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APPENDICES

Appendix A Synthesis of Polybenzoxazine

Polybenzoxazine can be synthesized via a solvent method proposed by Ishida (1996) through the Mannich condensation reaction of phenol, formaldehyde, and amine, having water as by-product. Figure A1 shows the synthesis reaction and thermal polymerization of polybenzoxazine.

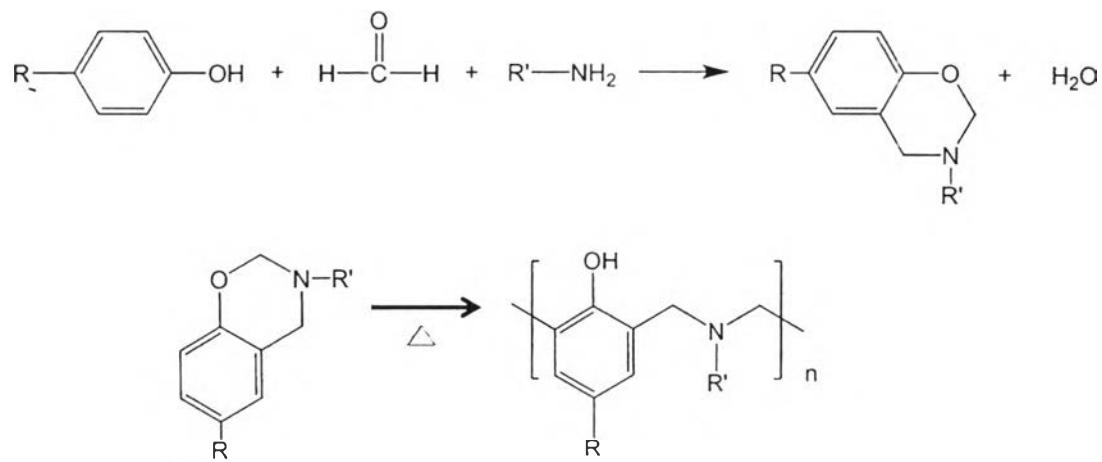


Figure A1 Synthesis reaction of polybenzoxazine.

Appendix B Peak Positions and Assignments in the FTIR Spectrum of Benzoxazine

The chemical structure of the benzoxazine, main-chain type benzoxazine polymer derived from bisphenol A, formaldehyde, and triethylenetetramine (teta), was confirmed by FTIR technique. The intensity of trisubstituted benzene ring at 1504 cm⁻¹ and out-of-plane bending vibrations of the benzene ring at 936 cm⁻¹ confirmed that the main-chain type benzoxazine precursor was successfully obtained. After the precursors were fully cross-linked, the intensities of these two bands significantly decreased, indicating that the ring-opening of oxazine ring took place.

Table B1 Peak positions and assignments in the FTIR spectrum of resulting benzoxazine precursors

Frequency (cm ⁻¹)	Assignments	Benzoxazine precursors	Poly benzoxazine	Carbon xerogels
1230	asymmetric stretching of C-O-C	✓	✓	✗
1185	asymmetric stretching of C-N-C	✓	✓	✗
1325-1328	CH ₂ wagging of oxazine ring	✓	✓	✗
1504	trisubstitute benzene ring out-of-plane bending of benzene ring	✓	↓	✗
936	to which oxazine ring is attached	✓	↓	✗

Notes: ✓ = peak was detected, ✗ = peak was not detected, and ↓ = peak was decreased

Appendix C Peak Positions and Assignments in the $^1\text{H-NMR}$ spectrum of Benzoxazine

The chemical structure of the benzoxazine, main-chain type benzoxazine polymer derived from bisphenol A, formaldehyde, and triethylenetetramine (teta), was confirmed by $^1\text{H-NMR}$ technique. The $^1\text{H-NMR}$ spectrum indicates the oxazine ring formation, as can be seen from the characteristic peaks at 4.82 ppm ($\text{O-CH}_2\text{-N}$) and 3.94 ppm ($\text{Ar-CH}_2\text{-N}$), representing the protons of methylene bridge in oxazine ring. The peak at 1.55 ppm corresponds to the protons of methyl groups in bisphenol-A. Moreover, the proton resonance belonging to aliphatic amine is found at 2.86 ppm and the resonance at 3.70 ppm represents the protons of $-\text{CH}_2-$ of the opened oxazine ring, indicating that MCBP(BA-teta) was in a partially-cured state.

Table C1 Peak positions and assignments in the $^1\text{H-NMR}$ spectrum of resulting benzoxazine precursors

Peak position (ppm)	Assignments
4.82	$\text{O-CH}_2\text{-N}$
3.94	$\text{Ar-CH}_2\text{-N}$
1.55	$-\text{CH}_3$ in Bisphenol-A
2.86	aliphatic amines
3.70	$-\text{CH}_2-$ of opened oxazine ring

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Publications:

1. Thubsuang, U.; Ishida, H.; Wongkasemjit, S.; and Chaisuwan, T. (2012) Novel template confinement derived from polybenzoxazine-based carbon xerogels for synthesis of ZSM-5 nanoparticles via microwave irradiation. Microporous and Mesoporous Materials, 156, 7-15.
2. Thubsuang, U.; Ishida, H.; Wongkasemjit, S.; and Chaisuwan, T. (2014) Self-formation of 3D interconnected macroporous carbon xerogels derived from polybenzoxazine by selective solvent during the sol-gel process. Journal of Materials Science, 49(14), 4946-4961.
3. Thubsuang, U.; Ishida, H.; Wongkasemjit, S.; and Chaisuwan, T. (2014) Effects of non-ionic and cationic surfactant on porous structure of polybenzoxazine-based carbon xerogels. Journal of Colloid and Interface Science, submitted.
4. Thubsuang, U.; Ishida, H.; Wongkasemjit, S.; and Chaisuwan, T. (2014) Improvement in the pore structure of polybenzoxazine-based carbon xerogels through a silica templating method. Journal of Porous Materials, accepted, in press. DOI 10.1007/s10934-014-9786-7

Presentations:

1. Thubsuang, U.; Wongkasemjit, S.; and Chaisuwan, T. (2010, March 21-25) Effects of solvents and concentrations on morphology of carbon aerogel derived from polybenzoxazine. Paper presented at The 239th ACS National Meeting & Exposition Conference 2010, San Francisco, USA.
2. Thubsuang, U.; Wongkasemjit, S.; and Chaisuwan, T. (2010, July 10) Synthesis of polybenzoxazine-based carbon aerogels and their applications. Oral presentation at Chula-Inha Joint Symposium on Advanced Polymers and Nanomaterials, Bangkok, Thailand.
3. Thubsuang, U.; Ishida, H.; Wongkasemjit, S.; and Chaisuwan, T. (2012, June 10-14) Polybenzoxazine-based porous carbon: Pore structure design and its application. Oral presentation at The 4th International Conference “Smart Materials, Structures and Systems” (CIMTEC 2012), Montecatini Terme, Italy.
4. Thubsuang, U.; Ishida, H.; Wongkasemjit, S.; and Chaisuwan, T. (2012, August 26-31) Textural properties of polybenzoxazine-based porous carbon and its application for synthesis of ZSM-5 nanoparticles and catalytic activity. Oral presentation at International Union of Materials Research Society-International Conference in Asia 2012 (IUMRS-ICA 2012), Busan, Republic of Korea.
5. Thubsuang, U.; Wongkasemjit, S.; and Chaisuwan, T. (2012, November 11-13) Development of polybenzoxazine-based carbon xerogels as a candidate adsorbent for wastewater treatment. Paper presented at The First International Symposium on Advanced Water Science & Technology (ISAWST-1), Nagoya, Japan.
6. Thubsuang, U.; Ishida, H.; Wongkasemjit, S.; and Chaisuwan, T. (2013, May 7-8) Novel template confinement derived from polybenzoxazine-based carbon xerogels for synthesis of ZSM-5 nanoparticles via microwave irradiation. Paper presented at The 2nd Thailand National Research University Summit (NRU Summit II), Bangkok, Thailand.