption pathway. The adsorptive desulfurization technology demonstrated herein, surmised to be

based on the use of activated-carbon-based adsorbents, is adaptable for biobased fuels, in addition to the conventional Cauvery Basin Refinery. Integration of the adsorption process with solar energy harvesting accelerates desulfurization [33–35].

CONCLUSION

A new approach of activation, which is a unique combination of nitric acid treatment and Ar activation, of activated carbon-based adsorbents to induce appropriate surface functionality, polarity, phase structure, and pore texture, was employed for the first time. Subsequent Ar activation of nitric-acid-treated carbon adsorbents is required to derive the best oxidative changes in carbon surface chemistry. A breakthrough in the fields of energy and the environment may result from the first-ever use of carbon, carbon, and their customized forms as adsorbents for organosulfur compounds. We will soon share our findings regarding the application of the activated carbon materials created in this work.

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