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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 were 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.

Keywords: Activated carbon, cauvery basin refinery (CBR), conventional refining technologies, desulfurization, diesel

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INTRODUCTION

A Web of Science search conducted with the specific keywords, namely, “adsorptive desulfurization” yields hardly 47 results as on 28th January 2026 [1–2]. An order of increase in the results, to 298, is observed when the keywords were rephrased as “desulfurization and adsorption”. This number shrinks to only 40 results, when the publications in the past 5 years (2025–2021) were scanned through [1–30]. Apart from the transportation sector, the chemical industry too has its own share of contamination of environment with S-containing compounds [4]. Adsorption-based techniques are widely adopted for both liquid and gaseous streams contaminated with sulfur. Theoretical methods based on density functional theory (DFT) facilitates establishing the structure–property–function relationships of various carbon-based adsorbents and to screen out the ideal candidates for the desulfurization application based on the understanding of the reaction mechanism [5, 12, 31]. Biobased materials like nanocellulose were explored as adsorbents for desulfurization aimed at attaining sustainability development goals, like the good health and well-being (SDG 3), clean energy (SDG 13) and climate action (SDG 17) [3–6].

Among various adsorbents developed for the desulfurization of gasoline and diesel, activated carbons remain a low-cost and highly innovative option. The objective of this investigation is to develop a regenerable activated carbon-based adsorbent to reduce sulfur in straight-run (SR) Cauvery Basin Refinery (CBR) diesel from about 737 ppm to less than 200 ppm. The feed employed is the SR feed of the 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 Cauvery Basin Refinery (CBR) India, is taken up and 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 x 40 mesh), IG 12 Indo (12 x 40 mesh) and IG 8 (8 x 30 mesh) were purchased from German Carbon Limited, India; AC 4 (4 x 8 mesh, coconut-shell-based), AC 6 (6 x 12 mesh) and AC 12 (12 x 30 mesh) were purchased from Active Carbon India Pvt. Ltd., India; Activated carbon materials 60 CTC 12 x 30 and 60 CTC 4 x 8 were purchased from Sud Chemie India Pvt. Ltd.

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

Activation with Concentrated HNO_3

Treatment with HNO_3 changes the surface chemistry of carbon materials. Such oxidative treatment results in the formation of oxygen-containing surface functional groups (carbonyl and carboxyl). The presence of such surface functional groups, in most cases, enhances the adsorption capacity of carbon materials [12–19]. Two commercial activated carbon materials, namely, the adsorbent carbon (A) and the 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 refluxing conditions in a 2-liter RB flask. The contents were then cooled to room temperature, washed with water, and dried at 110 °C for 2 h.

Activation Under Ar Atmosphere

Ar activation involved the thermal activation of nitric-acid-treated carbon materials A and B at a temperature of 800 °C under Ar atmosphere for 2 h in a quartz tube. The carbon samples after activation were termed as 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 CuKα radiation ($\lambda = 1.5418$ Å) at a scan rate of 1°/min was used to record XRD patterns of different materials. The scan range (2θ) of 5 to 90° was used to produce the diffraction profiles.

The Bragg Equation is Used to Determine the Interlayer Spacing, As Demonstrated Below:

$d = n\lambda/2\sin\theta$

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

θ is the diffraction angle for the peak position.

Table 1. Properties of SR diesel from CBR distillation unit used in the studies.

Property	Value
Total Sulphur 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 size values along the c-axis (stacking axis), Lc, and along the a-axis, La, were calculated using the Scherrer equation (shown below).

$L = K\lambda/B\cos\theta$ where L = Lc or La

B is the half-width of the peak in radians and K is the form factor. The lattice dimension affects the form factor K. Lc and La values were calculated using K values of 0.9 and 1.84, respectively.

RESULTS AND DISCUSSION

The physicochemical properties of the CBR diesel are summarized in Table 1.

The essential criteria for an ideal adsorbent include: The adsorbent should be capable of desulfurizing the feed at mild operating conditions, e.g., low pressure (5–10 bar) and low temperatures (less than 250 °C). Besides, the adsorbent should also be easily regenerable under modest conditions of temperature and pressure by either solvent or hydrogen or air.

Several commercially available activated carbon materials (IG 18 x 40, IG 12 x 10, IG 8 x 30, AC 4 x 8, AC 6 x 12, AC 12 x 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 SR CBR diesel [32]. Prior to actual testing of the activated carbon material for the said application, a variety of the activated carbon materials were systematically characterized by XRD to obtain knowledge on the structural features of the material which will establish the structure–property–function relationship.

Structural (Crystallographic) Properties of Carbon Materials

XRD Analysis

XRD patterns of adsorbent carbon as received, adsorbent carbon treated with Conc. HNO_3 and adsorbent carbon treated with conc. HNO_3 and subsequently activated in Ar environment were shown in Fig. (1)

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

The phase structure of adsorbent carbon remained unaltered on nitric acid treatment (Figures 1(a) and (b). But in the case of adsorbent carbon treated with nitric acid followed by activation in Ar atmosphere, an additional intense and narrow diffraction peak is seen 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 adsorbent carbon as-received or adsorbent carbon treated with nitric acid alone. Thus, Ar activation at 1073 K improved the crystallinity of nitric-acid-treated adsorbent carbon.

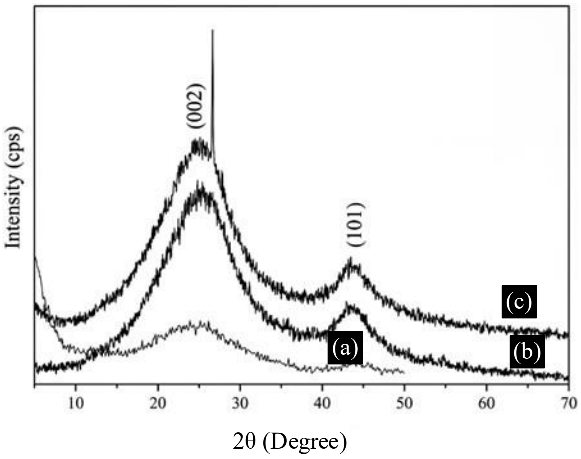

Figure 1. XRD patterns of (a) adsorbent carbon as-received, (b) adsorbent carbon treated with HNO_3 and (c) adsorbent carbon treated with HNO_3 and activated with Ar.

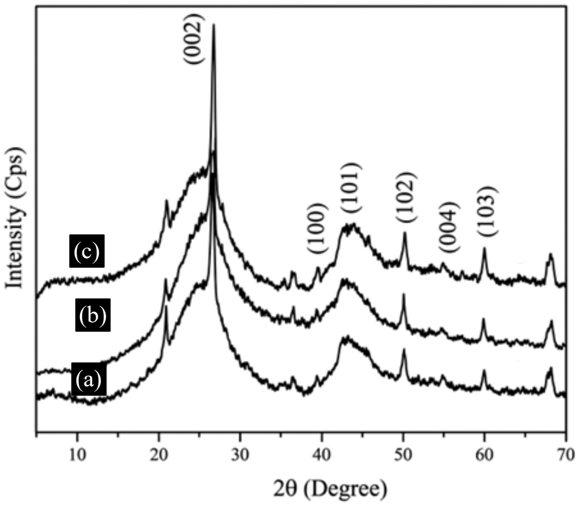

Figure 2. XRD patterns of (a) Calgon carbon as-received, (b) Calgon carbon treated with HNO_3 and (c) Calgon carbon treated with HNO_3 followed by Ar activation.

Figure 2 displays the X-ray diffraction patterns of Calgon carbon as-received, Calgon carbon treated with HNO_3, and Calgon carbon treated with HNO_3 followed by Ar activation in Figure 2(a), (b), and (c) in that order.

The diffraction peaks arising from each of these carbon samples are all indexed and are typical of 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 much influence on the phase structure of Calgon carbon.

There is a marked difference in the structural order between adsorbent carbon and Calgon carbon. No diffraction peaks resulted from 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 to biobased fuels, current research efforts are devoted toward the desulfurization of biogas and other biofuels through the adsorption pathway. The adsorptive desulfurization technology demonstrated herein, surmised based on the use of activated-carbon-based adsorbents, is adaptable for biobased fuels also apart from the conventional Cauvery Basin Refinery (CBR). Integration of adsorption process with solar energy harvesting will accelerate the desulfurization process [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 is employed for the first time. It is shown that subsequent Ar activation of nitric-acid-treated carbon adsorbents is required to derive the best out of oxidative change of carbon surface chemistry. A breakthrough in the fields of energy and the environment may result from the first-ever use of adsorbent carbon, Calgon carbon, and their customized forms as adsorbents for organosulfur compounds. We will soon share the findings about the application of the activated carbon materials created in this work.

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