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Template Synthesis of Boron Nitride Nanotubes Over Iron Impregnated Mesoporous Silica MCM-41 by Chemical Vapor Deposition Technique

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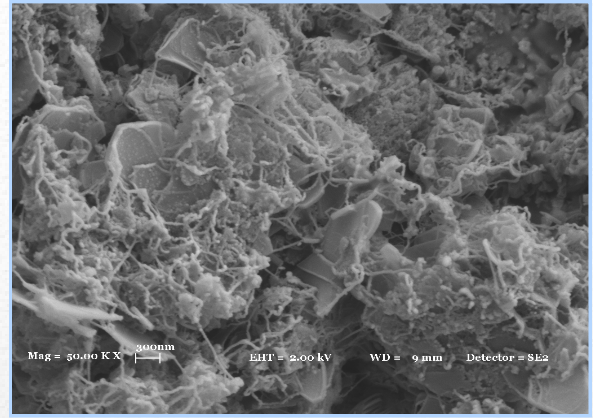
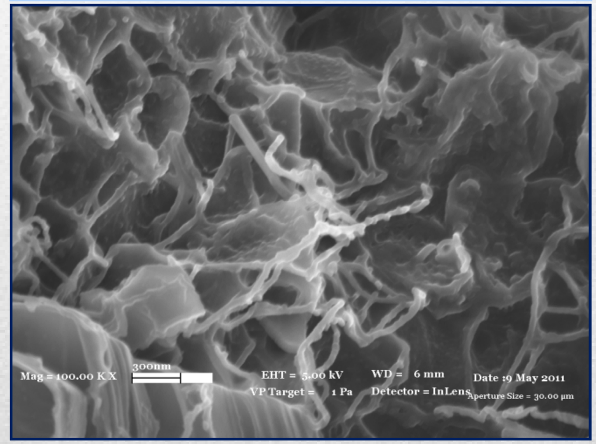
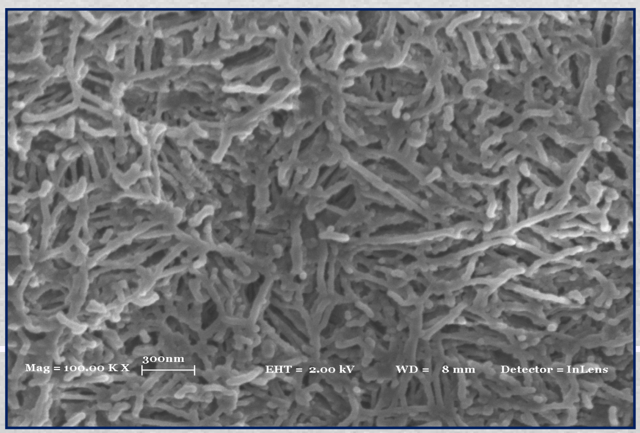
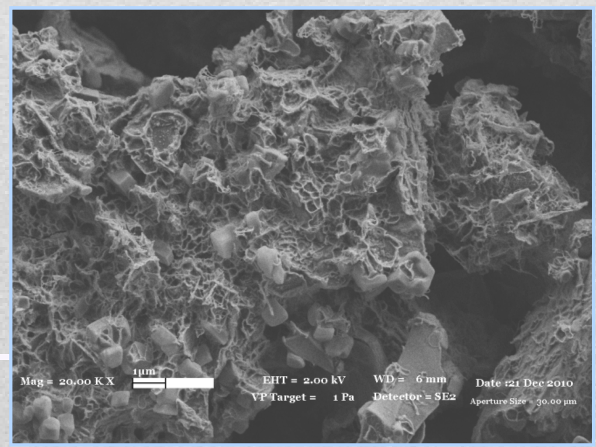
OUTLINE

- Introduction
- Synthesis Methods of BNNTs
- Synthesis and Characterization of
  - ➔ MCM-41
  - ➔ Boron Nitride Nanotubes (BNNTs)
- Hydrogen Uptake of Synthesized BNNTs
- Conclusion

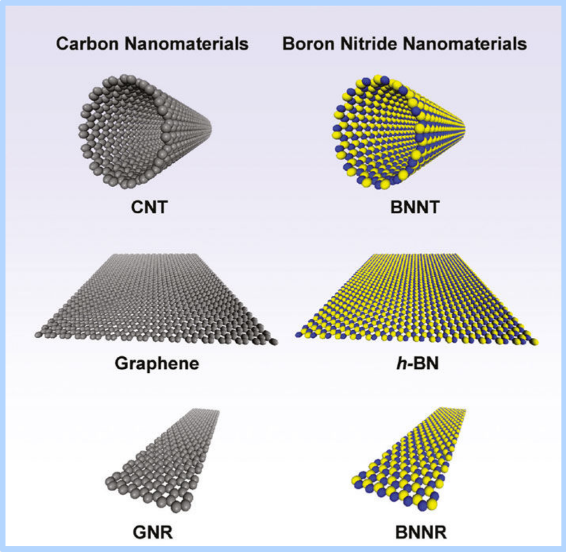








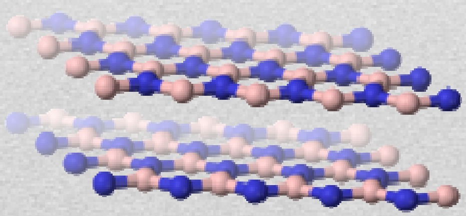
INTRODUCTION

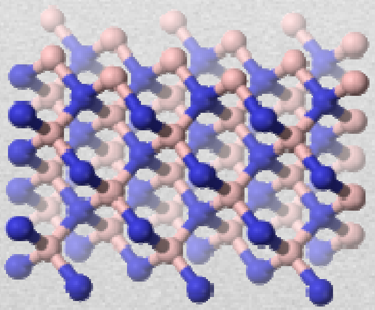


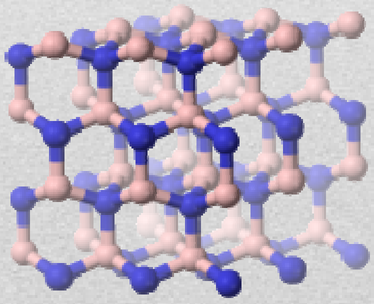
- There are numerous works about the synthesis and characterization of BNNTs because of

  - High mechanical strength
  - Good resistance to corrosion
  - Low density
  - Excellent thermal and electrical properties
  - Suited for high temperature

- Boron nitride (BN) structures can be synthesized in crystallographic forms such as cubic (c-BN), hexagonal (h-BN), wurtzite (w-BN), and rhombohedral (r-BN)







INTRODUCTION

○ Boron nitride nanotubes were theoretically predicted in 1994 and experimentally discovered in 1995.

○ For the first time, multi walled-BNNTs and single walled-BNNTs have been achieved by using an adapted arc discharge technique.

○ Goldberg et al. synthesized pure BNNTs by laser ablation method which was also applied for the synthesis of fullerenes.

○ Tang et al. demonstrated the synthesis of multi walled-BNNTs from a mixture of boron and iron oxide powders placed into an alumina crucible at 1350^{o}C.

○ Bando et al. synthesized the nanotubular BN materials via chemical vapor deposition (CVD) method using B-N-O precursors at a high temperature of 1700^{o}C.

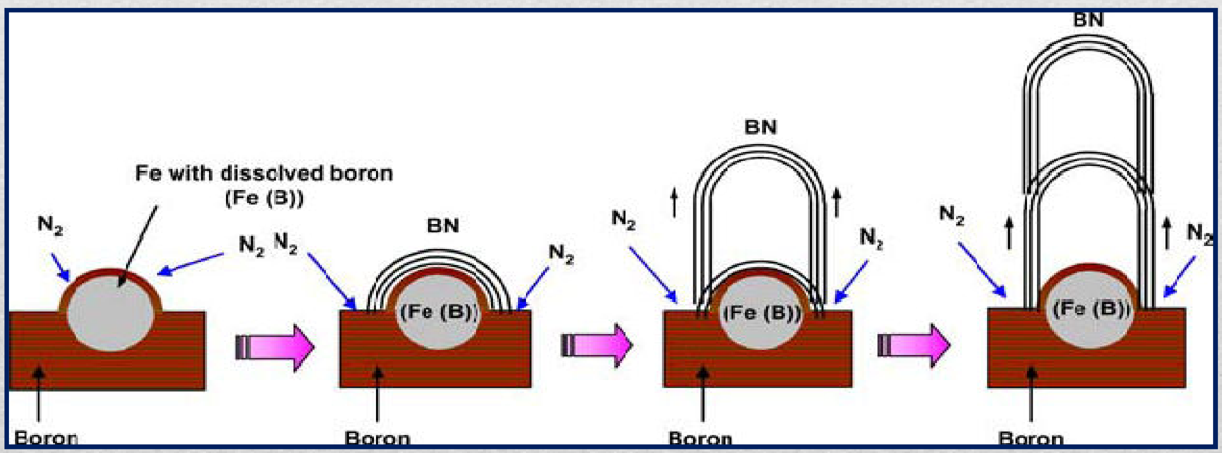
○ Cai et al. reported a convenient synthesis route to BNNT by the reaction of boron powder, iron oxide, and ammonium chloride at 600^{o}C for 12 h.

GROWTH OF BNNT OVER TEMPLATES

o Wang et al. prepared BNNTs, BN-bamboos and BN-fibers from borazine oligomer under the confinement of alumina anodic membrane as a template.

o Li et al. produced BNNTs with a uniform diameter of about 7 nm using BCl_3 and NH_3 at relatively low temperatures (650–850^{o}C) within the channels of mesoporous silica SBA-15.

o High-quality and high-yield BNNTs can be synthesized over mesoporous silica templates by CVD method.



APPLICATIONS

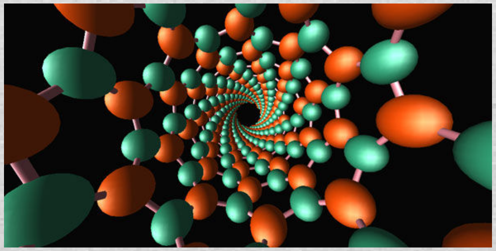
BNNTs with the unique material properties become promising candidate in various technical applications

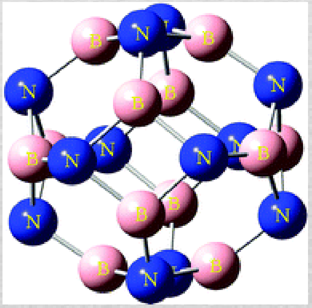
- Insulating nanomaterials

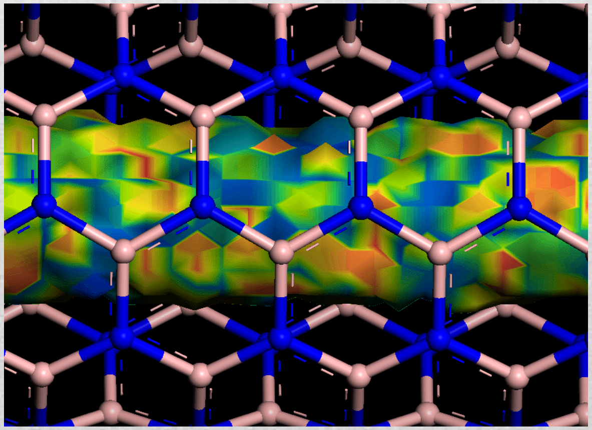
- Deep-UV photoelectronic devices

- Nanovectors to carry electrical/mechanical signals within a cellular system

- Hydrogen storage medium







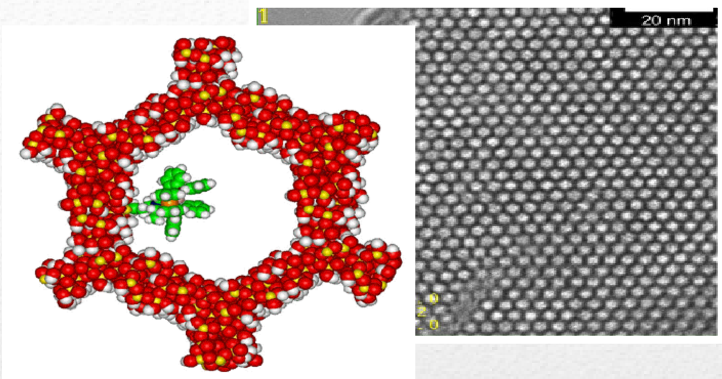
OBJECTIVE

- High-quality and high-yield BNNTs can be synthesized over mesoporous silica templates by CVD method.

- Mesoporous MCM-41 as a template which has a regular hexagonal array of uniform pore openings with diameters between 2 and 10 nm is a good candidate.

- A simple and shorter synthesis technique for the production of BNNT over iron impregnated mesoporous silica MCM-41 at a relatively low reaction temperature by CVD method was aimed.

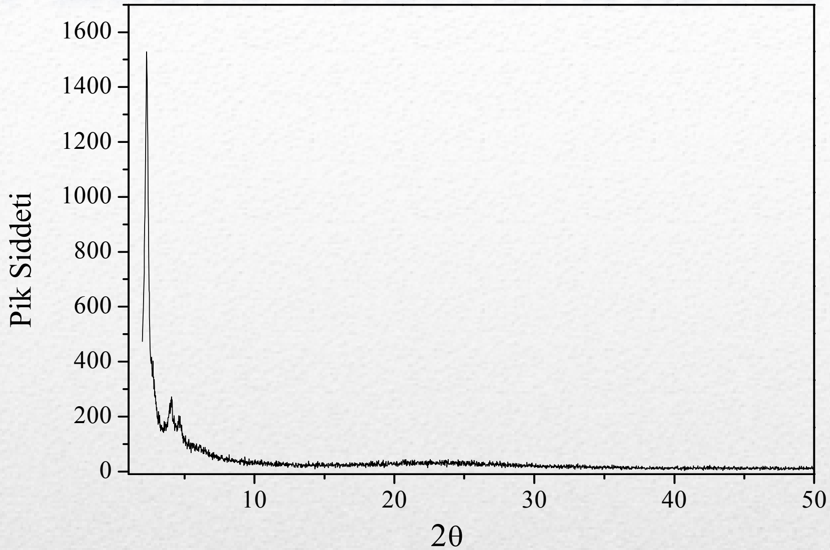
SYNTHESIS OF IRON IMPREGNATED MCM-41

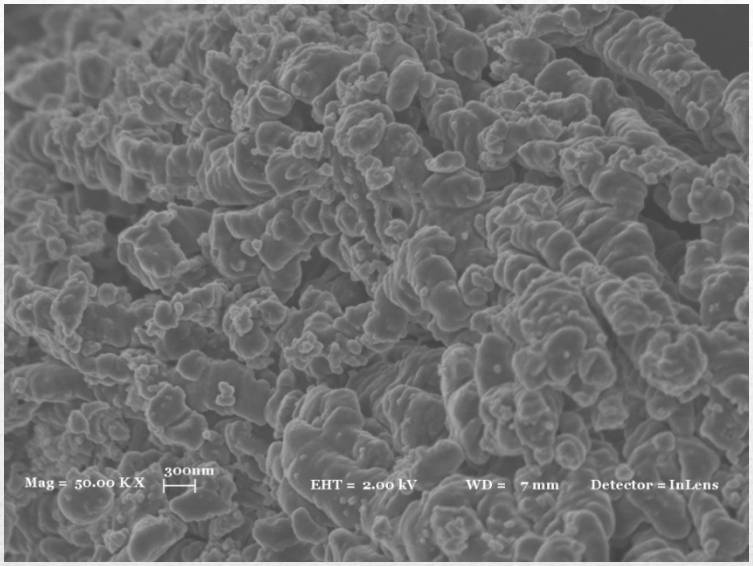


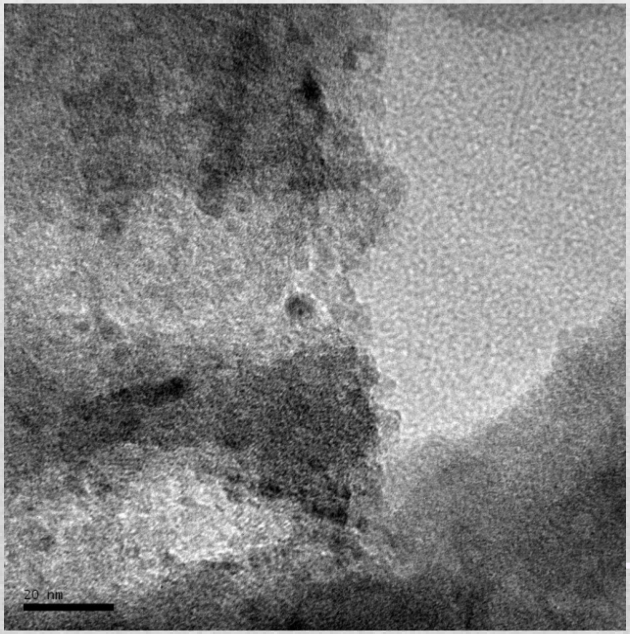
- ✓ Narrow pore size distribution (2-10 nm)
- ✓ High surface areas (1500 m^2/g)
- ✓ High pore volume (1 cm^3/g)
- ✓ Controllable size and morphology
- ✓ Designable chemical composition and functionalizable surface

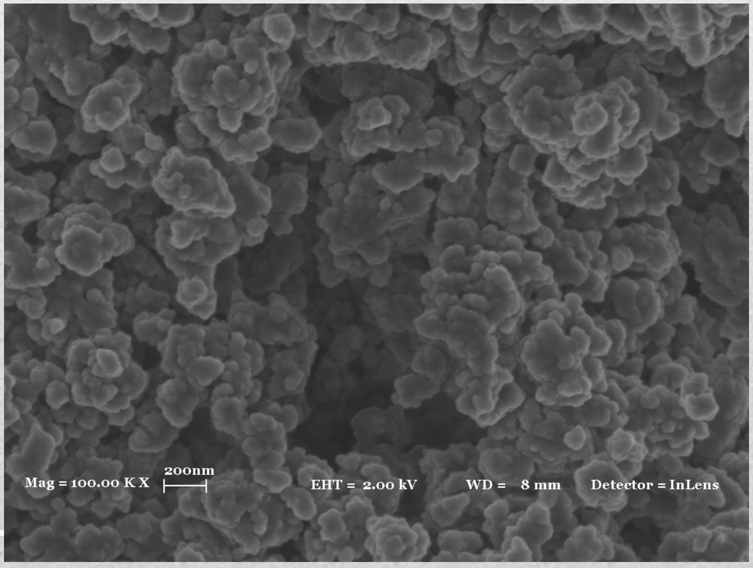
○ Iron impregnated MCM-41 with different metal/Si ratios were obtained by microwave-assisted direct synthesis method.

CHARACTERIZATION OF IRON IMPREGNATED MCM-41









BET CHARACTERIZATION

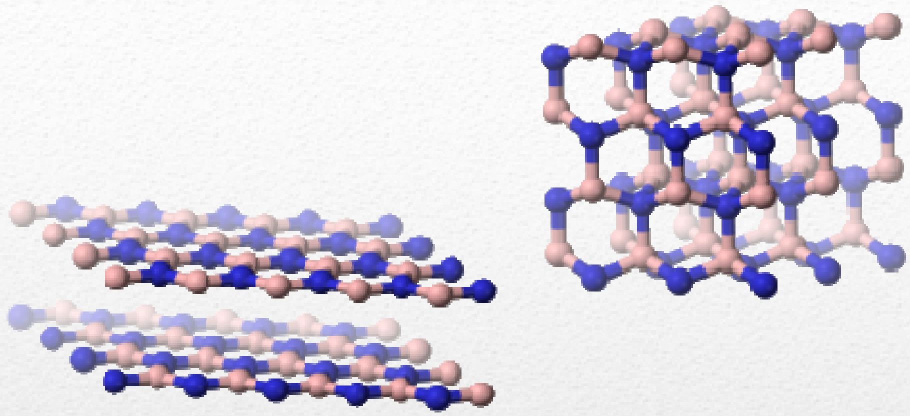
Si/Metal mol ratio	Si/Metal mol ratio (EDX)	BET Surface Area (m^2/g)	BJH Des. Pore volume (cm^3/g)	BJH Des. Pore diameter "d_p" (nm)	d_{100} (nm)	Lattice parameter "a" (nm)	Pore wall thickness "δ" (nm)
Fe-DS-25	0,06	1253	0,53	4,0	3,9	4,50	0,70
Fe-DS-50	0,04	1582	0,59	3,9	3,9	4,50	0,71
Fe-DS-75	0,03	1289	1,32	3,5	3,5	4,04	0,73
Fe-DS-100	0,02	1108	0,82	3,5	3,5	4,09	0,74

✓ The determination was based on the measurements of the adsorption isotherms of nitrogen at 77 K.

✓The specific surface areas were evaluated with the Brunauer–Emmett–Teller (BET) method in the P/P_0 range of 0.05–0.35.

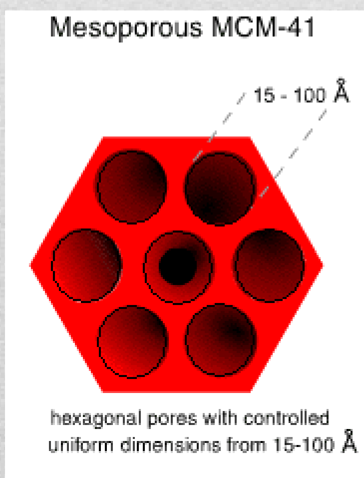
SYNTHESIS OF BORON NITRIDE NANOTUBES

Production of Iron Impregneted MCM-41 by using Microwave heating

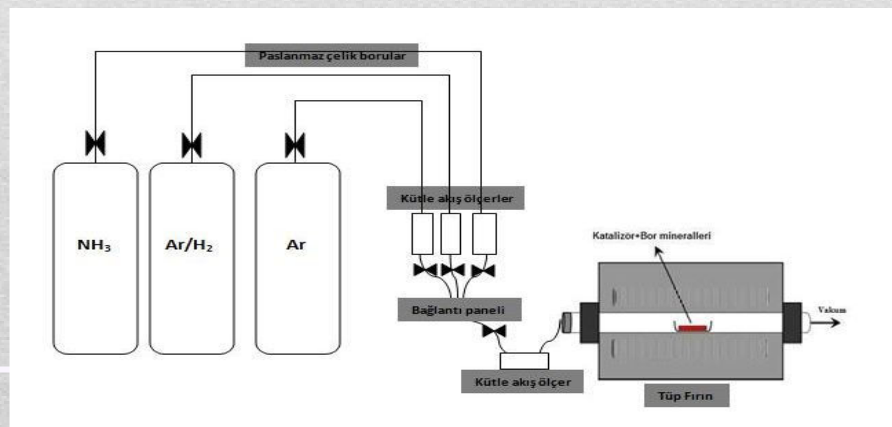


Characterization of BNNTs

Characterization of Iron Impregneted MCM-41



Production of BNNTs by using Iron Impregneted MCM-41 as the catalyst



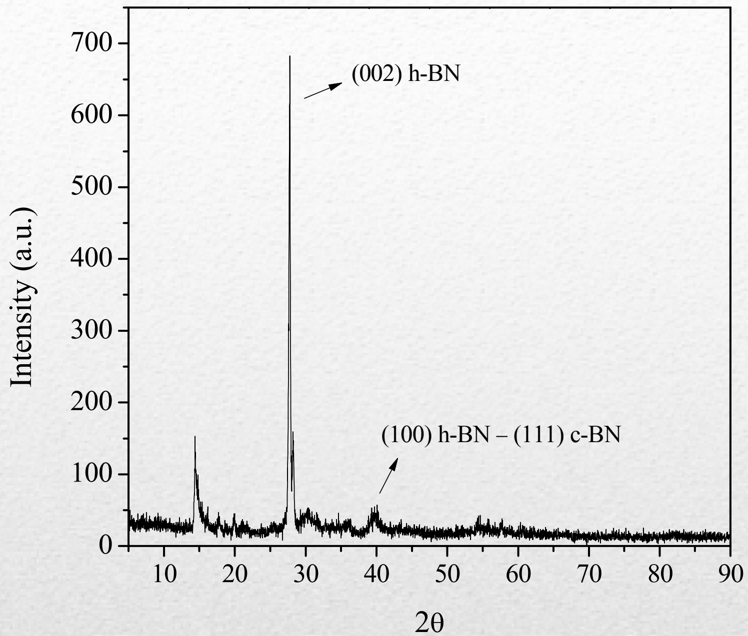
PURIFICATION OF BORON NITRIDE NANOTUBES

The sample obtained from the CVD treatment was mixed with about 50 mL of 4 M HCl solution and kept for 4 hours at room temperature.

After HCl treatment, 50 mL of 1 M HNO_3 solution was poured to the reaction mixture and stirred for 24 hours at 50°C

At the end of purification process, the solution was filtered through filter paper with 0.45 µm pore size and washed with distilled water

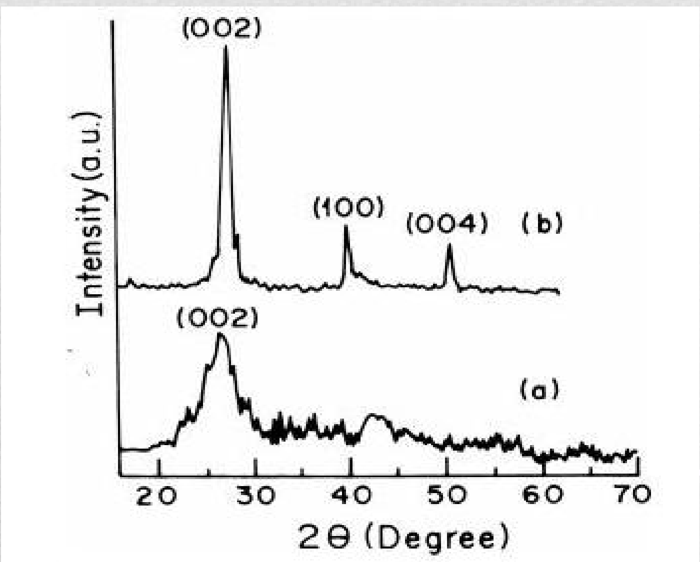
XRD CHARACTERIZATION

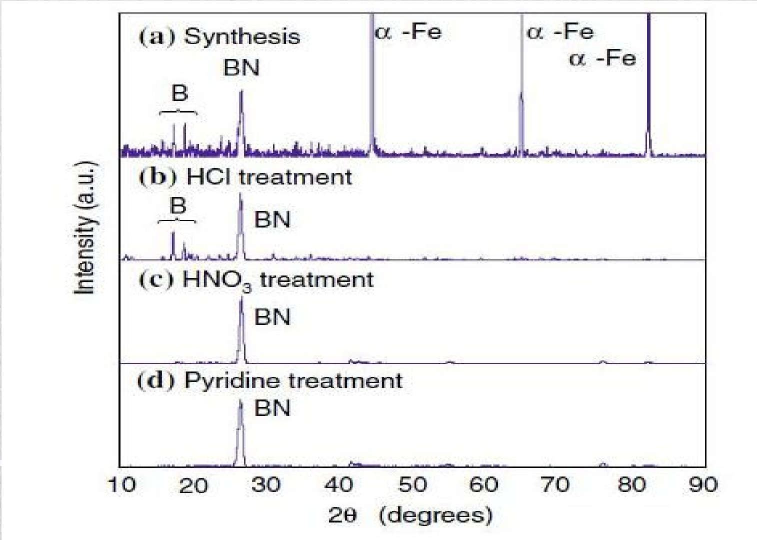


✓ The characteristic peaks of hexagonal-BN were observed at about 2θ=27.3° (002) and 2θ=41.2° (100).

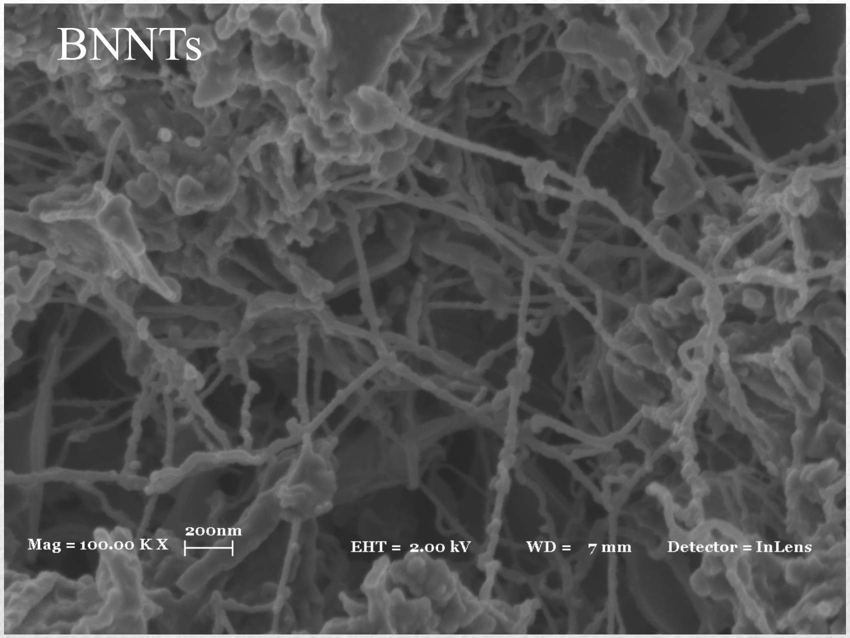
✓ The small and broad peak near 2θ=41.2° was assigned to (111) peak of cubic-BN overlapped with (100) hexagonal-BN peak.

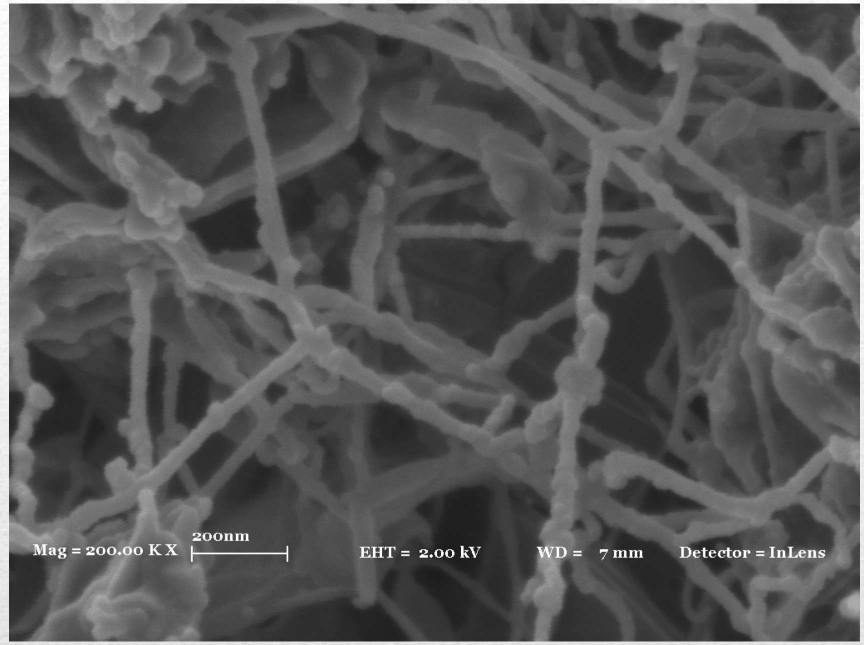
✓ These noticeable BN peaks showed that most of side products were removed successfully by the separation steps.

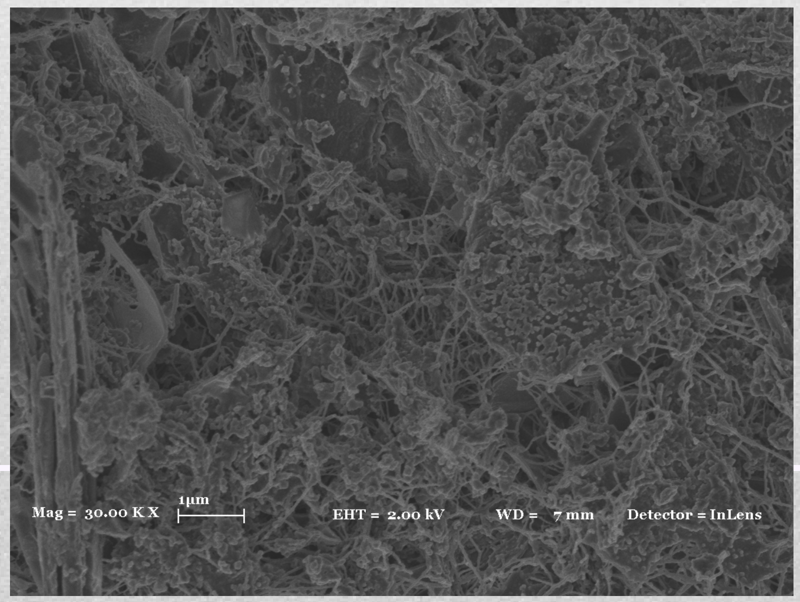




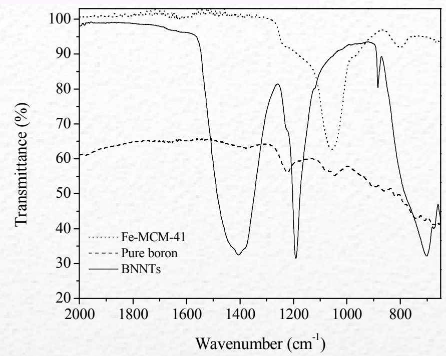
SEM CHARACTERIZATION

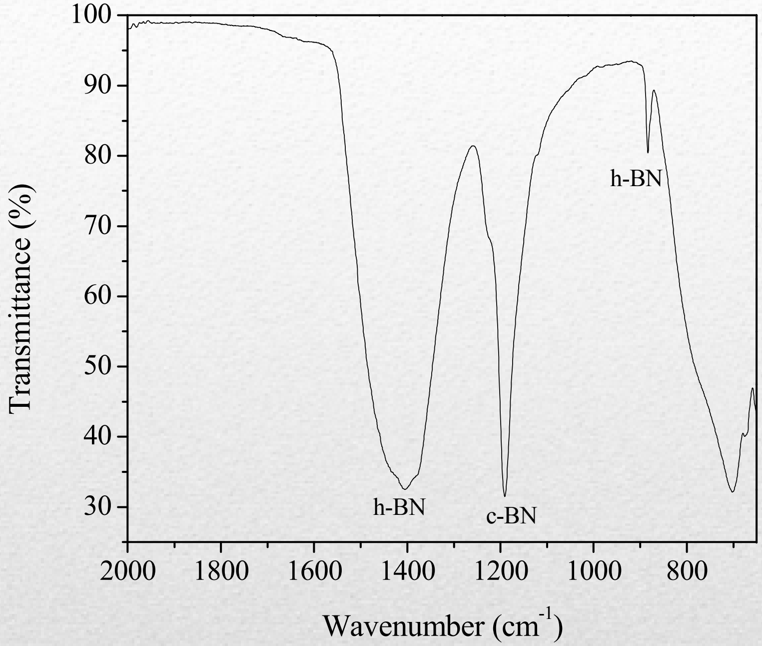






FTIR ANALYSES OF BNNT

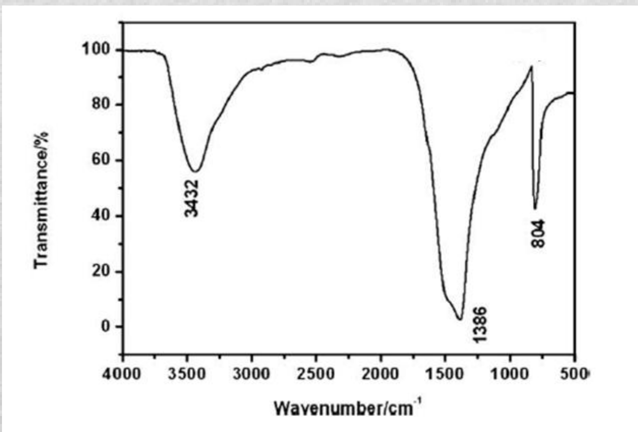


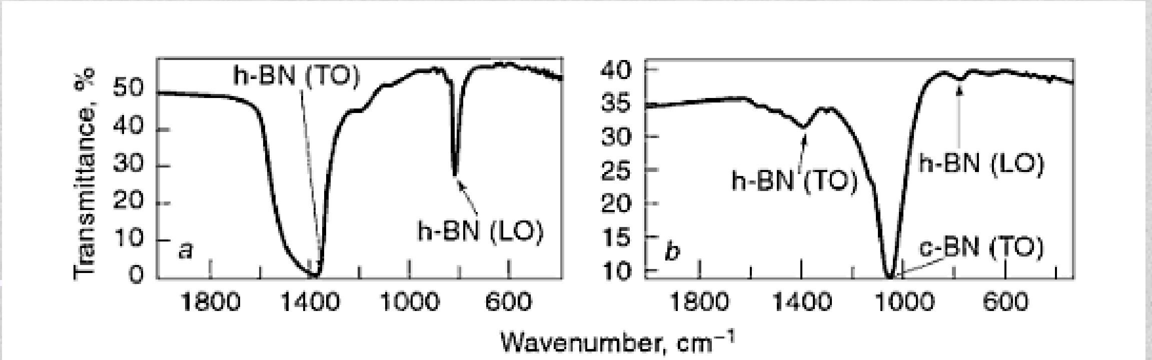


✓ These spectra are dominated by 1110 cm-1 band due to the c-BN structures and, 1383 cm^{-1} and 813 cm^{-1} bands due to h-BN structures.

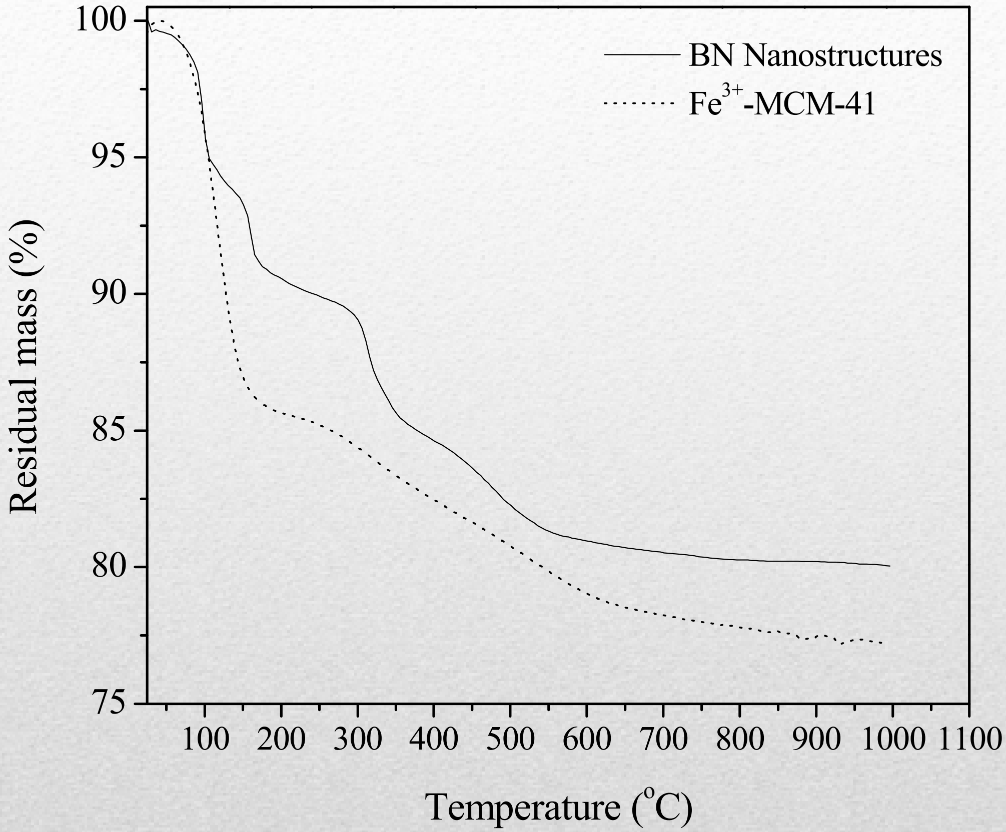
✓ The FTIR spectrum contained a strong and broad peak near 1400 cm^{-1} due to in-plane sp^2 bonded B-N stretching vibrations.

✓The peak near 850 cm^{-1} assigned to the B-N-B out-of-plane bending vibration encountered in h-BN formation.





THERMAL GRAVIMETRIC ANALYSIS



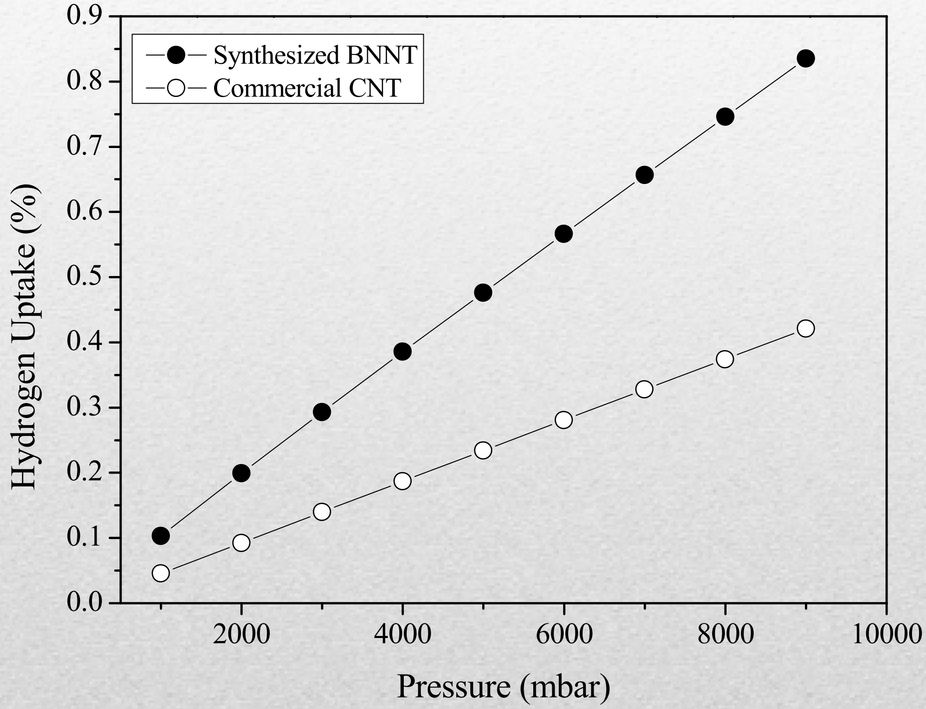
✓ Iron impregnated MCM-41 seemed to be very stable at temperatures higher than 200°C.

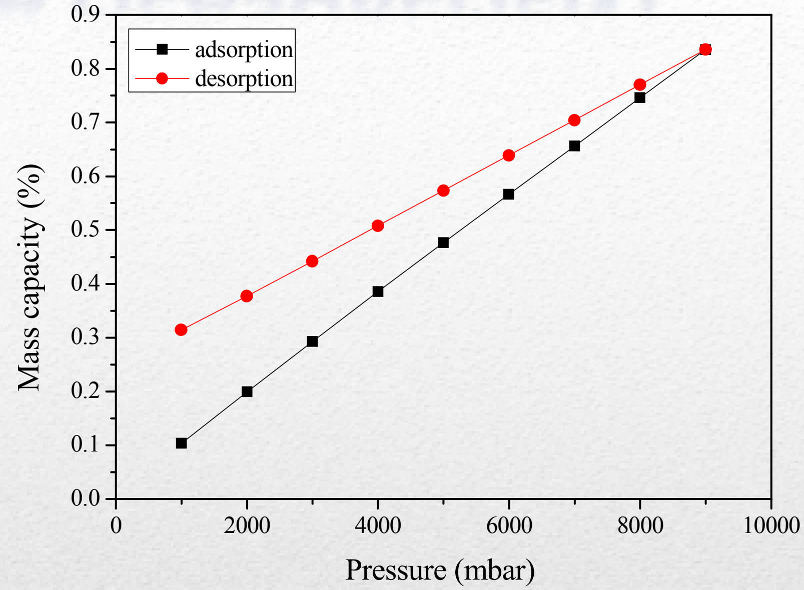
✓ At 150°C moisture present in the sample was lost. As the temperature reached to 350°C and 500°C two sets of oxidative reactions occurred.

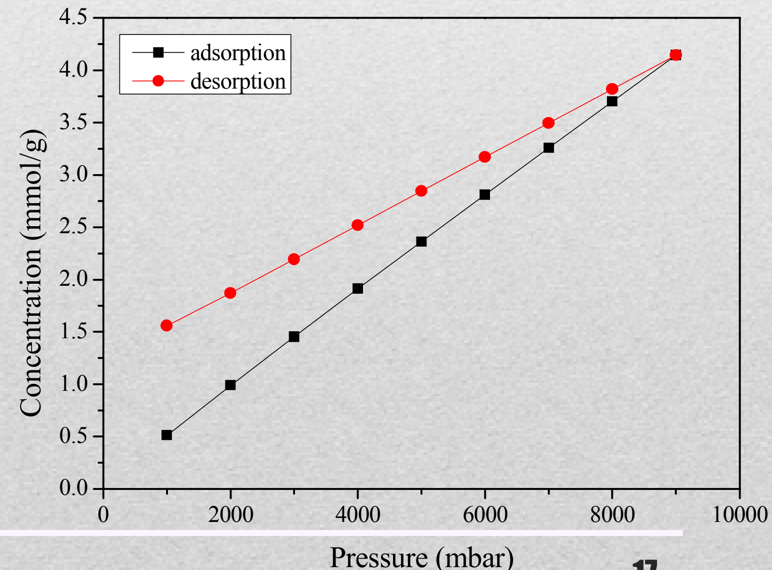
✓At both of the temperatures about 5% of mass of the BNNTs were lost.

✓Beyond 550°C the BNNTs seemed to be very stable up to 1000°C.

HYDROGEN STORAGE- IGA MEASUREMENT







CONCLUSION

- BN nanotubes were successfully grown over iron impregnated MCM-41 at a relatively low temperature of 750^{o}C for 1 hour by CVD technique.

- BN nanotubes were obtained after the purification procedure including HCl and HNO_3 treatments to remove impurities.

- SEM image showed the formation of nano-fibrous network BN structures in the diameter range of 20 nm to 40 nm.

- Both XRD and FTIR characterization results supported the formation of h-BN and c-BN nanostructures.

- Oxidative TGA results indicated that the synthesized BN nanostructures were thermally stable at temperatures higher than 550^{o}C.

- Hydrogen storage measurements via IGA showed that BNNTs could adsorb 0.85 wt% hydrogen which was two times larger than for commercial CNTs.

ACKNOWLEDGEMENTS

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- Thank you!!