CHAPTER IV
RESULTS AND DISCUSSION

The results of this study were composed of preparation and characterization of polyHIPEs.

4.1 Preparation of PolyHIPEs

When the aqueous phase (colourless and clear solution) was added to oil phase (yellow and clear solution), the mixture became white emulsion and the viscosity increased. After polymerization, the emulsion turned to be high porous solid called polyHIPE which was white porous solid, brittle and chalky.

4.1.1 Effect of Monomer Ratio on PolyHIPEs Properties

The SEMs of polyHIPEs with different DVB/VBC ratios are shown in Figure 4.1. When the amount of DVB in the monomer ratio increased, the void diameter decreased due to DVB cross-linking agent. When the amount of DVB increased, the polymer structure became denser with smaller void diameter. When the amount of DVB increases, the surface area is decreased as shown in Table 4.1.

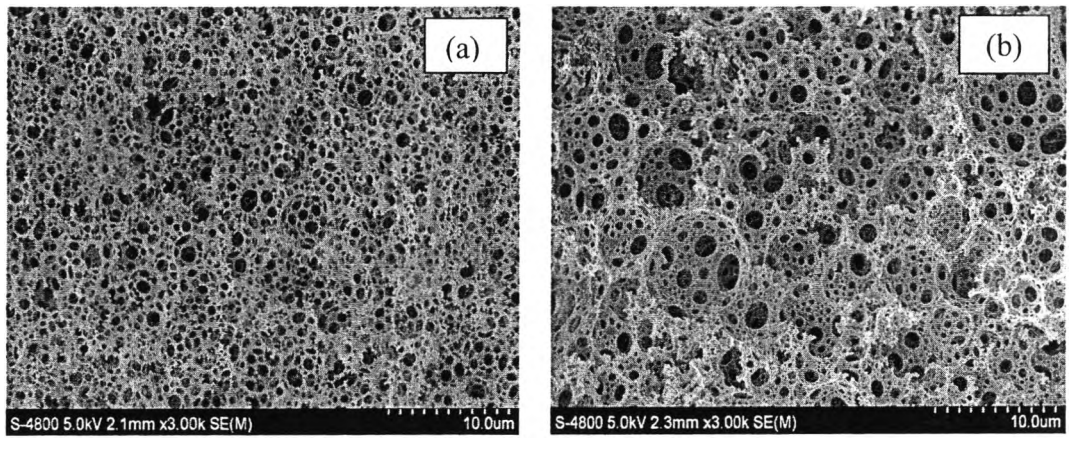

Figure 4.1 SEMs of polyHIPE without PEI loading and different DVB/CBV ratio of (a) 100/0, (b) 90/10, (c) 80/20, (d) 70/30, (e) 60/40 and (f) 50/50.

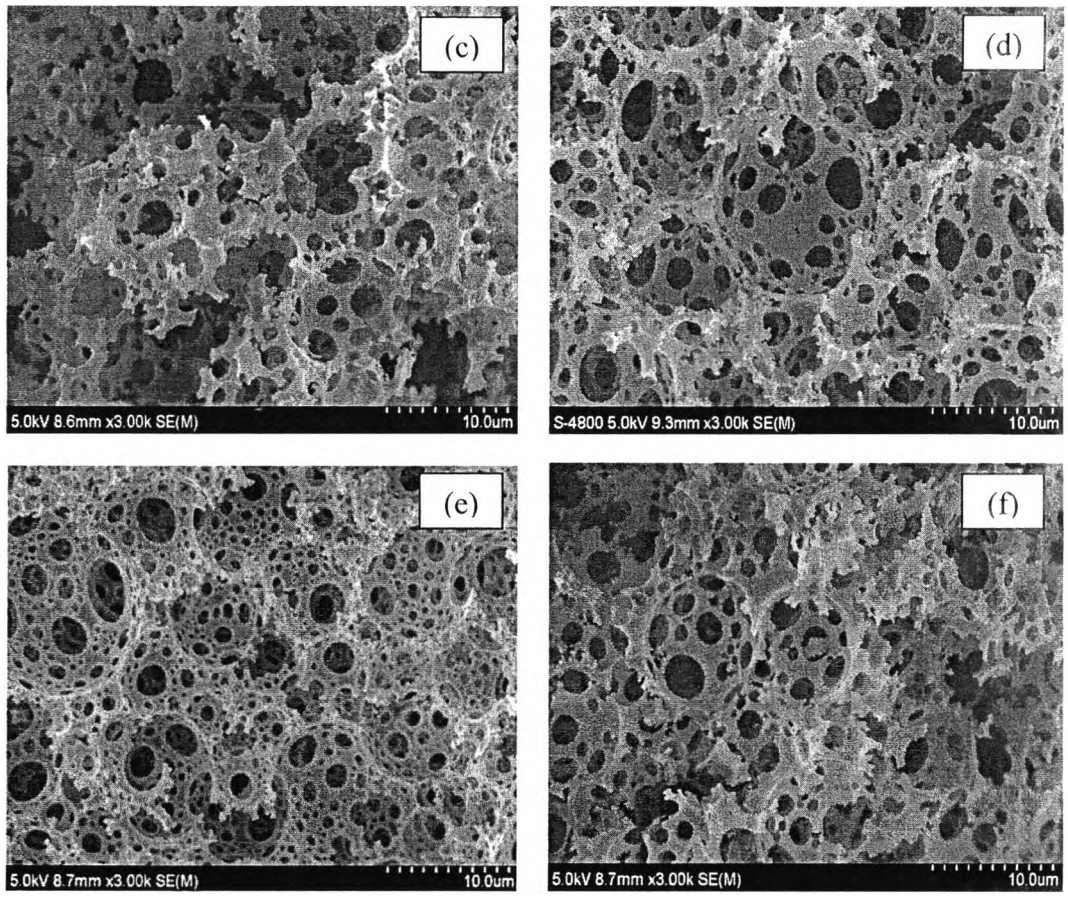

Figure 4.1 (cont.) SEMs of polyHIPE without PEI loading and different DVB/CBV ratio of (a) 100/0, (b) 90/10, (c) 80/20, (d) 70/30, (e) 60/40 and (f) 50/50.

Table 4.1 Surface area of polyHIPEs at different monomer ratio

PolyHIPEs (DVB/VBC)	Surface area (m^2/g)
100/0	303.0
90/10	210.0
80/20	180.0
70/30	142.5
60/40	93.2
50/50	57.3

When 10 wt% of PEI was loaded into the emulsions with different monomer ratios, the SEMs illustrated in Figures 4.2 and 4.3, with magnification of 3k and 10k respectively, show that PEI is hardly seen in the monomer ratio of 100/0 whereas in the other ratios, PEI is observed clearly. FTIR results also confirmed the results of SEM. All FTIR spectra were obtained from solid samples in KBr pellets. The characteristic peak of PEI in solid sample is N-H bending peak at 1650-1550 cm^{-1} which is sharp and easy to identification. In Figure 4.4c) polyHIPE with 100% DVB and 10% PEI, there was almost no N-H bending peak because PEI cannot form a chemical bond with DVB. When the polyHIPE was washed with ethanol, PEI was also washed out. However, there was very small amount of PEI remaining in the polyHIPE probably due to the inadequate soxhlet extraction time to wash out all PEI.

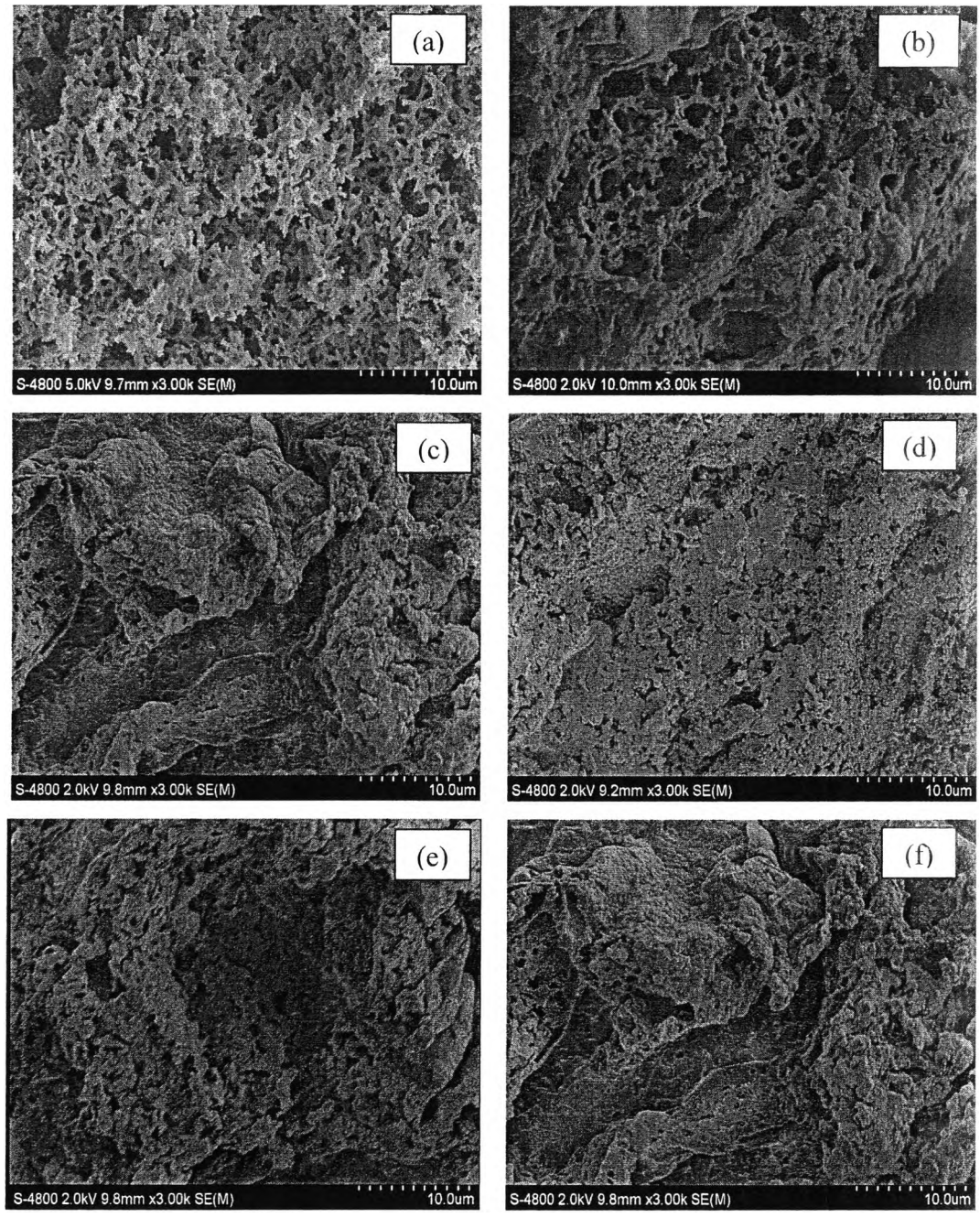

Figure 4.2 SEMs with 3k magnification of polyHIPE with 10 wt% PEI and DVB/VBC ratio (a) 100/0, (b) 90/10, (c) 80/20, (d) 70/30, (e) 60/40 and (f) 50/50.

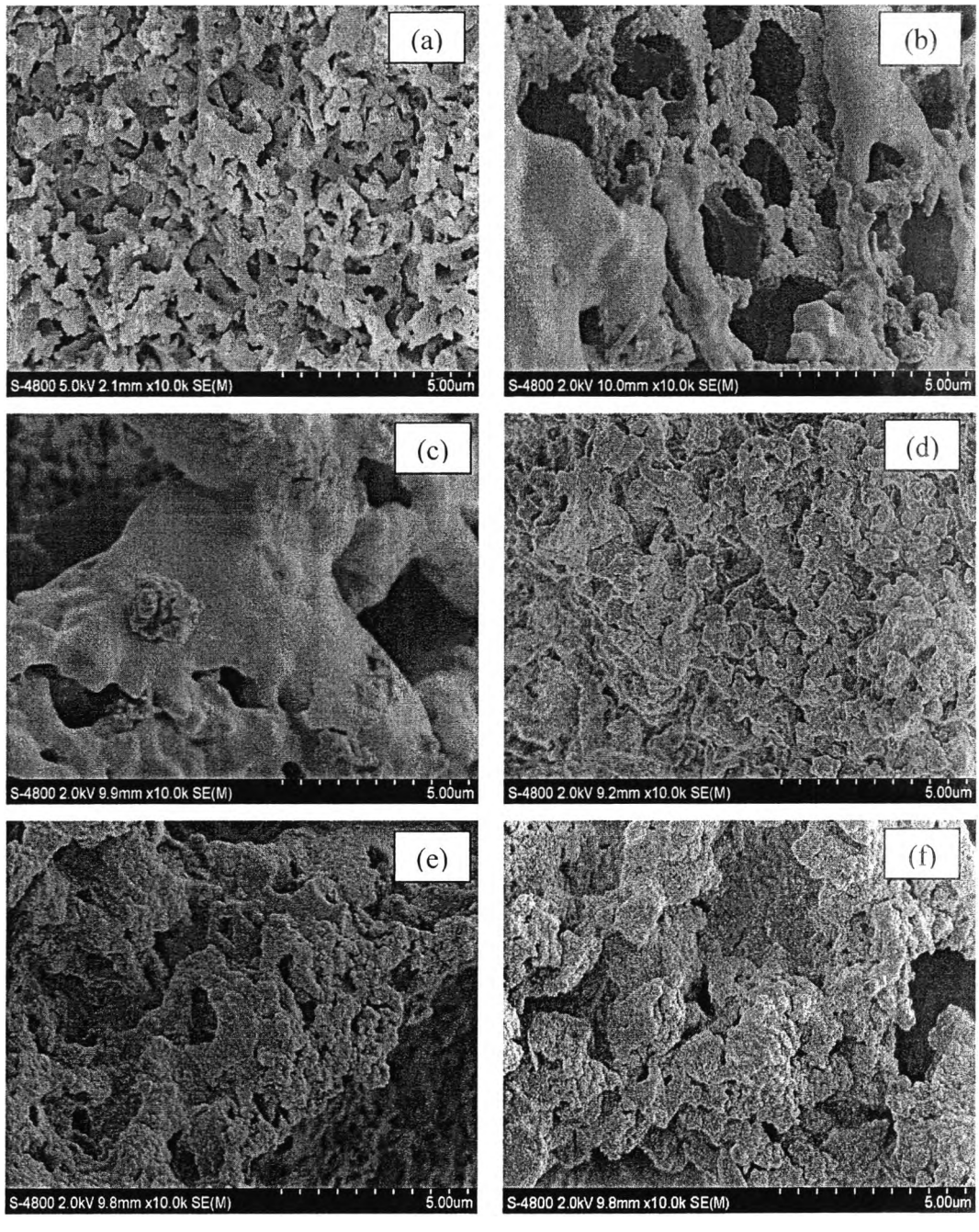

Figure 4.3 SEMs with 10k magnification of polyHIPE with 10 wt% PEI and DVB/VBC ratio (a)100/0, (b) 90/10, (c) 80/20, (d) 70/30, (e) 60/40 and (f) 50/50.

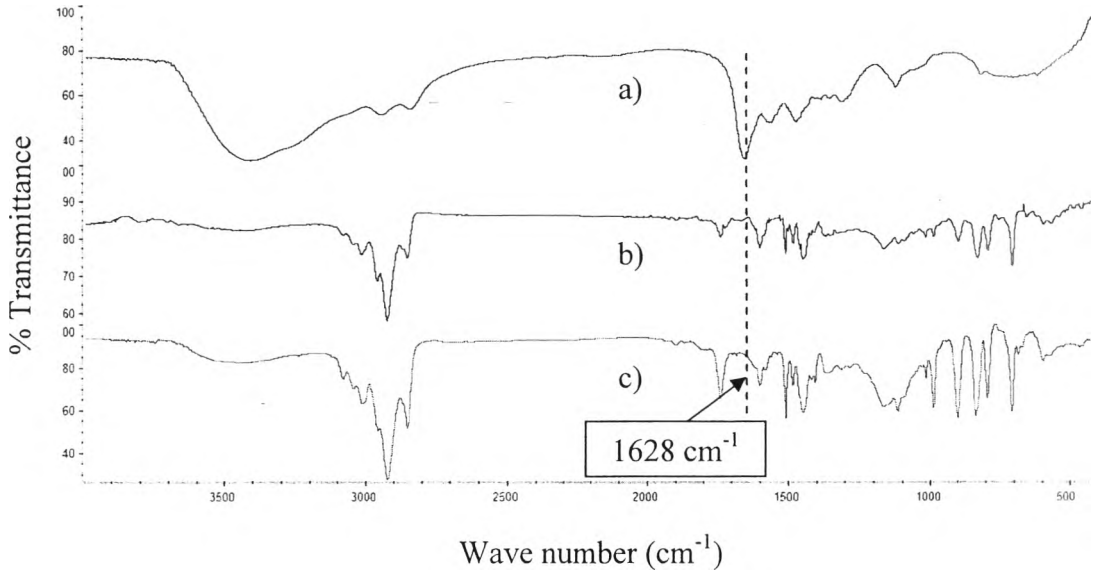

Figure 4.4 FTIR spectra of solid samples prepared by KBr pellets a) PEI, b) polyHIPE with 100/0 DVB/VBC and 0 wt% PEI, c) polyHIPE with 100/0 DVB/VBC and 10 wt% PEI.

FTIR spectra of PEI and PEI in polyHIPEs at 50/50 and 90/10 monomer ratios as compared to no PEI loading are shown in Figure 4.5. In Figure 4.5 b) and d), there were no N-H bending peak. However, there was C-Cl peak of VBC appearing at 1268 cm^{-1}. In Figure 4.5 c) and e), are polyHIPE with monomer ratio 50/50 and 90/10 with 10% PEI, there were N-H bending peaks of PEI. This confirmed that PEI could be loaded into polyHIPE.

The amount of PEI loading was measured by UV-VIS spectroscopy technique at the absorption peak 255 nm which detected the amount of salicylaldehyde remaining in the solution. The absorbance of this peak, as shown in Figure 4.6, will be converted to percent of amine in polyHIPE by using a calibration curve of UV-VIS absorbance against salicylaldehyde concentration which is shown in Figure 4.7. The UV-VIS results are shown in Table 4.2, at monomer ratio 100/0, very small amount of PEI can be loaded into the polyHIPE. However, approximate the same amount of PEI was loaded into the polyHIPE with other monomer ratios.

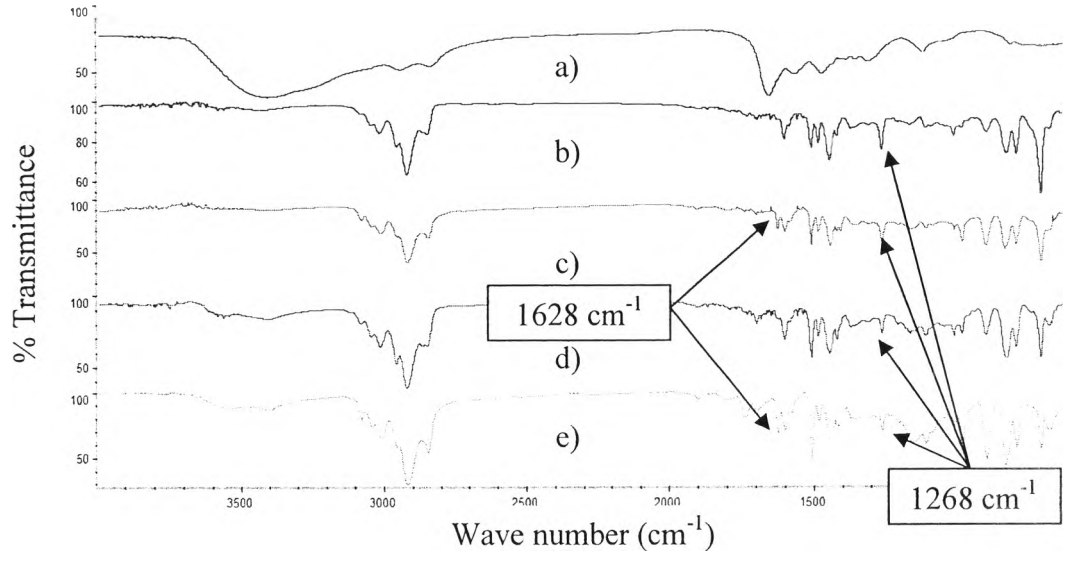

Figure 4.5 FTIR spectra of solid samples in KBr pellets a) PEI, b) polyHIPE with 50% DVB/50% VBC and 0 wt% PEI, c) polyHIPE with 50% DVB/50% VBC and 10wt% PEI, d) polyHIPE with 90% DVB/10% VBC and 0 wt% PEI and e) polyHIPE with 90% DVB/10% VBC and 10 wt% PEI.

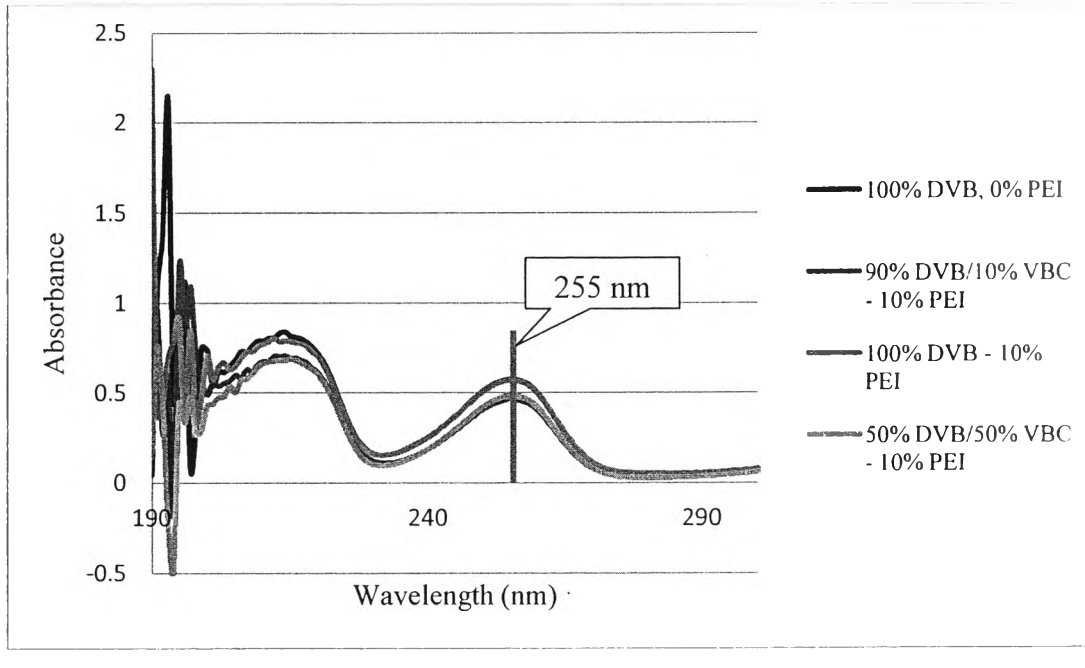

Figure 4.6 UV-VIS spectra.

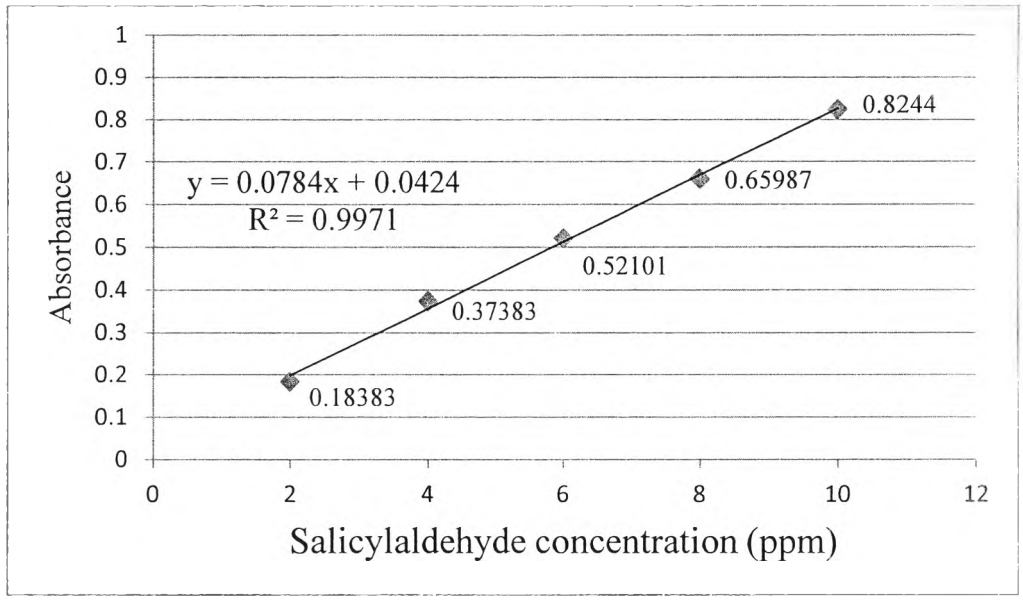

Figure 4.7 Calibration curve of salicylaldehyde concentration measured by UV-VIS spectrophotometer at wavelength 255 nm.

Table 4.2 UV- VIS results of PEI in polyHIPEs

Solution	Absorbance at 255 nm	Percent of amine in polyHIPE (%)	Theoretical Value (%)	Loading Efficiency (%)
100/0, 10% PEI	0.570	0.11	10	1.1
90/10, 10% PEI	0.468	1.71	10	17.1
80/20, 10% PEI	0.470	1.68	10	16.8
70/30, 10% PEI	0.475	1.60	10	16.0
60/40, 10% PEI	0.468	1.71	10	17.1
50/50, 10% PEI	0.474	1.61	10	16.1

From the calculation, 10 wt% of PEI reacts completely with 7.5 %v/v of VBC, therefore, the amount of PEI loading depends on the amount of VBC. However, when the amount of VBC increases, the viscosity of the emulsion increases. Moreover, when PEI is added into the emulsion, the emulsion viscosity is increased. Thus, the monomer ratio of 60/40 was selected for the study of different amount of PEI loading due to the optimum viscosity.

4.1.2 Effect of Different Percent PEI Loading

The percent of PEI loading into a polyHIPE with monomer ratio 60/40 was varied from 0-30 wt%, however, when the percent of PEI was more than 20 wt%, the solution could not be stirred due to too high viscosity of the emulsion. The amount of PEI loading as shown in Table 4.3 indicated that when the concentration of PEI solution increases, the amount of PEI loading increases up to 2.57 wt%. SEMs, as shown in Figure 4.8, also indicated that when the concentration of PEI solution increased, the porous structures of the polyHIPE were covered by PEI leading to decrease of surface area. BET results are shown in Table 4.4.

Table 4.3 UV-VIS results

Solution	Absorbance at 255 nm	Percent of Amine in PolyHIPE (%)	Theoretical Value (%)	Loading Efficiency (%)
60/40, 5% PEI	0.488	1.39	5	27.8
60/40, 10% PEI	0.462	1.80	10	18.0
60/40, 15% PEI	0.446	2.05	15	13.7
60/40, 20% PEI	0.413	2.57	20	12.8

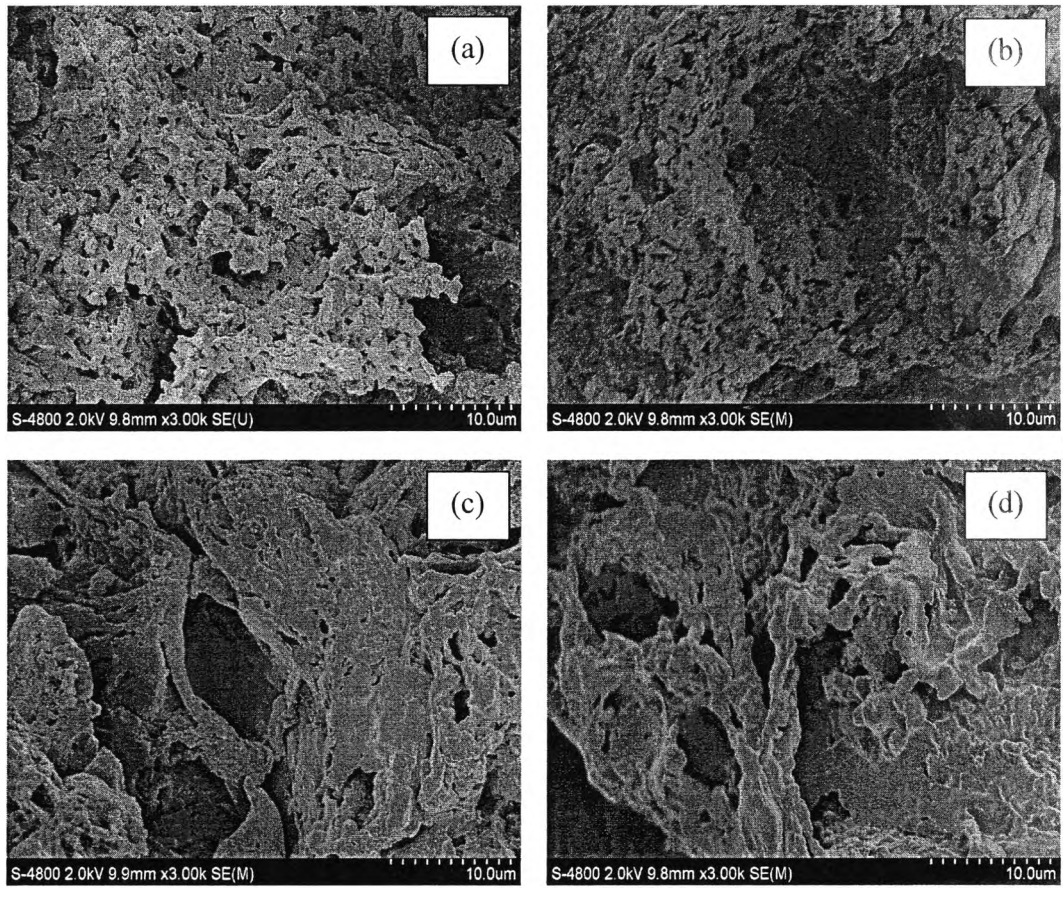

Fig. 4.8 SEMs with 3k magnification of polyHIPE with DVB/VBC ratio 60/40 and with percent of PEI loading (a) 5 wt%, (b) 10 wt%, (c) 15wt% and (d) 20 wt%.

Table 4.4 BET results

PolyHIPEs (DVB/VBC)	**Surface area (m^2/g)**
60/40, with 5% PEI	30.4
60/40, with 10% PEI	21.2
60/40, with 15% PEI	11.1
60/40, with 20% PEI	6.3