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APPENDICES

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จุฬาลงกรณ์มหาวิทยาลัย

APPENDIX A

A. Degree of Polymerization of Poly(diethylbenzalmalonate vinyl ether)

The weight average molecular weight (\bar{M}_w) of poly(diethylbenzalmalonate vinyl ether) obtained by gel permeation chromatography technique (GPC) was 1943.

The average degree of polymerization was calculated by the following equation:

$$\text{Average degree of polymerization} = \frac{\bar{M}_w \text{ of polymer}}{\text{MW of monomeric unit}}$$

Since MW of monomeric units was 290.13,

$$\begin{aligned} \text{Therefore, the average degree of polymerization of this compound} &= \frac{1943}{290.13} \\ &= 6.7 \end{aligned}$$

Structure of synthesized poly(diethylbenzalmalonate vinyl ether) can therefore, be expressed as follows:

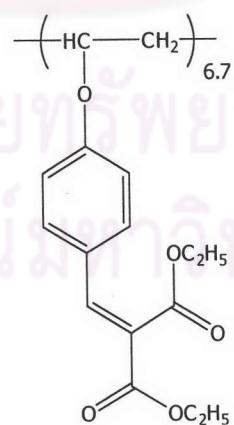


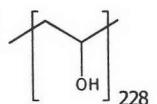
Figure A.1 Structure of poly(diethylbenzalmalonate vinyl ether).

B. Substitution Degree of Cinnamoyl Groups in Poly[(Vinyl 2,4,5-Trimethoxy cinnamate)(Vinyl Alcohol)] Copolymer

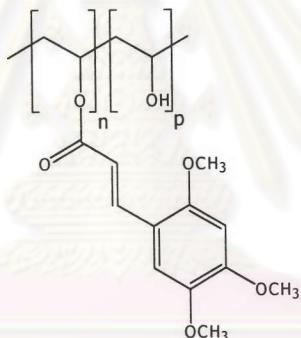
B.1 Calculation from Weight Average Molecular Weight (\bar{M}_w) Data

The GPC analysis of poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer (90°C grafted compound) gave weight average molecular weight (\bar{M}_w) of 31557. From the molecular weight of the grafted product and the weight average molecular weight of the starting material (PVA; $\bar{M}_w = 10024$), the average degree of cinnamoyl substitution of this compound could be calculated.

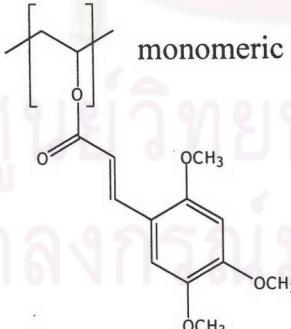
- One mole of PVA ($\bar{M}_w = 10024$) consists of 228 hydroxyl units



Given general structure of poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] as follows:



Where, MW of monomeric unit is 264 and



MW of monomeric unit is 44,

We can obtain $(264 \times n) + (44 \times p) = 31557$ ----- (1)

$$n + p = 228; p = 228 - n \quad \text{----- (2)}$$

$$(1) \text{ and } (2) \quad [(264 \times n) + 44 \times (228 - n)] = 31557$$

$$220n = 21569$$

$$\therefore n \sim 98, p = 228 - 98 = 130$$

From the above calculation, poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer (90°C grafted product) can be depicted as follows:

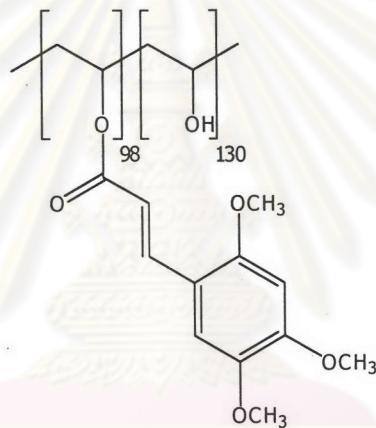


Figure A.2 Structure of poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer.

B.2 Calculation from UV Absorption Data

Since molar absorptivity (ϵ) of 2,4,5-trimethoxycinnamoyl moiety are $12,400 \text{ M}^{-1}\text{cm}^{-1}$ ($\lambda_{\max} = 290 \text{ nm}$) and $14,200 \text{ M}^{-1}\text{cm}^{-1}$ ($\lambda_{\max} = 349 \text{ nm}$)³¹ and molar absorptivity (ϵ) of the grafted product are $1,180,000 \text{ M}^{-1}\text{cm}^{-1}$ ($\lambda_{\max} = 284 \text{ nm}$) and $1,220,000 \text{ M}^{-1}\text{cm}^{-1}$ ($\lambda_{\max} = 342 \text{ nm}$), calculation of degree of substitution can be done as follows:

$$\text{Average degree of cinnamoyl substitution} = \frac{\epsilon's \text{ of a grafted polymer}}{\epsilon's \text{ of 2,4,5-trimethoxycinnamoyl moiety}}$$

$$\text{Therefore, calculated at UVB region} = \frac{1,180,000}{12,400} \sim 96 \text{ unit}$$

$$\text{And when calculated at UVA region} = \frac{1,220,000}{14,200} \sim 86 \text{ unit}$$

From the above calculation, poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer (90°C grafted product) can be expressed as follows:

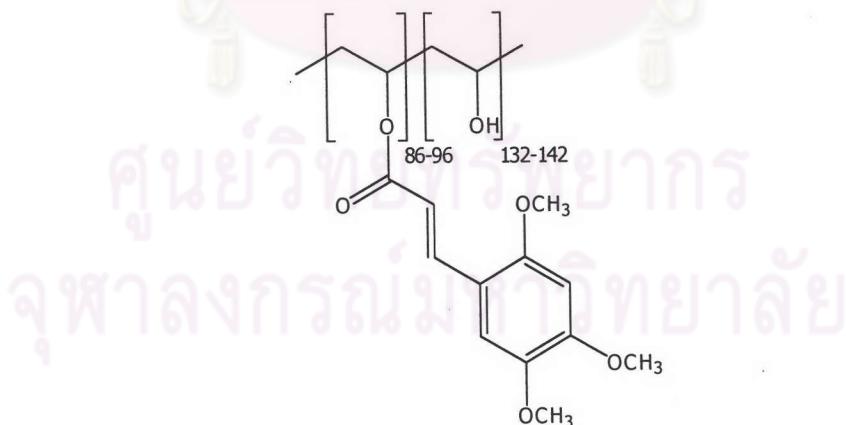


Figure A.3 Structure of poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer.



Figure B.1 ^1H -NMR spectrum of poly(diethylbenzalmalonate vinyl ether).

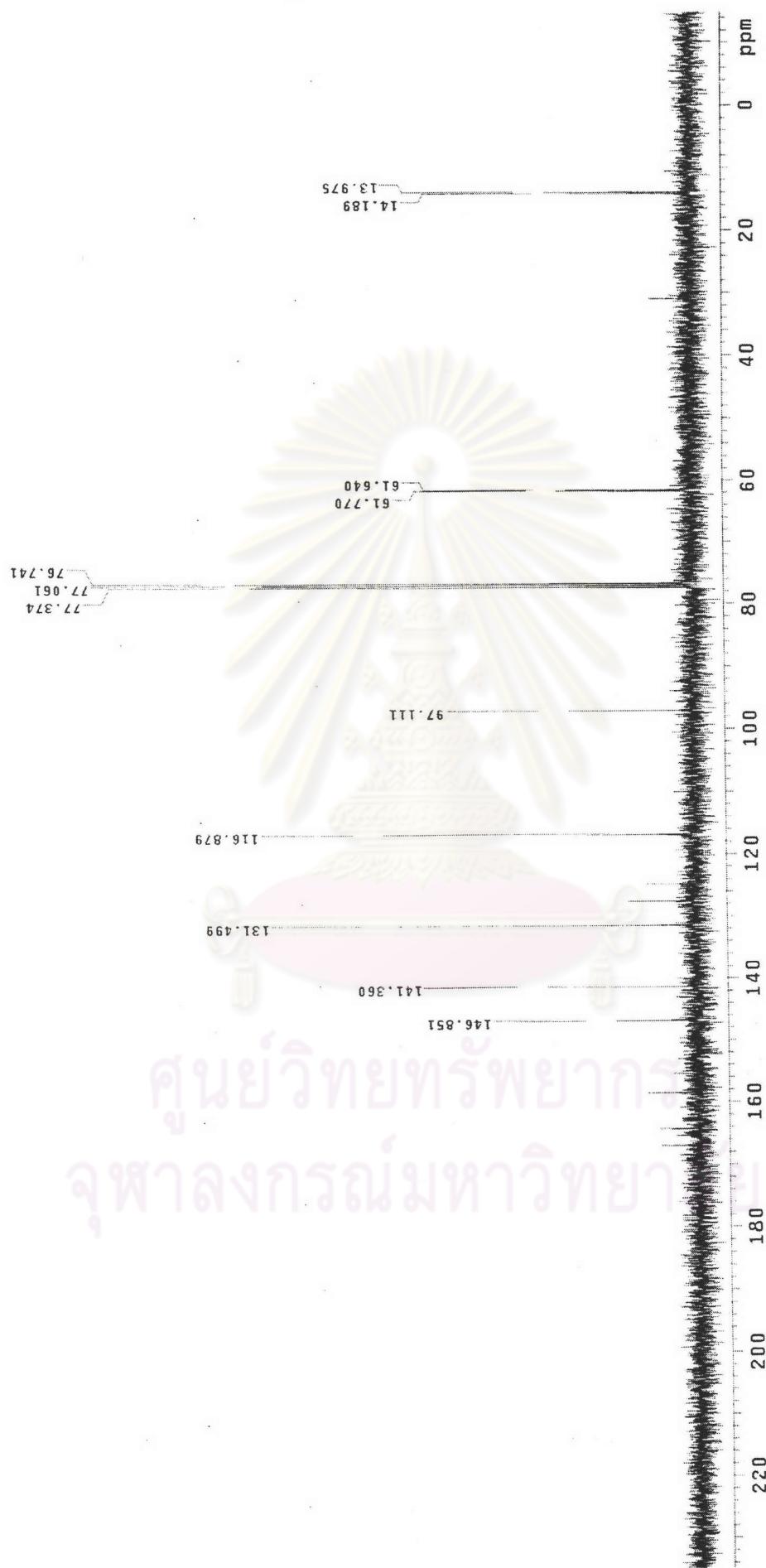


Figure B.2 ^{13}C -NMR spectrum of poly(diethylbenzalmalonate vinyl ether).

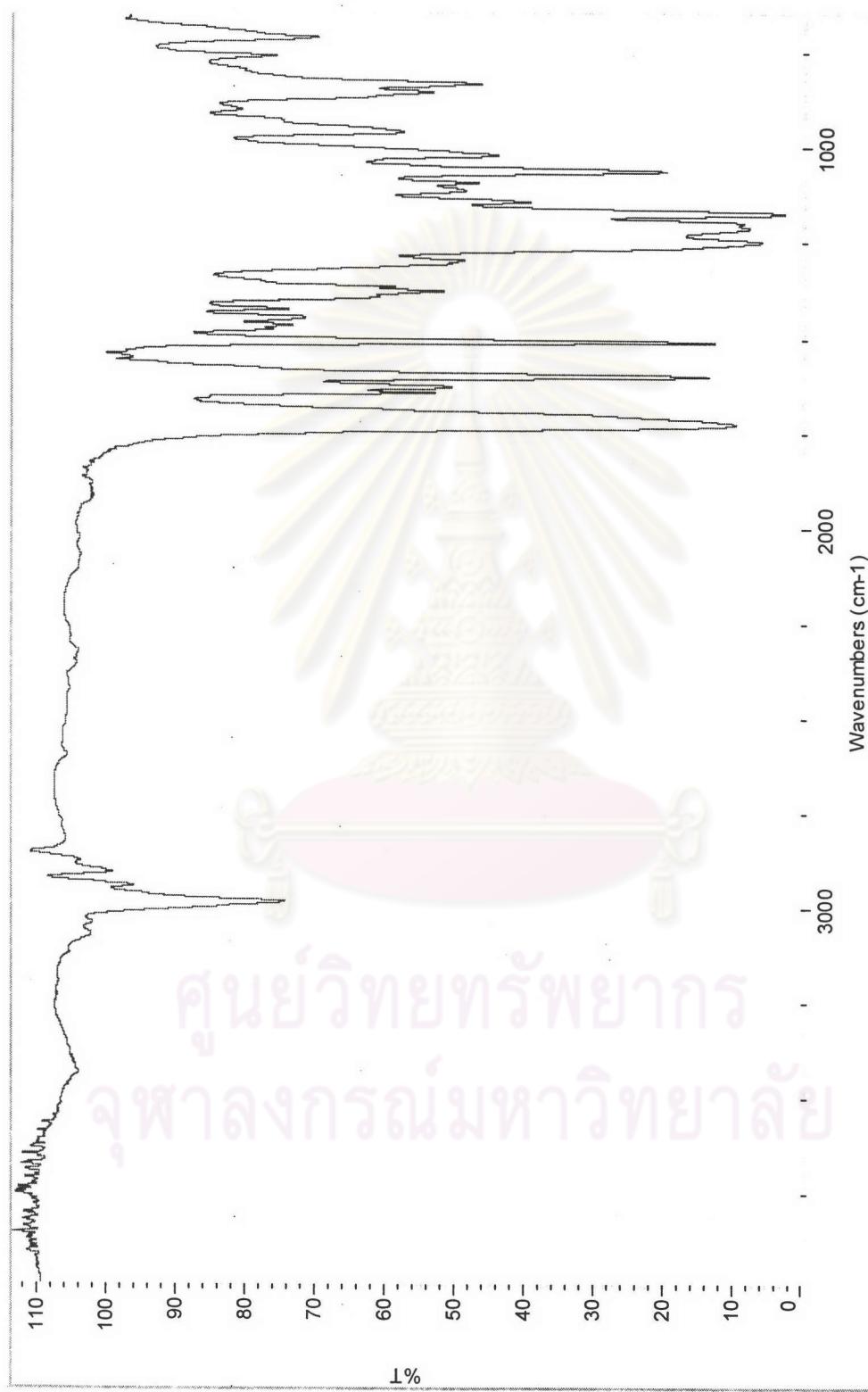


Figure B.3 IR spectrum of poly(diethylbenzalmalonate vinyl ether).

Sample Information

Sample Name poly(diethylbenzalmalonate vinyl ether)
 Vial 1
 Injection Volume 100.00 μ l
 Channel SATIN
 Run Time 22.00 Minutes

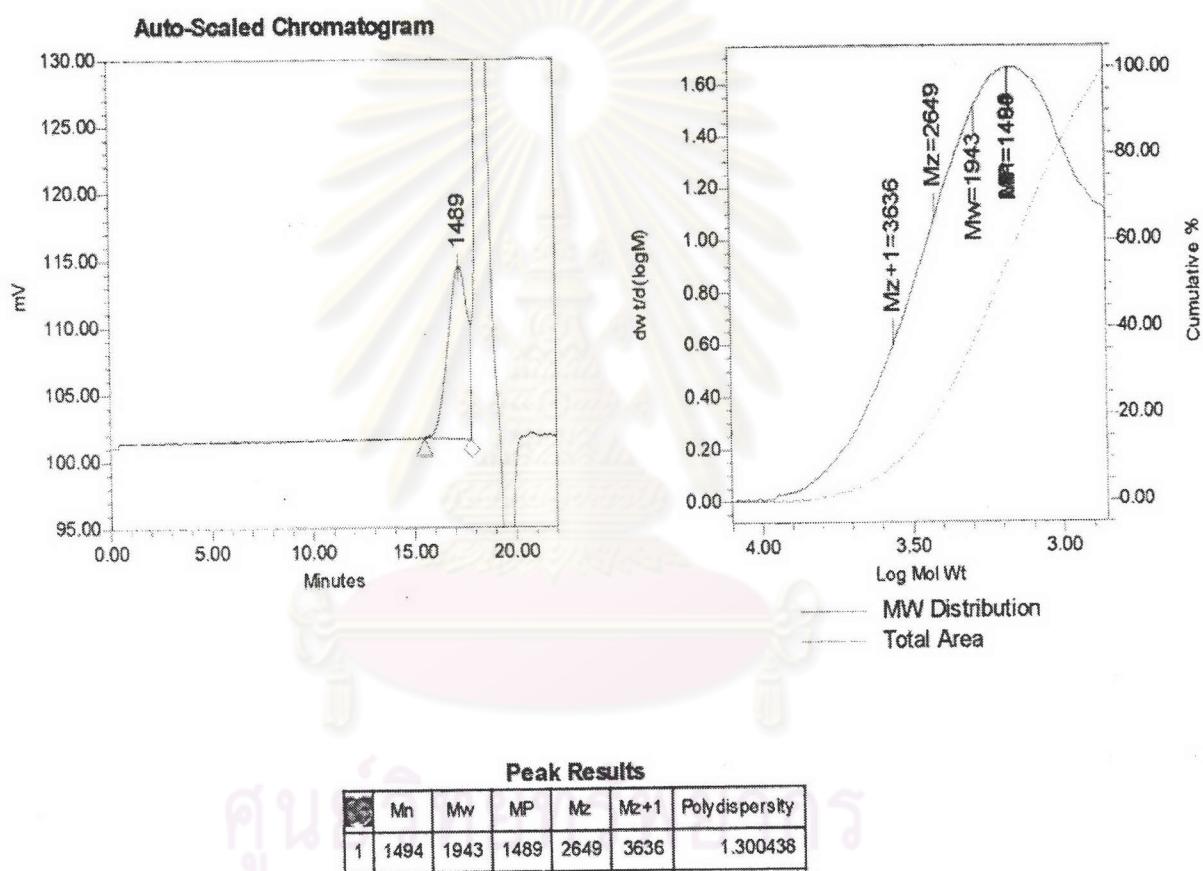


Figure B.4 GPC chromatogram of poly(diethylbenzalmalonate vinyl ether).



Figure B.5 $^1\text{H-NMR}$ spectrum of poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer.

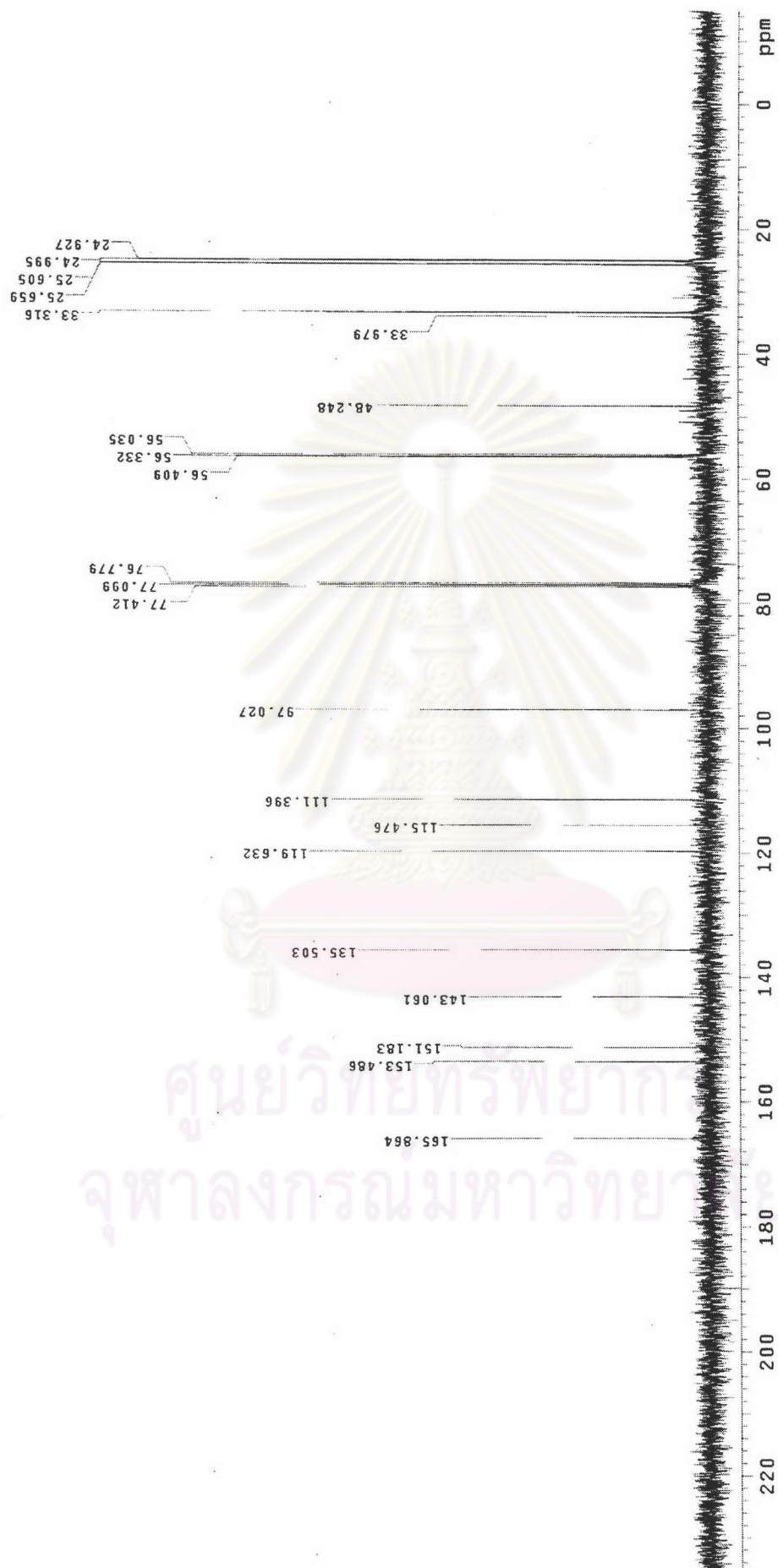


Figure B.6 ^{13}C -NMR spectrum of poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer.

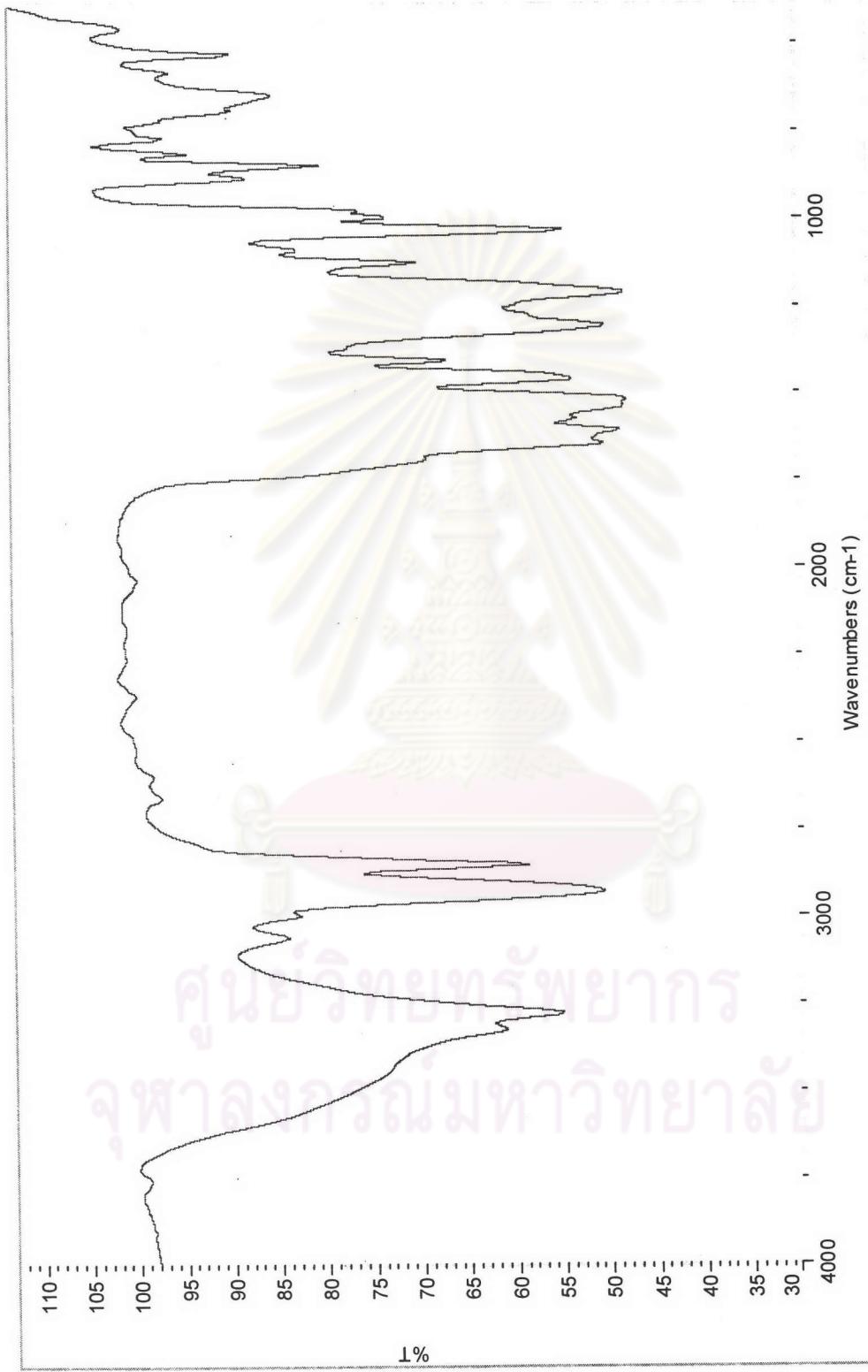
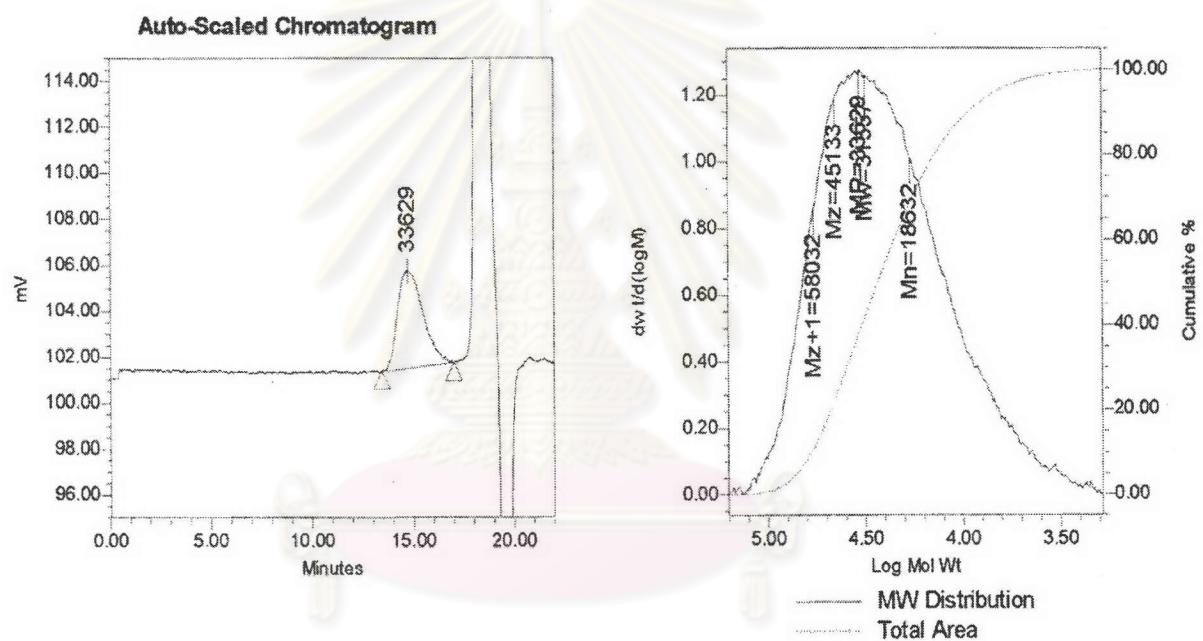


Figure B.7 IR spectrum of poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer.

Sample Information

Sample Name poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer
 Vial 2
 Injection Volume 100.00 μ l
 Channel SATIN
 Run Time 22.00 Minutes



Peak Results						
	Mn	Mw	Mp	Mz	Mz+1	Polydispersity
1	18632	31557	33629	45133	58032	1.693749

Figure B.8 GPC chromatogram of poly[(vinyl 2,4,5-trimethoxycinnamate)(vinyl alcohol)] copolymer.

VITA

Miss Piyawan Hirunsupachot was born on April 17, 1981 in Bangkok. She got a Bachelor Degree of Science in Chemistry from Chulalongkorn University in 2002. After that, Miss Hirunsupachot has been a graduate student pursuing a Master Degree in Organic Chemistry at Chulalongkorn University. During her study towards the Master's Degree, Miss Hirunsupachot was awarded a teaching assistant scholarship by the Faculty of Science during 2003-2005. She was also awarded a research grant from the Graduate School, Chulalongkorn University.

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