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APPENDICES

Appendix A Determination of Functional Groups in PPV Precursor, PPV, and Doped PPV by Fourier Transform Infrared Spectroscopy

The PPV Precursor, PPV, and doped PPV were characterized by FT-IR spectroscopy in order to identify functional groups. Optical grade KBr (Carlo Erba Reagent) was used as the background material. Ten-mg sample was mixed with 50-mg KBr. An FT-IR spectrum was observed by using an FT-IR spectrometer (Bruker, model EQUOX55/S) in the absorption mode with 32 scans at a resolution of 4 cm^{-1} .

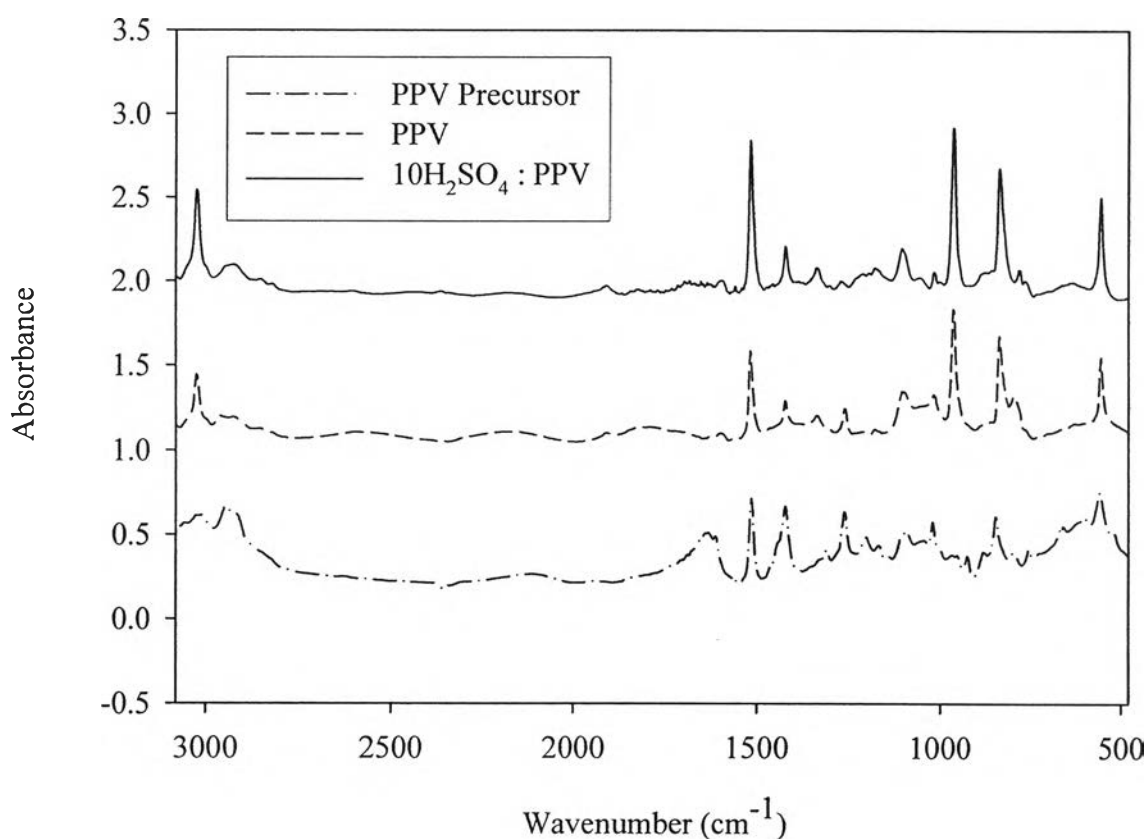


Figure A1 The FT-IR spectra of PPV Precursor, PPV, and doped PPV with a mole ratio of sulfuric acid to monomer unit equal to 10:1.

The assignments of peaks in the spectrum are shown in Table A1. PPV Precursor, PPV and sulfuric acid doped PPV were examined by FTIR spectroscopy. The presence of the absorption band near 960 cm^{-1} , resulting from C-H out-of-plane bending, is a characteristic of the trans configuration of the vinylene group (Çirpan, 2002). The absorption band around 3022 cm^{-1} is due to the trans vinylene C-H stretching mode. The absorption band around 550 cm^{-1} is attributed to the phenylene out-of-plane ring-bending. The bands at 830 cm^{-1} and 1511 cm^{-1} are assigned to para-phenylene ring C-H out-of-plane bending and C-C ring stretching, respectively. The bands at 2872 and 2960 cm^{-1} represent the CH_3 symmetric and CH_3 asymmetric deformation (Çirpan, 2002). After the heat treatment under vacuum, the intensity of these two bands decrease. The intensity of the absorption band near 3022 cm^{-1} increase due to the elimination of the tetrahydrothiopenyl group and HCl. The absence of the C-S linkage peak at 632 cm^{-1} from tetrahydrothiophene indicate full conversion of the precursor after pyrolysis (Çirpan, 2002). Upon oxidation of PPV, the infrared spectrum shows band at 1177 cm^{-1} . The emergence of this band in the spectra is related with the formation of quinoid structure. The quinoid structure is a result of a break of symmetry of the polymeric chain. The presence of vibrational bands from hydrogensulfate anion the counter ion of the oxidized polymer backbone, at 1200 , 1050 , and 650 cm^{-1} are due to S=O asymmetric stretching, S=O symmetric stretching, and S-O stretching, respectively (Fernandes, 2004).

Table A1 Peak positions from FT-IR spectra of PPV Precursor, PPV, and doped PPV with a mole ratio of sulfuric acid to monomer unit equal to 10:1

Functional groups	Wavenumber (cm ⁻¹)			References
	PPV Precursor	PPV	doped PPV	
Phenylene out of plane ring bending	550±10 (560)	550±10 (557)	550±10 (557)	Çirpan <i>et al.</i> ,(2002)
C-S stretching	632±10 (637)	–	–	Fernandes <i>et al.</i> ,(2004)
S-O stretching	–	–	650±10 (640)	Fernandes <i>et al.</i> ,(2004)
Para-phenylene ring C-H out of plane bending	830±10 (840)	830±10 (837)	830±10 (837)	Çirpan <i>et al.</i> ,(2002)
C-H out of plane bending	960±10 (950)	960±10 (963)	960±10 (963)	Çirpan <i>et al.</i> ,(2002)
S=O symmetric stretching	–	–	1050±10 (1056)	Fernandes <i>et al.</i> ,(2004)
Quinoid ring C=C stretching	–	–	1170±10 (1177)	Fernandes <i>et al.</i> ,(2004)
S=O asymmetric stretching	–	–	1200±10 (1204)	Fernandes <i>et al.</i> ,(2004)
C-C ring stretching	1517±10 (1513)	1517±10 (1517)	1517±10 (1517)	Çirpan <i>et al.</i> ,(2002)
CH ₃ symmetric stretching	2872±10 (2880)	2872±10 (2882)	2872±10 (2880)	Çirpan <i>et al.</i> ,(2002)
CH ₃ asymmetric stretching	2960±10 (2950)	2960±10 (2961)	2960±10 (2962)	Çirpan <i>et al.</i> ,(2002)
Trans vinylene C-H stretching	–	3022±10 (3023)	3022±10 (3023)	Çirpan <i>et al.</i> ,(2002)

Appendix B The thermogravimetric thermogram of Poly(p-phenylene vinylene), PPV and Salicylic acid doped poly(p-phenylene vinylene), SA doped PPV

A thermal gravimetric analyzer (DuPont, model TGA 2950) was used to determine amount of moisture content and dopant, and decomposition temperature of PPV and SA doped PPV with the temperature scan from 30 to 800°C with a heating rate of 10°C/min. The samples were weighted in the range of 5-10 mg and loaded into a platinum pan, and then heated it under an oxygen gas flow. The TGA thermogram of the synthesized PPV shows only one degradation step at 450-550 °C, corresponding to the degradation reaction of the PPV main chain only (Cirpan *et al.*, 2003). The thermal behavior of salicylic acid doped PPV shows two steps weight loss. The first step, 130-180 °C, is due to the loss of counterion belonging to the dopant. The last step, 480-580 °C, is related to the degradation of polymer. The TGA results of PPV and doped PPV show that the doped PPV has higher thermal stability; after doping the degradation temperature of the salicylic acid doped PPV is higher than that of the pristine PPV.

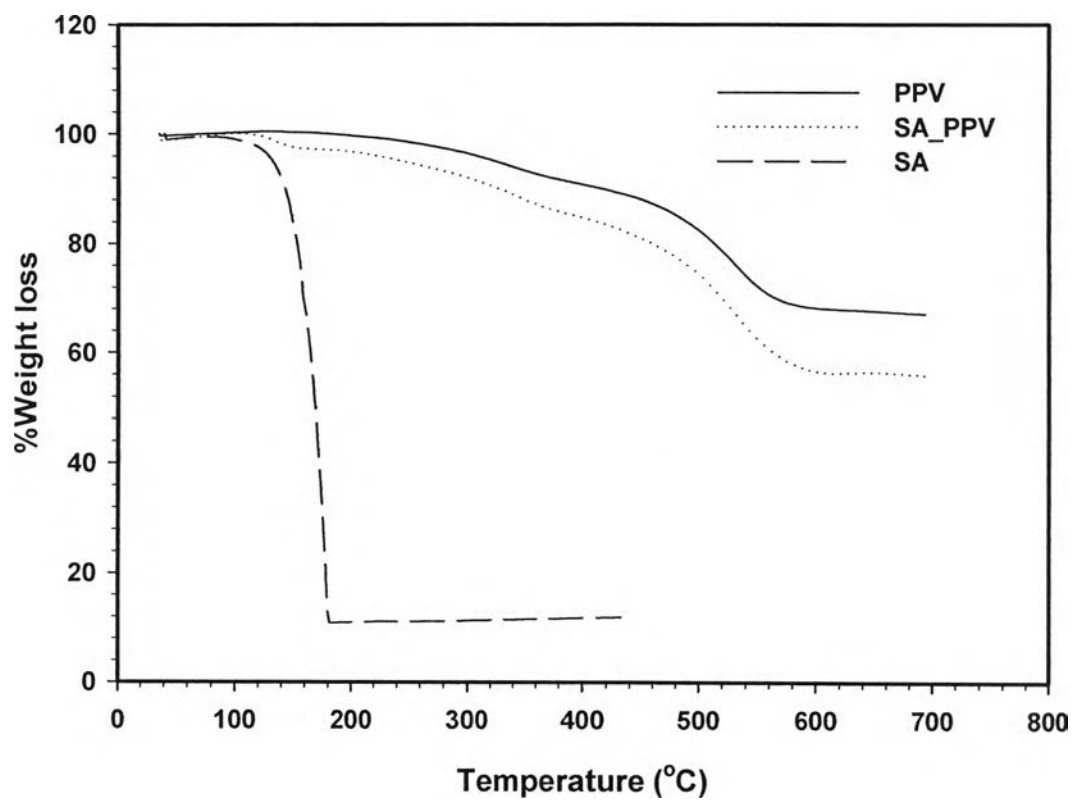


Figure B1 The TGA thermogram of PPV, SA doped PPV and SA.

Appendix C Determination of particle sizes of undoped and salicylic acid doped PPV**Table C1** Summarized the particles diameter of undoped and doped PPV

sample	Particles diameter (μm)
Undoped PPV	44.0 ± 3.84
SA doped PPV	46.0 ± 4.34

Table C2 The raw data from particle size analysis of undoped PPV

Size		Poly(p-phenylene vinylene)					
Low (μm)	High (μm)	In%	Under%	In%	Under%	In%	Under%
0.5	1.32	0.05	0.05	0.09	0.09	0.08	0.08
1.32	1.6	0.25	0.31	0.29	0.37	0.26	0.34
1.6	1.95	0.4	0.71	0.42	0.8	0.38	0.72
1.95	2.38	0.5	1.2	0.49	1.29	0.42	1.14
2.38	2.9	0.55	1.76	0.51	1.8	0.42	1.56
2.9	3.53	0.63	2.39	0.56	2.36	0.44	2
3.53	4.3	0.77	3.16	0.7	3.05	0.55	2.55
4.3	5.24	1.03	4.19	0.99	4.04	0.83	3.37
5.24	6.39	1.45	5.64	1.48	5.52	1.32	4.69
6.39	7.78	2.02	7.66	2.13	7.66	2.02	6.71
7.78	9.48	2.68	10.34	2.9	10.56	2.9	9.61
9.48	11.55	3.49	13.83	3.74	14.29	3.87	13.48
11.55	14.08	4.31	18.14	4.54	18.83	4.83	18.31
14.08	17.15	4.99	23.13	5.21	24.04	5.63	23.93
17.15	20.9	5.59	28.73	5.82	29.86	6.32	30.25
20.9	25.46	6.36	35.09	6.54	36.4	7.05	37.3
25.46	31.01	7.49	42.59	7.56	43.96	8.01	45.31
31.01	37.79	8.87	51.45	8.88	52.84	9.16	54.47
37.79	46.03	10.14	61.59	10.13	62.98	10.17	64.64
46.03	56.09	10.61	72.2	10.61	73.58	10.44	75.08
56.09	68.33	9.63	81.83	9.72	83.3	9.47	84.55
68.33	83.26	7.3	89.12	7.5	90.79	7.26	91.81
83.26	101.44	4.56	93.69	4.75	95.55	4.51	96.32
101.44	123.59	2.25	95.95	2.32	97.87	2.1	98.94
123.59	150.57	0.76	96.7	0.7	98.57	0.51	98.95
150.57	183.44	0.16	96.87	0	98.58	0	98.95
183.44	223.51	0.24	97.11	0	98.88	0	98.96
223.51	272.31	0.57	97.68	0.3	99.43	0.01	99.22
272.31	331.77	0.76	98.43	0.55	99.91	0.26	99.59
331.77	404.21	0.7	99.14	0.48	100	0.37	99.89
404.21	492.47	0.51	99.65	0.09	100	0.3	99.99
492.47	600	0.35	100	0	100	0.11	100

Table C2 The raw data from particle size analysis of SA doped PPV

Size		Salicylic acid doped poly(p-phenylene vinylene)					
Low (μm)	High (μm)	In%	Under%	In%	Under%	In%	Under%
0.5	1.32	0.05	0.05	0.09	0.09	0.08	0.08
1.32	1.6	0.29	0.31	0.29	0.37	0.26	0.34
1.6	1.95	0.42	0.71	0.42	0.8	0.38	0.72
1.95	2.38	0.49	1.2	0.49	1.29	0.42	1.14
2.38	2.9	0.51	1.76	0.51	1.8	0.42	1.56
2.9	3.53	0.63	2.39	0.56	2.36	0.44	2
3.53	4.3	0.77	3.16	0.7	3.05	0.55	2.55
4.3	5.24	1.03	4.19	0.99	4.04	0.83	3.37
5.24	6.39	1.45	5.64	1.48	5.52	1.32	4.69
6.39	7.78	2.02	7.66	2.13	7.66	2.02	6.71
7.78	9.48	2.68	10.34	2.9	10.56	2.9	9.61
9.48	11.55	3.49	13.83	3.74	14.29	3.87	13.48
11.55	14.08	4.31	18.14	4.54	18.83	4.83	18.31
14.08	17.15	4.99	23.13	5.21	24.04	5.63	23.93
17.15	20.9	5.59	28.73	5.82	29.86	6.32	30.25
20.9	25.46	6.36	35.09	6.54	36.4	7.05	37.3
25.46	31.01	7.49	42.59	7.56	43.96	8.01	45.31
31.01	37.79	8.87	51.45	8.88	52.84	9.16	54.47
37.79	46.03	10.14	61.59	10.13	62.98	10.17	64.64
46.03	56.09	10.61	72.2	10.61	73.58	10.44	75.08
56.09	68.33	9.63	81.83	9.72	83.3	9.47	84.55
68.33	83.26	7.3	89.12	7.5	90.79	7.26	91.81
83.26	101.44	4.56	93.69	4.75	95.55	4.51	96.32
101.44	123.59	2.25	95.95	2.32	97.87	2.1	98.94
123.59	150.57	0.76	96.7	0.7	98.57	0.51	98.95
150.57	183.44	0.16	96.87	0	98.58	0	98.95
183.44	223.51	0.24	97.11	0	98.88	0	98.96
223.51	272.31	0.57	97.68	0.3	99.43	0.01	99.22
272.31	331.77	0.76	98.43	0.55	99.91	0.26	99.59
331.77	404.21	0.7	99.14	0.48	100	0.37	99.89
404.21	492.47	0.51	99.65	0.09	100	0.3	99.99
492.47	600	0.35	100	0	100	0.11	100

Appendix D Determination of the Correction Factor (K)

The electrical conductivity of sample was measured by a two-point probe meter. The meter consists of two probes, making contact on the surface of thin layer sample. These probes were connected to a source meter (Keithley, Model 6517A) for a constant voltage source and reading the resultant current.

The geometrical correction factor was taken into account of geometric effects, depending on the configuration and probe tip spacing

$$K = \frac{w}{l} \quad (\text{D.1})$$

where K is geometrical correction factor

w is width of probe tip spacing (cm)

l is the length between probe (cm)

In this measurement, the constant K value was determined by using a standard sheet with a known resistivity value; we used silicon wafer chips (SiO₂). K was calculated by using Equation M.2.

$$K = \frac{\rho}{R \times t} = \frac{I \times \rho}{V \times t} \quad (\text{D.2})$$

where K = geometric correction factor

ρ = resistivity of standard silicon wafer which were calibrated by using a four point probe at King Mongkut's Institute Technology Lad Krabang ($\Omega \cdot \text{cm}$)

t = film thickness (cm)

R = film resistance (Ω)

I = measure current (A)

V = voltage drop (V)

Standard Si wafer were cleaned to remove organic impurities prior to be used according to the standard RCA method (Kern, 1993).

Materials

Acetones (Scharlau, 99.5%), Methanol (CARLO ERBA, 99.9%), Ammonium hydroxide (Merk, 99.9%), Hydrogen peroxide (CARLO ERBA, 30% in water), and dilute (2%) Hydrofluoric acid

Experiment

The cleaning procedures contain 3 steps: the solvent clean, the RCA01 and the HF dip. The first step is the solvent clean step, employed to remove oils and organic residues that appeared on Si wafer surface. The Si wafer was placed into the acetone at 55°C for 10 min, removed and placed in methanol for 2-5 min, subsequently rinsed with deionized water and blown dried with nitrogen gas. Second step is the RCA clean, to remove organic residues from silicon wafers. This process oxidized the silicon wafer and left a thin oxide on the surface of the wafer. RCA solution was prepared with 5 parts of water (H₂O), 1 part of 27% ammonium hydroxide (NH₄OH), and 1 part of 30% hydrogen peroxide (H₂O₂). 65 ml of NH₄OH (27%) was added into 325 ml of deionized water in a beaker and then heated to 70 ± 5°C. The mixture would bubble vigorously after 1-2 min, indicated that it was ready to use. Silicon wafer was soaked in the solution for 15 min, consequently overflowed with deionized water in order to rinse and remove the solution. The third step is the HF dip, which was carried out to remove native silicon dioxide from wafer. 480 ml of deionised water was added to the polypropylene bottle and then added to 20 ml HF. Wafer was soaked in this solution for 2 min, removed and checked for hydrophobicity by performing the wetting test. Deionized water was poured onto the surface wafer; the clean silicon surface would show that the beads of water would roll off. Clean Si wafer was further blown dried with nitrogen and stored in a clean and dry environment.

Table D1 Determination the correction factor of probe A and B

Probe	K (correction factor)				
	1	2	3	Average	SD
A	1.780E-04	1.843E-04	1.936E-04	1.853E-04	7.830E-06

Table D2 Determination the correction factor of probe with standard Si wafer (specific resistivity 0.014265 Ω .cm, thickness 0.0724 cm, 27-28°C, 60%R.H)

Applied Voltage (V)	Measured Current (A)		
	Probe A		
	1	2	3
0.005	4.78E-06	4.22E-06	3.93E-06
0.01	9.49E-06	8.32E-06	7.65E-06
0.015	1.19E-05	1.04E-05	9.56E-06
0.02	1.43E-05	1.25E-05	1.15E-05
0.025	1.68E-05	1.45E-05	1.35E-05
0.03	2.16E-05	1.89E-05	1.75E-05
0.035	2.65E-05	2.33E-05	2.16E-05
0.04	2.89E-05	2.57E-05	2.45E-05
0.045	3.39E-05	3.05E-05	2.81E-05
0.05	3.65E-05	3.28E-05	3.01E-05
0.055	4.17E-05	3.77E-05	3.46E-05
0.06	4.71E-05	4.29E-05	3.92E-05
0.065	4.98E-05	4.55E-05	4.17E-05
0.07	5.53E-05	5.10E-05	4.64E-05
0.075	5.78E-05	5.40E-05	4.89E-05
0.08	6.36E-05	5.95E-05	5.43E-05
0.085	6.95E-05	6.55E-05	5.97E-05
0.09	7.24E-05	6.86E-05	6.24E-05
0.095	7.87E-05	7.49E-05	6.85E-05
0.1	8.17E-05	7.81E-05	7.12E-05

(Temperature 26 ± 1 °C, Humidity 55 ± 1 %)

Appendix E Conductivity Measurement

The specific conductivity, which is the inversion of specific resistivity (ρ) of undoped PPV and doped PPV pellets were measured by using the two-point probe connected to a source meter (Keithley, Model 6517A) for a constant voltage source and reading resultant current . The thickness of pellets was measured by a thickness gauge. The applied voltage was plotted versus the current change to determine the linear ohmic regime of each sample. The applied voltage and the current change in the linear ohmic regime were converted to the electrical conductivity of the polymer by using equation (E.1) as follows:

$$\sigma = \frac{1}{\rho} = \frac{1}{R_s \times t} = \frac{I}{K \times V \times t} \quad (\text{E.1})$$

where

- σ = specific conductivity (S/cm)
- ρ = specific resistivity ($\Omega \cdot \text{cm}$),
- R_s = sheet resistivity (Ω)
- I = measured current (A)
- K = geometric correction factor
- V = applied voltage (voltage drop) (V)
- t = pellet thickness (cm).

Table E1 Determination the specific conductivity (S/cm) of undoped and doped PPV.

Sample	Specific conductivity (S.cm)	STD
PPV	2.21E-07	1.88E-09
SA doped PPV	2.93E-03	1.53E-04
Polydimethylsiloxane ($N_c/N_m = 0.01$)	3.49E-08	4.58E-10

Appendix F Scanning electron micrograph of undoped PPV, SA doped PPV, poly(p-phenylene vinylene)/polydimethylsiloxane blend and polyacrylamide hydrogel

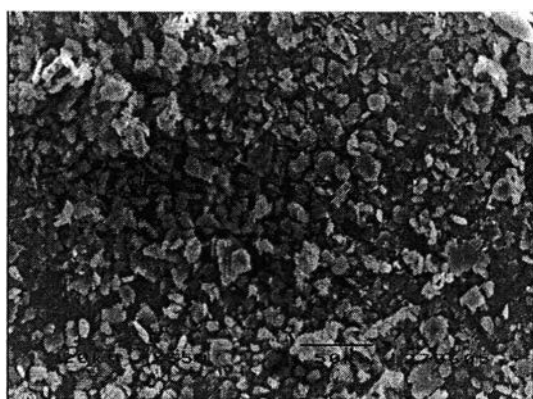
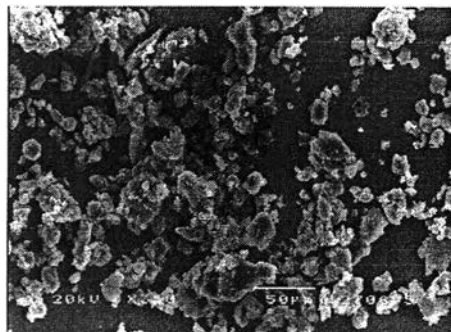
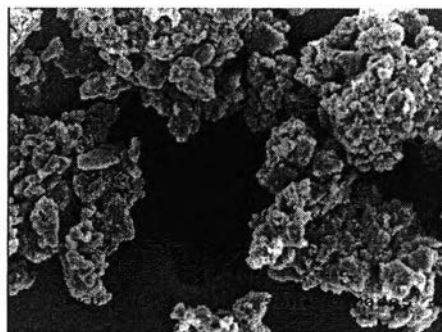


Figure F1 The morphology of undoped poly(p-phenylene vinylene) (PPV) powder at magnification 350.



(a)



(b)

Figure F2 The morphology of doped PPV powder with H₂SO₄ as various doping ratio at magnification 1500; a) 10:1 and b) 100:.

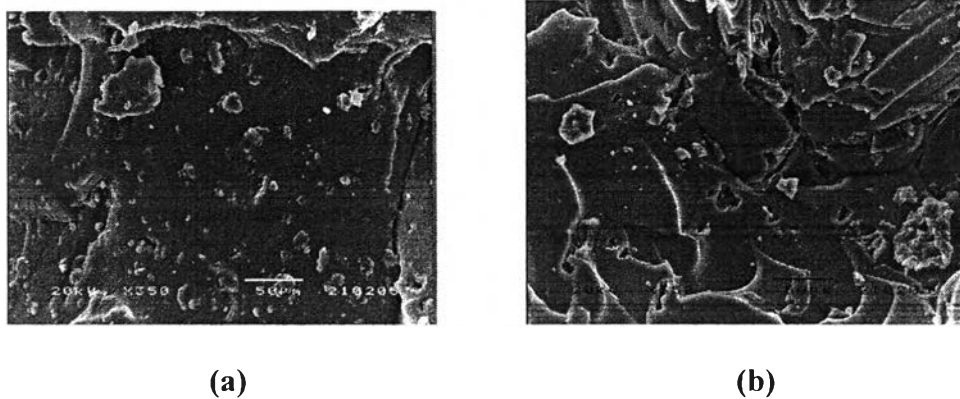


Figure F3 The morphology of poly(p-phenylene vinylene)/polydimethylsiloxane blend; a) PPV_0/PDMS90; and b) PPV_100/PDMS90 at magnification of 350.

Appendix G Electrorheological properties measurement of pure polydimethylsiloxane at various degree of crosslinking

The electrorheological properties of pure polydimethylsiloxane at various degree of crosslinking were measured by the melt rheometer (Rheometric Scientific, ARES) under oscillatory shear mode and applied electric field strength varying from 0 to 2 kV/mm. In these experiments, the dynamic moduli (G' and G'') were measured as functions of frequency and electric field strength. Strain sweep tests were first carried out to determine the suitable strain to measure G' and G'' in the linear viscoelastic regime as showed in Table II.

Table G1 Summarized the linear viscoelastic regime of polydimethylsiloxane at various degree of crosslinking

Systems	Crosslinking ratio (N_{ini}/N_m)	Linear Viscoelastic Range (% Strain)
PDMS 01	0	700
PDMS 02	0.01	1
PDMS 03	0.03	1
PDMS 04	0.05	1
PDMS 05	0.1	1

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