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From Boron to Borophene: Experimental Insights into 2D Material Synthesis and Characterization

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Borophene, a two-dimensional (2D) allotrope of boron, has emerged as a promising material due to its exceptional electronic, mechanical, and thermal properties. In this study, we report a facile and effective synthesis of borophene via an ultrasonic exfoliation process, enabling the formation of few-layer boron nanosheets under controlled conditions. The influence of exfoliation parameters on the yield and stability of the resulting borophene was systematically investigated. Comprehensive characterization was carried out to evaluate the morphological, compositional, structural, and thermal properties of the synthesized material. Scanning Electron Microscopy (SEM) revealed sheet-like structures with layered morphology, while Energy Dispersive X-ray Spectroscopy (EDX) confirmed the elemental composition and purity of boron. X-ray Diffraction (XRD) analysis indicated characteristic crystalline features associated with borophene phases. Fourier Transform Infrared Spectroscopy (FTIR) provided insights into bonding characteristics and surface functional groups, and Thermogravimetric Analysis (TGA) demonstrated the thermal stability of the material. The combined results confirm the successful synthesis of borophene and highlight the effectiveness of ultrasonic exfoliation as a scalable method for producing 2D boron materials. This study provides important insights into the structure-property relationships of borophene and supports its potential application in advanced nanomaterials and energy-related technologies.

Keywords: 2D Materials, boron, borophene, ultrasonic exfoliation

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Two-dimensional (2D) materials have attracted significant scientific interest due to their unique physical and chemical properties, which differ markedly from their bulk counterparts [1]. Following the discovery of graphene, research has expanded toward other elemental 2D materials, including borophene, a monolayer allotrope of boron. Borophene exhibits remarkable properties such as high anisotropy, metallic conductivity, mechanical flexibility, and potential for energy storage applications [2]. However, unlike graphene, borophene is more complex due to boron's electron deficiency, resulting in diverse polymorphic structures and challenging synthesis pathways. Despite its promising properties, the experimental realization of borophene remains limited and challenging. Conventional synthesis techniques, such as molecular beam epitaxy, often require ultra-high vacuum conditions and specific substrates, making them costly and less scalable [3]. Additionally, achieving stable, high-quality borophene with controlled morphology and minimal defects remains a significant challenge. There is a clear need for simpler, cost-effective, and scalable synthesis methods, alongside comprehensive characterization to better understand the structure-property relationships of borophene [4].

In this study, borophene was synthesized via ultrasonic exfoliation, offering a relatively simple and scalable alternative to traditional methods. Bulk boron material was subjected to controlled ultrasonic treatment to produce few-layer boron nanosheets. The synthesized material was then systematically characterized using a range of analytical techniques. Scanning Electron Microscopy (SEM) was employed to investigate surface morphology, while Energy Dispersive X-ray Spectroscopy (EDX) confirmed elemental composition. Structural properties were analyzed using X-ray Diffraction (XRD), and bonding characteristics were examined through Fourier Transform Infrared Spectroscopy (FTIR). Thermogravimetric Analysis (TGA) was further conducted to evaluate thermal stability. The experimental findings indicate the successful formation of borophene-like nanosheets with layered and sheet-like morphology, as observed through SEM analysis. EDX results confirmed the presence of boron as the primary constituent with minimal impurities. XRD patterns revealed crystalline features consistent with reported borophene structures, while FTIR analysis provided evidence of bonding characteristics and possible

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surface functionalization. TGA results demonstrated that the synthesized material possesses appreciable thermal stability under controlled conditions. Overall, the results highlight the effectiveness of ultrasonic exfoliation in producing borophene and emphasize the relationship between synthesis conditions and material properties.

Future work should focus on optimizing the ultrasonic exfoliation parameters to improve the yield, uniformity, and structural integrity of borophene sheets. Advanced characterization techniques, such as transmission electron microscopy (TEM) and atomic force microscopy (AFM), could provide deeper insights into thickness and atomic structure. Additionally, surface modification and functionalization strategies may enhance the stability of borophene under ambient conditions. Further exploration of its application potential in electronics, catalysis, and energy storage systems will be essential to realize the capabilities of this emerging 2D material.

EXPERIMENTAL SECTION

Material

Boron (B) powder (CAS No. 7440-42-8, 99% purity) and isopropyl alcohol (IPA, $(CH_3)_2CHOH$) (CAS No. 67-63-0, ≥ 99%) were purchased from Merck, Germany. All chemicals were of analytical grade and used without further purification.

Method

The overall procedure is outlined in **Fig. 1.** First, a boron suspension was prepared in IPA at a concentration of 0.6 mg/mL and sonicated in an ultrasonic bath for 15 minutes. To control temperature during further processing, the suspension was then placed in an ice bath. Subsequently, probe ultrasonication was carried out in pulse mode (5 s on/off cycles) for a total duration of 6 hours. The temperature of the ice bath was carefully monitored throughout the process, and ice was replenished as needed to maintain stable conditions and minimize solvent evaporation. The entire sonication procedure was performed in a sealed environment to reduce potential contamination. After sonication, the upper dispersion was collected and centrifuged at 4200 rpm for 7 minutes. This step was repeated three times to remove any remaining bulk boron and aggregated particles. Due to the low yield of the exfoliated material, the resulting borophene was finally dried at a mild temperature of 45 °C.

Characterizations

The microstructure and surface morphology of the borophene were characterized using scanning electron microscopy (SEM, Tescan Vega 3, Czech Republic). Elemental composition and spatial distribution were investigated by energy-dispersive X-ray spectroscopy (EDX, Oxford Instruments). Phase composition and crystallographic structure were identified using X-ray diffraction (XRD). Measurements were carried out on a D/teX Ultra2 diffractometer (Rigaku, Japan) with Cu Kα radiation (λ = 0.154 nm), scanning over a 2θ range of 0°-90° at a rate of 2° per minute under texture mode.

Figure 1. Schematic of the liquid-phase sonochemical exfoliation process: boron powder (B) is dispersed in IPA, treated via probe ultrasonication, and centrifuged to separate unexfoliated material from the borophene nanosheet supernatant to extract exfoliated borophene.

RESULTS AND DISCUSSION

Morphological Analysis

The surface morphology of the synthesized borophene obtained via ultrasonic exfoliation was analyzed using SEM **Fig. 2 (a-b)** and FESEM **Fig. 2 (c-d)**, as presented. At lower magnification in **(Fig. 2a)**, SEM analysis reveals that the material exhibits a highly agglomerated structure composed of irregularly distributed particles, indicating partial exfoliation of bulk boron with clusters containing both unexfoliated material and emerging layered features. As the magnification increases **(Fig. 2b)**, flake-like and plate-like structures become more prominent, suggesting the initiation of layer separation due to ultrasonic cavitation. The presence of voids and loosely packed regions further supports the breakdown of bulk boron into thinner sheets. In **(Fig. 2c),** which is analysed by using advance instrument (FESEM) a higher density of thin, irregular nanosheets and fragmented flakes is observed, revealing a more layered morphology, although the coexistence of micro-sized plates and smaller particles indicates non-uniform exfoliation. At the highest magnification **(Fig. 2d)**, images clearly show elongated sheet-like and ribbon-like nanostructures with relatively smooth surfaces and sharp edges, which are characteristic of few-layer borophene. These features confirm effective delamination into quasi-two-dimensional structures. The progression from agglomerated particles to well-defined nanosheets demonstrates the success of the ultrasonic exfoliation process, although some degree of agglomeration and size variation suggests the need for further optimization to achieve more uniform borophene layers.

The EDX spectrum and elemental mapping of the synthesized borophene (**Fig. 3a-c**) confirm the elemental composition and purity of the sample. The EDX results indicate ~98% boron and ~1.97% oxygen, demonstrating high boron purity with minor oxidation. Elemental mapping also shows the presence of carbon, which is attributed to the carbon tape used during sample preparation; therefore, the carbon signal is excluded from the material composition analysis.

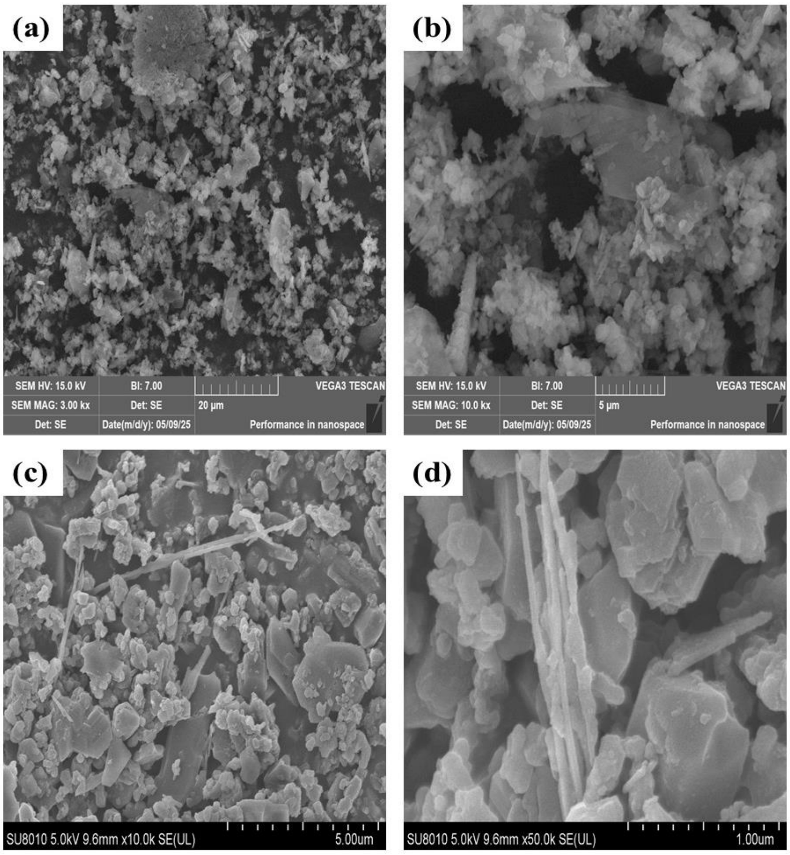

Figure 2. (a-b) SEM and (c-d) FESEM images of borophene synthesized via ultrasonic exfoliation: (a) agglomerated bulk particles; (b) partially exfoliated flake-like structures; (c) layered nanosheets with fragmented particles; and (d) sheet-like and ribbon-like borophene structures at higher magnification.

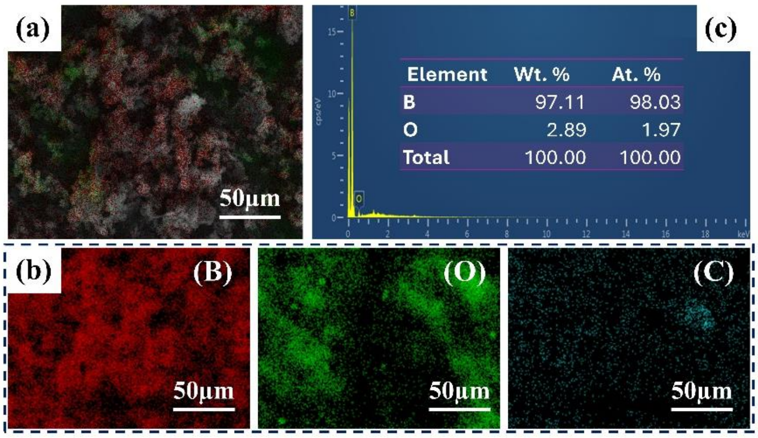

Figure 3. (a-b) represent the elemental mapping of the synthesized borophene, and (c) represents EDX analysis of the materials.

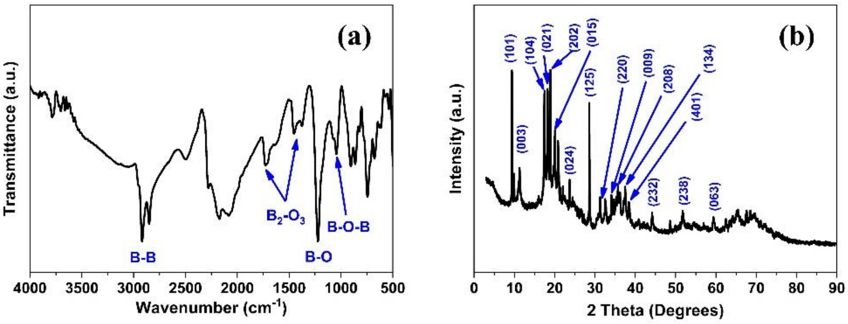

Figure 4. (a) FTIR spectrum of synthesized borophene. (b) XRD pattern with preserved crystallinity after exfoliation.

Structural and Crystallographic Analysis

The structural characteristics of the synthesized borophene were analyzed using FTIR and XRD, as shown in **Fig. 4(a-b)**. The FTIR spectrum (**Fig. 4a**) exhibits characteristic absorption bands corresponding to B-B (3023 $cm^{-1}$and 600 cm^{-1}) stretching vibrations, confirming the formation of boron frameworks [5]. Additional peaks assigned to B-O (1793 cm^{-1}) and B-O-B (1123 cm^{-1}) bonds indicate slight surface oxidation, along with minor B_2O_3 (1765 cm^{-1} and 1487 cm^{-1}) related features, which are commonly observed due to the high reactivity of borophene under ambient conditions. In the spectra few others peak (2200 cm^{-1}-2300 cm^{-1}) also observed which represent some trace contamination specifically the peak 897 cm^{-1} belongs to B-Al bonding. The XRD pattern (**Fig. 4b**) depicts the crystalline nature of the material and reveals a B_{12} boron crystal structure. The diffraction peaks are indexed to the β-rhombohedral boron phase (JCPDS 00-031-0207), belonging to the $R\bar{3}m$ (No. 166) space group. This finding is consistent with previously reported studies indicating that the applied exfoliation method effectively preserves the crystallinity of the bulk boron precursor while enabling the formation of borophene-like nanosheets [6].

Light Absorption and Degradation Analysis

Thermal stability of the synthesized borophene was evaluated using TGA, as presented in **Fig. 5(a)**. The curve shows relatively stable behavior at lower temperatures as we run the sample 35-900 °C, lowed by slight weight variations at higher peratures (after 600 °C), which can be attributed to oxidation or structural changes. Overall, the limited weight loss indicates good thermal stability of the material under the tested conditions.

The UV-Vis spectrum shown in **Figure 5(b)** demonstrates the optical response of the synthesized borophene. A relatively smooth absorption profile with slight variation in intensity suggests consistent electronic behavior across the measured wavelength range [7]. This indicates the potential of borophene for optoelectronic applications, although no sharp absorption edge is observed, which is typical for metallic or semi-metallic 2D materials.

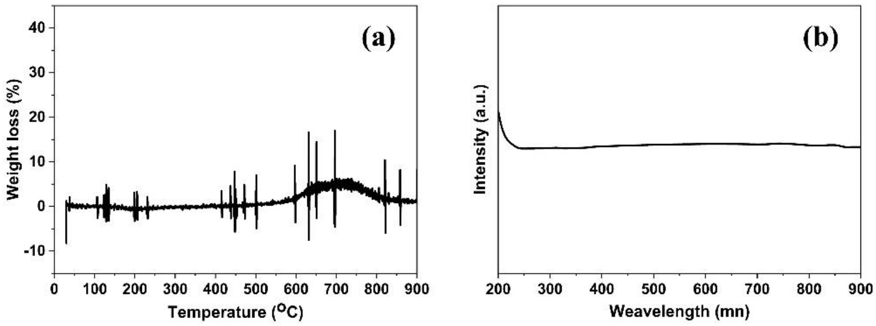

Figure 5. (a) thermal degradation (TGA) spectrum of synthesized borophene, (b) light absorption (UV-Vis) pattern of synthesized borophene.

CONCLUSION

In this work, borophene was successfully synthesized via an ultrasonic exfoliation process, demonstrating a simple, cost-effective, and potentially scalable approach for producing two-dimensional boron materials. The study confirmed that controlled ultrasonic treatment can effectively generate few-layer borophene-like nanosheets from bulk boron. Comprehensive characterization using SEM, EDX, FESEM, and XRD validated the morphological, compositional, structural, and thermal properties of the synthesized material. The results revealed layered sheet-like structures, high boron purity, identifiable crystalline features, and satisfactory thermal stability, indicating the successful formation of borophene. Overall, this study provides valuable insights into an alternative synthesis route for borophene and establishes a foundation for further optimization and application of 2D boron materials. The findings contribute to ongoing efforts toward scalable production and broaden the potential for borophene in advanced technological applications.

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