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Nmr, Maldi-TOF, Rheology, and XPS Spectra of Dichlorophenylacetylene (Dpb) Reagent and the Associated Carbonization Products

Introduction

Figure S1 compares ^{1}H NMR spectra between dichlorophenylacetylene borane (DPB) and the starting 1-phenyl-2-trimethylsilylacetylene reagent. The ^{1}H NMR spectrum of DPB shows the complete disappearance of the trimethylsilane protons that are present in the starting material. In addition, due to the lower electronegativity of boron (2.04) compared to carbon (2.55), we also observe a slight up field shift in the aromatic protons due to the presence of boron in the resulting DPB compound. Figure S2 compares ^{13}C NMR spectra of the same set of dichlorophenylacetylene borane (DPB) and the starting 1-phenyl-2-trimethylsilylacetylene reagent. The ^{13}C NMR spectrum of DPB compound clearly shows the presence of the alkyne group and the disappearance of the trimethylsilyl carbons (0 ppm) present in the starting material. Furthermore, Figure S3 compares ^{11}B NMR spectra between dichlorophenylacetylene borane (DPB) and BCl_3 reagent. The up field chemical shift, from 47 ppm for BCl_3 to about 43 ppm for DPB, shows a decrease in the acidity of boron due to the π-electrons conjugation with phenylacetylene moiety.

Figure S7 shows high resolution XPS spectra (with fitting curves) to examine B species. Evidently, there are three boron compounds, including boron oxide, boron carbide, and an intermediate boron species between boron oxide and boron carbide, formed immediately after exposing CB_x material to high temperatures in air. Once the CB_x material is heated to 750ºC for 5 hours, there is only boron oxide detected on the surface that serves as a protective layer.

As shown in Table S1, the amount of boron content on the surface of the CB_x compound increases with an increase in either the temperature or time of exposure to air. The boron species is in the form of an oxide and thus we also see an increase in the oxygen content. The carbon content gradually decreased because the surface of the CB_x material eventually becomes

Table S1. A summary of elemental compositions on the surface of CB_x material after exposing to air at 700ºC for 10 min, 1 hr and 3 hrs, and at 750ºC for 5 hrs, respectively

Exposure in Air		B 1s (%)	C 1s (%)	O 1s (%)	Ratio of O1s/B1s
Temperature	Time				
700 oC	10 min	2.37	87.92	9.71	4.12
700 oC	3 hrs	11.41	64.80	23.79	2.08
750 oC	5 hrs	30.40	12.28	57.32	1.89

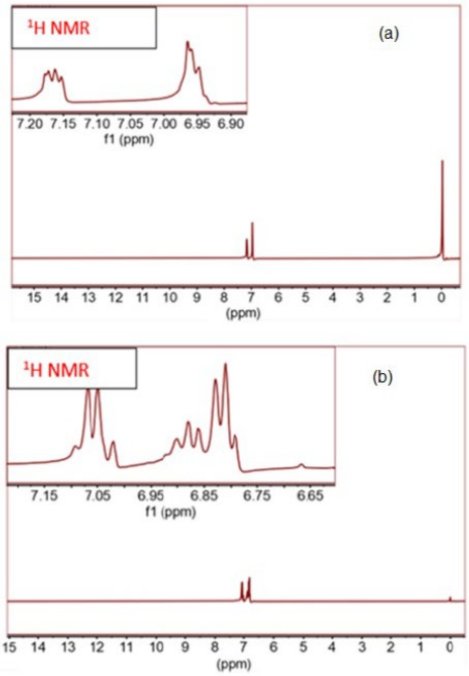


Figure S1. ^{1}H NMR spectra of (a) the starting 1-phenyl-2-trimethylsilylacetylene reagent and (b) dichlorophenylacetylene borane (DPB)

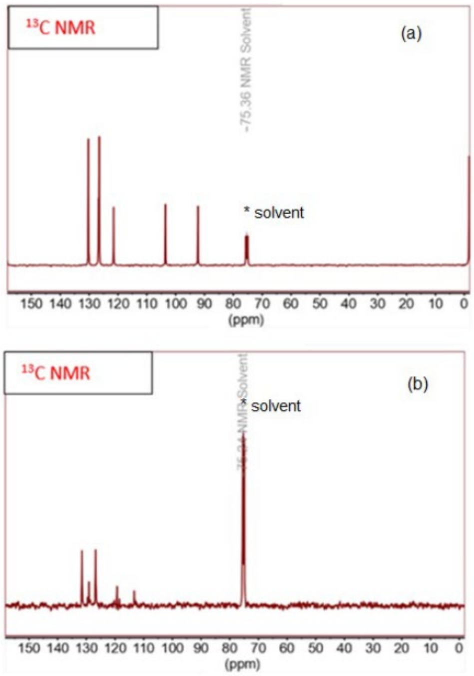
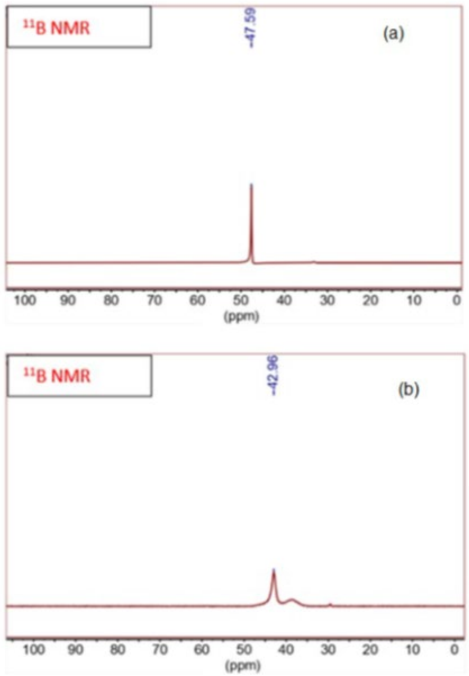


Figure S2. ^{13}C NMR spectra of (a) the starting 1-phenyl-2-trimethylsilylacetylene reagent and (b) dichlorophenylacetylene borane (DPB)



coated with boron oxide, which is thick enough to prevent exposure of

Figure S3. ^{11}B NMR spectra of (a) BCl_3 and (b) dichlorophenylacetylene borane (DPB)

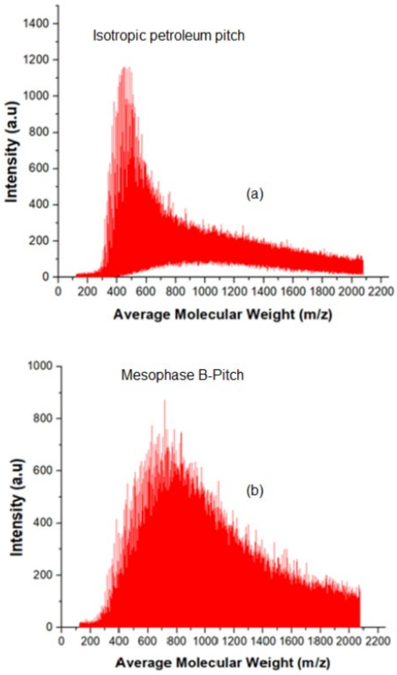
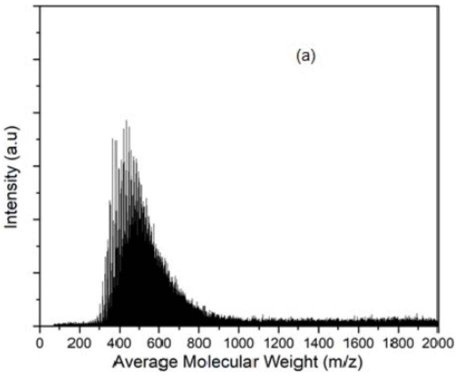


Figure S4. Maldi-TOF MS plots of (a) the starting isotropic petroleum pitch and (b) the resulting mesophase B-Pitch with B-content= 4.7 wt.%



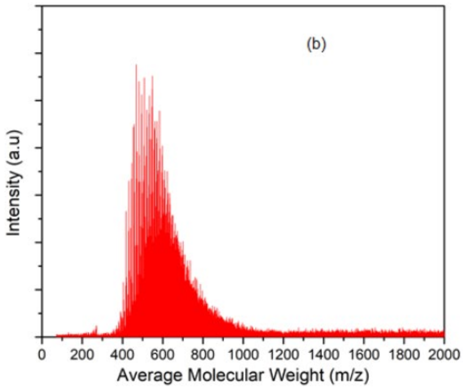


Figure S5. Maldi-TOF MS plots of (a) a mixture (1/1 wt. ratio) between isotropic petroleum pitch and mesophase B-Pitch and (b) the resulting mixture after heating at 350ºC under N_2 for 1 hr

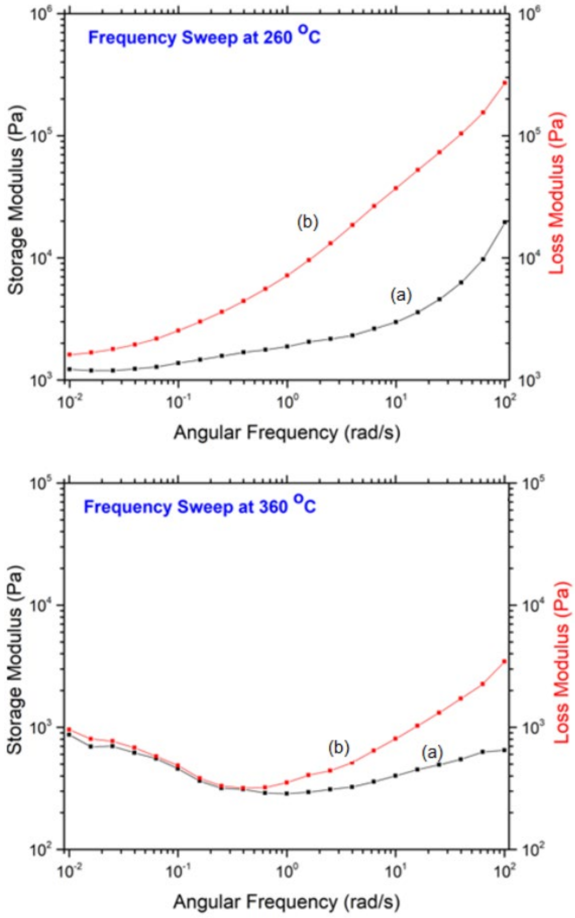
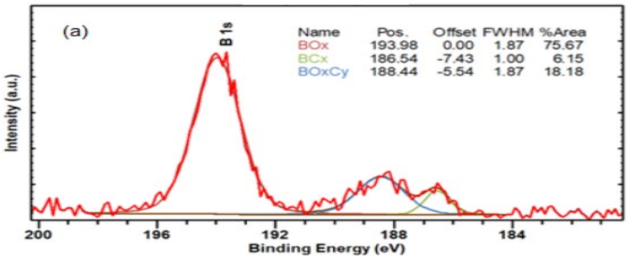
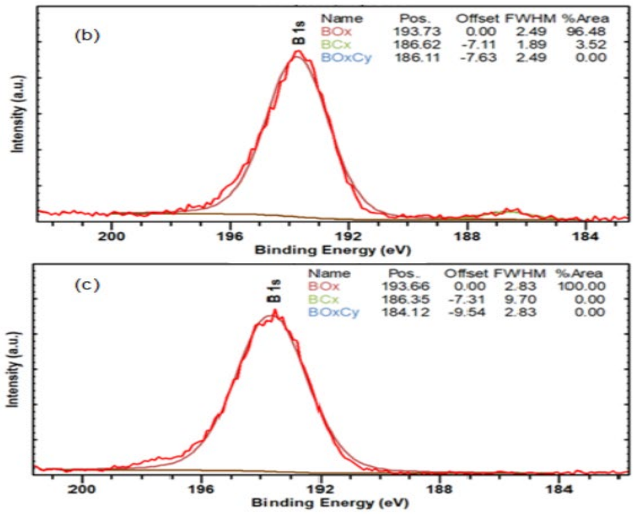


Figure S6. Frequency sweep of precursor-3 with (a) storage and (b) loss modulus vs frequency plots at 260 oC (top) and 360 oC (bottom)





Name	Pos.	Offset	FWHM	%Area
BOx	193.73	0.00	2.49	96.48
BCx	186.62	-7.11	1.89	3.52
BOxCy	186.11	-7.63	2.49	0.00

Name	Pos.	Offset	FWHM	%Area
BOx	193.66	0.00	2.83	100.00
BCx	186.35	-7.31	9.70	0.00
BOxCy	184.12	-9.54	2.83	0.00

Figure S7. High resolution XPS spectra (with fitting curves) for various boron species formed on the surface of CBx material after being exposed to air at 700ºC for (a) 10 min and (b) 3 hrs, and at 750ºC for 5 hrs, respectively