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Cerium hexaboride catalyst synthesized via microwave-assisted synthesis for aerobic oxidative desulfurization

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Abstract

In this study, the catalytic performance of CeB_6, synthesized by a rapid microwave-assisted method, was investigated in the aerobic oxidative desulfurization of dibenzothiophene (DBT), a representative model refractory sulfur compound. Structural characterization confirmed the formation of phase-pure CeB_6 with well-developed crystalline morphology. The CeB_6 catalyst demonstrated high conversion (up to 95%) at a relatively low temperature of 150 °C using only air as an oxidant. These findings provide new insights into the redox functionality of lanthanide hexaborides and their potential as a catalyst for oxidative desulfurization processes.

Keywords: cerium hexaboride; lanthanide; desulfurization; catalyst; microwave synthesis

1. Introduction

Lanthanide hexaborides (LnB_6) exhibit a wide range of technologically significant properties. They represent a distinct group of refractory materials combining metallic and ceramic characteristics – high hardness, high melting points, electrical conductivity, and remarkable chemical stability.

Magnetic[1,2] and electronic behaviors of LnB_6 compounds (notably CeB_6, PrB_6, NdB_6, EuB_6, and GdB_6) have been extensively studied due to complex *4f–2p* interactions, leading to phenomena such as Kondo lattice effects, heavy-fermion magnetism, and mixed valence states[3,4]. SmB_6, in particular, is a mixed-valent semiconductor often regarded as a potential topological Kondo insulator.[5,6]

Lanthanide hexaborides find use in various electronic and optical applications. LaB_6 and CeB_6 are classical thermionic electron emitters with low work functions and low vapor pressures,[7–9] widely employed in electron microscopy. Surface plasmon resonance (SPR) in nanostructured LaB_6 and CeB_6 has enabled their application in near-infrared (NIR) light shielding, solar absorber coating, solar-thermal conversion,[10–13] and photocatalysis.[14] LaB_6-based plasmonic materials also demonstrated efficiency in photothermal therapy.[15,16]

From an energy perspective, several lanthanide hexaborides have been proposed as anode materials for lithium-ion batteries (e.g., CeB_6), exhibiting high electrical conductivity and stability during cycling.[17] They have also been incorporated into composite coatings for mechanical reinforcement and arc-welding performance enhancement, particularly LaB_6–Ni–Cu systems.[18] Moreover, CeB_6 and GdB_6 have shown potential for environmental catalysis, including efficient removal of azo and cationic dyes from aqueous media.[19,20]

In heterogeneous catalysis, the boron sublattice in LnB_6 provides a high density of delocalized electrons, enabling charge transfer to adsorbed species and promoting bond activation. Recent studies demonstrated that CeB_6, LaB_6, NdB_6, and EuB_6 can enhance hydrogenation/dehydrogenation reactions and hydrogen storage performance due to their electronic conductivity and resistance to oxidation.[21,22] Additionally, the ability of mixed valence states Ln^{3+}/Ln^{4+} along with electron-rich surface can facilitate oxidation or reduction of organic substrates.

Despite these promising characteristics, the catalytic potential of CeB_6 remains largely unexplored in catalysis, especially in desulfurization chemistry. Deep removal of sulfur from fuel fractions is critical for meeting environmental regulations and preventing sulfur oxides emissions. Widespread conventional hydrodesulfurization (HDS) processes demand high temperature and hydrogen pressure. Among other methods, oxidative desulfurization (ODS) using air or peroxides as mild oxidants has emerged as a promising alternative.[23,24]

Commonly, lanthanide hexaborides have been synthesized by high-temperature solid-state reactions, such as borothermal,[25–27] carbothermal,[28] boron carbide,[28–32] and metallothermic[33–35] reductions. They require high-temperature heating in vacuum or inert atmosphere. Some works use mechanical activation.[36–38] However, these processes are energy-intensive and often produce coarse particles or boron-deficient phases. Other methods include molten-salt synthesis,[39–41] gas phase mediated techniques CVD and PVD,[42,43] self-propagating high-temperature synthesis (SHS),[44,45] spark-plasma sintering (SPS).[27,46] Recently, a rapid microwave-assisted solid-state synthesis of rare-earth hexaborides was reported by us enabling pure phase LnB_6 formation within minutes at markedly lower apparent temperatures, yielding fine, homogeneous powders.[47]

In this study, the catalytic performance of CeB_6 is investigated in the oxidative desulfurization of dibenzothiophene (DBT), a representative model compound of refractory sulfur species. Structural and surface characteristics as well as catalytic performance are discussed.

2. Materials and synthesis

As starting reagents CeO_2 (99.5%, p.a., Carl ROTH GmbH+Co.KG) and B (99.5%, average particle size 2 μm, Sigma-Aldrich) were used. Stoichiometric amounts of raw materials were weighed according to the equation $3\ CeO_2 + 22\ B = 3\ CeB_6 + 2\ B_2O_3$, and mixed in a glass beaker using a magnetic stirrer for 30 min, then homogenized in an agate mortar. The mixture was then placed in a continuous flow quartz reactor and purged by helium flow (25 mL min^{-1}). Then it was irradiated in a 2.5 GHz, 900 W household microwave oven for about two minutes until incandescence and kept for over 10 min to complete the reaction. The temperature in the incandescent region reached approximately 900–1050 °C measured by an optical pyrometer (Dostmann HT-1800). The synthesis product was washed several times with 5% NaOH solution and deionized water to remove boron oxide. Further, it was dried in an oven at 105 °C for 12 h.

3. Results and Discussion

3.1. Characterization

The X-ray diffraction (XRD) pattern was recorded using a Rigaku Rotaflex D/max-RC instrument equipped with $CuK\alpha_1$ radiation ($\lambda = 0.154$ nm). Data were collected over the 2θ range of 10–80°. Phase identification was performed using MDI/JADE by comparison with reference patterns from the JCPDS database.

Figure 1 shows the XRD pattern of the synthesized cerium hexaboride powder. The diffraction data confirm that the powder is mainly composed of the CeB_6 phase, and are in good agreement with the corresponding reference pattern (PDF # 38-1455). No detectable secondary phases were observed. Diffraction peaks are sharp and well resolved, with full width at half maximum (FWHM) of about

0.2°. Such narrow peaks indicate a high degree of crystallinity and relatively large particles exceeding several tens of nanometers.

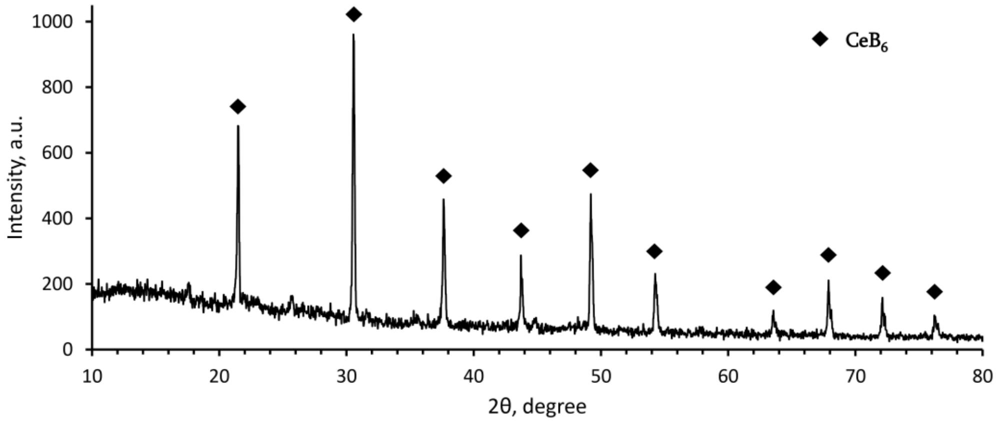

Figure 1. XRD pattern of synthesized CeB_6.

The morphology and microstructure of the sample were examined by scanning electron microscopy (SEM) using a Carl Zeiss NVision 40 microscope equipped with an Oxford Instruments X-Max energy-dispersive X-ray spectroscopy (EDS) system (80 mm^2).

The SEM image of the synthesized CeB_6 is shown in Figure 2. Well-developed crystal growth is clearly observed, and the particles have well-formed cubic morphology. The particle size ranged approximately from 1 to 10 μm.

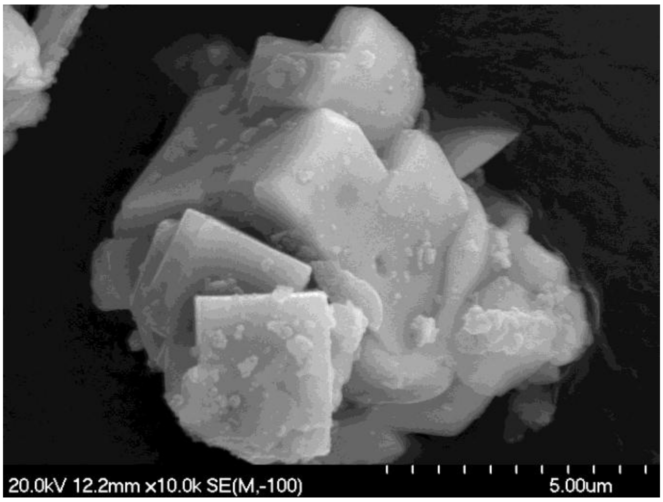

Figure 2. SEM image of synthesized CeB_6.

The surface chemical composition of the sample was characterized using EDS analysis. The EDS elemental mapping reveals a homogeneous distribution of cerium and boron across the surface. The results are consistent with the XRD data (Figure 1), which indicate the formation of pure single-phase hexaboride. The XRD, SEM and EDS data are in good agreement and confirm the successful formation of phase-pure cerium hexaboride.

3.2. Catalytic test

The oxidative desulfurization (ODS) activity of the synthesized CeB_6 catalyst was evaluated using a model reaction mixture of dibenzothiophene (DBT). A 30 mL portion of the model fuel consisting of dibenzothiophene solution in decalin with initial sulfur content of 500 ppm was placed in a roundbottom flask equipped with a reflux condenser. Various amounts of CeB_6 catalyst (6–104 mg, equivalent to 0.022–0.387 wt%) were added and the mixture was heated to a desired temperature with continuous stirring and air flow bubbling (6 L h^{-1}) as an oxidant. Reaction mixtures were analyzed by gas chromatography with flame ionization detection (GC-FID) using a Kristal 2000 M gas chromatograph equipped with a capillary column (ZB-1 stationary phase, 30 m × 0.32 mm × 0.25 µm). Conversion was calculated as $X = (C_0 - C_t)/C_0 \times 100\%$, where C_0 and C_t are the initial substrate concentration and the concentration after reaction, respectively. DBT and the other sulfur-containing substrates were quantified using the absolute calibration method. Figure 3 illustrates the influence of the catalyst amount on DBT conversion at 130 °C for one hour. The conversion increased almost linearly up to 26 mg catalyst loading, however further catalyst additions resulted in a decrease in efficiency gain, indicating the onset of catalytic saturation.

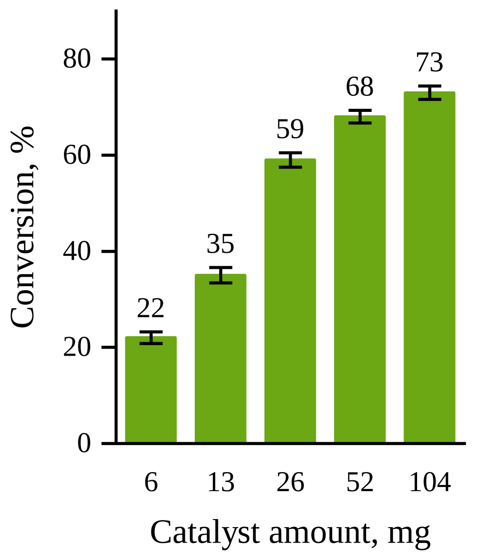

Figure 3. Effect of catalyst dosage on DBT conversion at 130 °C. Values are means of six experimental replicates (n = 6); error bars represent ±1 SD.

Under the same experimental conditions, 26 mg of cerium hexaboride exhibited substrate-dependent activity toward organosulfur compounds having the same initial sulfur content of 500 ppm (Table 1).

Table 1. Conversion of various sulfur substrates (initial sulfur content: 500 ppm).

Substrate	Conversion, %
Methylphenyl sulfide	98
Dibutyl sulfide	87
Benzothiophene	32
Dibenzothiophene	59
Methyldibenzothiophene (positional isomer not specified)	47

To elucidate the role of thermal activation, a series of experiments were conducted in the range 90–150 °C, in 10 °C increments. As can be seen from Figure 4, the catalytic activity was observed starting at 100 °C, and the conversion increased steadily with temperature, reaching 95% at 150 °C.

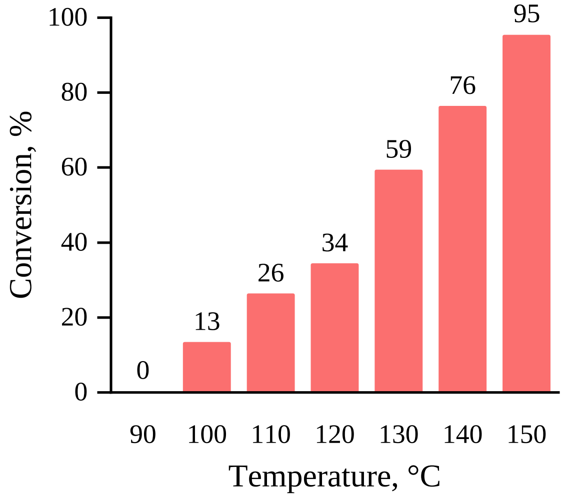

Figure 4. Temperature dependence of DBT conversion (reaction time: 1 h; initial sulfur: 500 ppm; catalyst mass: 26 mg; air flow: 6 L h^{-1}).

4. Conclusion

Pure-phase cerium hexaboride was synthesized by microwave irradiation within a short period. The synthesized CeB_6 demonstrated high catalytic activity in the aerobic oxidative desulfurization of dibenzothiophene, achieving high conversion under mild conditions. These results demonstrate the potential of CeB_6 as a catalyst for oxidative desulfurization, although further investigation is required to establish the structural and electronic factors governing its activity.

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ՀԱՅԱՍՏԱՆԻ ՀԱՆՐԱՊԵՏՈՒԹՅԱՆ ԳԻՏՈՒԹՅՈՒՆՆԵՐԻ ԱԶԳԱՅԻՆ ԱԿԱԴԵՄԻԱ

ԱՄՓՈՓԱԳԻՐ
Միկրոալիքային ճառագայթմամբ սինթեզված ցերիումի հեքսաբորիդ կատալիզատոր՝ աէրոբ օքսիդատիվ ծծմբազերծման համար

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Աշխատանքում ուսումնասիրվել է արագ միկրոալիքային մեթոդով սինթեզված ցերիումի հեքսաբորիդի (CeB_6) կատալիտիկ արդյունավետությունը դիբենզոթիոֆենի (DBT) աէրոբ օքսիդատիվ ծծմբազերծման գործընթացում, որը հանդիսանում է դժվար օքսիդացվող ծծմբային միացությունների մոդելային ներկայացուցիչ: Կառուցվածքային բնութագրումը հաստատել է բարձր բյուրեղայնությամբ CeB_6-ի մաքուր ֆազի առաջացումը: Սինթեզված CeB_6 կատալիզատորը ցուցաբերել է բարձր ակտիվություն՝ ապահովելով մինչև 95% փոխակերպում համեմատաբար ցածր ջերմաստիճանում (150 °C)՝ օգտագործելով միայն օդը որպես օքսիդացնող միջավայր: Ստացված արդյունքները նոր պատկերացում են տալիս լանթանիդային հեքսաբորիդների օքսիդավերականգնող ֆունկցիոնալության վերաբերյալ և ընդգծում դրանց ներուժը՝ որպես արդյունավետ կատալիզատորներ օքսիդատիվ ծծմբազերծման գործընթացներում:

Բանալի բառեր՝ ցերիումի հեքսաբորիդ, լանթանիդ, ծծմբազերծում, կատալիզատոր, միկրոալիքային սինթեզ