

# Boron Nitride Nanoplatelets as a Solid Radical Initiator for the Aerobic Oxidation of Thiophenol to Diphenyldisulfide

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Boron nitride (BN) nanoplatelets suspended in ethanol were obtained through the sonication of BN powders prepared through biopolymer-templated pyrolysis and air calcination of  $(NH_4)_3BO_3$ -chitosan powders. AFM measurements indicate that BN nanoplatelets have an average height of 1.5 nm and length and width dimensions between 30 and 100 nm. BN nanoplatelets exhibit higher catalytic activity for the molecular oxygen-mediated oxidation of aromatic thiols to the corresponding disulfides than do boron and nitrogen-codoped graphene or

commercial bulk hexagonal BN. The reaction scope includes substituted aromatic and heteroaromatic thiols, whereas aliphatic thiols are considerably less reactive. The hot filtration test and radical quenching by *N-tert*-butyl- $\alpha$ -phenylnitrone support an autoxidation mechanism involving thiyl radicals. BN nanoplatelets are deactivated during the course of the reaction owing to aggregation. The present study uncovers the activity of BN nanoplatelets used as an insoluble radical initiator of S–H bonds.

## Introduction

There is a considerable interest in finding alternatives to noble and "critical" transition metals in catalysis. One of the possibilities to avoid the use of transition-metal-containing catalysts is carbocatalysis, in which the active sites are embedded in a carbon matrix.<sup>[1-4]</sup> Because graphene and related materials have become widely available, carbocatalysis is experiencing a considerable growth and it appears that many different types of organic reactions, particularly oxidations, can be catalysed by graphene.<sup>[5]</sup> As a consequence, the use of single- or few-layer graphene and related nanomaterials as catalysts is becoming an active area of research aimed at exploring the potential of this type of materials to compete with conventional transition-metal catalysts. In this context, one 2D nanomaterial that has not been reported as an insoluble radical initiator till date is boron nitride (BN). Single-layer BN is composed of a one-atom-thick sheet of alternating sp<sup>2</sup> boron and nitrogen atoms in a hexagonal arrangement.<sup>[5-10]</sup> Single-layer BN has the

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same structure as graphene, but has different physical properties, such as the lack of electrical conductivity, complete transparency, and emission in the deep UV region. BN is chemically more robust than graphene, and this robustness is, in principle, advantageous for its use in catalysis.

Perhaps the main reason why, in contrast to graphene, BN has not yet been explored as a catalyst is that single- or fewlayer BN suspensions are still difficult to prepare, particularly in conventional, not highly viscous solvents.<sup>[8-11]</sup> This is due to the difficulty in exfoliation of bulk hexagonal BN (h-BN) particles that are chemically inert and, in contrast to graphite, cannot be oxidised to promote exfoliation. Alternative procedures for the preparation of thin-walled BN samples are based on the synthesis of BN from aminoborane along with the generation of bubbles through the evaporation of ethanol.<sup>[12]</sup> We have recently developed a new procedure that can be used to prepare BN either as a film or as a type of powder that can be easily exfoliated, thus making available BN suspensions.<sup>[13]</sup> This methodology makes it possible to explore the catalytic activity of few-layer BN nanoplatelets that up to now have remained undisclosed. Although by now there are sufficient reports on the catalytic activity of graphene and related carbon materials,<sup>[5]</sup> the catalytic activity of BN remains unknown. Herein, we report that single- and few-layer BN nanoplatelets suspended in ethanol promotes the oxidative coupling of thiophenol (1) to diphenyldisulfide (2).

Disulfides have been used extensively in chemical and biological studies.<sup>[14-17]</sup> They are also often used as reagents in organic syntheses<sup>[18,19]</sup> and as a protecting group for thiols.<sup>[20]</sup> One of the most commonly used methods for the synthesis of disulfides is the oxidative coupling of thiols with permanganate,<sup>[21]</sup> halogens,<sup>[22]</sup> iron(III) chloride,<sup>[23]</sup> and peroxides<sup>[24]</sup> as reagents. Furthermore, transition-metal complexes have been



used as catalysts for the conversion of thiols to disulfides.[25-27] The aerobic oxidation of thiols to disulfides is a green alternative to those using other oxidising reagents such as organic peroxides in stoichiometric amounts. In this context, the aerobic oxidation of thiols to disulfides has been reported with heterogeneous catalysts such as frameworks,<sup>[28]</sup> metal–organic graphite oxide,<sup>[29]</sup> neutral alumina,<sup>[30]</sup> copper nanoparticles supported on diamond nanoparticles,[31] and gold supported on CeO<sub>2</sub>.<sup>[32]</sup> The selection of this reaction to check the catalytic activity of BN was based on the number of precedents showing that graphene can promote the oxidation because of the presence of defects,<sup>[33-35]</sup> such as thiols,<sup>[29]</sup> and also because some



**Figure 1.** a and b) AFM images of BN platelets of two different samples obtained following the procedure indicated in Scheme 1, showing length and width dimensions ranging from 30 to 80 nm. c) Height of three representative BN nanoplatelets. d) Statistical distribution of the height of these platelets, showing that more than 80% of the particles comprise less than four BN layers.

transition metals are deactivated in the process of thiol oxidation owing to the strong interaction of the metal with thiolate groups.<sup>[36]</sup> We anticipated that the chemical inertness of BN would make the use of thiols as substrates possible.

## **Results and Discussion**

Suspensions of BN nanoplatelets were obtained through the pyrolysis of dry  $(NH_a)_3BO_3$ -chitosan powders at 900 °C under



shows the hexagonal symmetry of the layers. AFM (Figure 1) shows that BN suspensions are composed of BN nanoplatelets with an average height of 1.5 nm (approximately three layers) and length and width dimensions between 30 and 80 nm.

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These BN nanoplatelets were tested as catalysts for the oxidation of thiols to disulfides. In the first stage, **1** was used as a model substrate to optimise the reaction conditions. The

**Scheme 1.** Procedure for the preparation of h-BN nanoplatelets: i) precipitation of the solution of  $H_3BO_3$  + chitosan in  $NH_4OH$  (solution 25 %), ii) pyrolysis at 900 °C, iii) calcination at 1000 °C, and iv) exfoliation of BN nanoplatelets.

argon flow and subsequent air calcination at  $1000^{\circ}$ C (Scheme 1). The rationale of this procedure is derived from the reported formation of graphene through the pyrolysis of chitosan<sup>[37,38]</sup> and the comparison of graphene and BN structures in such a way that chitosan pyrolysis is templating BN formation. Subsequently, combustion of graphene in air results in BN particles that can be easily exfoliated and suspended in water, ethanol, dichloromethane, and other volatile organic solvents.

aerobic oxidation of 1 to 2 in the absence of a catalyst resulted in 6% conversion. In contrast, the presence of BN nanoplatelets resulted in complete conversion of 1 after 8 h under atmospheric oxygen pressure with a selectivity towards 2 higher than 99%. The time-conversion plot for the conversion of 1 to 2 with BN as a promoter is shown in Figure 2. To put the activity of BN nanoplatelets into context, a series of other related materials such as bulk h-BN and doped graphenes were screened under the same reaction conditions. Thus, boron and

BN nanoplatelets were characterised by X-ray photoelectron

spectroscopy analysis, and the peaks of boron and nitrogen

present in a 1:1 atomic ratio at 190.75 and 398.20 eV binding

energy were recorded, which were consistent with the values reported in the literature for single-layer BN (Figure S1).<sup>[39]</sup> In Raman spectroscopy and XRD, the characteristic peaks of BN

at 1380 cm<sup>-1</sup> and 27° were recorded (Figure S2).<sup>[39]</sup> High-resolu-

tion TEM shows the structural ordering at the microscopic scale of the material, and selected area electron diffraction



**Figure 2.** Time–conversion plot for the aerobic oxidation of 1 to 2 with a) BN, b) boron and nitrogen-codoped graphene, c) commercial h-BN, and d) blank reaction without any catalyst.

nitrogen-codoped graphene exhibited 51% conversion of 1 after 8 h under identical reaction conditions. In contrast, the conversion of 1 was only 23% after 8 h with commercial h-BN as a promoter under identical reaction conditions. The available kinetic data indicate that the initial reaction rate and the final conversion of 1 with these materials were lower than those achieved with BN nanoplatelets (Figure 2). These experimental data reveal that BN lacking a transition metal can promote chemical reactions. The enhanced activity of BN as compared with other catalysts is believed to be due to its platelet morphology, a thin-layer structure with fine distribution of boron and nitrogen atoms; the nitrogen atoms are preferentially located at the periphery of the nanoplatelets. The lack of such preferential location of nitrogen atoms at the periphery and the larger sheet dimensions of boron and nitrogen-codoped graphene can be responsible for its lower activity than that of BN. As expected, replacing oxygen by nitrogen drastically reduced the conversion of 1, which still undergoes some oxidation owing to the incomplete removal of residual oxygen from the reaction medium. Furthermore, the conversion of 1 was reduced if the reaction was performed in the presence of air at atmospheric pressure instead of pure oxygen. These data clearly indicate the active role of the molecular oxygen as the oxidant in this reaction. In contrast, 12% conversion of 1 was observed if the aerobic oxidation was performed at room temperature. In addition, a control experiment of the aerobic oxidation of 1 to 2 with BN as a promoter was performed in the presence of the radical scavenger *N*-tert-butyl- $\alpha$ phenylnitrone, and only 29% conversion of 1 was observed; this result confirms that the process involves radicals as intermediates that can be guenched by the phenylnitrone.

The effect of the amount of BN as a promoter under the general reaction conditions was investigated. As shown in Figure 3, the temporal profile of the reaction at  $70^{\circ}$ C in ethanol depends on the amount of BN platelets, and the conversion at a given time decreases as the amount of BN present in the system decreases from 10 to 3 mg. The initial reaction rate estimated at 2 h from the time–conversion plot follows a quasi-linear relationship with the amount of BN present in



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Figure 3. Effect of the amount of BN present in the system on the aerobic oxidation of 1 to 2 in the presence of a) 10, b) 5, and c) 3 mg of BN.

Table 1. Aerobic oxidation of 1 to 2 promoted by various materials. <sup>[a]</sup>							
Entry	Promoter	<i>Т</i> [°С]	Conversion [%]	Selectivity [%]			
1	-	70	6	99			
2	BN	70	100	99			
3	commercial h-BN	70	23	99			
4	(B,N)-G <sup>[b]</sup>	70	51	99			
5	BN <sup>[c]</sup>	70	29	97			
6	BN <sup>[d]</sup>	70	27	99			
7	BN <sup>[e]</sup>	70	31	99			
8	BN	RT	12	99			
[a] Reaction conditions: <b>1</b> (1 mmol), ethanol (3 mL), oxygen purged, pro- moter (10 mg), $t=8$ h; [b] Boron and nitrogen-codoped graphene; [c] Re- action performed in the presence of 100 mg of <i>N-tert</i> -butyl- $\alpha$ -phenyl- nitrone; [d] Reaction under inert atmosphere; [e] Reaction under air at-							

the system. Similarly, the conversion of 1 to 2 was found to depend on the oxygen pressure in the system (cf. entries 2, 6, and 7 in Table 1).

The heterogeneity of the reaction was addressed by using the hot filtration test. After achieving approximately 31% conversion of 1, BN was removed from the reaction mixture through filtration while it was hot and the resultant reaction mixture was allowed to react further in the absence of solid (Figure 4). Notably, the conversion of 1 reaches to 70% after 8 h. This result indicates that the process follows an autoxidation mechanism with a radical chain involving thiyl radicals generated by the promoter. Thus, these experiments clearly establish that BN platelets act as radical initiators rather than as true catalysts. However, the effect of the amount of BN on the conversion indicates that there is the population of thiyl radicals generated that depends quasi-linearly on the amount of BN present in the system.

BN was reused for the aerobic oxidation of **1** to **2** under identical conditions. Unfortunately, the sample showed a considerable decrease in the conversion of **1** from 100 to 28% in the second run. The recovered solid was further analysed by using TEM. A comparison of the TEM images of the fresh and used BN materials is shown in Figure 5. As shown in the figure,

mosphere



Figure 4. Time-conversion plot for the oxidation of 1 at 70  $^{\circ}$ C a) in the presence of BN and b) in the hot filtration test in which the reaction is initiated in the presence of BN up to 3 h and then the solid removed while hot and the reaction continued in the absence of BN.



Figure 5. TEM images at two different magnifications of a and b) fresh and c) used BN platelets in the reaction of 1 to 2.

fresh BN platelets show the expected layered morphology. Upon the use of BN as a promoter, its morphology changes drastically and the material becomes agglomerated, also showing the appearance of some nanoparticles. This drastic change in morphology explains the origin of the loss of the activity of BN used as an oxidation promoter.

The scope of BN as a radical initiator was studied by testing the reactivity of different aromatic and aliphatic thiols under optimal conditions (Table 2). 4-Fluoro-, 4-chloro-, 4-methyl-, and 4-methoxythiophenol reached higher than 90% conversion with 99% selectivity towards the corresponding disulfide. Even heterocyclic 2-mercaptopyridine showed 99% conversion to the corresponding disulfide with very high selectivity, although it requires long time. 2-Napthylthiol showed complete conversion and selectivity towards the corresponding disulfide

<b>Table 2.</b> Aerobic oxidation of various thiols (R-SH; see R in the table) to their corresponding disulfides promoted by BN platelets. <sup>[a]</sup>								
Entry	R	t [h]	Conversion [%]	Selectivity [%]				
1	C <sub>6</sub> H₅	8	100	99				
2	$4-CH_3-C_6H_4$	5	99	99				
3	4-CI-C <sub>6</sub> H <sub>4</sub>	8	100	99				
4	4-OCH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>	9	99	99				
5	$4-F-C_6H_4$	24	90	97				
6	2-pyridyl	22	99	99				
7	2-naphthyl	6	100	99				
8	benzyl	23	54	99				
9	1-hexyl	48	32	99				
[a] Reaction conditions: thiol (1 mmol), ethanol (3 mL), oxygen purged, BN (10 mg), $T = 70 ^{\circ}$ C.								

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under the general reaction conditions in only 6 h. As expected, in view of the relative stability of the corresponding thiyl radicals, aliphatic thiols were less reactive than aromatic thiols. For example, benzyl mercaptan exhibited 54% conversion after 23 h; in contrast, 1-hexanethiol showed 32% conversion after 48 h.

## Conclusions

Boron nitride (BN) platelets of 30-80 nm in size are prepared by using the pyrolytic preparation method based on the templating effect of chitosan; these BN nanoplatelets are readily suspended in ethanol and conventional organic solvents, which enables us to study their activity. Similarly to graphene that has the same structure, BN platelets were found to promote the aerobic autoxidation of thiols to disulfides, a reaction that typically leads to poisoning of transition-metal catalysts. The evidence indicates that the activity of BN as an initiator derives from the dispersion of BN as nanometric platelets and is lost under the reaction conditions owing to massive agglomeration. As the activity of BN used to promote reactions remains unexplored, our study can open new avenues to delineate the potential of BN in catalysis and to determine the nature of the active sites that can be present in this 2D nanomaterial.

## **Experimental Section**

### Synthesis of h-BN nanoplatelets

h-BN powders were obtained through the precipitation of chitosan (1.12 g dissolved in 25 mL of 0.3  $\times$  HAcO) and H<sub>3</sub>BO<sub>3</sub> (0.45 g) solution in a NH<sub>4</sub>OH aqueous solution (25%). The resulting spheres were pyrolysed under argon flow (increasing the temperature up to 900°C; heating rate: 0.9°Cmin<sup>-1</sup>) and subsequently calcined in air up to 1000°C (heating rate: 8°C min<sup>-1</sup>). Then, h-BN powders were subjected to exfoliation by sonication. Exfoliation was performed by suspending h-BN powders in water and sonicating them with an ultrasound source (400 W) for 3 h. The resulting dispersion was left to stand for approximately 24 h to allow any unstable aggregates to form and precipitate. Subsequently, the sus-



pension was centrifuged at 1500 rpm for 30 min to obtain a homogeneous dispersion that was indefinitely persistent.

#### **Characterisation techniques**

The Raman spectra were recorded on a Renishaw in Via Raman spectrometer equipped with a charge coupled device detector at ambient temperature using a 514 nm laser excitation. The TEM images were acquired with a Philips CM300 FEG system operating at a voltage of 100 kV. The X-ray photoelectron spectra were recorded on a SPECS spectrometer equipped with a PHOIBOS 150 MCD-9 detector using a non-monochromatic X-ray source (Al and Mg) operating at 200 W. The samples were evacuated in the prechamber of the spectrometer at  $1 \times 10^{-9}$  mbar (1 mbar = 0.1 kPa). Some of the samples were activated in situ under  $N_2$  flow at 450  $^\circ$ C for 3 h followed by evacuation at 10<sup>-8</sup> mbar. The measured intensity ratios of components were obtained from the area of the corresponding peaks after a non-linear Shirley-type background subtraction and corrected by the transmission function of the spectrometer. The XRD patterns were obtained with a Philips X'Pert X-ray diffractometer using CuK<sub> $\alpha$ </sub> radiation ( $\lambda = 1.541178$  Å). AFM measurements were performed with a Veeco AFM apparatus operating in contact mode in air at ambient temperature. Notably, AFM measurements were not performed in a clean room and therefore films on glass substrates might have contained dust that could be detected by these techniques.

#### **Reactivity tests**

A 50 mL round bottom flask was charged with the solid (10 mg) in ethanol (3 mL) and subjected to sonication (150 W) for 30 min. Thereafter, the substrate (1 mmol) was added to the dispersion and the flask immersed into an oil bath preheated at the reaction temperature, 70 °C in most of the experiments. This reaction mixture was purged with oxygen from an inflated balloon. Then, it was continuously stirred magnetically. The reaction was monitored periodically by analysing the sample using GC until the completion of the reaction. The aliquots were filtered through a 0.2  $\mu m$  nylon filter and analysed immediately without delay. The mass balances of the final reaction mixture accounted for more than 97% of the initial substrate, as confirmed by GC using nitrobenzene as the external standard. The yields of the product were determined with nitrobenzene as the external standard, considering the response factors to be unity. In most of the cases, the expected disulfide with an acceptable purity was obtained directly from the reaction mixture without further purification.

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Boron Nitride Nanoplatelets as a Solid Radical Initiator for the Aerobic Oxidation of Thiophenol to Diphenyldisulfide



Aerobics is fun: Boron nitride nanoplatelets prepared through pyrolysis and sonication demonstrate higher catalytic activity for the aerobic oxidation of thiophenol.