

Article

Towards Hybrid 2D Nanomaterials: Covalent Functionalization of Boron Nitride Nanosheets

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Abstract

In contrast to the typically electrically conductive nanocarbon systems, boron nitride nanosheets (BNNs) are particularly attractive for the fabrication of polymers that require high thermal conductivity while maintaining electrical insulation. However, their tendency to aggregate and the weak interfacial interaction with the polymer matrix limit their effectiveness in enhancing thermal conductivity. To address these challenges, BNNs can be chemically modified to improve dispersion and compatibility within the matrix. Nonetheless, the inherent chemical inertness of boron nitride poses a significant obstacle to functionalization. In this work, we demonstrate the successful covalent modification of BNNs using three different silane coupling agents: (3-aminopropyl)dimethylmethoxysilane, (3-aminopropyl)diethoxymethylsilane, and (3-aminopropyl)trimethoxysilane. FT-IR, SEM/EDX, and WAXS confirm the successful functionalization and reveal that the number of alkoxy groups in the silane strongly influences siloxane network formation and the extent of surface coverage on BNNs.

Keywords: 2D nanomaterials; boron nitride nanosheets; surface functionalization; alkoxy silanes



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1. Introduction

With the rapid development of 2D materials, their demand for diverse applications is growing significantly. Alongside graphene and its derivatives, boron nitride nanosheets (BNNs), also called “white graphene”, have attracted intensive research interest. In contrast to graphene, which is typically electrically conductive, BNNs offer complementary properties as electrically insulating 2D nanomaterials. Their unmatched properties, such as a wide bandgap (about 6 eV) [1,2], outstanding thermal stability and conductivity [3–6], good chemical stability [7], great mechanical strength, and unique adsorption capability [8–10], make this material very promising for a variety of applications including high-performance electronic devices, thermal management, sensors, catalysts, dielectric substrates, and many more [11–18]. Moreover, BNNs can be superior to graphene and other 2D materials in various applications. For instance, the excellent thermal conductivity of graphene can be used in a variety of applications; however, its outstanding electrical conductivity can be of disadvantage in some. An example would be the polymer electronic packaging materials. With the rapid development of microelectronics, the high power of the integrated circuits presents severe heat dissipation issues. Thus, ensuring high thermal conductivity in the packaging materials is of critical importance. Moreover, these materials play an important role in the protection of the integrated circuits, and hence they should

also possess good mechanical properties, be lightweight, and have good electrical insulation characteristics. Graphene would be an ideal candidate, due to its light nature, excellent thermal conductivity, and good mechanical properties, but the electrical conductivity of graphene is not compatible with the desired insulation characteristics that are required for the electronic packaging process [19]. In this case, BNNSs can be used as an electrical insulating filler to increase the thermal conductivity of such material. Nonetheless, its poor dispersion within the polymer matrix and the development of weak interaction with the matrix present a major challenge. Similar issues have long been observed in graphene oxide and carbon nanotube systems, where surface functionalization strategies have been widely developed to improve compatibility and performance. Drawing on these parallels, BNNSs can likewise be chemically modified [19], for instance, through silane coupling agents, to reduce agglomeration and improve distribution within polymers. However, many factors play a significant role in the functionalization of boron nitride nanosheets. On the one hand, the inertness of this material makes chemical modification generally challenging [20–22]. On the other hand, factors such as oxidation under harsh conditions [23–30], reaction temperature, and the precise amount of water [31,32]—sufficient to initiate silane hydrolysis but not so high as to trigger undesired polymerization—further complicate the functionalization of BNNSs. Sun et al. reported the functionalization of boron nitride nanosheets with 3-aminopropyltriethoxysilane [33]. They characterized the surface chemical composition of the nanosheets via FT-IR and XPS. FT-IR showed peaks that were attributed to B-N, O-H, Si-O, and CH₂ vibrations, whereas XPS showed binding energies corresponding to B-N, B-O, C-O, C-N, C-Si, Si-OH, and Si-O-B, as well as Si-O-Si, proving the successful modification of boron nitride nanosheets with 3-aminopropyltriethoxysilane [33]. While these studies confirm successful surface modification, they do not systematically investigate how the number of alkoxy groups in the silane influences the formation of B–O–Si bonds versus Si–O–Si polymerization, which is critical for controlling surface coverage and siloxane network formation. Consequently, there is a lack of understanding of how silane structure—specifically, mono-, di-, or tri-alkoxy functionality—affects the functionalization efficiency.

This gap was addressed in this work by exploring three silanes with varying numbers of alkoxy groups, and we present the successful functionalization of BNNSs with three different alkoxysilanes: (3-aminopropyl)dimethylmethoxysilane (containing one alkoxy group, NH₂(CH₂)₃Si(CH₃)₂OCH₃), (3-aminopropyl)diethoxymethylsilane (two alkoxy groups, NH₂(CH₂)₃SiCH₃(OCH₂CH₃)₂), and (3-aminopropyl)trimethoxysilane (three alkoxy groups, NH₂(CH₂)₃Si(OCH₃)₃). The number of reactive groups influencing covalent functionalization and siloxane network formation was investigated. In addition to FT-IR, other techniques such as SEM/EDX, DLS, and WAXS were used to characterize the functionalized boron nitride nanosheets.

2. Materials and Methods

2.1. Materials

The boron nitride nanosheets (BNNSs) were produced via the liquid phase exfoliation (LPE) method, with a yield of 44% [34]. The hexagonal boron nitride bulk material (*h*-BN, 99.7%) was purchased from ESK Ceramics (3M Deutschland GmbH, Neuss, Germany) and was exfoliated in 2-propanol (99.9%), which was purchased from Carl Roth GmbH + Co. KG (Karlsruhe, Germany). The nitric acid (69%), which was used for the oxidation of the BNNSs, was purchased from VWR Chemicals (Vienna, Austria), and the sonication bath was the Emmi 40 from EMAG AG (Mörfelden-Walldorf, Germany), with a total maximum power of 580 W, ultrasonic frequency of 40 kHz, and a voltage of 230 V. (3-Aminopropyl)dimethylmethoxysilane (97%) was purchased from Fisher Scientific (Austria) GmbH (Vienna, Austria), (3-aminopropyl)diethoxymethylsilane (97%) from Sigma-Aldrich

Chemie GmbH (Steinheim, Germany), and (3-aminopropyl)trimethoxysilane (97%) was purchased from Alfa Aesar GmbH & Co KG (Karlsruhe, Germany).

2.2. Preparation of the Oxidized BNNSs

A total of 20 mg of BNNSs was dissolved in 10 mL nitric acid (69%) and sonicated in a sonication bath for 24 h at a bath temperature of 15 °C. The sample was then centrifuged for 10 min at 4500 rpm, and the nitric acid removed. The BNNSs were washed with distilled water twice, followed by two washes with dry toluene.

2.3. Preparation of the Functionalized BNNSs

A total of 40 mg of the oxidized boron nitride nanosheets (BNNS-OH) was dispersed in 40 mL dry toluene and heated up to 110 °C. Subsequently, 0.4 mL silane, which is approximately 1 wt% (based on the weight of the nanosheets) [35], was added to the solution, and the reaction was run for 4 h under reflux and inert gas atmosphere (N₂). Standard high-purity nitrogen from Linde Gas GmbH Austria (Purity > 99.9%), sufficient for typical Schlenk-type chemistry, was used to provide the inert atmosphere. After cooling to room temperature, the solvent was removed via a 10 min centrifugation at 2000 rpm, and the product was washed twice with fresh dry toluene. The centrifuge that was used was the Rotanta 460 with an angle rotor at 45° and an 18 cm radius from Hettich GmbH & Co. (Tuttlingen, Germany). The functionalized BNNSs were dried under nitrogen gas flow and stored in an inert gas atmosphere.

2.4. Characterization

The characterization techniques used in this study are commonly applied across a wide range of 2D nanomaterials, including graphene-based systems. The Fourier transform infrared spectroscopy (FT-IR) analyses were carried out on an ALPHA spectrometer from Bruker (Bruker Optik GmbH, Ettlingen, Germany), where KBr was used as the transmission standard. The FT-IR spectra were recorded from 4000 cm^{−1} to 400 cm^{−1}. The WAXS measurements were performed on the SAXSpoint 2.0 (Anton-Paar GmbH, Graz, Austria), which comprised a Primux 100 micro microfocus X-ray source operating at a wavelength of $\lambda = 0.154$ nm (Cu K α). The samples were measured as a dry powder, with an acquisition time of 60 s, 10 times. SEM micrographs and EDX spectra were collected using Tescan VEGA 3 SEM (Oxford Instruments plc, Abingdon, UK) with a tungsten source filament working at 20 kV. SEM images were obtained using the standard secondary electron (SE) detector, with a resolution of 5 μ m and a working distance of 15 mm, while EDX spectra were collected at a 0–10 keV scale. Before analysis, the samples were sputter-coated with gold. The DLS instrument included a goniometer, a diode laser working at $\lambda = 532$ nm (Coherent Verdi V5) with single fiber detection optics (OZ from GMP, Zürich, Switzerland), an ALV 7004 digital multi-tau real-time correlator (ALV, Langen, Germany), and an ALV/SO-SIPD/DUAL photomultiplier in pseudo-cross-correlation mode. The correlation functions were recorded and stored by using the AVL software package. The measurements were performed at a scattering angle of 90° and a temperature of 25 °C (10 measurements of 30 s each). The hydrodynamic radius was calculated using the optimized regulation technique software [36].

3. Results and Discussion

3.1. Oxidation

Graphene shares many structural and two-dimensional morphological similarities with boron nitride nanosheets (BNNSs), which makes functionalization strategies developed for nanocarbons directly relevant. For example, the oxidation of graphene through

methods such as the Hummers' process has been widely applied to introduce hydroxyl, epoxy, and carboxyl groups, enabling further chemical modifications and colloidal stabilization [37]. Mechanistic studies by Dimiev et al. [38] further illuminate how oxygen groups are incorporated into graphene's lattice at electron-deficient sites. In parallel, Goni et al. [34] demonstrate that sonication-assisted liquid-phase exfoliation—commonly used for graphene—is effective for processing other 2D materials, underscoring the broader applicability of such methods across nanocarbon-like systems. Although BNNSs are electrically insulating rather than conductive, their structural and chemical parallels suggest that similar oxidative and exfoliation approaches can be strategically adapted, with the primary obstacle being BN's greater chemical inertness.

To increase the reactivity of boron nitride, a pre-treatment step is necessary. The chemical inertness makes the oxidation step challenging, and it therefore requires harsh conditions. Based on previous work on boron nitride nanotubes [39], the BNNSs were treated with concentrated nitric acid (69%) in a sonication bath for approximately 24 h, followed by a washing process with distilled water. A strong oxidation with nitric acid allowed the OH groups to be chemically linked to the boron atom. Due to the high electronegativity of the nitrogen atom, the negatively charged OH groups would bond to the electron-poorer boron atom, which served as a Lewis acid. After washing with water, the water was then removed via centrifugation, and the BNNS-OH was washed twice with toluene. The insolubility of water in toluene (0.03% at 25 °C) protected the nanosheets from adsorbing too much water from the environment, as this first step of the functionalization was performed under atmospheric conditions. Figure 1 illustrates the FT-IR spectrum of the oxidized boron nitride nanosheets (BNNS-OH) in comparison to the untreated BNNSs.

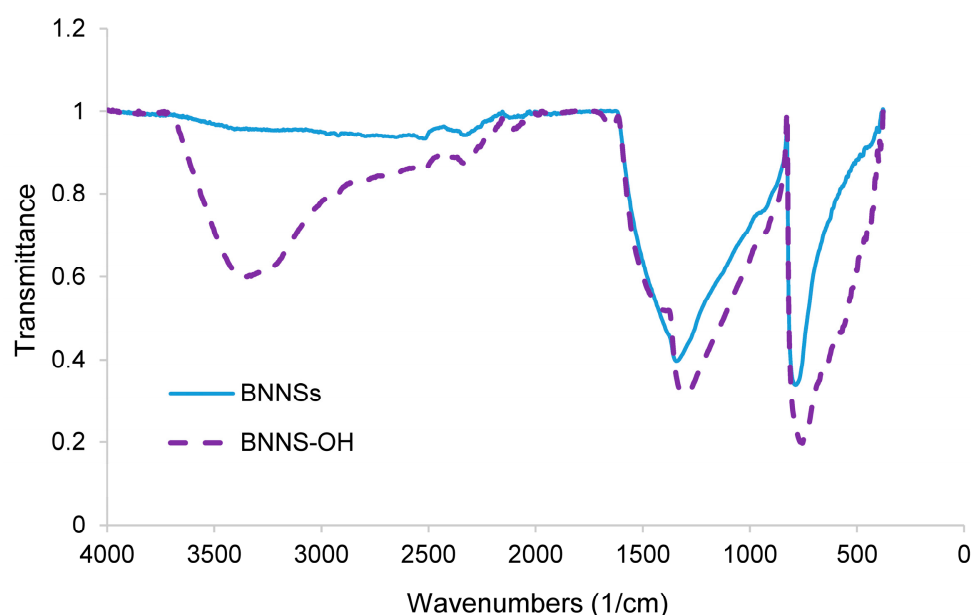


Figure 1. FT-IR spectrum of oxidized boron nitride nanosheets (BNNS-OH) in comparison to the untreated BNNSs. The broad band at around $3100\text{--}3500\text{ cm}^{-1}$ indicates the presence of OH groups.

The intense peaks at approximately 1350 cm^{-1} and 780 cm^{-1} corresponded to the B-N in-plane (stretching) and out-of-plane (bending) vibrations, respectively [40]. These peaks were clearly visible in both the untreated as well as the oxidized BNNSs, as expected. However, the FT-IR spectrum of BNNS-OH showed a new broad band that was quite evident at around $3100\text{--}3500\text{ cm}^{-1}$. This vibration is typical for hydroxyl groups, as previously reported in the literature [41,42]. Although this band might also appear due to traces of water that might still be in the system, the visible B-O vibration, though not as

apparent as the latter, is an indicator of the B-OH bond. The absorption band associated with the B-O stretching vibration was identified at 1370 cm^{-1} as well as at 570 cm^{-1} . These values were consistent with the spectroscopic data reported by Shurvell and Faniran [43]. The B-O vibrations were not as apparent as the B-N and the O-H, since they overlapped with the very intense B-N vibrations at 1350 cm^{-1} and 780 cm^{-1} . The small peaks at 2100 cm^{-1} , 2300 cm^{-1} , and 2500 cm^{-1} were seen in both spectra, and because they could not be assigned to any known vibrations within the BNNS-OH molecule, they were possibly artifacts. The FT-IR spectrum illustrated in Figure 1 clearly shows the successful oxidation of the boron nitride nanosheets. Furthermore, scanning electron microscopy (SEM) was used to determine the form and shape of the nanosheets. Since the sample was in its dry form and at a high concentration, the boron nitride nanosheets appeared to have flocculated, as can be observed in the SEM image in Figure 2. Nevertheless, their two-dimensional shape was well visible. Furthermore, the SEM image showed the polydispersity of the sample, demonstrating different radii and thicknesses of the boron nitride nanosheets. Monolayers were difficult to find due to their very thin nature, but few-layered as well as flocculated nanosheets are illustrated in Figure 2.

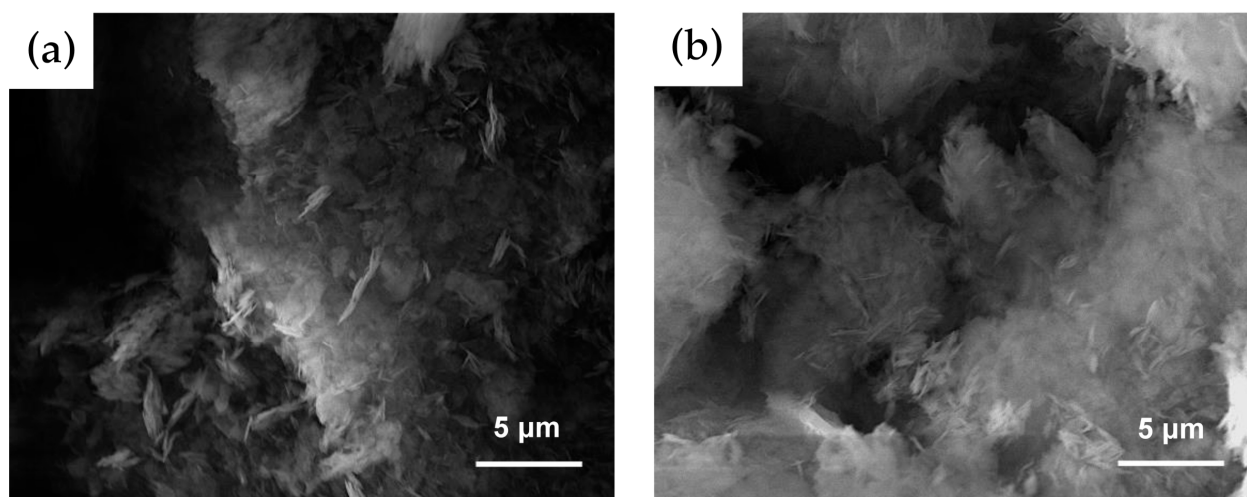


Figure 2. SEM image of (a) boron nitride nanosheets (BNNSs) and (b) oxidized boron nitride nanosheets (BNNS-OH).

A few nanoscrolls [44] were also observed. However, there is no explanation why this occurs. It might be due to a specific diameter-to-thickness ratio, or some external stimulus. The nanoscrolls observed in the BNNS image (Figure 2a) were still visible even after oxidation, as shown in Figure 2b. Furthermore, Figure 2b illustrates the different sizes of the nanosheets, with the few-layered and thinner BNNS-OH being distinguished from the rest due to the very weak contrast between them and the background. They were slightly visible, but difficult to detect nonetheless. Figure 2 demonstrates the two-dimensional character of BNNSs, which remained unaltered even after oxidation. As illustrated in Figure 2b, the oxidized BNNSs also exhibited a 2D shape. Their flocculation appeared to be stronger, but this might have also been due to a higher concentration of the sample on the carbon tape.

In addition to SEM, energy-dispersive X-ray spectroscopy (EDX) was also performed. EDX spectra of the non-modified BNNSs as well as the oxidized ones were collected to confirm that there were no other elements contained other than boron and nitrogen, as well as oxygen in the case of BNNS-OH.

Figure 3 demonstrates the elements that are contained in BNNSs and BNNS-OHs, with boron and nitrogen exhibiting the most intense peaks, and oxygen (Figure 3b), which came

from the OH groups that were attached to the boron atom, being detected in the oxidized boron nitride. The EDX data are in good agreement with the FT-IR results, indicating the successful activation of the boron nitride nanosheets.

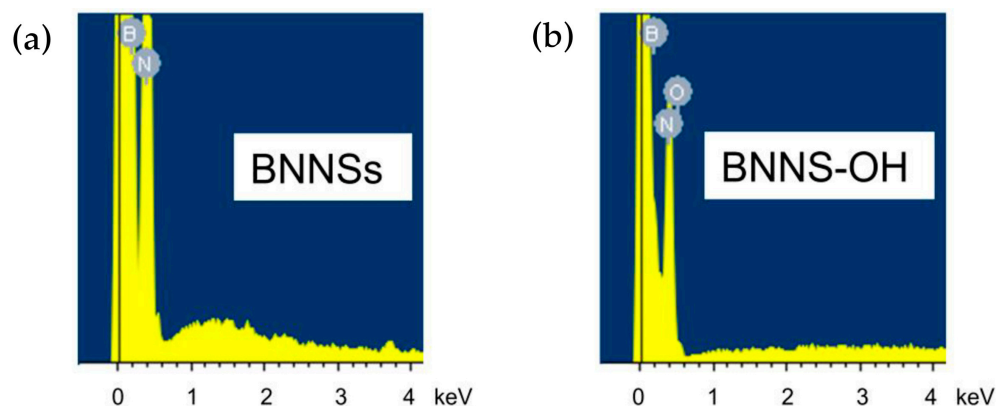


Figure 3. (a) EDX spectrum of BNNSs. Boron and nitrogen that are contained in the boron nitride nanosheets were clearly detected. (b) EDX spectrum of BNNS-OH. In addition to boron and nitrogen, oxygen was also detected.

3.2. Silanization

Since graphene and boron nitride share a hexagonal lattice, functionalization challenges such as low reactivities, edge/surface modifications, and dispersibilities are directly comparable. The hydroxyl groups that were introduced to the BNNSs make this material more reactive and enable further functionalization with silane agents. Three different alkoxy silanes were used for the chemical modification of the BNNSs:

- (a) (3-aminopropyl)dimethylmethoxysilane (also called Methoxy for simplicity reasons),
- (b) (3-aminopropyl)diethoxymethylsilane (also called Diethoxy),
- (c) (3-aminopropyl)trimethoxysilane (also called Trimethoxy).

These variable silanes were chosen to determine the effect that the different number of alkoxy groups has on the functionalization of the boron nitride. The more alkoxy groups, namely, the more reactive sites the silane has, the easier it is for it to polymerize and form siloxane networks, hence prohibiting the covalent bonding of the silane to the boron nitride. The amount of water in the system also plays a crucial role in the formation of these networks. The more water there is, the higher the probability that the silanes, especially those with two or three alkoxy groups, will react with each other, forming the siloxane network rather than reacting with the oxidized BNNSs. Therefore, the reaction was performed under an inert gas atmosphere, and the same reaction conditions were used for all three silanes.

Figure 4 is a model representation of the BNNS functionalization. The BNNSs are illustrated as multi-layered nanosheets to be in accordance with the polydispersity of the samples.

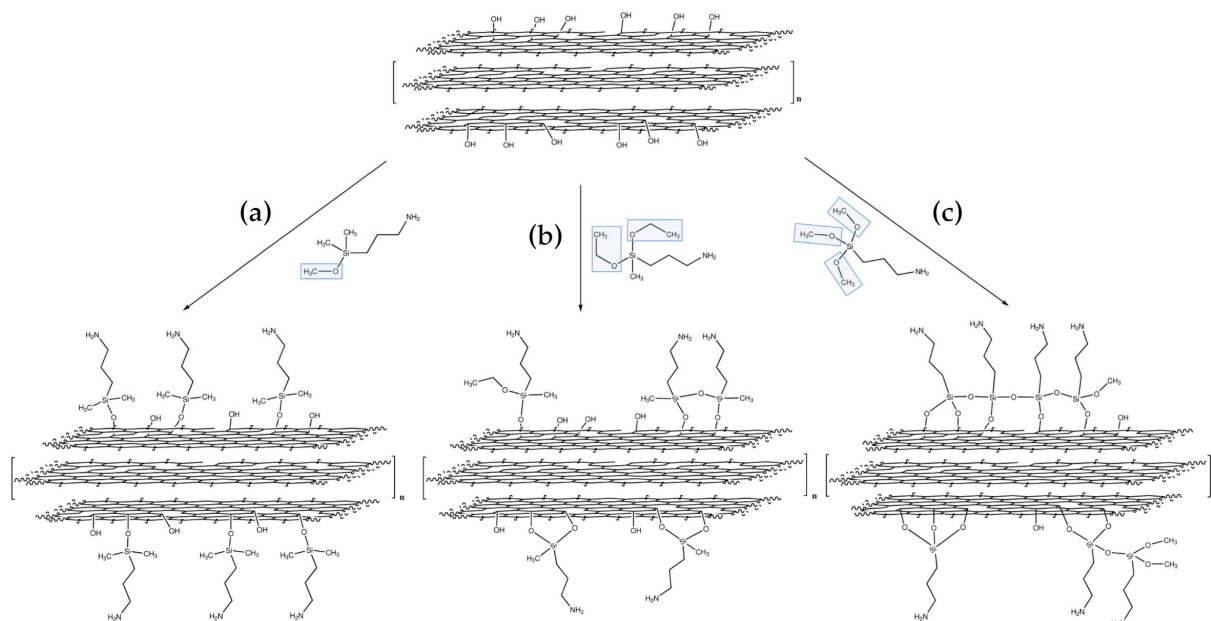


Figure 4. Functionalization of BNNSs with (a) (3-aminopropyl)dimethylmethoxysilane, (b) (3-aminopropyl)diethoxymethylsilane, and (c) (3-aminopropyl)trimethoxysilane.

The silane had to be hydrolyzed first before it could covalently bond to the surface of the boron nitride. The hydrolysis was enabled by a small amount of water molecules that were transported into the system with the BNNS-OH, which was stored in the atmosphere before the silanization reaction. This water was necessary to hydrolyze the alkoxy silanes into silanols. Nevertheless, the water content should be controlled, and therefore the reaction was performed under dry conditions in an inert gas atmosphere so that no more water could be adsorbed from the air. As it has been previously reported, the silanization reaction is highly sensitive to water [31,45–49]. The silanols were then bound to the boron atom through elimination of water. Some disiloxanes that might have been formed during the reaction were ultimately removed by washing the product with toluene, which was then dried under nitrogen gas flow. Figure 4a shows the functionalization of the oxidized BNNSs with (3-aminopropyl)dimethylmethoxysilane. In this case, there was only one active methoxy group and therefore only one way the silane could bond to the BNNSs. Due to having a single reactive site, this silane forms a covalent B-O-Si bond efficiently, minimizing siloxane network formation and maximizing functionalization at the BNNS surface. On the other hand, when the silane contained more than one reactive alkoxy group (Figure 4b,c), there were a few ways that it could bond to the boron nitride nanosheets. In Figure 4b, two sites of the (3-aminopropyl)diethoxymethylsilane formed B-O-Si bonds to the boron nitride. However, due to the higher number of reactive alkoxy groups, it was highly possible that the silanes would also form Si-O-Si bonds with each other, leading to the formation of disiloxanes on the surface of the boron nitride. In the case of the (3-aminopropyl)trimethoxysilane, there were three reactive methoxy groups. The oxygen could bond either to the boron or to the silicon of another silane, whose oxygens could also bond to yet another silane, and so on. This could lead to polymerization and formation of a siloxane network. If this network was not covalently bound to the boron nitride through a B-O-Si linkage, it could be washed away after the reaction. However, if it was bound to the nanosheet, it could form a siloxane layer on top of the boron nitride. Another possibility is that all three oxygens could form covalent bonds with the boron atoms of the BNNSs, or only two of them would covalently bond to the boron nitride, but the third one could either form a siloxane bond to the next silane or exist still as an unreacted

methoxy group. There is more than one possibility of how the silane can bond to the boron nitride, from bonding completely with all three oxygens, as well as partially with only one or two oxygens, to forming disiloxanes or siloxane layers. The results indicate that (3-aminopropyl)dimethylmethoxysilane, with only one reactive methoxy group, is the most effective silane for the covalent functionalization of BNNSs, as it minimizes undesired siloxane formation and maximizes B-O-Si bonding.

The functionalized BNNSs were characterized by FT-IR, illustrated in Figure 5.

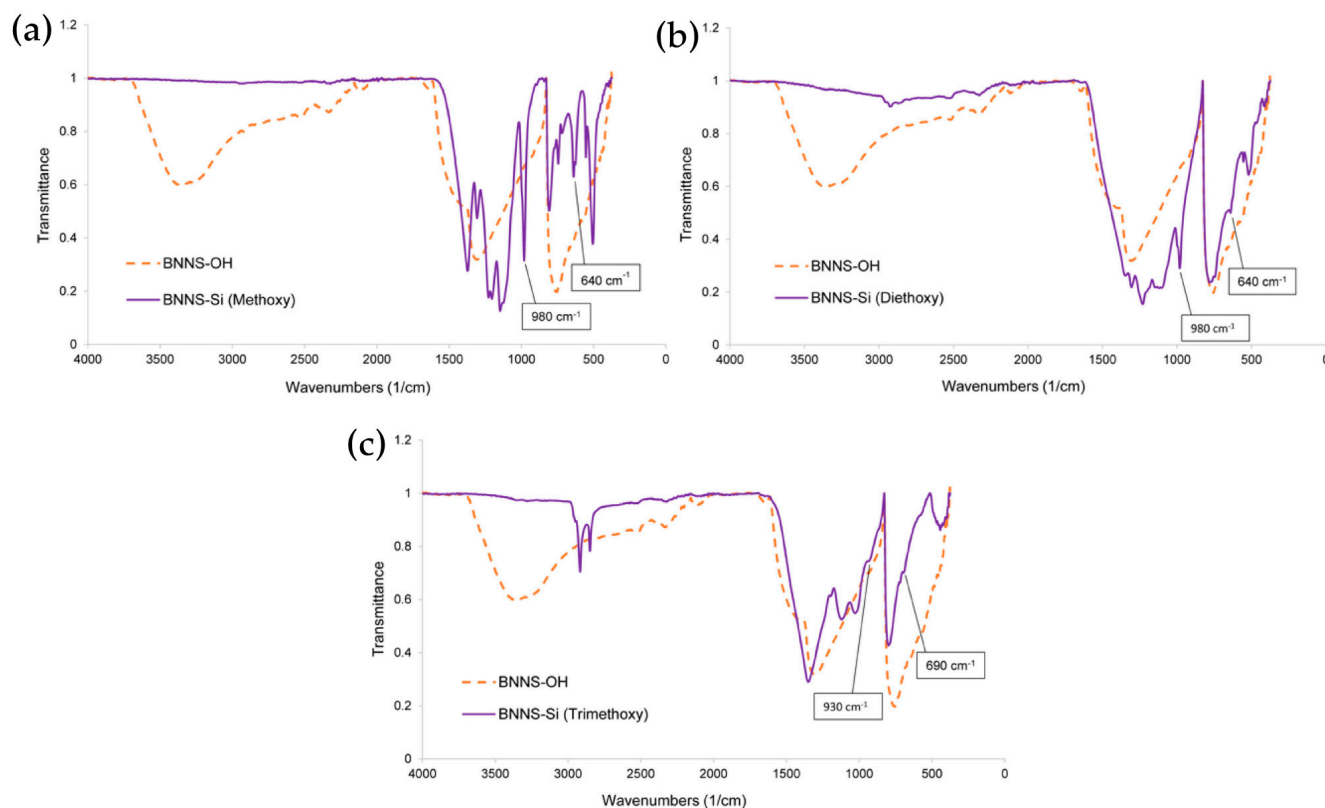


Figure 5. FT-IR spectra of the boron nitride nanosheets that are functionalized with (a) (3-aminopropyl)dimethylmethoxysilane, (b) (3-aminopropyl)diethoxymethylsilane, and (c) (3-aminopropyl)trimethoxysilane, in comparison to the BNNS-OH.

The vibrations demonstrated in Figure 5 are summarized in Table 1, shown below.

Table 1. FT-IR vibrations of BNNSs that are functionalized with (3-aminopropyl)dimethylmethoxysilane, (3-aminopropyl)diethoxymethylsilane, and (3-aminopropyl)trimethoxysilane.

FT-IR Vibrations/(cm ⁻¹)			
	BNNS-Si (Methoxy)	BNNS-Si (Diethoxy)	BNNS-Si (Trimethoxy)
B-N	1350, 780	1350, 780	1350, 780
Si-CH₃	1260, 750	1300	-
Si-CH₂	1220, 1200	1220	-
Si-O	1150	1140	1150
Si-O-Si	-	1100, 460	1120, 1020, 440
B-O	530, 505	530, 510	580
B-O-Si	980, 640	980, 640	930, 690

As expected, the B-N vibrations were visible at approximately 1350 cm^{-1} as well as 780 cm^{-1} [40]. In Figure 5a, the two small but sharp peaks at 1260 cm^{-1} and 750 cm^{-1} were associated with Si-CH₃ vibrations that stem from the methyl groups on the silane [50]. A further vibration that arises from the silane was that of the Si-CH₂ bond, and it was visible as a sharp band split into two components at around 1200 cm^{-1} and 1220 cm^{-1} , corresponding to the propyl chain [50]. The strong band at around 1150 cm^{-1} was attributed to the Si-O vibration [51], and the vibration bands at 530 cm^{-1} and at 505 cm^{-1} were associated with the B-O bond [43]. However, the main indicator that the silane was bound covalently to the boron nitride and not just adsorbed on the surface was the sharp band at 980 cm^{-1} and the one at 640 cm^{-1} , which corresponded to the B-O-Si vibration [52–55]. The high intensity of this band showed the successful functionalization of BNNSs with (3-aminopropyl)dimethylmethoxysilane. The FT-IR spectrum demonstrated in Figure 5b showed a broad band at approximately 1100 cm^{-1} , which masked many of the other bands near that area. This strong band was attributed to a Si-OCH₂CH₃ vibration [50] or a Si-O-Si bond. The Si-O-Si absorption was also observed at 460 cm^{-1} as a weak band. This indicated that the silanes bond to the boron nitride, as well as to each other, forming siloxanes. Furthermore, the presence of the Si-OCH₂CH₃ vibration showed that not all alkoxy groups had reacted to bond to the BNNSs or another silane. The Si-CH₃ and Si-CH₂ vibrations appeared at 1300 cm^{-1} and 1220 cm^{-1} , respectively [50], and the B-N vibrations were visible at 1350 cm^{-1} and 780 cm^{-1} , as expected [40]. Moreover, Figure 5b showed the B-O absorption at 510 cm^{-1} and 530 cm^{-1} [43], as well as a weak band at around 2920 cm^{-1} , which was attributed to the primary amine. The B-O-Si bond was demonstrated by a sharp absorption band at 980 cm^{-1} as well as a weaker one at approximately 640 cm^{-1} [52–55]. The B-O-Si vibration once again showed the successful functionalization of BNNSs with (3-aminopropyl)diethoxymethylsilane. In contrast to Sun et al., whose FT-IR did not display the peaks corresponding to the B-O-Si vibration [33], we were able to identify this vibration in all three syntheses. In Figure 5c, the amine vibration was shown at 2900 cm^{-1} , together with a sharp band at 2840 cm^{-1} , which could be attributed to the Si-O-CH₃ vibration [50], indicating the presence of some unreacted methoxy groups. The weak band that is almost masked by the broad B-N vibration at 1150 cm^{-1} was attributed to the Si-O bond, and the apparent bands at 1120 cm^{-1} , 1020 cm^{-1} , and 440 cm^{-1} were attributed to the siloxane Si-O-Si bond [50]. The B-O-Si vibration that represented the covalent functionalization of the BNNSs was displayed as a weak band appearing as a shoulder at 930 cm^{-1} and 690 cm^{-1} [52–55]. In comparison to the other two reactions, this FT-IR spectrum showed that the silane was bound more in the form of a siloxane network on top of the boron nitride nanosheet, which was expected due to the high number of the reactive methoxy groups. This led to a slight increase in size, as demonstrated by dynamic light scattering (DLS). Figure 6 illustrates a comparison of the hydrodynamic radius of the BNNSs, the oxidized boron nitride nanosheets (BNNS-OH), as well as the functionalized BNNSs with all three different alkoxysilanes.

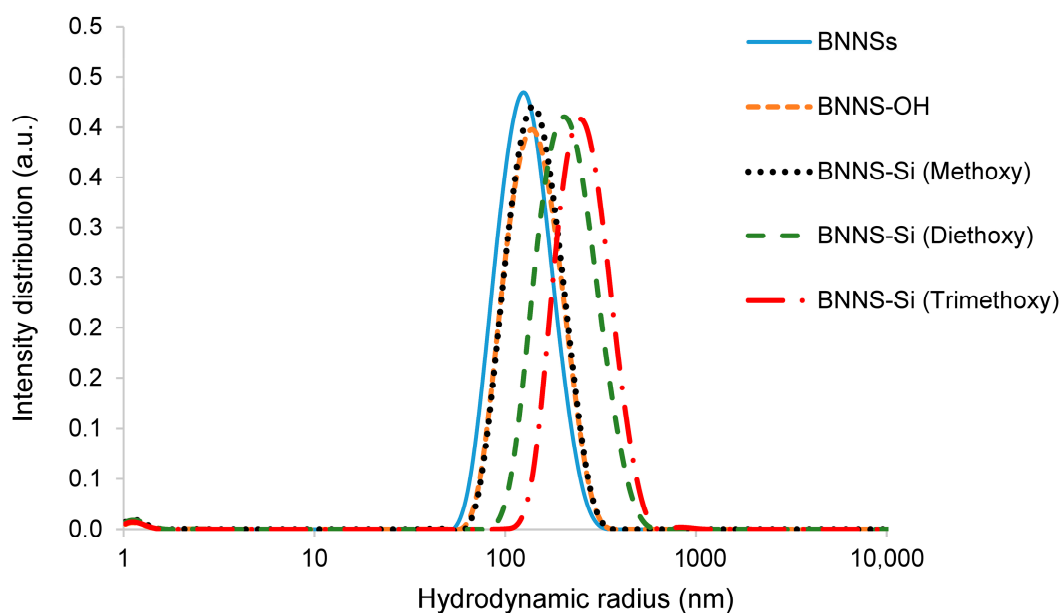


Figure 6. A comparison of the hydrodynamic radius of the BNNSs, the oxidized boron nitride nanosheets (BNNS-OH) and the functionalized BNNSs (BNNS-Si), as determined by DLS.

The average hydrodynamic radius of the boron nitride nanosheets that were used for the functionalization of the BNNSs was around 140 nm. The hydrolysis of the BNNSs did not lead to agglomeration and hence an increase in size, as the average hydrodynamic radius of BNNS-OH was also around 140 nm. These nanosheets were then used for the reaction with three different alkoxy silanes, which led to three different sizes of the functionalized BNNSs. The BNNSs that were functionalized with (3-aminopropyl)dimethylmethoxysilane (BNNS-Si (Methoxy)) showed an average hydrodynamic radius of approximately 140 nm, as illustrated in Figure 6, which was the same as the non-modified boron nitride. On the other hand, the functionalization with the other two silanes led to a slight increase in the size of the nanosheets. The BNNSs that were functionalized with (3-aminopropyl)diethoxymethylsilane (BNNS-Si (Diethoxy)) exhibited an average hydrodynamic radius of approximately 230 nm, whereas those functionalized with (3-aminopropyl)trimethoxysilane (BNNS-Si (Trimethoxy)) had an average hydrodynamic radius of approximately 270 nm. This increase in size was ascribed to the formation of a siloxane layer on the surface of the boron nitride, which was consistent with the results from infrared spectroscopy. The increase in size was more significant in the case of BNNSs that were functionalized with the silane with three methoxy groups, as these formed a bigger siloxane network than the one with only two ethoxy groups. This siloxane network was visible in the SEM images, as illustrated in Figure 7. There was no apparent difference between Figure 7a, which demonstrates the functionalized boron nitride nanosheets with (3-aminopropyl)dimethylmethoxysilane (BNNS-Si (Methoxy)), and Figure 7b, which demonstrates the functionalized BNNSs with (3-aminopropyl)diethoxymethylsilane (BNNS-Si (Diethoxy)). There were no visible spherical aggregates, but rather the nanosheets were flocculated and formed nanoscrolls. However, the BNNSs that were functionalized with (3-aminopropyl)trimethoxysilane (BNNS-Si (Trimethoxy)) displayed a different form, as illustrated in Figure 7c.

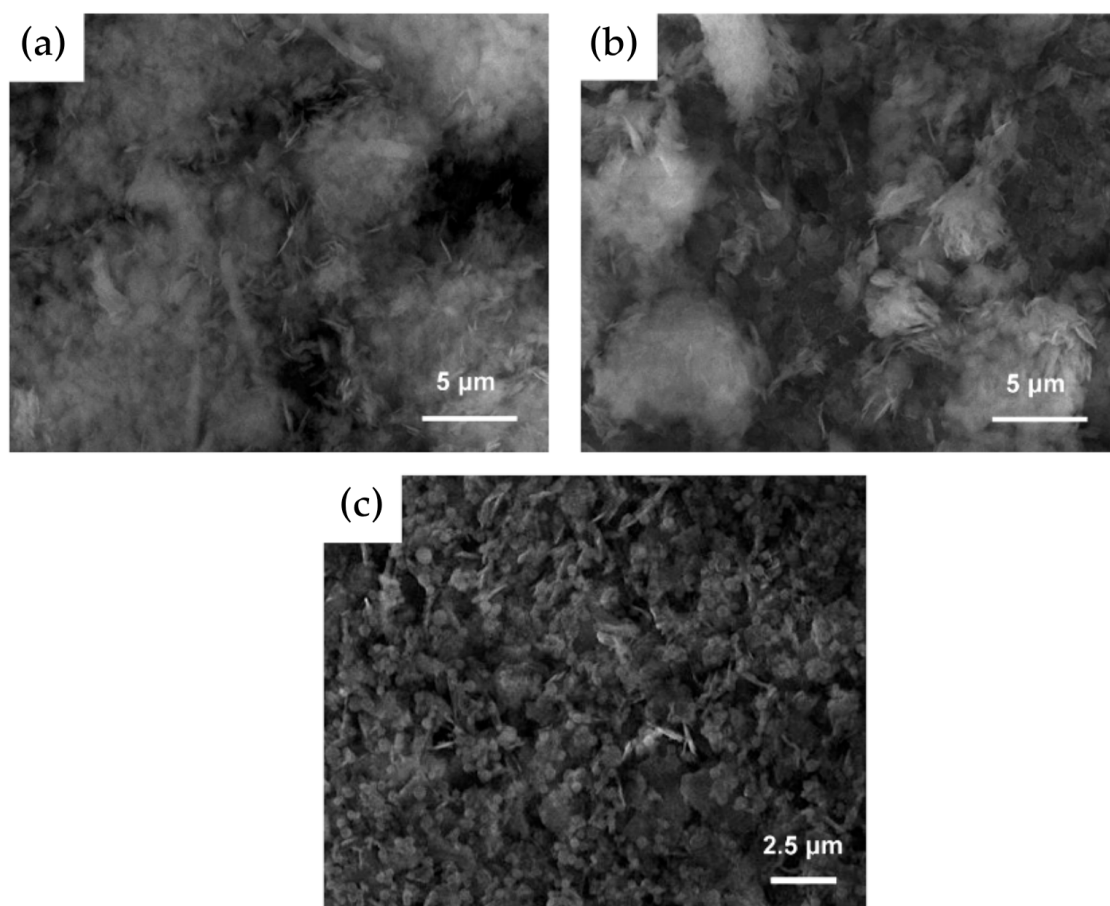


Figure 7. SEM images of the functionalized BNNSs with (a) (3-aminopropyl)dimethylmethoxysilane, (b) (3-aminopropyl)diethoxymethylsilane, and (c) (3-aminopropyl)trimethoxysilane.

Figure 7c demonstrates round-shaped nanoparticles on top of the 2D nanosheets. This was in good agreement with the previous results that showed the formation of siloxane networks. The high number of reactive alkoxy groups of the (3-aminopropyl)trimethoxysilane easily led to polymerization. These siloxane networks were bound to the surface of the BNNSs and were represented as small spheres on top of the nanosheets. Although the siloxane formation occurred during functionalization with (3-aminopropyl)diethoxymethylsilane (BNNS-Si (Diethoxy)) as well, the intense polymerization was only observed when the silane with three methoxy groups was used. This is consistent with previous FT-IR results that showed the formation of a siloxane network.

Furthermore, energy-dispersive X-ray spectroscopy (EDX) was also performed to characterize the compounds of the functionalized boron nitride.

In addition to the boron and nitrogen, all the elements that were contained in the alkoxy silanes that were used for the functionalization were detected. Carbon, oxygen and silicon peaks were visible in all three EDX spectra (Figure 8), clearly indicating the successful covalent functionalization of the boron nitride with silanes.

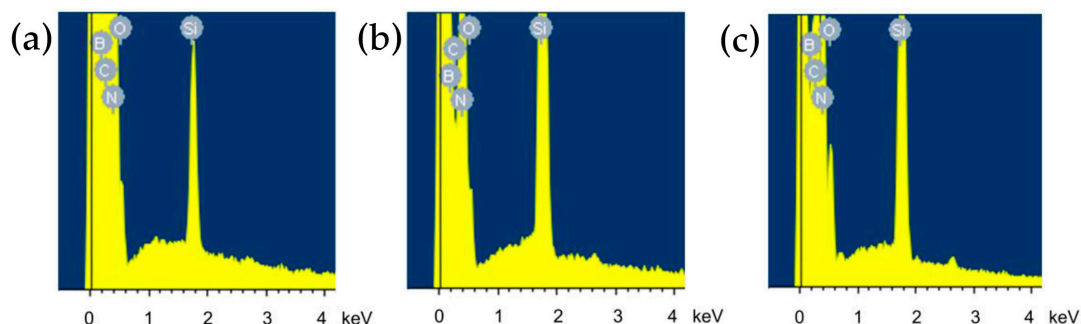


Figure 8. EDX spectra of boron nitride nanosheets that are functionalized with (a) (3-aminopropyl)dimethylmethoxysilane, (b) (3-aminopropyl)diethoxymethylsilane, and (c) (3-aminopropyl)trimethoxysilane. In addition to boron and nitrogen, oxygen, carbon, and silicon were also detected.

A further characterization technique that was used to determine the successful functionalization of BNNSs was wide-angle X-ray scattering (WAXS).

The peak at $q = 19.02 \text{ nm}^{-1}$, also called a Bragg peak, which was visible for both the unmodified boron nitride nanosheets and the oxidized ones (BNNS-OH), corresponds to an interlayer distance of 0.33 nm, which was in good agreement with the value reported in the literature [56]. The position of the Bragg peak's maximum (q_{peak}) indicates the distance (d_{Bragg}) between the highly ordered nanosheets by using *Bragg's law*:

$$d_{\text{Bragg}} = \frac{2 * \pi}{q_{\text{peak}}} \quad (1)$$

The value of q being 19.02 nm^{-1} correlates with the diffraction angle $2\theta = 26.96^\circ$, which corresponds to the (002) crystal plane of boron nitride, indicating a hexagonal crystal structure without any impurity phases [19,51,57,58]. The conversion from q to 2θ was calculated by following Equation (2):

$$q = \frac{4\pi}{\lambda_0} \sin \frac{2\theta}{2} \quad (2)$$

The hydrolysis of the boron nitride did not lead to a change in crystallinity, as there was no shift in the q value observed, nor any additional peaks. This implied a good BNNS quality even after the oxidation process. The interlayer spacing of the oxidized BNNSs remained unchanged, as demonstrated by the same diffraction peak position (002) in Figure 9a. Analogous to the XRD data previously reported in the literature [19,51,57,58], the Bragg peak at $q = 19.02 \text{ nm}^{-1}$, observed in Figure 9b, corresponds to the hexagonal crystal structure of the boron nitride. In addition to this peak, there were two others visible at $q = 12.92 \text{ nm}^{-1}$ and at $q = 22.36 \text{ nm}^{-1}$, which correlated with an interlayer spacing of $d = 0.49 \text{ nm}$ and $d = 0.28 \text{ nm}$, respectively. According to Hubbard, Swanson, and Mauer [59] the lattice constant of single-crystal silicon is around 0.5 nm. Although the silane that was used for the functionalization of the boron nitride was not in the form of a single crystal silicon, these data were in good agreement with the q value at 12.92 nm^{-1} . Furthermore, this peak was consistently observed in the WAXS spectra of the other two functionalized BNNSs as well, as illustrated in Figure 9c,d. The characteristic boron nitride peak for the hexagonal structure at $q = 19.02 \text{ nm}^{-1}$, the peak at $q = 12.95 \text{ nm}^{-1}$ for the (3-aminopropyl)diethoxymethylsilane-functionalized boron nitride nanosheets (BNNS-Si (Diethoxy)), and the one displayed at $q = 12.13 \text{ nm}^{-1}$ for the (3-aminopropyl)trimethoxysilane-functionalized boron nitride nanosheets (BNNS-Si (Trimethoxy)) that were attributed to silicon are apparent in Figure 9c,d. The diffraction peak at $q = 15.27 \text{ nm}^{-1}$, which is demon-

strated in Figure 9d, could be converted to $2\theta = 21.57^\circ$, which corresponded to the XRD pattern of siloxanes [60]. This was consistent with previous FT-IR results that showed the formation of a siloxane layer on the surface of the boron nitride. An additional small peak at $q = 10.08 \text{ nm}^{-1}$ that corresponds to an interlayer spacing of $d = 0.62 \text{ nm}$ was also apparent in Figure 9d. This and the one in Figure 9b at $q = 22.36 \text{ nm}^{-1}$, corresponding to the interlayer spacing $d = 0.28 \text{ nm}$, could not be attributed to any specific crystal structure due to the lack of literature data. Nevertheless, WAXS has illustrated a successful functionalization of the BNNSs, which was in good agreement with the previously shown data from the other techniques used to characterize the boron nitride. Furthermore, WAXS has also shown that the hexagonal crystalline structure of the boron nitride remains unchanged after functionalization, due to the same position of the characteristic boron nitride peak at $q = 19.02 \text{ nm}^{-1}$, exhibiting an interlayer spacing of 0.33 nm .

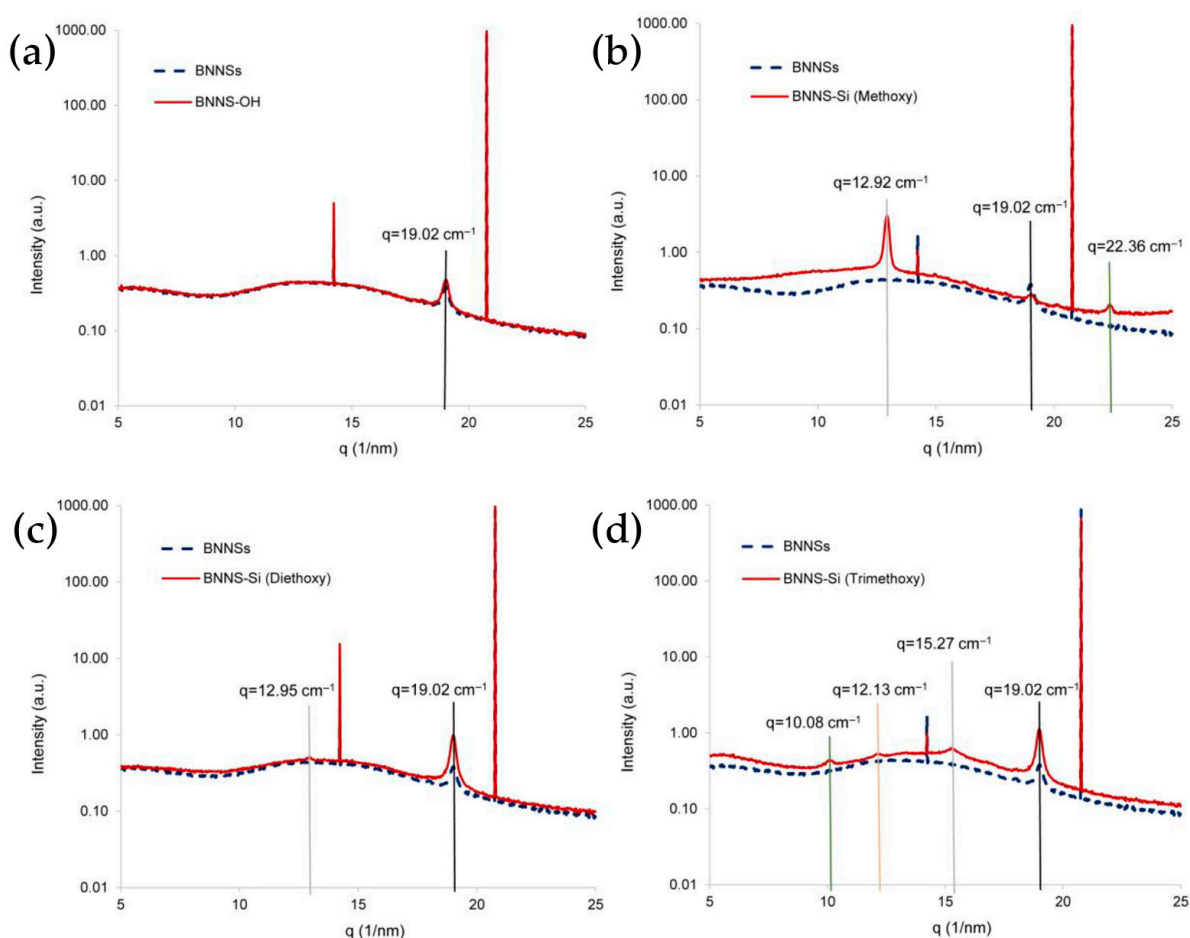


Figure 9. A comparison of the length of the scattering vector q of the untreated BNNSs and (a) the oxidized boron nitride nanosheets (BNNS-OH), (b) the BNNSs that are functionalized with (3-aminopropyl)dimethylmethoxysilane (BNNS-Si (Methoxy)), (c) the BNNSs that are functionalized with (3-aminopropyl)diethoxymethylsilane (BNNS-Si (Diethoxy)), and (d) the BNNSs that are functionalized with (3-aminopropyl)trimethoxysilane (BNNS-Si (Trimethoxy)).

4. Conclusions

This work demonstrates functionalization strategies that are directly applicable to the broader 2D nanocarbon field, enabling improved integration into hybrid nanomaterial systems and expanding the design space for electronic, thermal, and composite applications. To enable covalent functionalization, it was first necessary to activate the boron nitride nanosheets by increasing their surface reactivity. This was accomplished by introducing

OH groups through treatment with nitric acid (69%) in a sonication bath, followed by a washing process with water and then toluene. Their successful oxidation was confirmed by the appearance of the broad OH peak at around $3100\text{--}3500\text{ cm}^{-1}$, as illustrated in the FT-IR spectrum in Figure 1, as well as the presence of oxygen that was detected by EDX, as demonstrated in Figure 3b. After this activation step, the oxidized BNNSs were functionalized with alkoxysilanes. It was observed that the number of reactive alkoxy groups had an influence on the reaction and on how the silane was attached to the surface of the boron nitride. Among the three silanes tested, (3-aminopropyl)dimethylmethoxysilane—with only one reactive alkoxy group—proved to be the most effective for the covalent functionalization of BNNSs, minimizing undesired siloxane formation while maximizing B-O-Si bonding. The size and shape of the boron nitride nanosheets did not change after the functionalization, as is demonstrated by DLS and the SEM image. Moreover, the visible sharp IR bands at 980 cm^{-1} and 640 cm^{-1} , which are characteristic of the covalent B-O-Si bond, and the detection of silicon, oxygen, and carbon via EDX were proof of the successful covalent functionalization of the boron nitride nanosheets. There is only one way this silane can bond to the boron nitride because there is only one reactive alkoxy group that can form a covalent B-O-Si bond, as is demonstrated in Figure 4a. This, however, was not the case with the other two coupling agents. The more alkoxy groups a silane contained, the more difficult the functionalization became due to the polymerization into siloxanes. Although the functionalization of BNNSs with (3-aminopropyl)diethoxymethylsilane was successful—as was verified by the characteristic sharp bands of the covalent B-O-Si bond at 980 cm^{-1} and at 640 cm^{-1} and the detection of silicon, oxygen, and carbon via EDX—a slight increase in size was observed by DLS measurements due to the polymerization of the silane into disiloxanes. Furthermore, the formation of Si-O-Si bonds was supported by the appearance of the vibration band at 1100 cm^{-1} . However, this polymerization was not as intense as in the case of the silane with three alkoxy groups, which appeared in the SEM image as round-shaped particles. (3-aminopropyl)trimethoxysilane contains three active methoxy groups that, in the presence of water, can easily form siloxane networks on top of the boron nitride nanosheets. This was supported not only by the apparent round-shaped particles in the SEM image, but also by the increase in the hydrodynamic radius, as determined by DLS. Moreover, the FT-IR analysis showed bands at 1120 cm^{-1} , 1020 cm^{-1} , and 440 cm^{-1} , which contribute to the Si-O-Si vibration. These results are in good agreement with WAXS analysis, which showed a diffraction peak at $q = 15.27\text{ nm}^{-1}$ (converted to $2\theta = 21.57^\circ$) that corresponds to the XRD pattern of siloxanes, as demonstrated in Figure 9d. Regardless, the B-O-Si vibration bands at 930 cm^{-1} and at 690 cm^{-1} , although weakly displayed, confirmed the successful covalent functionalization of the boron nitride nanosheets. This was also supported by the detection of silicon, oxygen, and carbon via EDX. Furthermore, the crystallinity of BNNSs remained unaltered after oxidation as well as functionalization with all three alkoxysilanes. The diffraction peak at $q = 19.02\text{ nm}^{-1}$ was observed in all WAXS spectra, as is demonstrated in Figure 9. This corresponds to an interlayer spacing of 0.33 nm and a (002) crystal plane, which is characteristic of the hexagonal structure of the boron nitride. Ultimately, the BNNSs were successfully functionalized and characterized. This would be the first step towards enabling such materials to be incorporated into polymers, enhancing their thermal conductivity. Moreover, this functionalization approach can serve as a steppingstone towards further chemical modifications of 2D materials, including graphene-type systems, allowing such materials to be more widely applied.

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