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Amino Functionalization of Boron Nitride: Structural, Surface, and Colloidal Characterization

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ABSTRACT

Boron nitride (BN) has some advantageous characteristics such as good dispersion stability, low specific density, resilience to high temperatures, resistance to oxidation. Functionalized BN retains its crystalline structure, thermal and mechanical properties. Functionalized BN also shows some extra advantageous characteristics like more dispersion stability, surface reactivity, and biocompatibility due to the introduction of active functional groups. This study aimed to functionalize and characterize BN. BN was functionalized with amino groups using diazonium salt. The functionalized BN was characterized by FTIR, XRD, DLS and Zeta potential. FTIR ensured the formation of functionalized BN. XRD analysis ensured hexagonal structure with the crystal size 28.95 nm for BN and 31.33 nm for functionalized BN. DLS analysis showed the average particle size of 606 nm and 697 nm for BN and functionalized BN respectively. Zeta potential was measured -26.8 mV for BN and 25.3 mV for functionalized BN.

Keywords: Dispersion, Functionalization, Zeta potential

1. Introduction

Boron nitride (BN) is an advanced covalent material that has drawn significant attention from scientists and engineers due to its unique structural, physical, and chemical properties. It has a composition of 56.4% boron and 43.6% nitrogen. BN exists in four crystalline polymorphs. These are wurtzite, hexagonal , diamond shaped, and cubic [1]. Nanostructured forms such as boron nitride nanosheets and boron nitride nanotubes have opened exciting avenues for research, particularly in nanotechnology, electronics, and biomedical engineering [2]. In the 1990s, researchers found its distinct bonding between boron and nitrogen atoms which leads to extraordinary material properties. These include excellent thermal conductivity, wide bandgap, electrical insulation, strong oxidation resistance, chemical inertness, and neutron absorption capability [2]. As research advanced in the early 2000s, the chemical vapor deposition (CVD) method enabled large-scale synthesis of BN under high-temperature, high-pressure conditions. Then a detailed characterization was followed using analytical tools such as transmission electron microscopy (TEM), Raman spectroscopy, and Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM),. These studies provided critical insights into particle size, lattice structures, and bonding features [3-5]. Such progress has encouraged researchers to functionalize BN with different surface groups to enhance dispersibility in polar solvents and expand its potential applications. BN contains a combination of remarkable properties, which distinguish it from other ceramic and carbon-based nanomaterials. Its thermal stability allows BN to withstand extreme temperatures without losing structural integrity, making it highly suitable for thermal management systems. Unlike carbon-based nanostructures such as carbon nanotubes (CNTs), BN is an excellent electrical insulator, which is essential for microelectronic devices requiring both high thermal conductivity and dielectric stability [2]. BN also possesses a wide bandgap ranging from 5.5 to 6.2 eV, depending on its crystalline form, positioning it as an attractive candidate for optoelectronic and energy applications [3, 4]. Moreover, BN is resistant to oxidation at elevated temperatures: while single-layer BN nanosheets begin oxidizing near 850 °C, boron nitride nanotubes can tolerate up to 950 °C [2]. It is also able to absorb neutrons, making BN particularly relevant in nuclear shielding applications [6]. Collectively, these attributes underscore why BN has become a promising material across diverse research domains.

Over the past decades, multiple synthesis strategies have been developed for BN, each with specific benefits and limitations. These methods can be classified into two categories. One is top-down approaches. Such as mechanical or chemical exfoliation, molecular beam epitaxy, milling etc. The other is bottom-up approaches, Such as atomic layer deposition, physical vapor deposition, chemical vapor deposition, sol-gel Technique [5]. Among the bottom-up techniques, sol-gel, direct chemical synthesis, and co-precipitation methods are most widely studied. The sol-gel method provides better control over particle size, morphology, and crystallinity. It involves the transition of a colloidal solution (sol) into a gel phase through hydrolysis

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and condensation, followed by drying and thermal treatment to yield BN [7]. This method is operated at moderate temperatures and allows uniform dispersion of nanoparticles. Also, it is cost-effective, eco-friendly, and energy-efficient [8]. For instance, Kayani et al. synthesized BN thin films using sol-gel by employing boric acid (H_3BO_3), boron nitrate, ammonia (NH_3), and water, achieving stable BN structures [7]. Another approach, the direct chemical synthesis method, uses reactions between boron containing precursors and nitrogen sources at elevated temperatures. This method often requires high energy input and controlled reaction environments. The co-precipitation method is also significant. It enables large-scale synthesis of BN nanotubes through simultaneous precipitation of boron and nitrogen precursors, followed by calcination. Bi et al. successfully achieved this using boron-containing salts and ammonium-based precursors [9]. Though it is efficient, it often demands post-synthesis treatments to achieve desired purity and morphology. This makes the sol-gel highly attractive for BN synthesis at the laboratory scale. But pure boron nitride (BN) does not mix well with water or other organic solvents. So, researchers are trying to find ways to make BN dissolve better, stay stable, and good interaction with other materials. Functionalization has therefore become a key strategy [10, 11]. This can be done by bonding functional groups like amino, hydroxyl, or carboxyl to it directly. These are called covalent methods. Functionalization can also be done by non-covalent methods. These methods work without altering lattice structure. Non-covalent methods rely on π–π interactions, Van der Waals forces, or surfactant adsorption to modify BN. Between them covalent methods provide more stable and permanent modifications. Functionalized BN looks to be useful in a lot of ways. Like adding amino groups to BN nanosheets. Amino functionalization via diazonium salt chemistry has emerged as a particularly promising route. The introduction of amino groups not only improves solubility through hydrogen bonding but also provides reactive sites for further modification, enhances drug adsorption (e.g., doxorubicin in anticancer therapy), and shifts the zeta potential toward positive values, facilitating better stability and stronger interactions with biological membranes [10, 12]. In electronics, h-BN is valued as a dielectric substrate for graphene-based transistors, due to its atomic flatness and insulating behavior [4]. In biomedical science, BN nanostructures are being investigated as carriers for drug delivery, antimicrobial agents, and scaffolds for tissue engineering [7, 12]. Its antimicrobial potential stems from its ability to disrupt bacterial membranes through direct contact, suggesting its use in implant coatings and biomedical devices [7].

Wang et al. followed the covalent functionalization of boron nitride nanotubes (BNNTs) with aniline groups using in-situ generated diazonium salts, achieving a remarkable graft content of 71.4 wt.% and significantly improved dispersion in chloroform [10]. Their study proved amino surface functionalization provides better surface chemistry in boron nitride. Inspired by their work the present study followed amino functionalization through diazonium salt chemistry. BN was synthesized using sol-gel method in lab-scale [13]. Then characterization of BN nanoparticles in terms of size, morphology, and crystallinity using advanced techniques such as FTIR, XRD, DLS and zeta potential was conducted. Similar characterization techniques were followed for functionalized BN. By addressing these objectives, the study contributes to bridging the gap between fundamental BN research and practical applications, particularly in biomedicine and optoelectronics.

2. Materials and methodology

2.1 Materials

Boric acid (powder), liquid ammonia, ethanol, deionized water., phenylenediamine, sodium nitrite, iron powder, concentrated HCl.

2.2 Methodology

0.4 g of phenylene diamine was dissolved in 10 mL of deionized water in one beaker, while 0.25 g of sodium nitrite was dissolved in 10 mL of deionized water in a separate beaker. Both beakers were immersed in ice baths to ensure that the temperature remained below 5°C. Next, a total of 2.5 mL of hydrochloric acid (HCl) with a concentration of 35% was added gradually and in small amounts to the solution containing aniline. This addition resulted in the release of strong-smelling fumes. The mixture was then agitated at a speed of 800-1000 revolutions per minute for a duration of 30 minutes, leading to a decrease in temperature to 2°C. Afterwards, the sodium nitrate solution was cautiously added in little drops to the aniline mixture to avoid any disruption or bubbling, leading to the creation of a dark brown diazonium salt [10]. Individually, 0.1 g of boron nitride (BN) were evenly distributed in 10 ml of deionized water and subjected to sonication for a duration of 40 minutes. The solution of diazonium salt was gradually added to the dispersion of BN and agitated for 1 hour at a speed of 800-1200 rpm. Subsequently, a little quantity of HCl was added drop by drop. Following the stirring process, the mixture was sonicated for a duration of 10 minutes to inhibit agglomeration. Then the sample centrifuged for a total of 8 cycles, with each cycle lasting 10 minutes. This process continued until the pH of the sample, which initially started at 1.08, reached about 7. The silt was gathered and subjected to desiccation in an oven set at a temperature of 100°C for a duration of 16 hours [10]. Finally, the dried sample was put in mortar and pestle for milling. Finally, characterization test: FTIR, XRD, DLS and Zeta Potential.

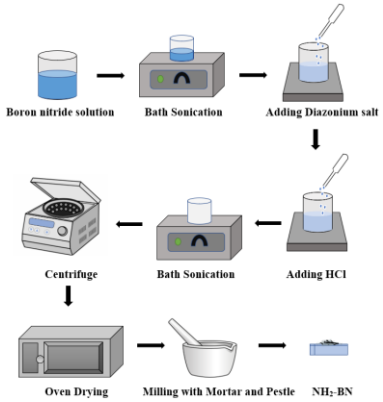

Fig. 1 Schematic of functionalization procedure**.**

3. Results and Discussion

The characterization result of boron nitride (BN) and amino-functionalized boron nitride (F-BN) are presented in this section. The samples were analyzed using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Dynamic Light Scattering (DLS), and zeta potential measurements. Key findings are detailed below with relevant equations and calculated values.

3.1 FTIR analysis:

Fourier Transform Infrared Spectroscopy (FTIR) is used to identify and study materials based on how they absorb infrared (IR) light. Different chemical bonds and functional groups absorb IR radiation at specific frequencies, producing a unique "fingerprint" spectrum for each material. FTIR spectroscopy was executed with NICOLET iSS, ThermoScientific.

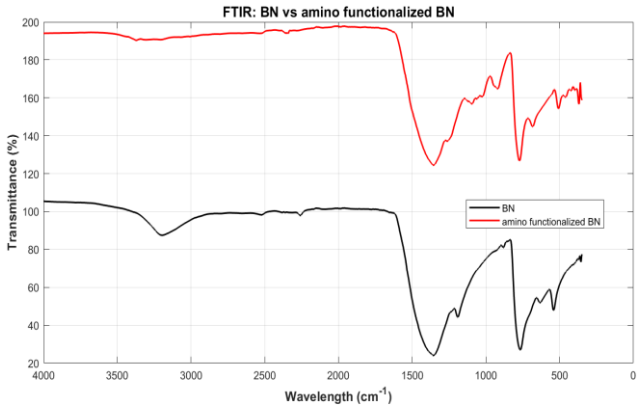

Fig. 2 FTIR analysis of BN and F-BN in same plot**.**

"Fig. 2" represents the characterization curve of FTIR spectra. The plot of BN showed two strong peaks at 1358 cm^{-1} and 772 cm^{-1}, which correspond to the stretching and bending vibrations of the B-N bond. Additionally, a broad peak around 3200 cm^{-1} indicated the presence of B-OH groups due to moisture absorption in both BN and F-BN plots. After functionalization, the FTIR spectra of F-BN exhibited a prominent new band at 3370 cm^{-1} corresponding to stretching vibrations of N-H group, and additional peaks at 1260 cm^{-1}, attributed to B-O-C which formed due to the reaction between anilino carbocation and B-OH. Another peak found in 684 cm^{-1} which corresponds to bending vibration of N-H group [14, 15]. The peak in 3064 cm^{-1} falls into C-H stretching of aromatic group. From the FTIR peaks, as shown in "Table 2" indicates better functionalization.

Table 1 Vibrational band assignments of BN**.**

FTIR peaks (cm^{-1})	Reference Values (BN) (cm^{-1}) [16]	Band assignments
3200	3200-3400	(O-H) stretching(oxidation)
1358	1378	B-N in-plane stretching
767	790	B-N-B out-of-plane bending

Table 2 Vibrational band assignments of F-BN

FTIR peaks (cm^{-1})	Reference Values (F-BN) (cm^{-1})[10]	Band assignments
3200	3200-3400	(O-H) stretching(oxidation)
3370	3500-3300	ν_{N-H} of amine group
3064	3000-3100	$\nu_{C=H}$ of benzene
684	600-750	γ_{N-H} of amine group
1260	1340-1250	γ_{C-N} of aromatic amine

3.2 XRD analysis and calculations:

The XRD belongs to Rigaku Smartlab with CuKα radiation at wavelength λ= 1.54 Å, scanning range 10°-80°, scanning speed 10°/min with 40 kV voltage and 40 mA applied current.

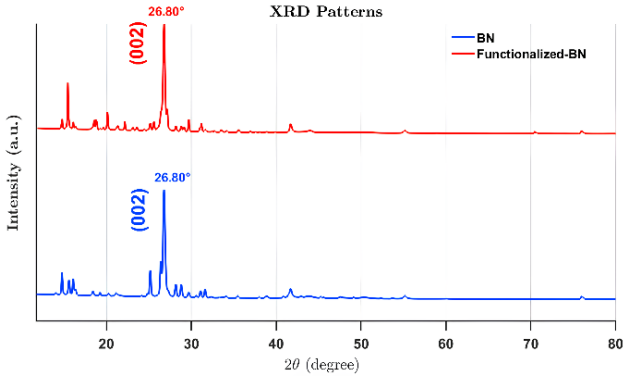

Fig. 3 XRD analysis curve for BN (blue) and F-BN (red) in a same plot.

In "Fig. 3", XRD spectra confirms that all diffraction peaks correspond to standard BN (JCPDS Card No. 85-1068), exhibiting a layered hexagonal structure. The maximum peak at 2θ = 26.8° matches the h-BN (002) plane, which is the strongest peak of h-BN (JCPDS Card No. 85-1068). The lattice constants of BN sample are in good agreement (As shown in "Table 3") with the reported values of h-BN, namely a = 2.51 Å and c = 6.6 Å (JCPDS No. 85-1068) [17, 18]. From "Fig. 3", maximum 2θ value is 26.80° for (002) crystal plane for both BN and F-BN. The full width at half maximum (FWHM) is 0.2944° or 5.14×10^{-3} radian for BN and 0.2723° or 4.75×10^{-3} radian for F-BN. The size of the crystallite was determined using the Scherrer equation, which is given by [19],

$$D = {k\lambda}/{\beta\cos\theta} \quad (1)$$

Here in Eq. (1), D is crystalline size, K is constant value of 0.94, β is the while width at half maximum in radian, θ is the Bragg angle of (002) peak, and λ is the wavelength of CuKα.

In Eq. (2), the degree of crystallinity is determined by the following formula [20],

$$\mathbf{X_C = 0.24/(\beta_{002})^2} \quad (2)$$

In Eq. (3), dislocation density (δ) [21],

$$\boldsymbol{\delta = 1/D^2} \quad (3)$$

In Eq. (4), the lattice constants a, c, and the inter-planner spacing can be calculated by [21],

$$\mathbf{\frac{1}{d_{hkl}} = \left[\frac{4}{3a^2}(h^2 + k^2 + hk) + \frac{l^2}{c^2}\right]^{-1/2}} \quad (4)$$

For, 002 plane (h, l, k)=(0, 0, 2). Eq. (4) can be written,

$$d_{hkl} = \frac{l}{c} \quad (5)$$

$$c = \frac{\lambda}{\sin\theta} \quad (6)$$

Thes train (ε) was given by in Eq. (7) [7],

$$\varepsilon = \frac{\beta\cos\theta}{4} \quad (7)$$

where β is in radian unit.
The required parameters of F-BN were calculated in the same way.

Table 3 Structural properties of BN and F-BN.

Parameters	BN	F- BN
Crystal dimensions, D (nm)	28.95	31.33
Degree of crystallinity, X_C	2.769	3.236
Dislocation density, $\delta\times10^4$ (nm^{-2})	12	10.2
Lattice constant, c (Å)	6.63	6.63
Interplanar spacing, d_{002} (Å)	0.3	0.3
Strain, $\varepsilon\times10^3$	1.25	1.16

3.3 DLS Analysis:

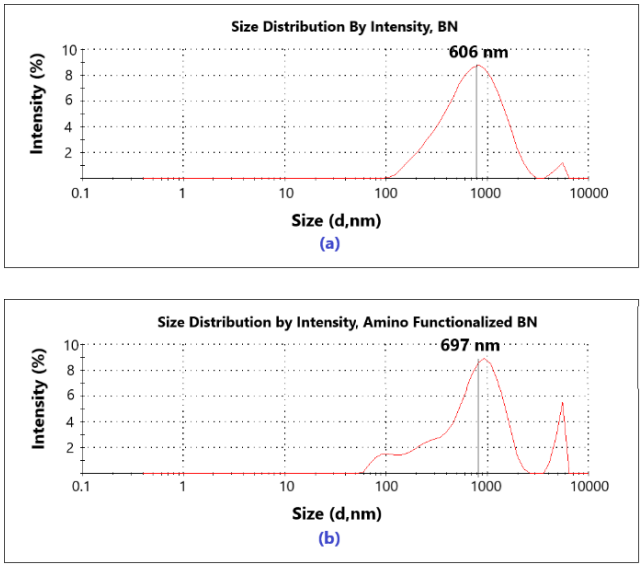

Fig. 4 (a) DLS analysis curve of BN, (b) DLS analysis curve of F-BN

"Fig. 4(a) and 4(b)" represent the DLS curve for BN and F-BN respectively. The hydrodynamic diameters of BN and F-BN were measured in aqueous suspension. BN exhibited an average size of 606 nm. After functionalization, the mean size increased to 697 nm. The increased size indicates the functionalization process. The bonding of functional groups like amino groups leads to an overall rise in the particle size. The functionalization has improved dispersion by reducing uncontrolled aggregation despite the slightly larger hydrodynamic size.

3.4 Zeta Potential Analysis:

The zeta potential (ζ) indicates the surface charge and stability of nanoparticles in suspension. Zeta potentials more than ±30 mV is considered for good physical stability of the ionic systems, whereas for non-ionic systems, values more than ±20 mV are considered adequate for colloidal stability of the systems [22]. "Fig. 5(a) and 5(b)" represent the zeta potential curve for BN and F-BN respectively. Pristine BN nanoparticles had a zeta potential of –26.3 mV. This indicates moderate colloidal stability and a slightly hydrophilic surface, although the basal planes of BN are mostly hydrophobic. Aggregation can still occur over time. After amino functionalization, the zeta potential shifted to +25.3 mV. This shows successful surface modification, increased hydrophilicity, and complete charge reversal. The positive charge improves dispersion in water and allows interaction with negatively charged biological membranes [23]. Functionalized BN is therefore useful for drug delivery and antimicrobial applications.

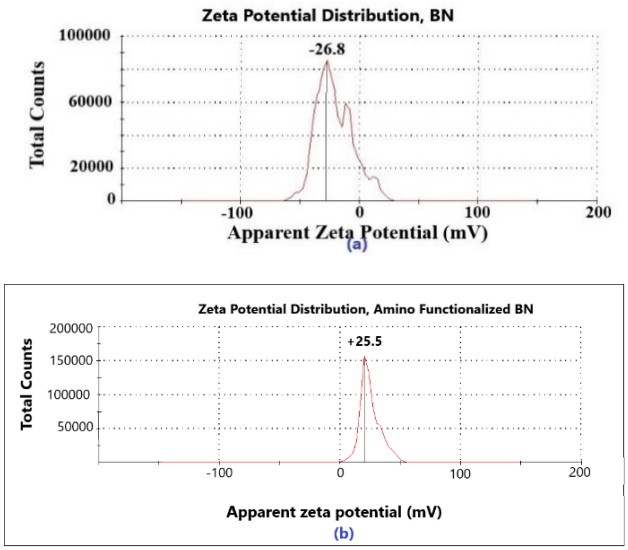

Fig. 5 (a) Zeta Potential Curve of BN, (b) Zeta Potential Curve of F-BN

3.5 Inside Mechanism of Diazonium salt Functionalization:

3.5.1 Protonation of p-phenylenediamine

$$NH_2\text{-}C_6H_4\text{-}NH_2 + HCl \rightarrow NH_2\text{-}C_6H_4\text{-}NH_3^+Cl^- \quad (8)$$

–NH_2 groups of p-phenylenediamine accepts a proton from HCl and becomes –NH_2.

3.5.2 Nitrosation reaction

$$NaNO_2 + HCl \rightarrow HNO_2 + NaCl \quad (9)$$

$$HNO_2 \rightarrow NO^+ + OH^- \quad (10)$$

Sodium nitrite reacts with HCl to form nitrous acid (HNO_2). Then HNO_2 decomposes to give nitrosonium ion (NO^+), which is the key reagent in diazotization.

3.5.3 Diazoniation reaction

$$NH_2\text{-}C_6H_4\text{-}NH_3^+Cl^- + NO^+ + OH^- \rightarrow NH_2\text{-}C_6H_4\text{-}N_2^+Cl^- + 2H_2O \quad (11)$$

Here, $-NH_2$ group reacts with NO^+ to form diazonium salt ($-N_2^+Cl^-$). This is the main intermediate in further reactions.

3.5.4 Functionalization reaction

$$C_6H_6(NH_2)_2 + NaNO_2 + 2HCl \rightarrow NH_2\text{-}C_6H_6\text{-}N{\equiv}N^+Cl^- + NaCl + H_2O \quad (12)$$

At low temperature (0-5°C), aromatic amine reacts with $NaNO_2$ and HCl to form diazonium chloride.

$$NH_2\text{-}C_6H_6\text{-}N{\equiv}N^+Cl^- \xrightarrow[\text{Acidic}]{\text{Hydrolysis}} NH_2\text{-}C_6H_6 + N_2 + Cl^- \quad (13)$$

The diazonium salt undergoes acidic hydrolysis, releasing nitrogen gas (N_2) and giving back an aromatic compound aniline.

$$BN + NH_2\text{-}C_6H_6 \rightarrow NH_2\text{-}C_6H_6\text{-}O\text{-}BN\text{-}NH_2\text{-}C_6H_6 \quad (13)$$

Finally, aniline bonds with BN. The oxygen in the product arises from the moisture content in BN sample.

4. Conclusion

This work was about making and improving hexagonal boron nitride (h-BN) particles in a simple and eco-friendly way. For this, the sol–gel method was chosen because it uses common and safe materials like boric acid, ammonia, and water. The method not only kept the cost low but also reduced harmful impact on the environment, which makes it suitable for both lab use and future large-scale applications. Since pure BN has low solubility and is not very active, we tried to modify it using diazonium salt. This step added amino groups to the surface, which improved its stability and ability to mix with water or other solvents. Different tests such as FTIR, XRD, DLS, and zeta potential confirmed that the functionalization was successful. The results of this work are not just for theory but also for real use. Functionalized BN can play an important role in medicine, for example in carrying drugs inside the body, fighting harmful bacteria, or even in gene-related treatments. It can also be useful in industries, like protecting metals from rust, managing heat in machines, or working as special coatings. In the end, this project reached its main goals and showed new possibilities. With more studies in the future like improving the production amount, trying other types of functional groups, and checking how safe it is for people and the environment, BN can become even more useful. This research proves that with simple methods and small changes, a common material can turn into something valuable for both science and industry.

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