REFERENCES

- Bailey, W. J., Sun, L. A., Katsuki, H., Endo, A., Iwama, H., Tsushima, R.,
 Saigo, K., and Brittito, M. M. (1977). "Ring-Opening Polymerization with Expansion in Volume", <u>American Chemical</u> Society Symposium Series, 59, 38.
- Cairn, T., Eglinton, G. (1965). "Hydrogen Bonding in Phenols. Part II. Alkyl Substituted Bis(hydroxyphenyl)alkanes (Dinuclear Novolaks)", Journal of Chemical Soceity, 5906-5913.
- Dunkers, J. P., Zarate, A., Ishida, H. (1996). "Crystal Structure and Hydrogen Bonding Characteristics of N,N-Bis(3,5-dimethyl-2hydroxybenzyl) methylamine : A Benzoxazine Dimer", <u>Journal</u> <u>of Physical Chemistry</u>, 100, 13514-13525.
- Holly, F. W., Cope, A. C. (1944). "Condensation Products of Aldehydes and Ketones with *o*-Aminobenzyl Alcohol and *o*-Hydroxybenzylamine", <u>Journal of American Chemical Society</u>, 66, 1875-1879.
- Ishida, H., Rodriguez, Y. (1995). "Curing Kinetic of a New Benzoaxine-Based Phenolic Resin by Differential Scanning Calorimetry", <u>Polymer</u>, 36, 3151-315.
- Ishida, H.; Allen, D. J. (1996). "Physical and Mechanical Characterization of Near-Zero Shrinkage Polybenzoxazines", <u>Journal of polymer</u> <u>Science: Part B: Polymer Physics</u>, 34, 1019-1030.

- Ishida,H., Hong, Y. L. (19970. "A Study on the Volumetric Expansion of Benzoxazine-Based on Phenolic Resin", <u>Macromolacules</u>, 30, 1099-1106.
- Ishida, H., Allen, D. J., Dhumrongvaraporn, S., Wirasate, S. (1998). "Molecular Origin of Unusual Physical and Mechanical properties in Novel Phenolic materials Based on Benzoxazine Chemistry", Journal of Applied Polymer Science, 70, 1299-1306.
- Knop, A., Pilato, L. A. (1985). <u>Phenolic Resin</u>. New York: Springer-Verlag.
- Ning, X., Ishida, H. (1994). "Phenolic Materials via Ring-Opening Polymerization of Benzoxazines ; Effect of Molecular Structure on Mechanical And Dynamic Mechanical Properties", <u>Journal of</u> <u>Polymer Science., Polymer Physical Education</u>, 32, 921-933.
- Ning, X., Ishida, H. (1994). "Phenolic Materials via Ring-Opening Polymerization: Synthesis and Characterization of Bisphenol-A Based Benzoxazines and Their Polymers", <u>Journal of Polymer</u> <u>Science: Part A: Polymer chemistry</u>, 32, 1121-1129.
- Riess, G., Schwob, J. M., Guth, G., Roche, M., Lande, B. (1986). <u>Advances in Polymer Science</u>. New York: (Eds. B.M. Culbertson and J.F. McGrath) Plenum.
- Rosato, D. (1985). Reinforced Plastics. <u>Encyclopedia of Polymer Science</u> <u>and Engineering</u>, 14, 362-365.
- Russell, B. M., Koening, J. L., Low, H. Y., Ishida, H. (1998). "Study of the Characterization and Curing of Benzoxazines Using ¹³C Solid-State Nuclear Magnetic Resonance", <u>Journal of Applied Polymer</u> <u>Science</u>, 70, 1413-1425.
- Schreiber, H. (1973). Ger. Offen. 2225504.

- Sollner, G. (1985). Tooling in Plastics. <u>Encyclopedia of Polymer Science</u> and Engineering, 16, 863-864.
- Sun, Chivin. (2000). <u>Synthesis of Spirosilicate Directly from Silica and</u> <u>Ethylene Gylcal / Ethylene Glycal Derivatives</u>. M.S. Thesis in Polymer Science, Petroleum and Petrochemical College, Chulalongkorn University.
- Yokosawa, T., Sato, M., Endo, T. (1990). "Preparation and Polymerization of Spiroorthoester Bearing the Perfluoroalkyl Group", <u>Journal of</u> <u>Polymer Science: Part A: Polymer chemistry</u>, 28, 1841-1846.

APPENDIX I

Theoretical calculation of % ceramic yield of purified monomers

From the combustion of a sample in TGA technique, it can be represented in eq. (A-1). Silica is the final product that left from the reaction, molecular weight of SiO_2 was used to calculate %ceramic yield, as shown in (A-2).

$$ightarrow SiO_2 + CO_2 + H_2$$
 (A-1)

%Ceramic yield =
$$\frac{\text{MW of SiO}_2}{\text{MW of sample}} \times 100$$
 (A-2)

The molecular weight of the sample can be calculated from the chemical formula obtained from chemical structure of product. For example, spirosilicate C2 product has chemical structure presented in eq. (A-1). The chemical formula of spirosilicate C2 is $Si(OCH_2CH_2O)_2$. Then %ceramic yield of spirosilicate C2

%Ceramic yield =
$$\frac{MW \text{ of } SiO_2}{MW \text{ of } Si(OCH_2CH_2O)_2} \times 100$$

$$= 60 \times 100 = 40.54$$

APPENDIX II

Calculation of Density of the Products

Density measurement for both monomers and polymers were studied using 25 ml pycnometer and distilled isooctane as a media at 25°C. The steps of density measurement are as followed;

the weight of sample	\equiv (A) g.
the weight of sample + isooctar	$he \equiv (B) g.$
the density of isooctane at	the set temperature obtaining from
reference	\equiv (C) g/cm ³
the weight of isooctane	= (B) - (A) $=$ D g.
the volume of isooctane	$= D / (C) = E cm^{3}$
the volume of sample	= the volume of the bottle - E
	$= G cm^3$
then the density of sample	= (A) / G g/cm ³

APPENDIX III

Finding the optimum conditions for curing the products

 Table A-1 %Ceramic yields of spirosilicate C2 with varying temperature.

Temperature (°C)		%Ceramic Yield	
	l st	2 nd	average
monomer	44.90	45.14	45.02
80	68.92	66.52	67.72
100	66.98	72.58	69.78
*120	75.17	75.09	75.13
140	77.94	75.74	76.84
160	77.43	80.53	78.98
180	80.05	78.83	79.44

*Suitable condition

Table A-2	%Ceramic yields of aminospirosilicate C3 with vary in
	temperature.

Temperature (°C)		%Ceramic Yield	
	l st	2 nd	average
monomer	33.59	31.23	32.41
80	54.69	53.31	54.00
100	64.96	67.62	66.29
120	69.63	67.47	68.55
*140	71.64	73.90	72.77
160	74.56	72.20	73.38
180	72.44	74.90	73.67

Table A-3 %Ceramic yields of aminospirosilicate C4 with vary in temperature.

Temperature (°C)		%Ceramic Yield	
	1 st	2 nd	average
monomer	25.47	28.05	26.76
80	33.27	30.75	32.01
100	40.98	39.16	40.07
120	45.96	46.74	46.35
140	54.92	53.02	53.97
*160	66.96	68.82	67.89
180	67.92	67.54	67.73

Time (hr)		%Ceramic Yield	
-	l st	2 nd	average
monomer	44.23	45.81	45.02
0.5	63.48	60.98	62.23
*1	74.55	73.07	73.81
2	74.22	72.76	73.49
3	74.47	76.51	75.49
4	76.68	75.00	75.84
5	75.59	75.65	75.62

Table A-4 %Ceramic yields of spirosilicate C2 with varying time at 120°C.

Table A-5%Ceramic yields of aminospirosilicate C3 with varying time at
140°C.

Time (hr)	%Ceramic Yield		
	l st	2 nd	average
monomer	44.23	45.81	32.41
0.5	63.48	60.98	50.62
*1	74.55	73.07	72.77
2	74.22	72.76	71.77
3	74.47	76.51	73.24
4	76.68	75.00	74.50
5	75.59	75.65	74.96

Time (hr)	0	%Ceramic Yield	
-	l st	2 nd	average
monomer	25.95	27.57	26.76
1	68.65	66.81	67.73
*2	71.05	72.99	72.02
3	73.32	71.08	72.20
4	71.44	73.62	72.53
5	71.84	72.54	72.19

Table A-6 %Ceramic yields of aminospirosilicate C4 with varying time at160°C.

Table A-7 Density results of the spirosilicate C2 product.

	Density (g/cm ²)	
Sample	Monomer	Polymer
1	1.7442	1.7478
2	1.7624	1.7887
3	1.6961	1.7187
average	1.7342	1.7517

	Density (g/cm ³)	
Sample	Monomer	Polymer
1	1.6076	1.6269
2	1.6145	1.6388
average	1.6111	1.6329

 Table A-8 Density results of the aminospirosilicate C3 product.

 Table A-9 Density results of the aminospirosilicate C4 product.

	Density (g	g/cm ³)
Sample	Monomer	Polymer
1	1.5437	1.5673
2	1.5636	1.5602
average	1.5537	1.5638

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