

Green synthesis of high-purity hexagonal boron nitride nanoparticles

Odiljon Abdurakhmonov^a, Mastura Aripova^a, Farrukh Erkinov^a,
Sherzod Abdurakhmonov^a, Utkirjon Sharopov^{b,*}, Mukhtorjon Karimov^c,
Muzaffar Kurbanov^c, Dilmurod Saidov^d, Zbigniew Pędzich^e, Dawid Kozien^e,
Tonni Agustiono Kurniawan^f, Ekaterina Bondar^g

^a Tashkent Institute of Chemical Technology, 100011, Tashkent, Uzbekistan

^b Physical-Technical Institute, Uzbekistan Academy of Sciences, 100084, Tashkent, Uzbekistan

^c Urgench State University, 220100, Urgench, Uzbekistan

^d Urgench Ranch University of Technology, 220100, Urgench, Uzbekistan

^e AGH - University of Krakow, 30-059, Krakow, Poland

^f College of Ecology and Environment, Xiamen University, Xiamen, 361102, Fujian, China

^g Institute of Physics and Technology, Satbayev University, 050013, Almaty, Kazakhstan

ARTICLE INFO

Handling Editor: Prof. L.G. Hultman

Keywords:

Nanoparticles
Hexagonal structure
Boron nitride
h-BN
Chemical synthesis
Heat treatment

ABSTRACT

Traditional methods, such as chemical vapor deposition (CVD), often require toxic and flammable organic reagents or result in a high carbon footprint, raising both health and environmental concerns. In this paper, we present an improved method for synthesizing hexagonal boron nitride (h-BN) nanoparticles using affordable and widely available precursors, boric acid, and urea. Our approach modifies the conventional techniques by introducing a two-step thermal treatment process, with nitrogen serving as an inert atmosphere and hydrogen as a diffusing agent. The resulting h-BN nanoparticles exhibit remarkable uniformity and high crystallinity. Characterization through scanning and transmission electron microscopy (SEM, TEM), high-resolution TEM (HR-TEM), selected-area electron diffraction (SAED), Fourier-transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD) confirmed the formation of amorphous nanoparticles with an average size of 10 nm during the initial heat treatment at 600 °C under a nitrogen atmosphere. Further heat treatment at 1000 °C in a nitrogen-hydrogen gas mixture led to the crystallization of highly uniform h-BN nanoparticles, reducing their size to approximately 6 nm. This method ensures high morphological homogeneity and product purity, making it effective for the large-scale production of h-BN nanoparticles.

1. Introduction

Boron nitride (BN) is a binary compound of boron and nitrogen, crucial for modern technologies. It exists in various allotropic forms, including amorphous (a-BN), hexagonal (h-BN), wurtzite (w-BN), cubic (c-BN or borazon), and turbostratic (t-BN) structures [1]. Hexagonal boron nitride (h-BN), akin to “white graphite,” shares a honeycomb hexagonal lattice with graphene [2]. h-BN exhibits high thermal [3] conductivity [4], mechanical strength [5], chemical inertness [6], thermal stability [7], and dielectric properties [8]. Applications range from insulators and steel mold rupture rings to solid lubricants [9], catalyst supports [10], and composite films [11]. Additionally, h-BN serves as an intermediate in cubic BN preparation [12]. Boron nitride

nanoparticles also have a wide range of applications in industries such as electronics [13,14], lubricants [15], coatings [16], hydrogen storage [17–20] and biomedical devices [21] due to their unique properties like high thermal stability, electrical insulation, and chemical inertness. A sustainable synthesis route can help meet the growing demand for these advanced materials in a more environmentally friendly manner [22].

Recent studies explore diverse morphologies of boron nitride, such as one-dimensional nanostructures [23], hollow spheres [24], nanocapsules [25], nanocages [26]. Methods for h-BN nanoparticle synthesis includes electric arc discharge [27], chemical vapor deposition (CVD) [28,29], sol-gel synthesis [30], mechanochemistry [31], electrodeposition [32], and laser ablation [33]. Traditional methods for synthesizing BNNPs often involve toxic chemicals and harsh reaction conditions [34].

* Corresponding author. “Solar thermal and power plants” laboratory, Physical-Technical Institute, Chingiz Aitmatov St., 2, 100084, Tashkent, Uzbekistan.
E-mail address: utkirstar@gmail.com (U. Sharopov).

<https://doi.org/10.1016/j.vacuum.2025.114386>

Received 16 January 2025; Received in revised form 30 April 2025; Accepted 4 May 2025

Available online 5 May 2025

0042-207X/© 2025 Elsevier Ltd. All rights are reserved, including those for text and data mining, AI training, and similar technologies.

Existing CVD methods rely on flammable and toxic organic reagents, posing risks to health and the environment [35]. Overcoming these limitations remains a critical goal for achieving safe, nanometer-sized h-BN particles [36]. By developing a sustainable route, this work aims to minimize the use of hazardous substances, making the production process safer for both the environment and human health [37].

A popular method for h-BN nanoparticle synthesis involves chemical vapor deposition (CVD) [38]. The CVD process includes heating a boron-nitrogen solution and nitrogen gas in a thermal reactor with ammonia and argon gases. This forms a reaction intermediate, which crystallizes to create a precursor for boron nitride nanoparticles on a metal substrate. However, this method relies on flammable and toxic organic reagents or have a high carbon footprint, posing risks to health and the environment. Traditional methods often involve expensive precursors and energy-intensive processes. While the work likely involves standard characterization techniques (SEM, TEM, XRD, FTIR), more in-depth studies on particle size distribution, morphology, crystallinity, and surface chemistry might be necessary to fully understand the nanoparticles' properties. Correlating the synthesis parameters with the resulting nanoparticle properties is crucial for tailoring the material for specific applications. Understanding how the choice of starting materials and process conditions influence the final product's characteristics is a key knowledge gap.

Recent advancements in boron nitride (BN) synthesis have explored various methods beyond conventional techniques, such as chemical vapor deposition (CVD) and thermal treatments. Ionothermal procedures and crystallization methods have emerged as innovative approaches, each offering unique advantages and limitations. The ionothermal synthesis method leverages ionic liquids as both solvents and reaction media, providing a low-temperature route for BN synthesis. This approach allows for the precise control of particle morphology and size distribution due to the unique properties of ionic liquids, such as low volatility and high thermal stability. For instance, H. Chen et al. reported the successful synthesis of h-BN nanostructures using an ionothermal method, demonstrating its potential for producing high-quality BN with tailored properties [39]. However, the use of ionic liquids can lead to higher production costs and potential challenges in large-scale applications. The crystallization method, on the other hand, offers a straightforward pathway for BN production through controlled nucleation and growth processes. H. Chen et al. demonstrated a novel crystallization technique to obtain BN nanosheets with exceptional structural uniformity [40]. While this method excels in achieving high crystallinity and purity, it often requires specialized conditions and prolonged reaction times, which may limit its scalability. Compared to these methods, the two-step thermal treatment process described in this work offers several advantages, including simplicity, cost-effectiveness, and scalability. By utilizing affordable and safe precursors such as boric acid and urea, along with an inert nitrogen atmosphere and hydrogen as a diffusing gas, this method minimizes environmental impact and ensures high product purity. Moreover, the controlled particle size and uniform morphology achieved in this process make it highly suitable for industrial applications. The comparative analysis highlights that, while ionothermal and crystallization methods contribute to the diverse toolkit for BN synthesis, the developed two-step thermal treatment method combines environmental safety, cost efficiency, and scalability, making it a robust alternative for large-scale production of h-BN nanoparticles.

Therefore, synthesis of h-BN nanoparticles at lower temperatures, reduction of synthesis time, and the use of available and safe reagents as starting materials with particle sizes in nanometer remains an urgent task.

The surface physicochemical properties of boron compounds play a crucial role in determining their applications in modern materials. According to the study by T. Hamieh et al. [41], key parameters such as surface free energy, polarity, and acid-base interactions significantly influence the behavior of boron-based materials. In the context of h-BN

synthesis, surface thermodynamic properties directly affect particle dispersion stability, phase interactions, and the control of crystallization. Understanding these properties enables optimization of h-BN nanoparticle morphology and structural uniformity, which are critical for their targeted applications.

To address the knowledge gaps, this study develops a more cost-effective synthesis route by using common starting materials and employs boric acid and urea as safer starting materials. To reduce the environmental impact of the production process, this work uses a two-stage heat treatment with nitrogen as an inert atmosphere and hydrogen as a diffusing gas. This results in uniform h-BN nanoparticles with sizes between 2 and 10 nm, offering scalability and safety [38].

By using boric acid and urea as safer starting materials, this work addresses the risks associated with toxic reagents, as the process likely reduces the use of hazardous or rare chemicals, minimizing waste and pollution. Developing a sustainable synthesis route for boron nitride nanoparticles (BNNPs) aligns with the principles of green chemistry, which emphasize reducing the environmental impact of chemical production. By using common and potentially less harmful starting materials, this work contributes to reducing the carbon footprint and chemical waste associated with nanoparticle synthesis [42]. If the synthesis process is optimized for energy efficiency, it can contribute to reducing greenhouse gas (GHG) emissions and energy consumption. If the synthesis process involves lower energy consumption or less GHG emissions, as compared to traditional methods, it contributes to climate action [43].

The key innovation lies in the two-stage heat treatment: nitrogen serves as an inert atmosphere, and hydrogen acts as a diffusing gas. This method yields uniform h-BN nanoparticles (2–10 nm) with reduced energy consumption and shorter synthesis time. Moreover, the use of nitrogen enhances safety, while hydrogen promotes uniform particle formation. If the synthesis process can be easily scaled up for industrial production, this would be a major breakthrough. Using common starting materials can potentially lower production costs, making boron nitride nanoparticles more accessible for various applications. This cost-effectiveness is crucial for scaling up production and integrating BNNPs into commercial products and technologies [41].

Additionally, the use of common starting materials suggests a departure from traditional, potentially hazardous or expensive precursors. While the use of common starting materials ensures a consistent supply chain for boron nitride nanoparticle production, a sustainable and efficient process could enable large-scale production, leading to economic growth and job creation. This aligns with the growing emphasis on sustainable and environmentally friendly synthesis methods in materials science.

The development of a sustainable synthesis method also may open up new possibilities for creating boron nitride-based materials with tailored properties. If the synthesized nanoparticles have specific size, shape, or structural characteristics that differ from those produced by other methods, this could represent a significant advancement. Developing a sustainable synthesis route involves optimizing reaction conditions and parameters, leading to advancements in materials processing technology. The sustainable route implies an efficient process that minimizes energy consumption, waste generation, and overall environmental impact. This could involve lower reaction temperatures, shorter reaction times, or higher product yields compared to existing methods [44].

To enable us to produce h-BN nanoparticles using available and safe starting materials, this study utilizes common starting materials such as boric acid and urea to reduce costs and environmental impact. The main difference of this sustainable method for synthesizing boron nitride nanoparticles is the use of a special two-stage heat treatment using nitrogen as an inert atmosphere and hydrogen as a diffusing gas. To optimize the synthesis process for achieving high yields and desired nanoparticle properties, heat treatment was performed at low temperatures, which reduces energy consumption and shortens synthesis time.

It is expected that this method results in the formation of dispersed h-

BN nanoparticles with a narrow size range between 2 and 10 nm, providing highly uniform morphology. The development of a novel synthesis method for BN nanoparticles adds valuable knowledge to the field of nanotechnology. It provides researchers with new techniques for producing nanoparticles with specific properties, which can be tailored for different applications. BNNPs are essential in cutting-edge technologies, including electronics, energy storage, and thermal management systems. A sustainable synthesis method could accelerate innovation in the sectors by providing a reliable and environmentally friendly source of high-quality nanoparticles.

Additionally, the use of nitrogen instead of ammonia while providing an inert atmosphere would make the process safer, and the diffusion of hydrogen in the grain-boundary regions of the crystals promotes the uniform formation of nanoparticles. This work supports broader environmental and economic goals by promoting the use of sustainable practices in nanomaterial production. It aligns with global efforts to reduce environmental impact while maintaining economic growth in high-tech industries. The features of the method make it possible to achieve the stated technical result, while scaling up the production of h-BN nanoparticles [45].

In the classical method for synthesizing h-BN nanoparticles, which typically involves the direct reaction of boric acid and urea with single-stage thermal treatment under an ammonia atmosphere, particle sizes are often larger than 50 nm [46]. The morphological uniformity and crystallinity of the product are limited, with crystallinity reaching approximately 40–50 % under standard conditions. Furthermore, the classical method generally requires higher temperatures (exceeding 1100 °C) for extended durations, leading to increased energy consumption and broader particle size distributions [46]. In contrast, our proposed method modifies the traditional approach by introducing a two-stage thermal treatment. During the first stage, nitrogen serves as an inert atmosphere, and in the second stage, hydrogen is employed as a diffusing agent. This modification enables enhanced control over particle growth and crystallization, resulting in smaller particle sizes (2–10 nm, with an average size of 6 nm), significantly higher crystallinity (up to 98 %), and improved morphological uniformity. Additionally, the proposed method achieves these outcomes with comparatively lower overall energy requirements due to optimized reaction conditions. This detailed comparison highlights the key advantages of the proposed method, including superior product quality, reduced energy demands, and potential for large-scale industrial application, demonstrating its clear superiority over conventional approaches.

2. Materials and experimental methods

2.1. Materials

In this work, all reagents were analytical grade without preliminary purification. The following reagents were used: H_3BO_3 , $(\text{NH}_2)_2\text{CO}$, N_2 (99.99 %), H_2 (99.99 %) and bidistilled water.

2.2. Methods

2.2.1. Characterization

To determine the dimensions and morphology of the nanoparticles, a JEM-1011 transmission electron microscope (TEM) from Jeol (Japan) and a high-resolution TEM model Talos F-200i from Thermo Fisher Scientific (USA) were employed. The study of nanostructures through scanning electron microscopy (SEM) was conducted using a JEOL JEM-6510LV device equipped with an energy-dispersive X-ray spectrometer SSD X-Max Inca Energy from Oxford Instruments (UK).

The phase analysis of the samples was conducted using X-ray diffraction on polycrystalline powders. The diffraction patterns were collected using a D2 Phaser instrument model Bruker-AXS (Germany) with $\text{CuK}\alpha$ radiation and a graphite monochromator ($\lambda = 1.54178 \text{ \AA}$). The instrument was operated in tube mode (Cu) with a current of 10 mA

and a voltage of 30 kV. The angle 2θ range was from 10° to 75° with a step of 0.02° . The slit was adjusted to 0.6 mm, and the exposure time at a given point was 1 s, with the energy discriminator ranging from 0.17 to 0.23 keV. Spectrum analysis and phase identification were performed using the JCPDS-ICDD library and specialized software. Infrared spectra of the samples were acquired using a Nicolet 380 FTIR spectrometer from Thermo Fisher Scientific (USA) within the range of $400\text{--}4000 \text{ cm}^{-1}$. The samples underwent heat treatment in a Protherm-250 tubular furnace (Turkey). Mass spectrometry (SIMS) was performed in a vacuum of $\sim 10^{-8}$ Torr using magnetron discharge pumps. The instrument detected negative ions generated by bombarding target samples with positive cesium ions. Further details can be found in works [47–50].

2.2.2. Synthesis method of h-BN nanoparticles

Numerous studies have explored the synthesis of h-BN nanoparticles from urea and boric acid [51]. The primary distinction of our method lies in a specialized two-stage thermal treatment process, utilizing nitrogen as an inert atmosphere and hydrogen as a diffusing gas. This low-temperature treatment significantly reduces energy consumption and shortens synthesis time, while ensuring the formation of well-dispersed h-BN nanoparticles with a narrow size distribution between 2 and 10 nm. The use of nitrogen instead of ammonia for creating an inert environment not only improves safety but also contributes to better process control. The diffusion of hydrogen in the grain boundary regions of the crystals facilitates uniform nanoparticle formation. These key aspects of the method allow us to achieve the desired technical outcomes, including enhanced morphological uniformity.

The proposed synthesis scheme is illustrated in Fig. 1.

Initially, the semi-finished product was obtained by dissolving H_3BO_3 and $(\text{NH}_2)_2\text{CO}$ in water in a ratio of 1:2 mol. The solution was stirred on a magnetic stirrer at a temperature of 80 °C for 2 h at 800 rpm. Then the solution was poured into a Petri dish and dried in a drying chamber at 120 °C for 12 h. The resulting samples were ground in a mortar after drying. Subsequently, a two-stage heat treatment was carried out. The first stage included firing the semi-finished product in a tubular furnace in a nitrogen flow. The heating process continued for 2 h at a heating rate of 5 °C/min until a temperature of 600 °C was reached. After this, the samples were held for 2 h and then left to cool. The samples were then ground. At this stage, secondary firing was performed in a flow of a mixture of N_2 and H_2 gases at a heating rate of 5 °C/min. After heating at 1000 °C for 2 h, the samples were left to cool freely. Before each stage of heat treatment, the tube furnace was evacuated to the level shown in the Table 1. After reaching the required vacuum level, the heat treatment process was carried out in a nitrogen or hydrogen atmosphere, which ensures minimal influence of residual gases on the synthesized material.

The final step involved washing the ground powder in 50 mL of distilled water on a magnetic stirrer at 80 °C and 600 rpm for 1 h, followed by centrifugation to precipitate the final product. The final product was then dried at 120 °C for 4 h.

3. Results and discussion

3.1. Characterization

Fig. 2 shows the SEM image of the obtained intermediate product containing boron (B) and nitrogen (N) after drying at 120 °C. The intermediate product was obtained by mixing boric acid (H_3BO_3) and urea ($(\text{NH}_2)_2\text{CO}$) in an aqueous medium, followed by crystallization during drying.

Fig. 2 shows the structure of the material, consisting of large micron-sized crystals with a dendritic morphology. The crystal size varies within a few microns, as evidenced by the scale mark in the image corresponding to 10 μm . The morphology of the crystals shows branched structures characteristic of dendritic crystals, with numerous branches emanating from a central axis.

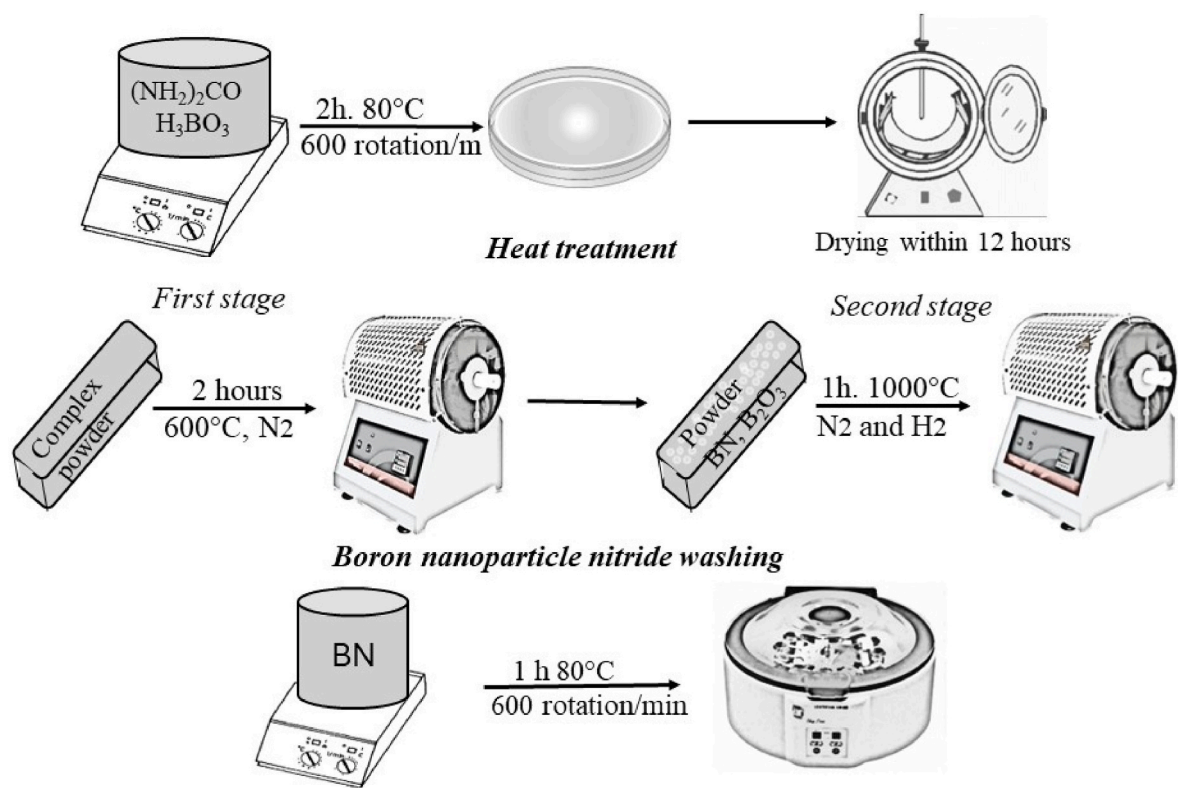


Fig. 1. Scheme for obtaining h-BN nanoparticles.

Table 1
High vacuum regimes.

Pressure	P (Torr)	<10 ⁻⁵
Particle number density	n (cm ⁻³)	<10 ¹¹
Mean free-path	λ (cm)	>10 ⁴
Impingement plate	Z _a (cm ⁻² s ⁻¹)	<10 ¹⁵
Vol.-related collision ratio	Z _v (cm ⁻³ s ⁻¹)	<10 ⁹
Monolayer-timea	τ (s)	>100
Type of gas flow		Molecular flow

This pattern of crystal growth may indicate specific crystallization conditions, which include controlled evaporation of water at 120 °C. These conditions favor the formation of crystals with a highly branched structure, which is typical for processes where diffusion and aggregation of molecules play a key role. Microstructural features of the intermediate product, such as large crystal sizes and their dendritic shape, can affect the physical and chemical properties of the product, including its mechanical properties, porosity, and reactivity. Using Fourier transform infrared spectrometry, the compounds obtained as a result of crystallization of the intermediate product and subsequent drying at 120 °C were

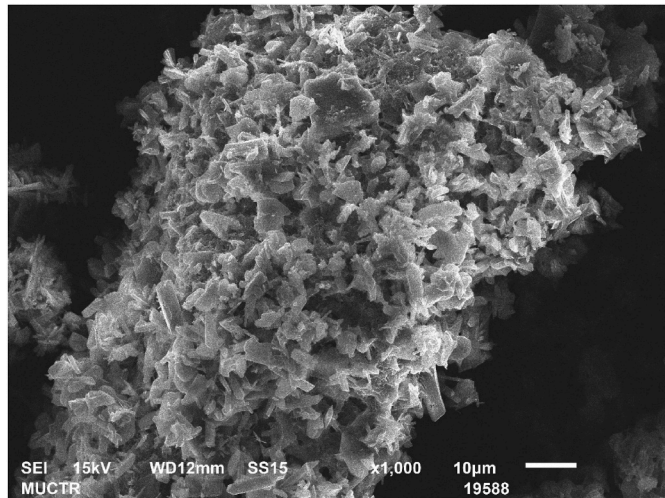


Fig. 2. SEM image of the obtained semi-product containing B and N after drying at 120 °C.

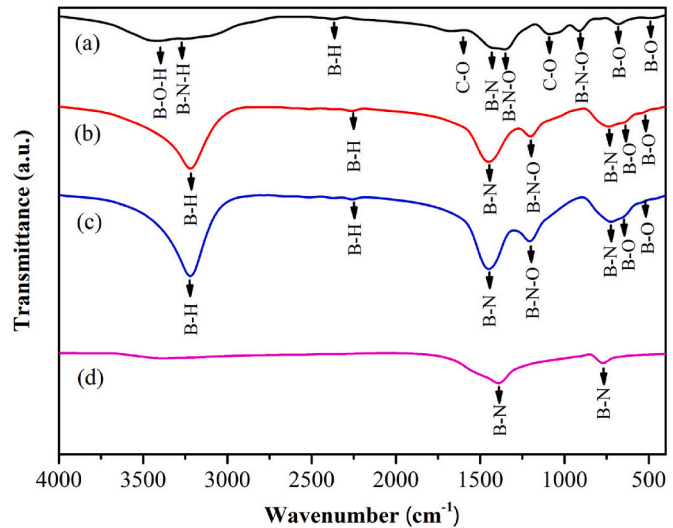


Fig. 3. FT-IR spectrum of the obtained products containing B and N: a - after drying at 120 °C, b - after the first stage of heat treatment at 600 °C, c - after the second stage of heat treatment at 1000 °C, d - after washing with water and drying.

studied (Fig. 3).

Fig. 3a (black line) shows the FT-IR spectrum of the obtained semi-products containing B and N. The characteristic vibration bands are observed: 3433 cm^{-1} , corresponding to B-O-H; 3255 cm^{-1} , corresponding to B-N-H; 2351 cm^{-1} , corresponding to B-H; 1667 cm^{-1} , corresponding to C-O; 1428 cm^{-1} , corresponding to B-N; 1347 and 923 cm^{-1} , corresponding to B-N-O; 1089 cm^{-1} , corresponding to C-O; 692 and 507 cm^{-1} , corresponding to B-O [45,52]. These spectral characteristics allow the identification of various functional groups and bonds in the obtained product, such as hydroxyl groups (B-OH), amino groups (B-NH₂), boron-hydrogen bonds (B-H), carbonyl groups (C-O), boron-nitrogen bonds (B-N), boron-nitrogen-oxygen (B-N-O), and boron-oxygen bonds (B-O).

Fig. 3b shows the FT-IR spectrum of the precursor obtained in the first step of heat treatment at $600\text{ }^{\circ}\text{C}$ under nitrogen atmosphere. Characteristic vibration bands are observed at 3217 cm^{-1} and 2261 cm^{-1} , corresponding to B-H; bands at 1438 and 883 cm^{-1} , corresponding to B-N; a band at 1195 cm^{-1} , corresponding to B-N-O; bands at 715 and 546 cm^{-1} , corresponding to B-O. However, the bands at 1667 cm^{-1} (C-O) and 1347 and 923 cm^{-1} (B-N-O) disappeared. This indicates that although the groups containing carbon and hydrogen were removed during the heat treatment at $600\text{ }^{\circ}\text{C}$ under nitrogen atmosphere, oxygen cannot be completely removed from the precursor structure.

Fig. 3c shows the FT-IR spectrum of the precursor after the second stage of heat treatment at $1000\text{ }^{\circ}\text{C}$ in a nitrogen and hydrogen atmosphere. All characteristic vibration bands are identical to those obtained after the first stage of heat treatment at $600\text{ }^{\circ}\text{C}$. However, the intensity of the bands at 1438 and 883 cm^{-1} , corresponding to B-N, increased.

Fig. 3d shows the FT-IR spectrum of the final product after water washing and drying. An increase in the intensity of the bands at 1438 and 883 cm^{-1} corresponding to B-N is noticeable. No peaks corresponding to other compounds determined at other processing stages were detected. This demonstrates the effectiveness of the washing in obtaining pure h-BN final product.

High vacuum ($<10^{-5}$ Torr) in the second step of heat treatment minimizes residual gases and hydroxyl groups, as evidenced by the decrease in H₂O partial pressure from 10^{-3} to 10^{-7} Torr [3]. The corresponding disappearance of the B-OH band at 3433 cm^{-1} in the FTIR spectra (Fig. 3c) indicates the effective removal of hydroxyl impurities.

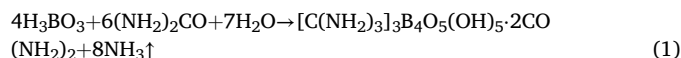
The combination of SEM and FTIR spectroscopy data allows us to trace the evolution of the material structure from intermediate products with dendritic morphology to the pure final product h-BN. Changes in the functional groups reflected in the FTIR spectra clearly correlate with changes in the crystal morphology at various stages of processing.

For complete identification of the obtained semi-finished product, X-

ray phase analysis was performed. Fig. 4 shows the diffraction pattern of the obtained semi-finished product after drying at $120\text{ }^{\circ}\text{C}$.

Based on the analysis of the diffraction pattern of the sample (black curve) obtained after drying, this implied that all the identified diffraction peaks correspond to the JCPDS card No. 45-1869 (red line). This card belongs to the crystalline complex *tris*(guanidinium)pentaoxopentahydroxy tetraborate bisurea guanidium hydroxide borate urea [C(NH₂)₃]B₄O₅(OH)₅·2CO(NH₂)₂.

The complex was obtained during the reaction of urea (NH₂)₂CO and boric acid H₃BO₃, which results in the release of ammonia (NH₃). An approximate mechanism for the formation of crystals of the complex is presented by the following reaction:



According to this mechanism, the interaction of boric acid and urea forms a complex accompanied by the release of ammonia as a result of urea degradation. The obtained diffraction analysis data confirm the identity of the product structure with the previously recorded crystalline complex.

The resulting intermediate product ([C(NH₂)₃]B₄O₅(OH)₅·2CO(NH₂)₂) for obtaining h-BN nanoparticles, a two-stage special heat treatment was carried out. The study of hexagonal boron nitride (h-BN) nanoparticles after the first stage of heat treatment at $600\text{ }^{\circ}\text{C}$ using TEM and particle size distribution analysis allows a deeper understanding of the formation process and characteristics of the material.

Fig. 5 shows the results of the first stage of heat treatment of BN nanoparticles at $600\text{ }^{\circ}\text{C}$.

The TEM image shown in Fig. 5a shows nanoparticles formed after the first stage of thermal treatment of the precursor at $600\text{ }^{\circ}\text{C}$. The nanoparticles have an irregular shape with sizes ranging from 5 to 14 nm. The average particle size is about 10 nm, indicating a narrow particle size distribution, which is confirmed by further analysis.

The nanoparticles in the image show a tendency to aggregate, which is typical for nanomaterials due to their high specific surface area and the associated interparticle interactions. However, the presence of particles with well-defined crystalline faces is also observed, indicating partial crystallinity of the material already at the first stage of thermal treatment. Such morphology may indicate nucleation and subsequent crystal growth at the nanoscale, which is typical for thermally treated materials.

The histogram in Fig. 5b shows the particle size distribution of the obtained BN nanoparticles after treatment at $600\text{ }^{\circ}\text{C}$. The distribution is close to normal, with the maximum proportion of particles with a diameter of about 9–10 nm. The peak of the distribution is in the region of 10 nm, with a symmetrical decrease in the proportion of particles both downwards (to 5–6 nm) and upwards (to 13–14 nm) in size.

Such a distribution indicates controlled particle growth during heat treatment, which is important for the synthesis of nanomaterials with a narrow range of particle sizes. The increase in particle size at the first stage of heat treatment can be associated with the processes of agglomeration and recrystallization of the precursor, as well as the onset of the formation of the h-BN structure.

The obtained TEM data and particle size distribution demonstrate that after the first stage of heat treatment at $600\text{ }^{\circ}\text{C}$, the BN nanoparticles have predominantly nanometer sizes with a narrow particle distribution. The average particle size is about 10 nm, indicating a controlled process of nanocrystal formation and growth. These data will be key for further analysis and improvement of heat treatment parameters in the next stages to obtain high-purity hexagonal boron nitride with optimal particle size and morphology.

The phase composition of the precursor product after the first stage of heat treatment at $600\text{ }^{\circ}\text{C}$ using X-ray diffraction (XRD) analysis enables a detailed characterization of the material structure. Fig. 6 shows the X-ray diffraction pattern of the precursor product after heat

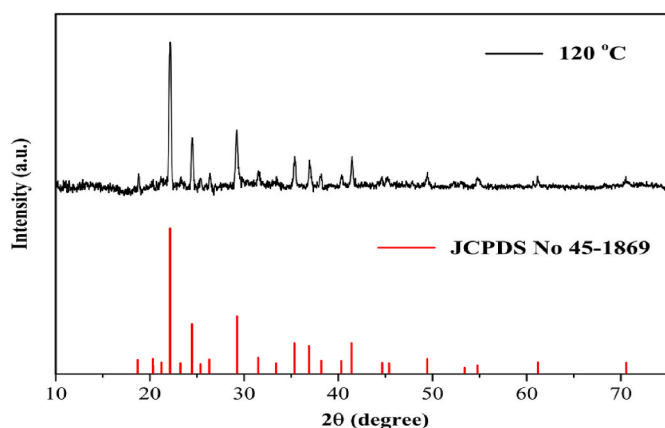


Fig. 4. Diffraction pattern of the obtained semi-product after drying at $120\text{ }^{\circ}\text{C}$ (black graph). JCPDS Card No. 45-1869 for the substance of the complex compound [C(NH₂)₃]B₄O₅(OH)₅·2CO(NH₂)₂.

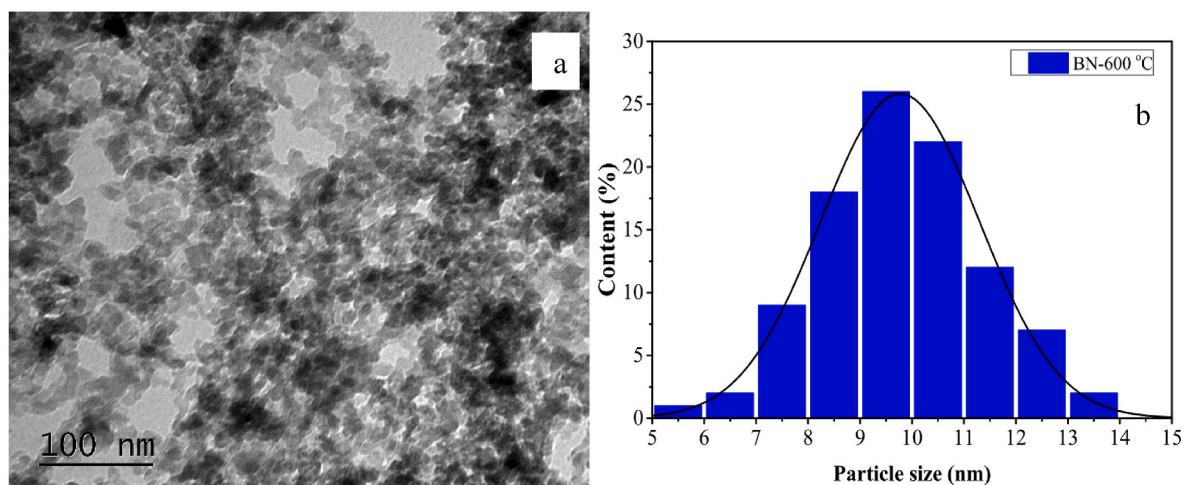


Fig. 5. Results of the first stage of heat treatment at 600 °C of BN nanoparticles: a) TEM image of the obtained BN nanoparticles; b) distribution of particle sizes of the obtained BN.

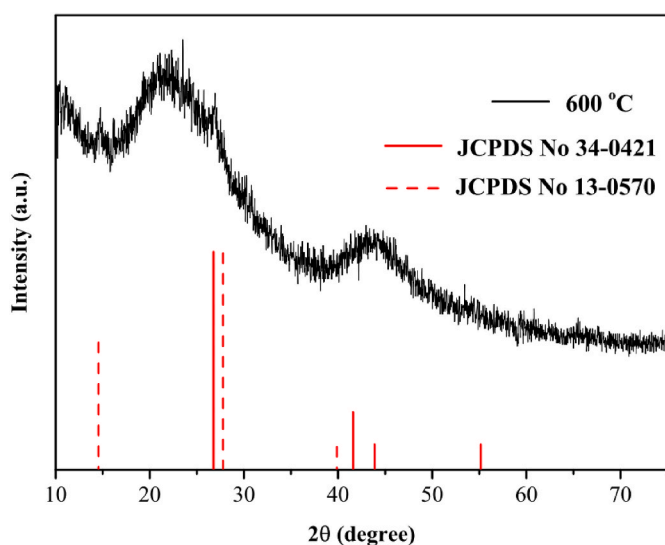


Fig. 6. Diffraction pattern of the obtained precursor nanoparticles after the first stage of heat treatment at 600 °C (black curve). JCPDS No. 34–0421 for the BN substance (red line) and JCPDS No. 13–0570 for the B₂O₃ substance (red dashed line). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

treatment at 600 °C in a nitrogen atmosphere (black curve). The graph also shows the reference data for hexagonal boron nitride (h-BN) according to JCPDS No. 34–0421 (red line) and for boron oxide (B₂O₃) according to JCPDS No. 13–0570 (red dotted line).

The diffraction pattern of the precursor product (black curve) contains peaks corresponding to the h-BN structure, which is confirmed by their coincidence with the positions of the peaks in the JCPDS No. 34–0421 map. These peaks indicate partial crystallinity of boron nitride in the sample after the first stage of heat treatment. However, the intensity of the peaks is low, which indicates a low degree of crystallinity of the material. Peaks corresponding to boron oxide are also observed in the diffraction pattern, which is confirmed by their coincidence with the data in JCPDS No. 13–0570. The presence of boron oxide indicates that boron was not completely converted into boron nitride during the heat treatment and is present as an oxide phase.

Broad peaks and noise in the diffraction pattern indicate a significant content of the amorphous phase in the material. This characteristic is typical for precursors that have undergone heat treatment at relatively

low temperatures, when crystallization is not completely completed and a significant part of the material remains amorphous. The degree of crystallinity χ_c was calculated using the integrated intensity of the peaks for the crystalline (A_c) and amorphous (A_a) regions according to Equation (2) [53,54]:

$$\chi_c = \frac{A_c}{A_c + A_a} \quad (2)$$

where χ_c is the crystallinity of the sample, A_c is the area of crystalline peaks, A_a is the area of amorphous peaks. The calculation showed that the degree of crystallinity of the precursor is 5 %, indicating the dominance of the amorphous phase in the material. This is consistent with TEM observations (Fig. 5a), where the irregular shape of the nanoparticles and their tendency to aggregate were noted.

XRD data show that after the first heat treatment step at 600 °C the precursor contains a small amount of crystalline h-BN along with boron oxide (B₂O₃). Most of the material remains in the amorphous phase, as evidenced by the broad peaks in the diffraction pattern and the low crystallinity level (5 %). These results are important for optimizing subsequent heat treatment steps to increase crystallinity and purify the product from oxides.

After the first stage of heat treatment of BN nanoparticles was completed, the second stage was carried out to obtain crystallized hexagonal boron nitride (h-BN) nanoparticles. In this stage of heat treatment, N₂ was used as an inert medium, and hydrogen (H₂) was used as a diffusing gas to implement grain boundary diffusion, which promotes the formation of fine crystallized h-BN nanoparticles.

Fig. 7 shows the findings of the second stage of heat treatment of BN nanoparticles at 1000 °C.

The TEM image shown in Fig. 7a demonstrates the high crystallinity of the obtained h-BN nanoparticles. The nanoparticles are predominantly spherical in shape, which is typical for particles formed at high temperatures under controlled conditions. The spherical shape of the particles also indicates the minimization of surface energy, which contributes to the thermodynamic stability of the material.

Clear and well-defined particle boundaries indicate complete crystallization, which confirms the effectiveness of the second stage of thermal treatment. The presence of such morphology indicates the potential application of these nanoparticles in areas requiring stable and uniform structures. The particle size distribution shown in Fig. 7b shows that the nanoparticle diameters range from 2 to 10 nm, with a peak distribution at around 6 nm. The narrow size distribution range indicates high homogeneity of the obtained product, which is critical for applications requiring high reproducibility and predictability of

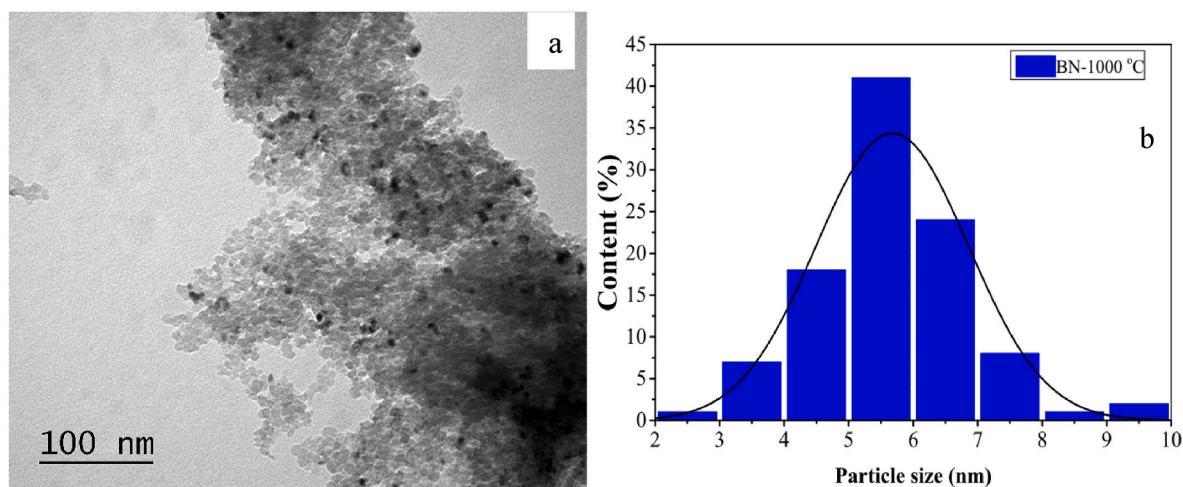


Fig. 7. a) TEM image of the BN nanoparticles; b) distribution of particle sizes of the BN.

material properties.

The particle size of h-BN obtained by the two-step thermal treatment method differs significantly from that of particles obtained by one-step thermal treatment in a nitrogen (N_2) atmosphere. In the two-step method, with the use of hydrogen as a diffusing gas, the particle size ranges from 2 to 10 nm, with an average size of 6 nm, ensuring high morphological uniformity and stability. In contrast, in the one-step method, the particles are larger, ranging from 5 to 14 nm, with an average size of 10 nm, due to the absence of hydrogen and less control over particle growth. Moreover, the crystallinity of the h-BN nanoparticles is only 5 % after the first stage, whereas it reaches 87 % after the second stage. Thus, the two-step thermal treatment allows for the production of smaller, more uniform, and crystalline h-BN particles.

TEM analysis (Fig. 7) shows that decreasing the heating rate and optimal precursor concentrations promote the formation of h-BN with a narrow particle size distribution. Smoother heating ensures controlled grain growth and reduces agglomeration, confirming the dependence of morphology on synthesis conditions.

The average diameter of 6 nm indicates that the conditions of the second stage of thermal treatment were carefully optimized to obtain particles in this size range. Particle sizes in the range of a few

nanometers are important for controlling the optical, electronic and catalytic properties of the material.

Additionally, to clarify the sizes of the obtained h-BN nanoparticles after the first and second stages of heat treatment, DLS analysis was carried out (Fig. 8).

During the DLS, the average particle size of h-BN was determined after the first and second stages of heat treatment: (a) after the first stage – 11.8 ± 4.2 nm, (b) after the second stage – 10.7 ± 3.9 nm.

For comparison, the average particle sizes determined by statistical analysis of TEM images (Figs. 5 and 7 a, b) are: after the first stage – 10 nm, after the second stage – 6 nm.

The discrepancy between the DLS and TEM results is due to the differences in the measurement principles. In DLS, the nanoparticles are dispersed in water and their hydrodynamic diameter is determined, which includes the contribution of the interfacial layer consisting of the adsorption (Stern) shell (hydroxyl ions, OH^-) and the diffuse Gouy layer (adsorbed water molecules) around the surface of the nanoparticles. Additionally, in h-BN solution, the nanoparticles tend to form soft agglomerates, which also increases the measured sizes in DLS. This effect is well documented in the literature and is consistent with previous studies [55].

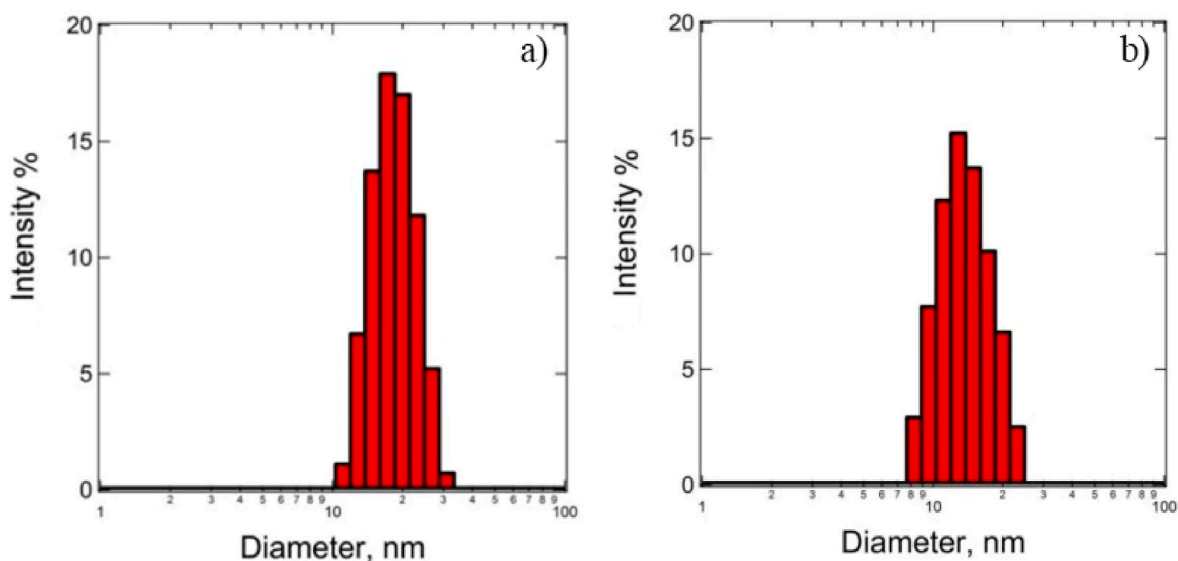


Fig. 8. The average diameter of h-BN nanoparticles calculated from DLS data is (a) after the first stage of heat treatment 11.8 ± 4.2 nm, (b) after the second stage of heat treatment 10.7 ± 3.9 nm, and, respectively.

The obtained data, presented in Fig. 7a and b, confirm the successful completion of the second stage of thermal treatment, which resulted in the formation of homogeneous, spherical h-BN nanoparticles with a narrow size distribution. These results indicate a high degree of control over the synthesis process and allow us to predict the successful application of the obtained nanoparticles in high-tech areas that require materials with high quality and stability standards. For a more detailed analysis of the obtained product, XRD analysis was performed. Fig. 9 shows the diffraction pattern of h-BN nanoparticles obtained after the second stage of thermal treatment at 1000 °C in a nitrogen and hydrogen atmosphere.

The diffraction pattern shown in Fig. 9 shows characteristic peaks corresponding to hexagonal boron nitride (h-BN) and boron oxide (B_2O_3). The black curve in the figure indicates the presence of both of these phases in the powder obtained after heat treatment. This was confirmed by comparing the experimental data with the data from the JCPDS standard cards: No. 34–0421 for h-BN (red line) and No. 13–0570 for B_2O_3 (red dotted line). Based on XRD analysis, the degree of crystallinity of the obtained product was found to be 87 %. This value indicates a high degree of ordering of the crystalline structure in the sample. The product consists of a mixture of h-BN and B_2O_3 nanoparticles. h-BN appears as the main phase, while B_2O_3 is present as a minor phase, probably as a result of reactions occurring at high temperatures under the conditions used in this experiment.

Considering that h-BN is a water-insoluble material, while B_2O_3 is water-soluble, it was decided to wash the product with water to remove B_2O_3 . This will allow obtaining a purer product consisting mainly of h-BN nanoparticles. This procedure will increase the purity of the material, which is important for its further use in areas where only h-BN is required.

The HR-TEM image shown in Fig. 10a shows BN nanoparticles after washing and drying. Despite the treatment, the nanoparticles retain their spherical shape. The spherical shape of the particles, as in the previous stages of the study, indicates the minimization of surface energy, which helps to maintain high thermodynamic stability of the material. The particle size distribution shown in Fig. 10b shows that the nanoparticle sizes range from 2 to 10 nm, with a peak distribution at about 6 nm. This confirms that the average particle diameter remains unchanged despite the treatment. The retention of particle size after washing and drying indicates the stability of the nanostructure and allows us to predict stable physicochemical properties of the final product.

The HR-TEM image shown in Fig. 10a illustrates the high-quality crystalline structure of h-BN nanoparticles. Lattice fringes are

observed in two different directions intersecting at an angle characteristic of the hexagonal crystal structure. The interplanar distances measured from the lattice fringes are 3.3 Å and 2.2 Å, corresponding to the (002) and (100) planes of h-BN, respectively. These values are consistent with the standard data for hexagonal boron nitride (JCPDS No. 34–0421).

Analysis of the angles between the lattice fringes confirmed the agreement with the [001] crystallographic zone axis. The vector product of the observed directions confirms the zone axis, demonstrating the agreement with the hexagonal symmetry of the h-BN structure.

The lattice fringes were identified using the Fourier transform of the HR-TEM image. This approach provided accurate measurements of interplanar spacings and angular relationships, avoiding projection or offset errors that might arise from analyzing only one set of bands. Detailed crystallographic analysis unambiguously confirmed the hexagonal phase of the nanoparticles. Visualization of grains along the zone axis eliminated possible compression errors, providing a reliable basis for phase identification.

To gain a deeper understanding of the structure of the final product, high-resolution transmission electron microscopy (HR-TEM) and selected area electron diffraction (SAED) imaging were analyzed. The results of these analyses are presented in Fig. 10c and d.

The HR-TEM image shown in Fig. 10c details the crystal structure of the h-BN nanoparticles. It is evident from the image that the structure consists of alternating layers of boron and nitrogen atoms, forming the characteristic layered structure of h-BN. The thickness of each layer is about 3.3 Å, which is consistent with the known literature data and confirms the correctness of the obtained results. The literature describes that the h-BN layers are stacked in an AA configuration and are linked to each other by weak van der Waals forces with an interlayer distance of about 3.33 Å [56].

The SAED image shown in Fig. 10d was used to confirm the crystal structure of h-BN. The SAED analysis highlights the (004) crystallographic planes, which show a strong contrast indicating the close packing of these planes in the hexagonal BN lattice. The presence of distinct diffraction spots confirms the high crystallinity of the material and the absence of amorphous phases, which is important for understanding the physicochemical properties of the final product [56].

The HR-TEM analysis further confirms the thermocrystallinity of the synthesized h-BN nanoparticles. The clarity and precision of the lattice bands enhance the reliability of the phase indicator, demonstrating the effectiveness of the synthesis method. The confirmed crystallographic zone axis provides further verification of the hexagonal phase of the material, which is consistent with the XRD and SAED results. These data accurately convey the quality and structure of the synthesized nanoparticles.

The final product was further characterized by XRD analysis. Fig. 11 shows the diffraction pattern of h-BN nanoparticles obtained after washing with water and drying the precipitated particles at 120 °C for 4 h.

In the diffraction pattern (Fig. 11, black curve), all the main peaks belong to h-BN. According to JCPDS No. 34–0421 (red line), h-BN has a hexagonal structure with the space group $P6_3/mmc$ (194) and the lattice parameters $a = b = 2.5$ and $c = 6.66$ Å [51]. The peaks found in the range of 2 θ : 26.76°, 41.60°, 43.87°, 55.17° can be attributed to the crystal structures with the arrangement (hkl): 002, 100, 101 and 004, corresponding to the h-BN phase. The degree of crystallinity of the final product was 98 %. No peaks related to other B or N compounds were found. This indicates that this method is effective for obtaining pure h-BN nanoparticles [57].

The developed two-stage method yields h-BN nanoparticles with 98 % crystallinity, a significant improvement from the initial 5 % after the first stage (Figs. 6 and 9). The SAED pattern (Fig. 10d) confirms this via (002) and (100) reflections, aligning with Li et al. [46], where similar crystallinity enhancement correlated with B_2O_3 removal during high-temperature annealing. This underscores the efficacy of our

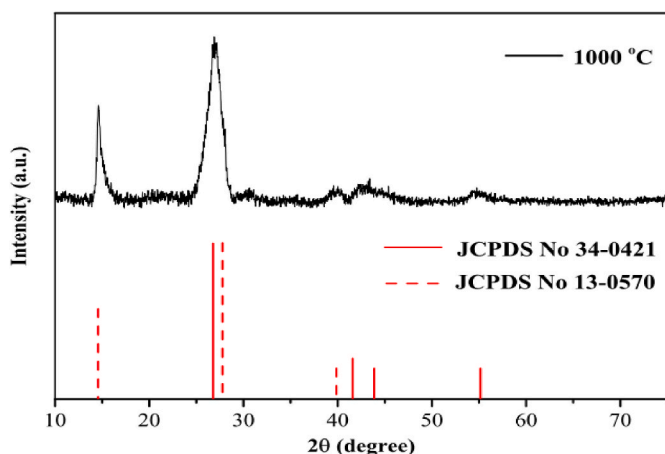


Fig. 9. Diffraction pattern of the obtained BN nanoparticles after the second stage of heat treatment at 1000 °C (black curve). JCPDS No. 34–0421 for the BN substance (red line) and JCPDS No. 13–0570 for the B_2O_3 substance (red dashed line). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

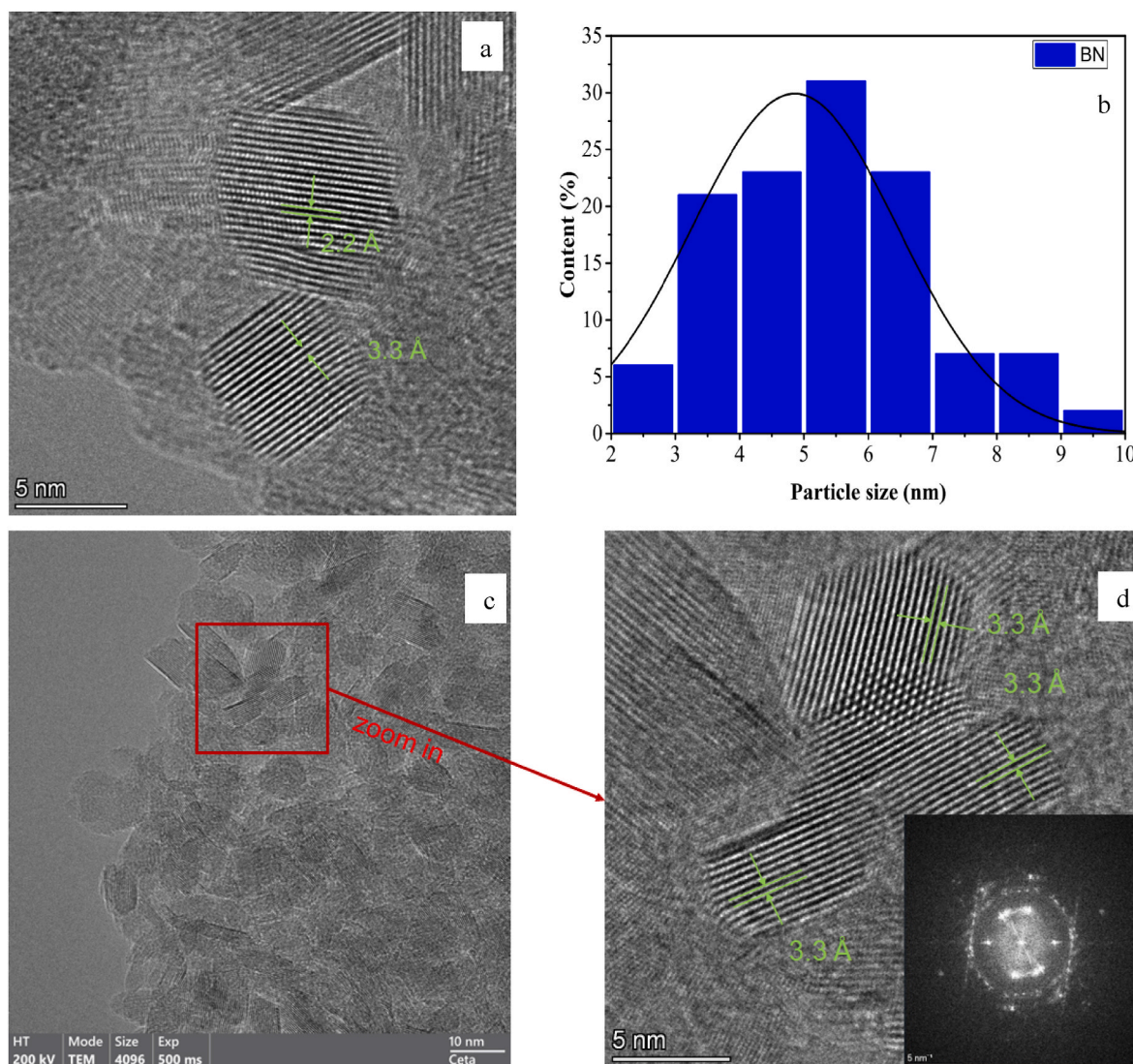


Fig. 10. a) TEM image of the obtained BN nanoparticles; b) Particle size distribution of the obtained BN; c) HR-TEM image with an enlargement of the interface area highlighted by the red square; d) HR-TEM image with an image of the selected area of the SAED electron diffraction in the corner. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

approach in achieving structural perfection.

Analysis of X-ray diffraction patterns (Fig. 11) shows that with a decrease in the heating rate, reflection intensity increases and their width decreases, which indicates an increase in the crystallinity of h-BN. Slow heating ensures ordered diffusion of atoms and the formation of a more perfect structure, whereas high heating rates limit this process, reducing crystallinity and partially amorphizing the material. Thus, the heating rate is a key parameter for controlling the structure of h-BN [38].

Also, for the sake of completeness of the study, a detailed study of the chemical composition of h-BN nanoparticles synthesized by a two-stage heat treatment method was carried out. Advanced analytical methods, including STEM-EDS, were used to determine the chemical composition of the samples (Fig. 12).

Fig. 12 shows the elemental mapping of the confined nanoparticles region using dark field (DF) detector and the distribution of elements C, B, N, Al, Si and O. The maps expand the distribution of the main elements B and N, which ensures the successful control of h-BN nanoparticles. The after-effects, burns and silicon were also detected. The presence of after-effects and silicon is due to the use of appropriate crucibles during the heat treatment, which is due to the presence of the

features of the STEM-EDS method, which uses carbon-coated grids.

Despite the limited accuracy of EDS and XRF, their combined application requires obtaining valuable data on the chemical compositions of the samples. This data became useful for analyzing the h-BN structure formation model and for better understanding the factors determining the stability and purity of the synthesized nanoparticles.

For a comprehensive understanding of the structural evolution and to reliably assess the efficiency of the synthesis process, it was necessary to investigate the sample obtained after the second stage of thermal treatment — both under non-vacuum and high-vacuum conditions. This approach enables a detailed evaluation of the degree of precursor decomposition, the removal of residual impurities, and the crystallinity improvement achieved during the final heat treatment step.

To further characterize the composition of the synthesized h-BN nanoparticles and assess the level of residual impurities and thermally transformed precursor products, secondary ion mass spectrometry (SIMS) analysis was performed (Fig. 13).

Fig. 13 presents comparative SIMS spectra of nanoparticles subjected to thermal treatment under different conditions: (a) in a gas flow without vacuum, and (b) under high vacuum conditions ($<10^{-5}$ Torr). The spectrum obtained without vacuum treatment (Fig. 13a) reveals

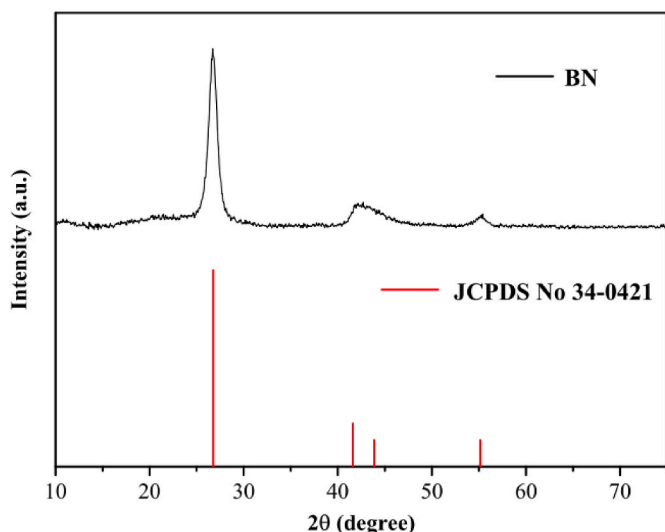


Fig. 11. Diffraction pattern of the obtained BN nanoparticles after washing with water and drying the precipitated particles at 120 °C for 4 h (black curve). JCPDS No. 34-0421 cards for BN substance (red line). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

intense peaks corresponding to light elements such as H^+ and O^- , indicating the presence of hydrogen- and oxygen-containing fragments like B–OH, B–O, and residual H_2O , which remain within the structure after drying and the secondary stage of thermal treatment. Notably, prominent peaks for CH^+ (13 amu), N^+ (14 amu) and B_2^+ (21.5 amu) ions are clearly visible, reflecting residual organic and nitrogen-containing species from incomplete decomposition of precursor components (boric acid and urea). Additionally, the B_2NO^+ ion ($m/z \approx 52$) is observed, serving as a marker of oxygen impurities incorporated into boron–nitrogen fragments, indicating partial oxidation or incomplete decomposition.

Both spectra also display well-resolved molecular ions containing boron and nitrogen (BN (25 amu)), confirming the formation of structural fragments characteristic of hexagonal boron nitride. The B_2N^+ ion ($m/z \approx 35$) appears with high intensity, associated with local fragments containing two coordinated boron atoms typically found at crystal edges or defect zones. The B_3N^+ ion ($m/z \approx 53$) is also distinctly observed, indicating the presence of larger clusters representing triangular boron–nitrogen units formed during initial crystallization stages. The B_3N_2^+ ion ($m/z \approx 66$) is prominent as well, characteristic of multi-atomic clusters formed during fragmentation of the hexagonal boron nitride network under ion bombardment.

The presence of these cluster ions— B_2N^+ , B_3N^+ , B_3N_2^+ —in SIMS spectra does not suggest material contamination, but rather reflects dissociation of the boron–nitrogen lattice in surface layers during ion sputtering. These fragments correspond to typical structural units of h-BN, confirming boron–nitrogen coordination. However, the noticeable presence of peaks corresponding to CH, N, B_2 and B_3 clusters in the non-vacuum sample highlights significant residual impurities.

After the second stage of thermal treatment at 1000 °C in a N_2/H_2 atmosphere under high vacuum ($<10^{-5}$ Torr, Fig. 13b), a dramatic shift in the SIMS spectrum occurs. Peaks corresponding to CH^+ , N^+ , BN^+ , and B_4^+ completely disappear, demonstrating effective removal of these impurities. There is a substantial reduction—by more than two orders of magnitude—in the intensity of H^+ and O^+ peaks, affirming significant removal of hydrogen- and oxygen-containing impurities. The previously detected B_3NO^+ peak also shows reduced intensity, indicative of improved purity.

In contrast, peaks corresponding to stable boron–nitrogen clusters (B_2N^+ , B_3N^+ , B_3N_2^+) remain prominent and dominate the vacuum-

treated sample spectrum, emphasizing the successful formation of structurally uniform and stable boron–nitrogen fragments. Additionally, new peaks such as B_3O^+ (48.5 amu), B_3NO^+ (62.5 amu), and B_4N^+ (57 amu) appear distinctly, indicating formation of stable complex oxide clusters that emerge under high-vacuum thermal treatment conditions, possibly due to localized residual oxidation.

According to the SIMS data (in vacuum, Fig. 13b), the presence of pronounced peaks of C^+ , O^+ , BO^+ , B_2O^+ , B_3O^+ , B_3NO^+ and indicates the residual presence of a small amount of oxygen-containing fragments and complex boron–nitrogen clusters, which may reflect traces of impurities or structural defects. The intensity of these peaks, with the exception of compounds directly related to B–N, in the distribution can correspond to approximately 2 % of the total composition, which is in good agreement with the minimum value of phase purity (98 %) obtained by X-ray diffraction (XRD).

However, it should be noted that SIMS is a very subtle surface technique that records fragments and surface defects, even if they are present in minimal concentrations, without necessarily affecting the bulk phase purity determined by XRD. Thus, the presence of minor traces of oxygen- and boron–nitrogen-containing clusters in the SIMS spectra does not contradict the declared bulk phase purity of 98 % obtained by the XRD method.

These SIMS results align well with FTIR analysis, which demonstrates the disappearance of the B–OH stretching band at 3433 cm^{-1} , and with XRD data, indicating a significant increase in crystallinity up to 98 %. Thus, the comprehensive SIMS analysis quantitatively confirms the enhanced purity and structural uniformity of the hexagonal boron nitride nanoparticles, while clearly elucidating key boron–nitrogen structural fragments formed under optimized vacuum thermal treatment conditions.

The conducted studies have shown that the presented method for obtaining h-BN nanoparticles is effective and allows obtaining particles in a narrow size range with the required properties. The use of accessible and safe reagents, such as boric acid and urea, in combination with two-stage heat treatment ensures a high degree of homogeneity and stability of the resulting nanoparticles. This method is also characterized by reduced energy consumption and reduced synthesis time, which makes it promising for scaling and industrial application.

3.2. Implications of this work on body of literature

The work has the potential to make significant contributions to the body of knowledge in several ways. The use of common starting materials and a two-stage heat treatment process represents a new approach to boron nitride nanoparticle production. The specific conditions (temperature, time, atmosphere) employed in the synthesis can provide valuable insights for further process optimization. The use of common, non-toxic starting materials contributes to a greener synthesis process. If the process is optimized for energy consumption, it can have a positive impact on environmental sustainability [58].

Using readily available and inexpensive starting materials can lower production costs. The success of the two-stage heat treatment process suggests potential for scaling up production while maintaining product quality [37]. The synthesized nanoparticles can serve as a basis for investigating the relationship between synthesis conditions and material properties [59]. The work can stimulate research into potential applications for the produced nanoparticles. Overall, this research contributes to the growing body of knowledge on boron nitride nanoparticles by offering a sustainable and scalable synthesis method.

To evaluate the advantages of the proposed method compared to traditional technologies (CVD, sol-gel method, mechanochemical synthesis), a comparative analysis of key synthesis parameters was conducted, including temperature conditions, process duration, energy consumption, raw material costs, hazard classification of reagents used, crystallinity, morphology, particle size, and equipment complexity. The results of this analysis are summarized in Table 2.

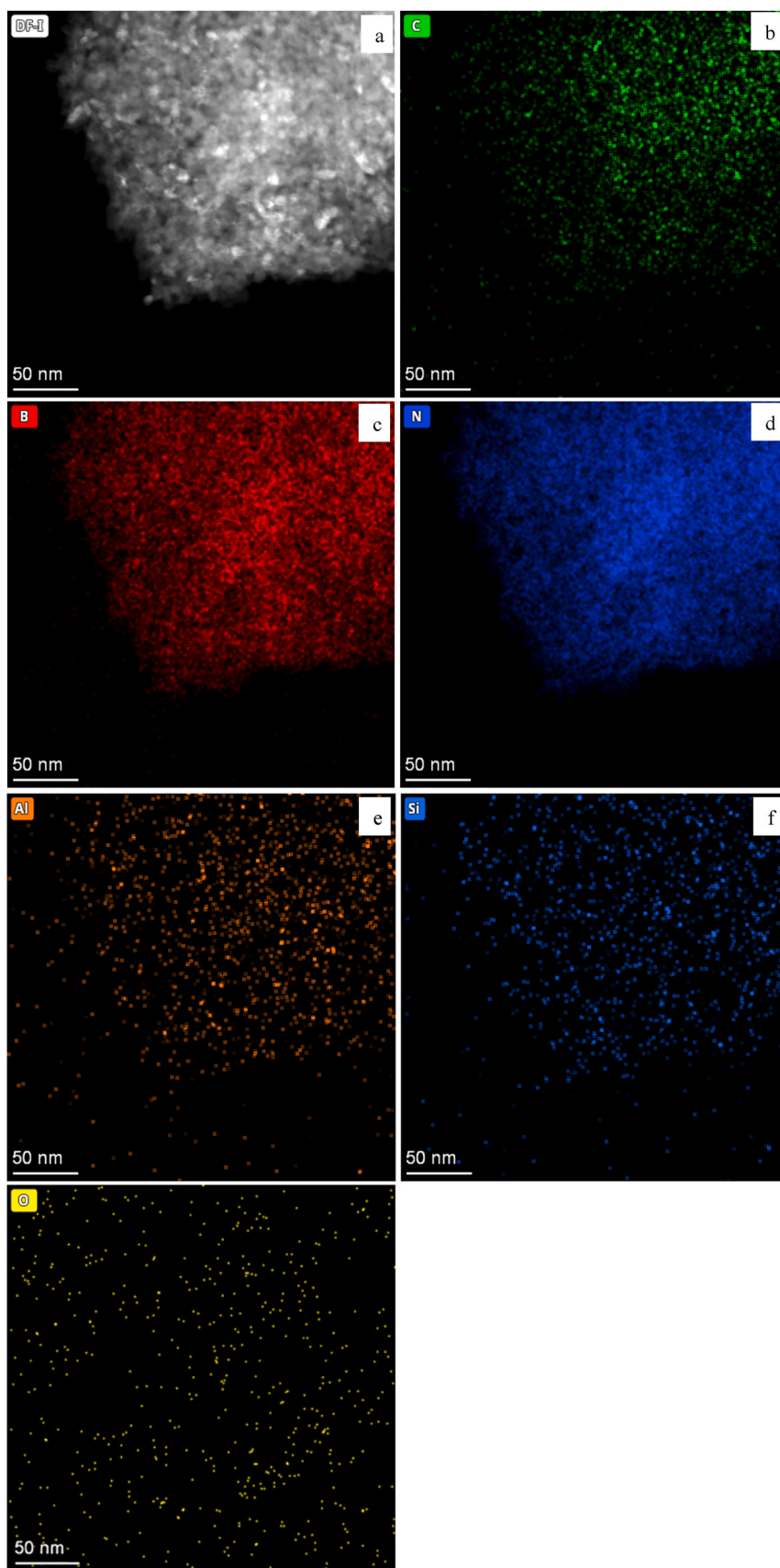


Fig. 12. STEM-EDS elemental mapping of the obtained h-BN nanoparticles region: DF image (a), and elemental maps of C (b), B (c), N (d), Al (e), Si (f), O (g).

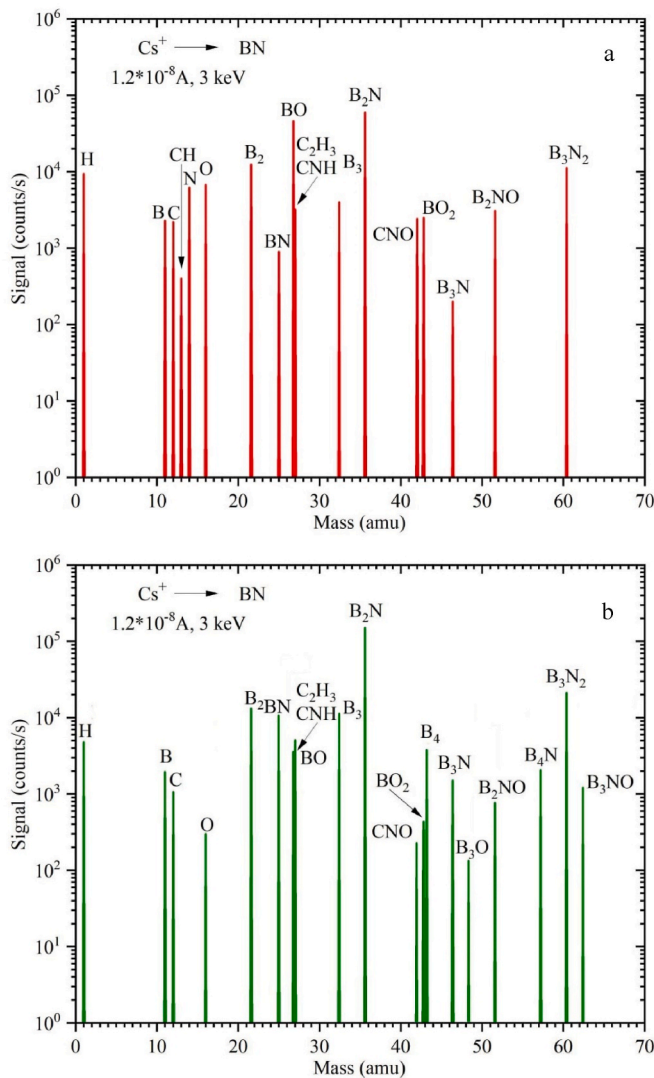


Fig. 13. SIMS spectra of the obtained h-BN nanoparticles after heat treatment under the following conditions: a - no vacuum; b - high vacuum ($<10^{-5}$ Torr).

The proposed method demonstrates several significant advantages. Unlike CVD, which requires complex and expensive equipment (reaction chambers with controlled atmosphere, gas reagent supply systems, and high-temperature installations), this approach is based on the use of accessible and less complex equipment, simplifying scaling and reducing capital costs. Additionally, the CVD process requires strict control of deposition parameters and growth conditions, increasing the complexity and energy consumption of the technological process. In contrast, the developed method allows for the production of nanostructures at similar temperatures but with lower energy costs and without the use of vacuum

systems.

The sol-gel method, commonly used for the synthesis of boron-containing nanomaterials, requires expensive precursors (BCl_3 , NH_3), whereas the proposed approach relies on affordable and cost-effective starting compounds (H_3BO_3 , $(\text{NH}_2)_2\text{CO}$), significantly reducing production costs.

The safety of the reagents used is also a critically important factor. CVD involves the use of toxic gaseous precursors (e.g., H_3NBH_3), while the sol-gel method utilizes easily hydrolyzable boron trichloride (BCl_3), requiring special storage and handling conditions. Unlike these methods, the proposed technique is based on low-toxicity reagents of the fourth hazard class (H_3BO_3 , $(\text{NH}_2)_2\text{CO}$), making it safer and more environmentally friendly.

Moreover, mechanochemical synthesis, despite its simplicity, often leads to the formation of particles with a wide size distribution and partially amorphous structures. In contrast, the proposed method ensures the production of nanoparticles with a narrow size distribution, high crystallinity, and controlled morphology, making it more suitable for technological applications.

Thus, the developed method combines high energy efficiency, economic feasibility, and safety, making it a promising candidate for further scaling and industrial implementation.

3.3. Limitations of this work and mitigation strategy

The controlled particle size, high crystallinity, and purity achieved through our synthesis method significantly enhance the performance of h-BN nanoparticles in several advanced technological fields. Specifically, these nanoparticles demonstrate great potential in the following applications:

Due to their high thermal conductivity, electrical insulation properties, and chemical stability, h-BN nanoparticles are ideal candidates for electronic devices, particularly in high-performance transistors and insulating layers. Recent studies have highlighted their use in enhancing thermal management for power electronics and high-frequency devices [62].

h-BN nanoparticles are widely utilized as fillers in polymer composites to improve mechanical strength, thermal resistance, and electrical insulation. Their high surface area and low density also make them suitable for enhancing the performance of lightweight structural materials [63]. These composites are particularly advantageous in the aerospace and automotive industries, where high-strength, heat-resistant materials are essential.

The unique lubricating properties of h-BN nanoparticles, especially at high temperatures, make them valuable for developing high-performance lubricants and coatings. They have been shown to significantly reduce friction and wear in mechanical systems, such as engine components and cutting tools [64,65]. Their exceptional stability under extreme conditions also makes them ideal for applications in space technology, where reliability and durability are critical.

The porous structure and high surface area of h-BN nanoparticles make them excellent support materials for catalytic reactions, such as hydrogenation and hydrodesulfurization processes. Their chemical

Table 2

Comparison of methods for synthesizing boron nitride nanostructures.

Parameter	Proposed method	CVD [60]	Sol-Gel [30]	Mechanochemical synthesis [61]
Temperature, °C	1000	1000	1200	Room temperature
Synthesis time, hours	4–8	6–12	4–8	12–24
Energy consumption, kWh/g	0.4–0.6	0.9–1.1	0.5–0.7	0.3–0.5
Raw material cost	Low (H_3BO_3 и $(\text{NH}_2)_2\text{CO}$)	High (H_3NBH_3)	High (BCl_3 и NH_3)	High ($\text{C}_3\text{H}_6\text{N}_6$, Mg, B_2O_3)
Hazard class of reagents	Class 4 (low hazard)	Class 3 (moderate hazard)	Class 2 (high hazard)	Class 4 (low hazard)
Crystallinity of BN, %	98	99.8	96–98	85–90
Morphology	Spherical	Layered	Fibrous	Nanosheets
Particle size	2–10 nm	25 μm (thickness)	50–200 nm	<100 nm
Equipment complexity	Simple	Complex	Complex	Simple

inertness in most environments further enhances their utility in catalysis [66].

These diverse applications demonstrate the versatility of h-BN nanoparticles in modern technology. The enhanced properties achieved through our modified synthesis method—including particle size control, high crystallinity, and exceptional purity—open new avenues for industrial use, particularly in electronics, advanced composites, lubrication systems, and catalysis.

While the use of common starting materials and a sustainable approach are promising, there are potential limitations to this work. They include particle size and distribution control, impurity levels, scale-up challenges, and characterization depth [67].

Achieving precise control over particle size and distribution might be challenging, affecting the material's properties and applications. The use of reagent-grade chemicals could introduce impurities that might impact the final product's performance. Transitioning from laboratory-scale synthesis to industrial production can introduce new challenges related to heat transfer, mass transfer, and equipment design. While TEM and XRD are valuable techniques, a more comprehensive characterization (e.g., XPS, Raman spectroscopy) might be necessary to fully understand the material's properties. Demonstrating the effectiveness of the synthesized nanoparticles in specific applications is crucial, but this might require additional research [59].

To mitigate them, optimization of synthesis parameters (temperature, time, reactant concentration) and the use of additives can help control particle size and distribution. Additionally, employing purification steps (washing, filtration) and using higher-purity starting materials can minimize impurities. Pilot-scale experiments and process modeling can aid in addressing scale-up challenges. Incorporating additional characterization techniques can provide a more detailed understanding of the material [68]. By addressing these limitations, the research can be further developed to produce high-quality boron nitride nanoparticles with a wider range of applications. The work not only advances the field of material science but also contributes to broader efforts to create more sustainable and environmentally responsible manufacturing processes.

The results demonstrate that the proposed method for synthesizing h-BN nanoparticles is both efficient and environmentally friendly. The two-step thermal process, using boric acid and urea as precursors, achieved a high degree of crystallinity (98 %) and particle uniformity with an average size of 6 nm. The primary mechanism of particle growth is attributed to controlled crystallization under specific thermal conditions and the use of gas atmospheres, which ensure uniform heat distribution and removal of reaction byproducts.

During the first thermal treatment at 600 °C, the intermediate product exhibited a significant amorphous phase. This can be attributed to the relatively low temperature, which was insufficient for complete crystallization. Partial crystallization was confirmed by XRD peaks corresponding to h-BN and B₂O₃. These results highlight the importance of further thermal treatment to complete the formation of a crystalline structure. The second stage of processing at 1000 °C in a nitrogen and hydrogen atmosphere resulted in the formation of homogeneous spherical h-BN nanoparticles. Hydrogen played a key role in grain boundary diffusion, enhancing crystallinity and reducing residual oxides. The high thermal stability and uniformity of the particles, as confirmed by TEM and HR-TEM analysis, were achieved under optimized processing conditions that minimized agglomeration and promoted controlled particle growth.

Atomic H diffuses along the grain boundary, promoting recrystallization and defect elimination [38]. This dual mechanism ensures the removal of oxygen impurities and improves the crystallinity of h-BN nanoparticles.

The proposed method avoids the use of toxic reagents, such as borazine and ammonia typically employed in conventional methods like CVD, thereby reducing environmental impact and improving process safety. Additionally, the high purity of the final product was achieved

through water washing to remove residual B₂O₃, as confirmed by XRD and SAED results. The layered structure of h-BN, with an interlayer spacing of approximately 3.3 Å, reflects strong van der Waals interactions between layers, contributing to the material's high mechanical strength and chemical inertness. These properties make the synthesized nanoparticles highly suitable for applications in electronics, catalysis, and composite materials.

4. Conclusion

The results of this study led to the development of an efficient method for the synthesis of hexagonal boron nitride (h-BN) nanoparticles using boric acid and urea as the starting precursors. The synthesis process yielded an intermediate product, which was a complex compound containing B, N, C, O, and H, which was confirmed by FTIR and XRD analyses. Scanning electron microscopy (SEM) analysis showed that the intermediate crystals were dendritic in shape and consisted of aggregated micrometer-sized particles. Amorphous BN nanoparticles with an average size of 10 nm were obtained. In the second step, at a temperature of 1000 °C in a nitrogen and hydrogen atmosphere, a crystalline BN product was obtained.

According to TEM, the obtained nanoparticles were spherical in shape with an average size of 6 nm. Further FTIR and XRD analyses showed that the product consisted of a mixture of h-BN and B₂O₃. B₂O₃ impurities were removed by washing with water. The final h-BN product was obtained from the washing solution by centrifugation and drying. The obtained product had a narrow particle size distribution, the average size of the h-BN nanoparticles was 6 nm. High-resolution TEM images (HR-TEM) demonstrated the layered structure of h-BN, consisting of alternating layers of boron and nitrogen atoms with a thickness of about 3.3 Å.

Surface electron diffraction (SAED) analysis confirmed that the atoms in the product structure have a hexagonal order. FTIR and XRD results also confirmed that the final product consists of BN with a hexagonal crystal structure and has a high purity. The crystallinity of the final product was 98 %, and no peaks associated with other boron or nitrogen compounds were detected. This method represents a significant step forward in h-BN nanoparticle production technology, offering a combination of high product purity, environmental safety and cost-effectiveness, making it promising for scalable industrial application.

CRediT authorship contribution statement

Odiljon Abdurakhmonov: Writing – original draft, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Mastura Aripova:** Validation, Supervision, Resources, Project administration. **Farrukh Erkinov:** Visualization, Validation, Resources, Methodology, Investigation. **Sherzod Abdurakhmonov:** Writing – original draft, Visualization, Validation, Methodology, Investigation, Data curation, Conceptualization. **Utkirjon Sharopov:** Writing – original draft, Visualization, Validation, Project administration, Methodology. **Mukhtorjon Karimov:** Writing – original draft, Resources, Project administration, Investigation, Funding acquisition. **Muzaffar Kurbanov:** Validation, Supervision, Software, Resources, Project administration, Methodology. **Dilmurod Saidov:** Validation, Supervision, Resources, Project administration, Methodology, Investigation. **Zbigniew Pędzich:** Writing – review & editing, Validation, Supervision, Software, Resources, Project administration. **Dawid Koziem:** Validation, Supervision, Software, Resources, Project administration. **Tonni Agustiono Kurniawan:** Writing – review & editing, Supervision, Software, Resources, Project administration. **Eka-terina Bondar:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources.

Informed consent

Not applicable.

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Research involving human participants and/or animals

Not applicable.

Funding

This research was supported by Ministry of Higher Education, Science and Innovation for financial support (project No. IL-402104388). This research received no other grant from any funding agency in the public, commercial, or not-for-profit sectors.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The analysis of samples by transmission electron microscopy was carried out at the Tashkent Chemical-Technological Institute in the “Scientific Laboratory of Physical and Chemical Analysis and Nanomaterials” using the HR-TEM Talos-F200i setup.

Data availability

Data will be made available on request.

References

- [1] C. Liu, Z. Zhou, J. Ji, Y. Gu, S. Yan, S. Yang, R. Zhang, Z. Liu, Y. Xue, C. Tang, Controllable preparation of boron nitride microspheres and their epoxy-based thermoconductive composites, *Ceram. Int.* 49 (2023) 27138–27146, <https://doi.org/10.1016/j.ceramint.2023.05.258>.
- [2] A. Kumar, G. Malik, K. Goyal, N. Sardana, R. Chandra, R.S. Mulik, Controllable synthesis of tunable aspect ratios novel h-BN nanorods with an enhanced wetting performance for water repellent applications, *Vacuum* 184 (2021) 109927, <https://doi.org/10.1016/j.vacuum.2020.109927>.
- [3] X. Zhou, C.H. Li, M. Jiang, T. Chen, W. Tang, Y. Ma, K. Xu, F. Yang, X. Zhang, L. Zhang, X. Zhang, Z. Zeng, B.S. Zhang, Large wafer-level two-dimensional h-BN with unintentional carbon doping grown by metalorganic chemical vapor deposition, *Vacuum* 213 (2023) 112083, <https://doi.org/10.1016/j.vacuum.2023.112083>.
- [4] K. Yang, Y. Feng, L. Liang, Z. Zhang, X. Chen, Q. Chen, Thermal conductivity and dielectric properties of EP composites filled by one-dimensional core-shell structured h-BN@SiO₂ fibers, *Ceram. Int.* (2024), <https://doi.org/10.1016/j.ceramint.2023.12.261>.
- [5] F. Wang, X. Zhang, Y. Du, Y. Ou, Q. Song, T. Liu, X. Wu, S. Tan, B. Wang, Overall fabrication of uniform BN interphase on 2.5D-SiC fabric via precursor-derived methods, *Vacuum* 230 (2024) 113727, <https://doi.org/10.1016/j.vacuum.2024.113727>.
- [6] Y. Shen, Z. Wu, Y. Li, Lightweight and thermally conductive thermoplastic polyurethane/hollow glass bead/boron nitride composites with flame retardancy, *Ceram. Int.* 48 (2022) 32748–32756, <https://doi.org/10.1016/j.ceramint.2022.07.199>.
- [7] N. Kostoglou, K. Polychronopoulou, C. Rebholz, Thermal and chemical stability of hexagonal boron nitride (h-BN) nanoplatelets, *Vacuum* 112 (2015) 42–45, <https://doi.org/10.1016/j.vacuum.2014.11.009>.
- [8] M. Zhu, Y. Shao, Y. Xin, D. Yang, X. Lu, H. Zhang, Anisotropic dielectric dispersions and thermal behaviors in highly textured BN thin films for heat self-dissipating electronics, *Vacuum* 225 (2024) 113207, <https://doi.org/10.1016/j.vacuum.2024.113207>.
- [9] S. Raadnu, S. Mahathanabodee, R. Tongsi, M. Morakotjinda, P. Wila, Comparison of dry sliding tribological behavior of SS 316L impregnated with MoS₂ vs h-BN solid lubricants: a statistical point of view, *Wear* 476 (2021) 203676, <https://doi.org/10.1016/j.wear.2021.203676>.
- [10] S.A. Aal, A.K. Alfuhaidi, Enhancement of hydrogen storage capacities of Co and Pt functionalized h-BN nanosheet: theoretical study, *Vacuum* 183 (2021) 109838, <https://doi.org/10.1016/j.vacuum.2020.109838>.
- [11] M. Mirzaee, A. Rashidi, A. Zolriasatein, M. Rezaei Abadchi, A simple, low cost, and template-free method for synthesis of boron nitride using different precursors, *Ceram. Int.* 47 (2021) 5977–5984, <https://doi.org/10.1016/j.ceramint.2020.10.171>.
- [12] A. Haque, J. Narayan, Conversion of h-BN into c-BN for tuning optoelectronic properties, *Mater. Adv.* 1 (2020) 830–836, <https://doi.org/10.1039/D0MA00008F>.
- [13] J. Liu, F. Jia, Y. Yang, L. Sang, T. Liu, X. Wang, H. Niu, B. Liu, BNQDs sensitized in-situ growth ZnO nanorod arrays for enhancing the gas sensing performances to acetone, *Vacuum* 216 (2023) 112443, <https://doi.org/10.1016/j.vacuum.2023.112443>.
- [14] R. Naresh Muthu, S.S.V. Tatiparti, Electrochemical behavior of cobalt oxide/boron-incorporated reduced graphene oxide nanocomposite electrode for supercapacitor applications, *J. Mater. Eng. Perform.* 29 (2020) 6535–6549, <https://doi.org/10.1007/S11665-020-05176-Z/FIGURES/7>.
- [15] W. Urbaniak, T. Majewski, I. Powazka, G. Smigielski, A.D. Petelska, Study of nano h-BN impact on lubricating properties of selected oil mixtures, *Materials* 15 (2022) 2052, <https://doi.org/10.3390/MA15062052>.
- [16] X. Yang, R. Zhang, J. Pu, Z. He, L. Xiong, 2D graphene and h-BN layers application in protective coatings, *Corrosion Rev.* 39 (2021) 93–107, <https://doi.org/10.1515/CORRREV-2020-0080/MACHINEREADABLECITATION/RIS>.
- [17] R.N. Muthu, S. Rajashabala, R. Kannan, Hydrogen storage performance of lithium borohydride decorated activated hexagonal boron nitride nanocomposite for fuel cell applications, *Int. J. Hydrogen Energy* 42 (2017) 15586–15596, <https://doi.org/10.1016/j.ijhydene.2017.04.240>.
- [18] R.N. Muthu, Synthesis of reduced graphene oxide/hexagonal boron nitride nanocomposite as efficient hydrogen-storage medium for fuel cell applications, *Phys. Chem. Res.* 12 (2024) 419–428, <https://doi.org/10.22036/pcr.2023.397652.2342>.
- [19] R.N. Muthu, S. Rajashabala, R. Kannan, Synthesis, characterization of hexagonal boron nitride nanoparticles decorated halloysite nanoclay composite and its application as hydrogen storage medium, *Renew. Energy* 90 (2016) 554–564, <https://doi.org/10.1016/j.renene.2016.01.026>.
- [20] R.N. Muthu, S. Rajashabala, R. Kannan, Facile synthesis and characterization of a reduced graphene oxide/halloysite nanotubes/hexagonal boron nitride (RGO/HNT/h-BN) hybrid nanocomposite and its potential application in hydrogen storage, *RSC Adv.* 6 (2016) 79072–79084, <https://doi.org/10.1039/C6RA13865A>.
- [21] S.K. Avinashi, N. Shweta, B. Bohra, R.K. Mishra, S. Kumari, Z. Fatima, A. Hussain, B. Saxena, S. Kumar, M. Banerjee, C.R. Gautam, Fabrication of novel 3-D nanocomposites of HAp-TiC-h-BN-ZrO₂: enhanced mechanical performances and in vivo toxicity study for biomedical applications, *ACS Biomater. Sci. Eng.* 10 (2024) 2116–2132, https://doi.org/10.1021/ACSBOMATERIALS.3C01478/SUPPL_FILE/AB3C01478_SI_001.PDF.
- [22] M. Llenas, L. Cuenca, C. Santos, I. Bdiqin, G. Gonçalves, G. Tobias-Rossell, Sustainable synthesis of highly biocompatible 2D boron nitride nanosheets, *Biomedicine* 10 (2022) 3238, <https://doi.org/10.3390/BIMEDICINES10123238/S1>.
- [23] N. Ploysongsri, V. Vchirawongkwin, V. Ruangpornvisuti, Hydrogen boride nanotubes and their C, N, O decoration and doping derivatives as materials for hydrogen-containing gases storage and sensing: a SCC-DFTB study, *Vacuum* 187 (2021) 110140, <https://doi.org/10.1016/j.vacuum.2021.110140>.
- [24] W. Han, J. Wang, S. Liu, C. Ge, S. Cao, B. Song, J. Wang, X. Zhang, Spectral properties of spherical boron nitride prepared using carbon spheres as template, *Ceram. Int.* 43 (2017) 3569–3575, <https://doi.org/10.1016/j.ceramint.2016.11.192>.
- [25] P. Arivazhagan, N.J. Usharani, M.J.S. Raju, S.S. Bhattacharya, Studies on drug carrier potential of spherical boron nitride nanoparticles in cancer therapy, *J. Mater. Sci.* 58 (2023) 8076–8091, <https://doi.org/10.1007/S10853-023-08543-Y/FIGURES/13>.
- [26] J.H. Oh, M. Kim, Y.H. Lee, S.H. Hong, S.S. Park, T.H. Kim, S. Choi, Synthesis of cobalt boride nanoparticles and h-BN nanocage encapsulation by thermal plasma, *Ceram. Int.* 46 (2020) 28792–28799, <https://doi.org/10.1016/j.ceramint.2020.08.042>.
- [27] A. Ben Belgacem, I. Hinkov, S. Ben Yahia, O. Brinza, S. Farhat, Arc discharge boron nitrogen doping of carbon nanotubes, *Mater. Today Commun.* 8 (2016) 183–195, <https://doi.org/10.1016/j.mtcomm.2016.08.001>.
- [28] B. Feng, J. Chen, Y. Yang, M. Yang, H. Wang, C. Zhong, Y. Hao, J. Yang, J. Jiao, Y. Yao, Fabrication of BN thin films by chemical vapor deposition on 4H-SiC (0001) single-crystalline surfaces, *Vacuum* 222 (2024) 113009, <https://doi.org/10.1016/j.vacuum.2024.113009>.
- [29] S. Dalui, A.K. Pal, Microstructural and optical properties of BN films deposited by inductively coupled plasma CVD, *Vacuum* 82 (2008) 1296–1301, <https://doi.org/10.1016/j.vacuum.2008.03.081>.
- [30] Y. Wang, H. Cheng, J. Wang, Effects of the single layer CVD BN interface on mechanical properties of oxide-oxide composites fabricated by a sol-gel process, *Ceram. Int.* 40 (2014) 7269–7277, <https://doi.org/10.1016/j.ceramint.2013.12.067>.

- [31] S.M. Queiroz, F.S. Medeiros, C.K.B. de Vasconcelos, G.G. Silva, H-BN nanosheets obtained by mechanochemical processes and its application in lamellar hybrid with graphene oxide, *Nanotechnology* 33 (2022) 035714, <https://doi.org/10.1088/1361-6528/ac20ff>.
- [32] E. Ünal, İ.H. Karahan, Production and characterization of electrodeposited Ni-B/hBN composite coatings, *Surf. Coat. Technol.* 333 (2018) 125–137, <https://doi.org/10.1016/j.surfcoat.2017.11.016>.
- [33] M.A. Domínguez-Crespo, E. Rodríguez, A.M. Torres-Huerta, I.J. Soni-Castro, S. B. Brachetti-Sibaja, R. Narro-García, A.B. López-Oyama, Production of BN nanostructures by pulsed laser ablation in liquids: influence of the applied Nd:YAG harmonics on the structural, optical and photoluminescence properties, *Ceram. Int.* 46 (2020) 21667–21680, <https://doi.org/10.1016/j.ceramint.2020.05.274>.
- [34] A.E. Naclerio, P.R. Kidambi, A.E. Naclerio, P.R. Kidambi, A review of scalable hexagonal boron nitride (h-BN) synthesis for present and future applications, *Adv. Mater.* 35 (2023) 2207374, <https://doi.org/10.1002/ADMA.202207374>.
- [35] R. Ma, Y. Bando, T. Sato, CVD synthesis of boron nitride nanotubes without metal catalysts, *Chem. Phys. Lett.* 337 (2001) 61–64, [https://doi.org/10.1016/S0009-2614\(01\)00194-4](https://doi.org/10.1016/S0009-2614(01)00194-4).
- [36] R.S. Al-Khafaji, F.Q. Mohammed, Effect of catalysts on BN NanoParticles production, *J. Mater. Res. Technol.* 9 (2020) 868–874, <https://doi.org/10.1016/J.JMRT.2019.11.026>.
- [37] R.S. Abdurakhmonov, S.A. Semenov, Y.V. Sokolova, Extraction of scandium from sulphuric acid-based red mud leach liquors, *Tsvetnye Met.* 2022 (2022) 33–39, <https://doi.org/10.17580/TSM.2022.04.04>.
- [38] Y. Chen, H. Liang, Q. Abbas, J. Liu, J. Shi, X. Xia, H. Zhang, G. Du, Growth and characterization of porous sp²-BN films with hollow spheres under hydrogen etching effect via borazane thermal CVD, *Appl. Surf. Sci.* 452 (2018) 314–321, <https://doi.org/10.1016/J.APSUSC.2018.04.217>.
- [39] H. Chen, Z. Yang, W. Guo, J.R. Dunlap, J. Liang, Y. Sun, K. Jie, S. Wang, J. Fu, S. Dai, From highly purified boron nitride to boron nitride-based heterostructures: an inorganic precursor-based strategy, *Adv. Funct. Mater.* 29 (2019) 1906284, <https://doi.org/10.1002/ADFM.201906284>.
- [40] H. Chen, Z. Yang, Z. Zhang, Z. Chen, M. Chi, S. Wang, J. Fu, S. Dai, Construction of a nanoporous highly crystalline hexagonal boron nitride from an amorphous precursor for catalytic dehydrogenation, *Angew. Chem. Int. Ed.* 58 (2019) 10626–10630, <https://doi.org/10.1002/ANIE.201904996>.
- [41] T. Hamieh, Study of surface thermodynamic properties of some boron compounds by inverse gas chromatography at infinite dilution, *Therm. Sci. Eng.* 6 (2023) 64, <https://doi.org/10.24294/TSE.V6I1.2052>.
- [42] J. Liu, Z. Zhang, C. Shi, Z. Ren, J. Peng, H. Zhou, Z. Liu, F. Meng, S. Zhao, Novel green chemical mechanical polishing of fused silica through designing synergistic CeO₂/h-BN abrasives with lubricity, *Appl. Surf. Sci.* 637 (2023) 157978, <https://doi.org/10.1016/J.APSUSC.2023.157978>.
- [43] F.O. Adeola, Global impact of chemicals and toxic substances on human health and the environment, https://doi.org/10.1007/978-3-030-05325-3_96-1, 2021.
- [44] O. Abdurakhmonov, U. Sharopov, S. Abdurakhmonov, Z. Kadirova, M. Karimov, M. Kurbanov, D. Saidov, Z. Iskandarov, S. Islamov, Production of High-Coercive nanostructured Nd-Fe-B alloy by chemical method, *J. Magn. Magn. Mater.* 600 (2024) 172130, <https://doi.org/10.1016/J.JMMM.2024.172130>.
- [45] P.M. Sudeep, S. Vinod, S. Ozden, R. Sruthi, A. Kukovec, Z. Konya, R. Vajtai, M. R. Anantharaman, P.M. Ajayan, T.N. Narayanan, Functionalized boron nitride porous solids, *RSC Adv.* 5 (2015) 93964–93968, <https://doi.org/10.1039/C5RA19091F>.
- [46] Q. Li, K. Zhang, X. Che, T. Gao, S. Wang, G. Ni, Preparation of BN nanoparticle with high sintering activity and its formation mechanism, *Molecules* 29 (2024) 3458, <https://doi.org/10.3390/MOLECULES29153458>.
- [47] U. Sharopov, Surface defects in wide-bandgap LiF, SiO₂, and ZnO crystals, <https://doi.org/10.1007/978-3-031-58850-1>, 2024.
- [48] R. Djabbarganov, B.G. Atabaev, Z.A. Isakhanov, U.B. Sharopov, Energy threshold of the atomic and cluster sputtering of some elements under bombardment with Cs, Rb, and Na ions, *J. Surface Invest.* 13 (2019) 640–643, <https://doi.org/10.1134/S1027451019040049/TABLES/2>.
- [49] U.B. Sharopov, O.A. Abdulkhaev, B.E. Egamberdiev, K.A. Samiev, N.M. Nazarova, M.K. Kurbanov, M.K. Karimov, D.S. Saidov, Z.I. Iskandarov, S.Y. Islamov, A. R. Kakhramonov, O.E. Abdurakhmonov, I.A. Pronin, A.S. Komolov, Defect Formation and displacement of atoms on the surface of a LiF crystal under bombardment with low-energy cesium ions, *J. Surface Invest.* 18 (2024) S302–S310, <https://doi.org/10.1134/S1027451024702215/TABLES/1>.
- [50] U. Sharopov, A. Abdusalomov, A. Kakhramonov, K. Rashidov, F. Akbarova, S. Turapova, M. Kurbanov, D. Saidov, B. Egamberdiev, A. Komolov, S. Pshenichnyuk, K. Kaur, H. Bandarenka, Comparative research fluorine and colloidal aggregate formation on the surface lithium fluoride thin films during electronic, ionic and thermal treatments, *Vacuum* 213 (2023) 112133, <https://doi.org/10.1016/J.VACUUM.2023.112133>.
- [51] H. Eslami-shahed, K. Nekouee, N. Ehsani, Synthesis of h-BN through spark plasma sintering with urea and boric acid as the starting materials, *Diam. Relat. Mater.* 124 (2022) 108915, <https://doi.org/10.1016/J.DIAMOND.2022.108915>.
- [52] O.E. Abdurakhmonov, M.E. Alisultanov, D.A. Vertaeva, A.G. Muradova, The effect of annealing temperature on crystallization of Nd₂O₃ nanoparticles synthesized by the deposition method, *Russ. J. Inorg. Chem.* 67 (2022) 1118–1124, <https://doi.org/10.1134/S0036023622070026>.
- [53] O.E. Abdurakhmonov, M.E. Alisultanov, Sh.E. Abdurakhmonov, A.G. Muradova, On the synthesis of α-Fe₂O₃ nanoparticles by the method of chemical deposition to obtain a magnetically hard Nd-Fe-B alloy, *Nanobiotechnol. Report.* 18 (2023) 226–232, <https://doi.org/10.1134/S2635167623700064>.
- [54] O.E. Abdurakhmonov, U.B. Sharopov, Sh.E. Abdurakhmonov, Z. Ch. Kadirova, New method of chemical synthesis for nanostructured Nd-Fe-B alloy, *J. Magn. Magn. Mater.* 589 (2024) 171562, <https://doi.org/10.1016/j.jmmm.2023.171562>.
- [55] Robert J. Hunter, *Foundations of Colloid Science*, Oxford University Press, 2001. https://books.google.com/books/about/Foundations_of_Colloid_Science.html?id=UVCAQAIAAJ. (Accessed 17 December 2024).
- [56] A. Suhail, I. Lahiri, Two-dimensional hexagonal boron nitride and borophenes, *Layer. 2D Adv. Mater. Allied Appl.* (2020) 303–336, <https://doi.org/10.1002/9781119655190.CH13>.
- [57] J. Men, B. Li, J. Li, G. Li, J. Chen, X. Hou, Amorphous liquid phase induced synthesis of boron nitride nanospheres for improving sintering property of h-BN/ZrO₂ composites, *Ceram. Int.* 46 (2020) 8031–8038, <https://doi.org/10.1016/j.ceramint.2019.12.027>.
- [58] O.E. Abdurakhmonov, D.A. Vertaeva, E.V. Yurtov, Chemical synthesis of magnetic materials of the Nd₂Fe₁₄B SiO₂ alloy of core-shell nanocomposites, *Lect. Notes Network Syst.* 252 (2023) 63–73, <https://doi.org/10.1007/978-3-030-78105-7/7/FIGURES/6>.
- [59] U. Sharopov, K. Samiev, A. To'raev, M. Kurbanov, M. Karimov, D. Saidov, F. Akbarova, S. Turapova, Z. Iskandarov, S. Islamov, A. Komolov, I. Pronin, H. Bandarenka, O. Abdurakhmonov, S. Abdurakhmonov, M. Srinivasan, K. Kaur, Exploring electron energy dependencies in the formation of surface charge on ZnO crystals, *Vacuum* 227 (2024) 113395, <https://doi.org/10.1016/J.VACUUM.2024.113395>.
- [60] N. Guo, J. Wei, Y. Jia, H. Sun, Y. Wang, K. Zhao, X. Shi, L. Zhang, X. Li, A. Cao, H. Zhu, K. Wang, D. Wu, Fabrication of large area hexagonal boron nitride thin films for bendable capacitors, *Nano Res.* 6 (2013) 602–610, <https://doi.org/10.1007/S12274-013-0336-4/METRICS>.
- [61] O. Torabi, M.H. Golabgir, H. Tajizadegan, A. Jamshidi, Mechanochemical behavior of magnesium–boron oxide–melamine ternary system in the synthesis of h-BN nanopowder, *Ceram. Int.* 42 (2016) 6450–6456, <https://doi.org/10.1016/J.CERAMINT.2016.01.084>.
- [62] S. Mateti, I. Sultana, Y. Chen, M. Kota, M.M. Rahman, Boron nitride-based nanomaterials: synthesis and application in rechargeable batteries, *Batteries* 9 (2023) 344, <https://doi.org/10.3390/BATTERIES9070344>.
- [63] M.G. Rasul, A. Kiziltas, B. Arfaei, R. Shahbazian-Yassar, 2D boron nitride nanosheets for polymer composite materials, *Npj 2D Mater. Appl.* 5 (1) (2021) 1–18, <https://doi.org/10.1038/s41699-021-00231-2>.
- [64] M.F. Bin Abdollah, H. Amiruddin, M.A. Azmi, N.A.M. Tahir, Lubrication mechanisms of hexagonal boron nitride nano-additives water-based lubricant for steel–steel contact, *Proc. IME J. J. Eng. Tribol.* 235 (2021) 1038–1046, <https://doi.org/10.1177/1350650120940173>.
- [65] W. Zhong, J. Dong, S. Chen, Z. Tong, The synergistic lubrication effects of h-BN and g-C₃N₄ nanoparticles as oil-based additives for steel/steel contact, *Materials* 16 (2023) 4979, <https://doi.org/10.3390/MA16144979>.
- [66] P. Rana, R. Dixit, S. Sharma, S. Dutta, S. Yadav, A. Sharma, B. Kaushik, P. Rana, A. Adholeya, R.K. Sharma, Enhanced catalysis through structurally modified hybrid 2-D boron nitride nanosheets comprising of complexed 2-hydroxy-4-methoxybenzophenone motif, *Sci. Rep.* 11 (1) (2021) 1–13, <https://doi.org/10.1038/s41598-021-03992-4>.
- [67] O. Hartal, A. Madinzi, S. Khattabi Rifi, C. Haddaji, T. Agustiono Kurniawan, A. Anouzla, S. Souabi, Optimization of coagulation-flocculation process for wastewater treatment from vegetable oil refineries using chitosan as a natural flocculant, *Environ. Nanotechnol. Monit. Manag.* 22 (2024) 100957, <https://doi.org/10.1016/J.ENMM.2024.100957>.
- [68] S.S. Albaser, H.E. Al-Hazmi, T.A. Kurniawan, X. Xu, S.A.M. Abdulrahman, P. Ezzati, S. Habibzadeh, H. Hollert, N. Rabiee, E.C. Lima, M. Badawi, M.R. Saeb, Microplastics in water resources: global pollution circle, possible technological solutions, legislations, and future horizon, *Sci. Total Environ.* 946 (2024) 173963, <https://doi.org/10.1016/J.SCITOTENV.2024.173963>.