

## CHAPTER IV

### RESULTS AND DISCUSSION

#### 4.1 Water Contents of the Solvents

The water contents of the solvents affect the extraction of increasing the extraction selectivity. Table 4.1 shows the water contents of the solvents before and after the preparation of the solvents by sodium sulfate anhydrous to keep the amount of water of the solvents at the same level. Ethylene Carbonate (EC) could not be determined the amount of water because of it crystallized at the test temperature.

**Table 4.1** Water contents of the solvents before and after the solvent preparation

Solvent	Water content (wt. %)	
	Before the preparation	After the preparation
Sulfolane	0.21	0.12
DMSO	0.45	0.12
EG	0.12	0.12
EC	N/A	N/A
3MOPN	0.17	0.12

#### 4.2 Critical Solution Temperature (CST)

##### 4.2.1 Single Solvent

###### 4.2.1.1 *Benzene/Hexane/Solvent Systems*

The critical solution temperature relates to the maximum operating temperature for extraction that should be 25°C lower than the critical solution temperature. For the benzene-hexane systems, there were some solvents that did not form clear solution up to 95°C which was the highest temperature under test, as shown in Table 4.2. This implies that extraction can be carried out at least 25°C lower than 95°C, that is at 70°C or lower. However, the boiling point of

n-hexane is 68.7 °C so the extraction temperature limit is at the boiling point of the n-hexane.

**Table 4.2** Critical solution temperatures of benzene/hexane/solvent systems

Solvent	Solvent-to-Feed ratio	CST (°C)
Sulfolane	3 to 1	not clear at 95
DMSO	3 to 1	75
EG	3 to 1	not clear at 95
EC	3 to 1	not clear at 95
3MOPN	3 to 1	42

#### 4.2.1.2 Toluene/Hexane/Solvent Systems

As mentioned earlier, for the systems of toluene/hexane/solvent, there are some systems that the system did not become homogeneous up to 95°C as shown in Table 4.3.

**Table 4.3** Critical solution temperatures of benzene/hexane/solvent systems

Solvent	Solvent-to-feed ratio	CST (°C)
Sulfolane	3 to 1	84
DMSO	3 to 1	83
EG	3 to 1	not clear at 95
EC	3 to 1	not clear at 95
3MOPN	3 to 1	26

#### 4.2.1.3 p-Xylene/Hexane/Solvent Systems

Table 4.4 shows the critical solution temperatures of p-xylene/hexane/solvent systems. There were some systems that did not become homogenous up to 95°C and there was a system that did not form two-phase solution even when the temperature was as low as 25°C.

**Table 4.4** Critical solution temperatures of *p*-xylene/hexane/solvent systems

Solvent	Solvent-to-feed ratio	CST (°C)
Sulfolane	3 to 1	83
DMSO	3 to 1	80
EG	3 to 1	not clear at 95
EC	3 to 1	not clear at 95
3MOPN	3 to 1	not clear at 25

#### 4.2.2 Mixed Solvents

##### 4.2.2.1 *Benzene/Hexane/Mixed Solvent Systems*

For the mixed solvent, the benzene/hexane/solvent systems performed points of turbidity as shown in Table 4.5.

**Table 4.5** Critical solution temperatures of benzene/hexane/mixed solvent systems

Solvent	Mixed ratio	Solvent-to-feed ratio	CST (°C)
EC /DMSO	50/ 50	3 to 1	not clear at 95
	10/ 90	3 to 1	not clear at 95
EG /3MOPN	70/ 30	3 to 1	not clear at 95
	50/ 50	3 to 1	not clear at 95
	30/ 70	3 to 1	82

##### 4.2.2.2 *Toluene/Hexane/Mixed Solvent Systems*

For the mixed solvent, the toluene/hexane/solvent systems performed turbidity as shown in Table 4.6.

**Table 4.6** Critical solution temperatures of toluene/hexane/mixed solvent systems

Solvent	Mixed ratio	Solvent-to-feed ratio	CST (°C)
EC /DMSO	50/ 50	3 to 1	not clear at 95
	10/ 90	3 to 1	not clear at 95
EG /3MOPN	70/ 30	3 to 1	not clear at 95
	50/ 50	3 to 1	not clear at 95
	30/ 70	3 to 1	76

#### 4.2.2.3 *p*-Xylene/Hexane/Solvent Systems

For the mixed solvent, the *p*-xylene/hexane/solvent systems perform turbidity as shown in Table 4.7.

**Table 4.7** Critical solution temperatures of *p*-xylene/hexane/mixed solvent systems

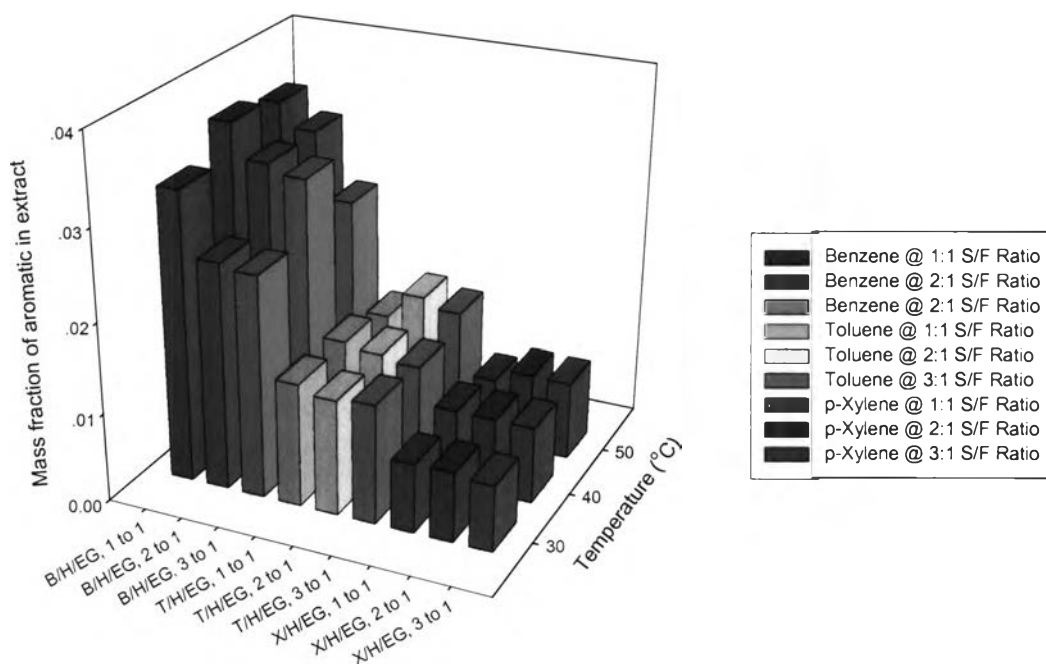
Solvent	Mixed ratio	Solvent-to-feed ratio	CST (°C)
EC /DMSO	50/ 50	3 to 1	not clear at 95
	10/ 90	3 to 1	not clear at 95
EG /3MOPN	70/ 30	3 to 1	not clear at 95
	50/ 50	3 to 1	not clear at 95
	30/ 70	3 to 1	81

### 4.3 Aromatics Extraction

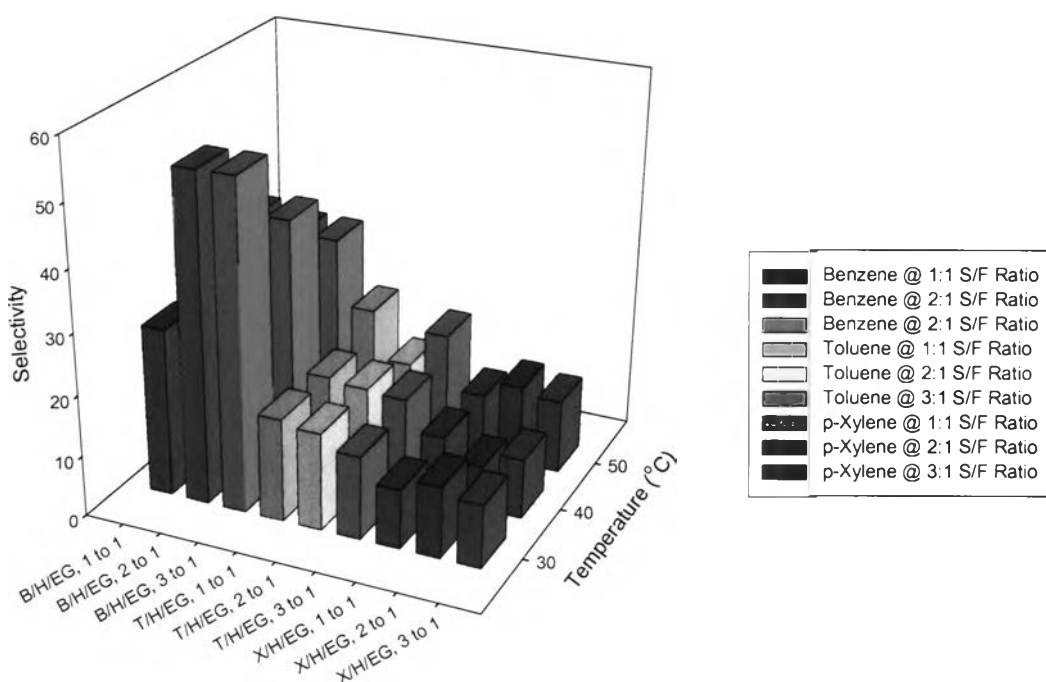
#### 4.3.1 Single Solvent

##### 4.3.1.1 *Ethylene Glycol (EG)*

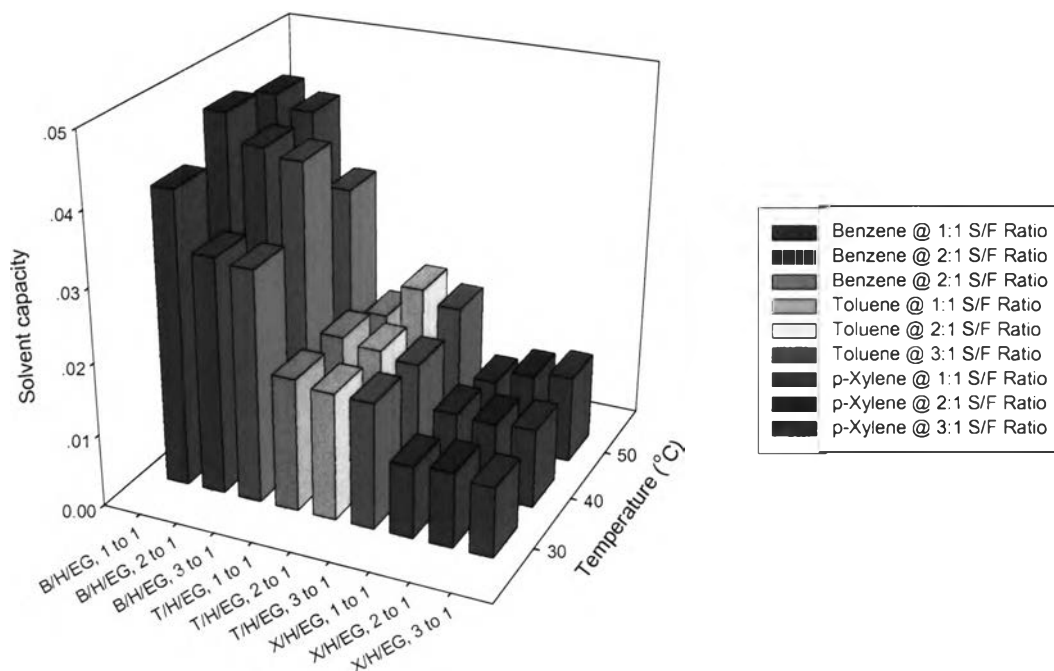
With ethylene glycol (EG) as a solvent for extraction, it was found that benzene could be much better extracted than toluene and *p*-xylene. As shown in Figure 4.1, the range of mass fraction of aromatic in the extract phase found within 0.024 to 0.036, 0.012 to 0.015, and 0.007 to 0.008 for benzene, toluene, and *p*-xylene, respectively. Moreover, the percentage of aromatic in the extract was found to decrease as the solvent-to-feed ratio increased. This is in line with the fact that as the amount of the solvent increases, the extract also increases, as shown in Figure 4.1. This kind of phenomena was also found in the extraction by tetraethylene glycol (Al-Sahhaf and Kapetanovic, 1996). Figure 4.2 shows that EG was observed to have higher selectivity for benzene than toluene and *p*-xylene. In Figure 4.3, solvent capacity of the extraction of EG had greater capacity for benzene compared to toluene and *p*-xylene, respectively. In addition, it was found that in the temperature range studied, the temperature did not play significant effects to the extractions. Sample of calculation of the terms above is shown in Appendix B.



**Figure 4.1** Mass fraction of the aromatic in extract phase of aromatic/hexane/EG systems at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



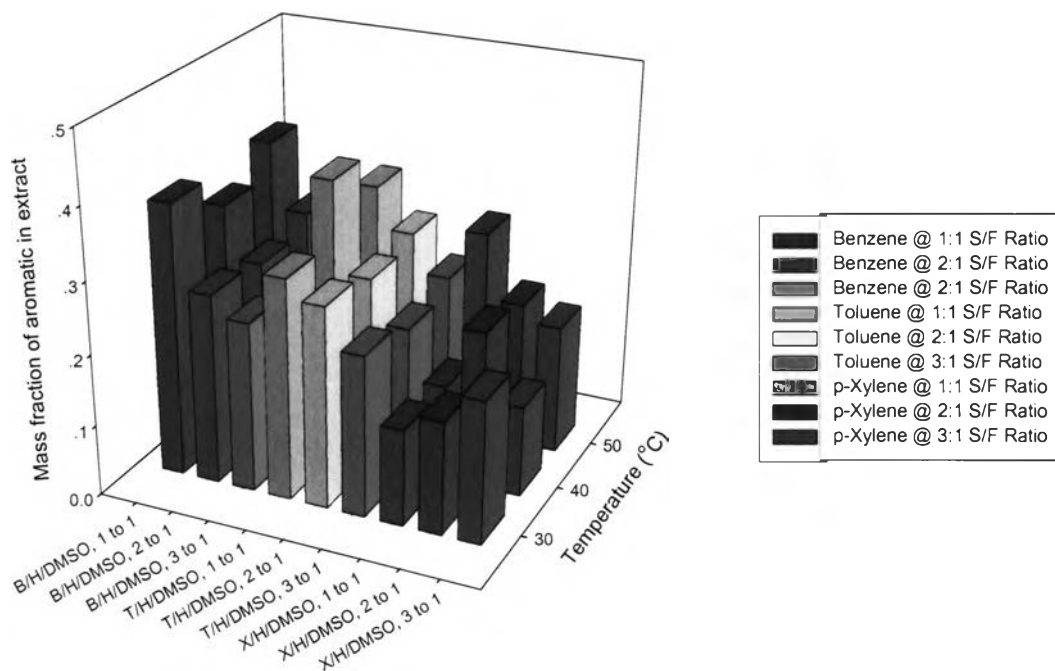
**Figure 4.2** Selectivity of the extraction of aromatic/hexane/EG systems at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



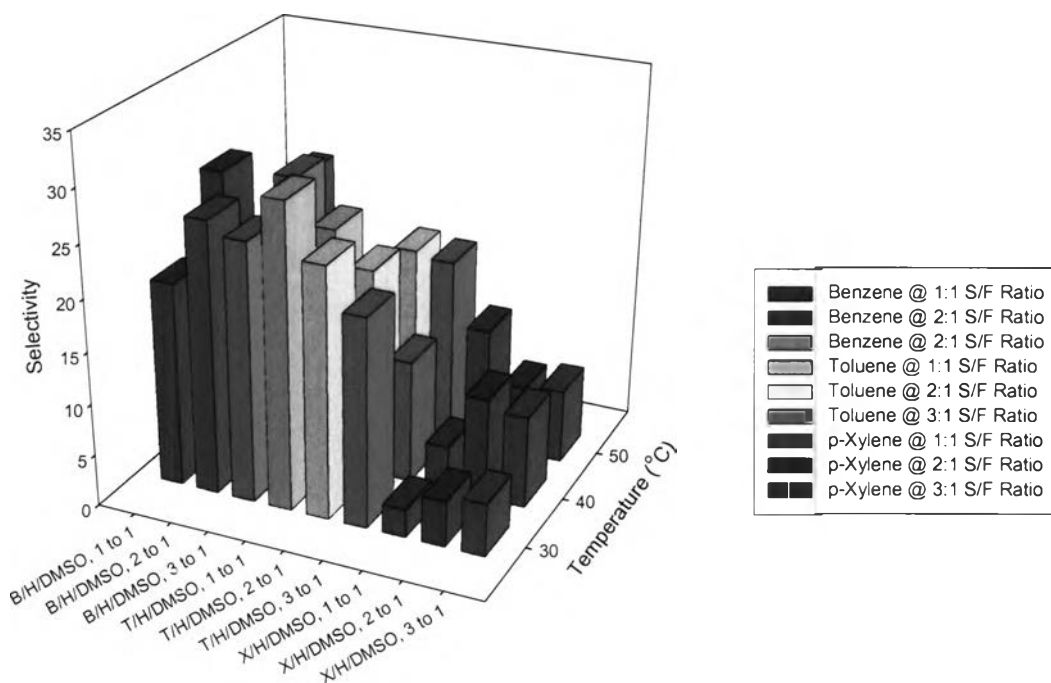
**Figure 4.3** Solvent capacity of the extraction of aromatic/hexane/EG systems at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.

#### 4.3.1.2 Dimethylsulfoxide (DMSO)

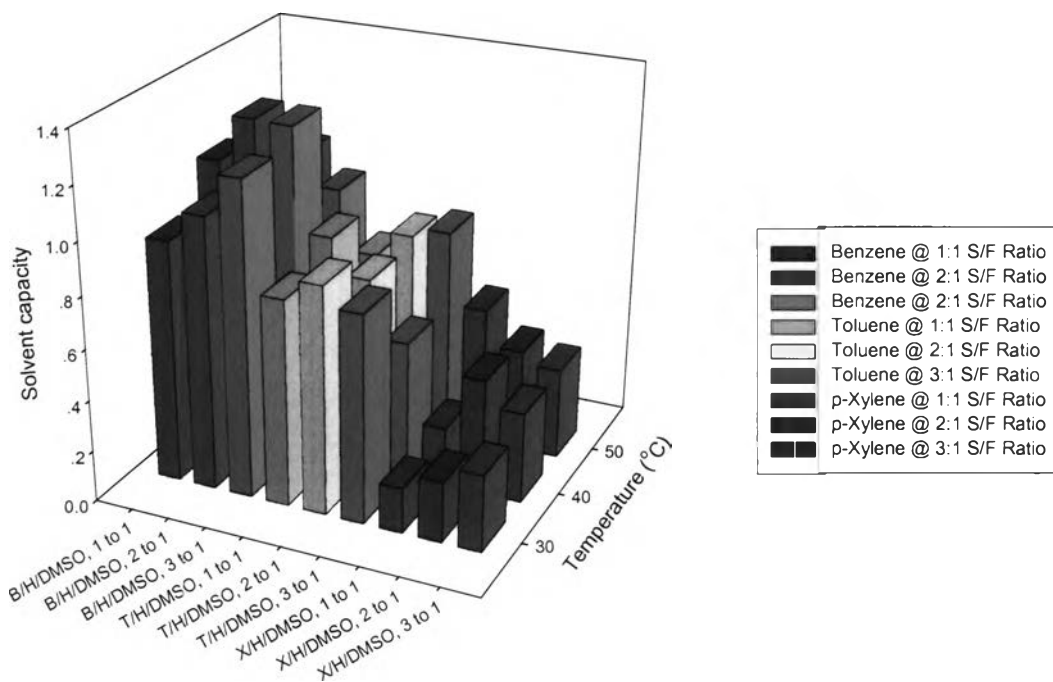
The extraction of aromatic from hexane employing DMSO, as shown in Figure 4.4 showed similar result with EG. As at higher solvent-to-feed ratio the mass fraction of the aromatic extracted decreased and benzene was better extracted than toluene and *p*-xylene. However, it was observed that, when using DMSO as the solvent compared to EG, the mass fraction of toluene extracted was closer to that of benzene. In Figure 4.5, the selectivity of the extraction of benzene was close to that of toluene; benzene in the range of 15.13 to 27.50 and toluene 11.85 to 29.26, respectively, while that for *p*-xylene are in the range of 2.80 to 11.67. The solvent capacity, as shown in Figure 4.6, again can be distinguished among the three aromatics studied that benzene was better capacitive than toluene and *p*-xylene, respectively. As mention earlier, the effect of temperature did not show significant effect in the range studied.



**Figure 4.4** Mass fraction of the aromatic in extract phase of aromatic/hexane/DMSO systems at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



**Figure 4.5** Selectivity of the extraction of aromatic/hexane/DMSO systems at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.

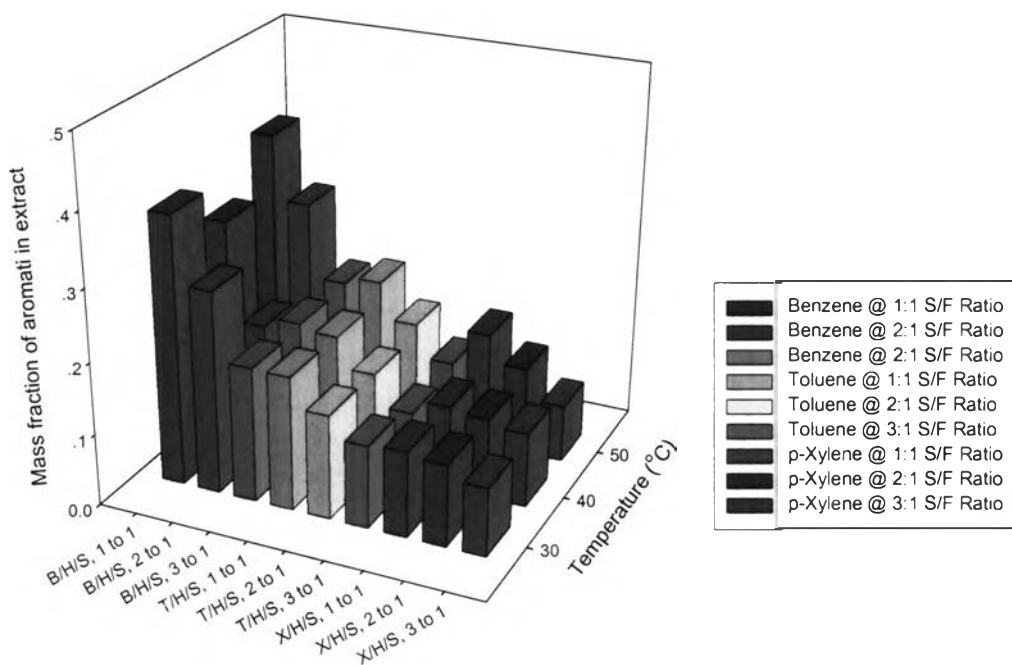


**Figure 4.6** Solvent capacity of the extraction of aromatic/hexane/DMSO systems at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.

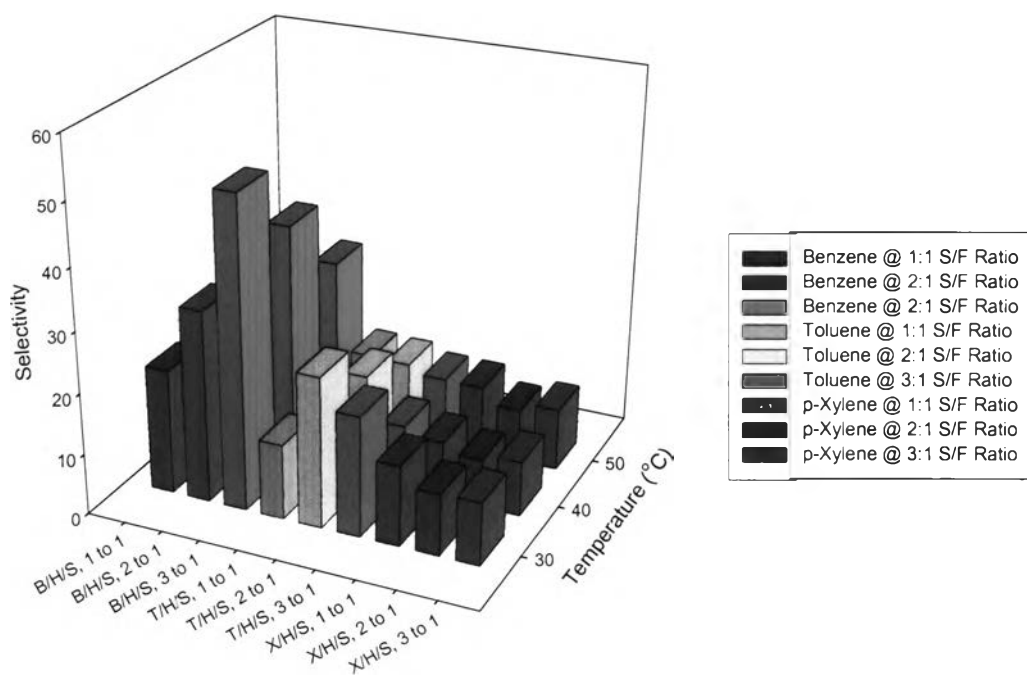
#### 4.3.1.3 Sulfolane

The extraction of benzene by sulfolane was found to be better than toluene and *p*-xylene especially at the same solvent-to-feed ratio; the mass-fraction range of benzene extracted are 0.130 to 0.293 while toluene and *p*-xylene were in the ranges of 0.090 to 0.166, and 0.085 to 0.144, respectively, as shown in Figure 4.7. In Figure 4.8, the selectivity of the extraction was found to be high in benzene as they are in the range of 16.76 to 50.85 and lower in toluene and *p*-xylene as the range are 10.71 to 24.81 and 7.90 to 13.37, respectively. The solvent capacity of benzene, as shown in Figure 4.9, was higher than those of toluene and *p*-xylene, respectively. Figure 4.9 shows the ranges of solvent capacity as they are 0.32 to 0.54 for benzene and 0.18 to 0.32 and 0.17 to 0.20 for toluene and *p*-xylene, respectively. For the effect of temperature, it was found that temperature also did not play significant effect to this extraction system.

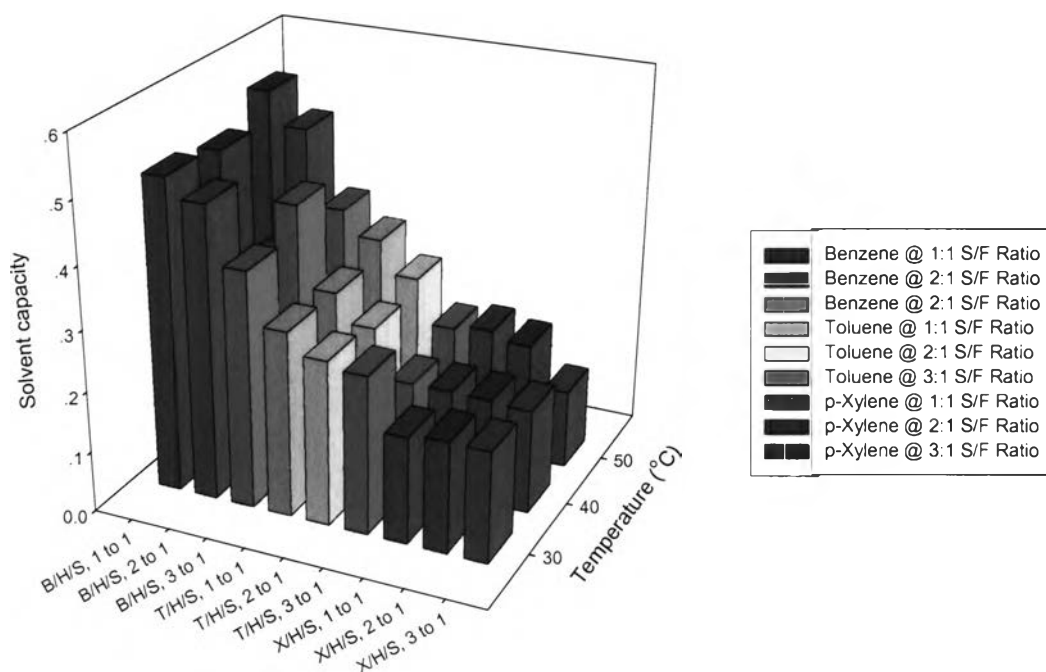




**Figure 4.7** Mass fraction of the aromatic in extract phase of aromatic/hexane/sulfolane systems at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



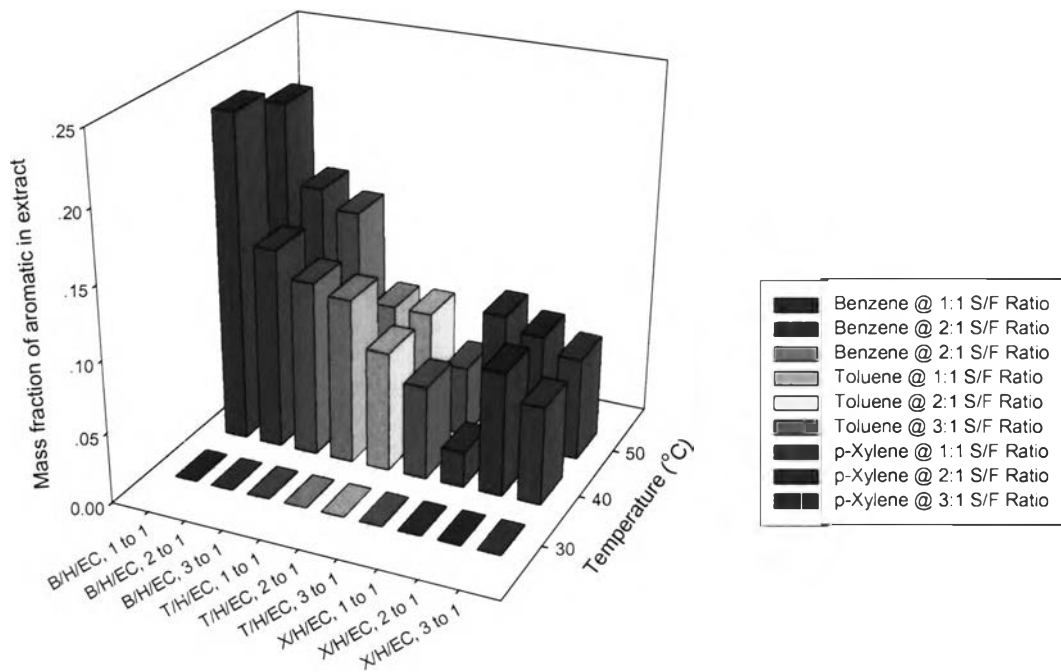
**Figure 4.8** Selectivity of the extraction of aromatic/hexane/sulfolane systems at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



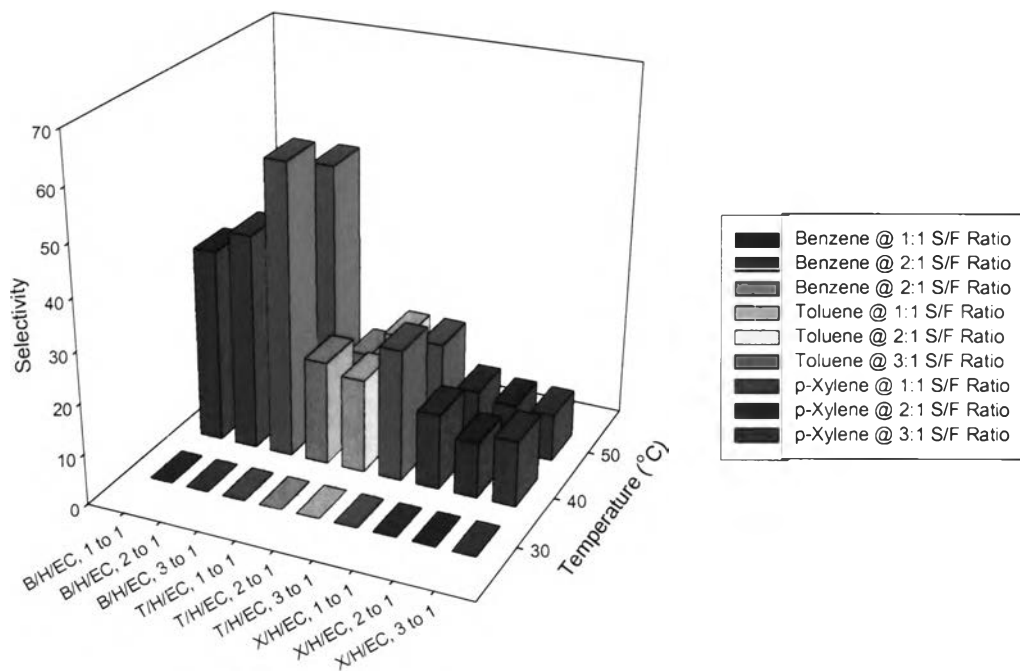
**Figure 4.9** Solvent capacity of the extraction of aromatic/hexane/sulfolane systems at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios. Li

#### 4.3.1.4 Ethylene Carbonate (EC)

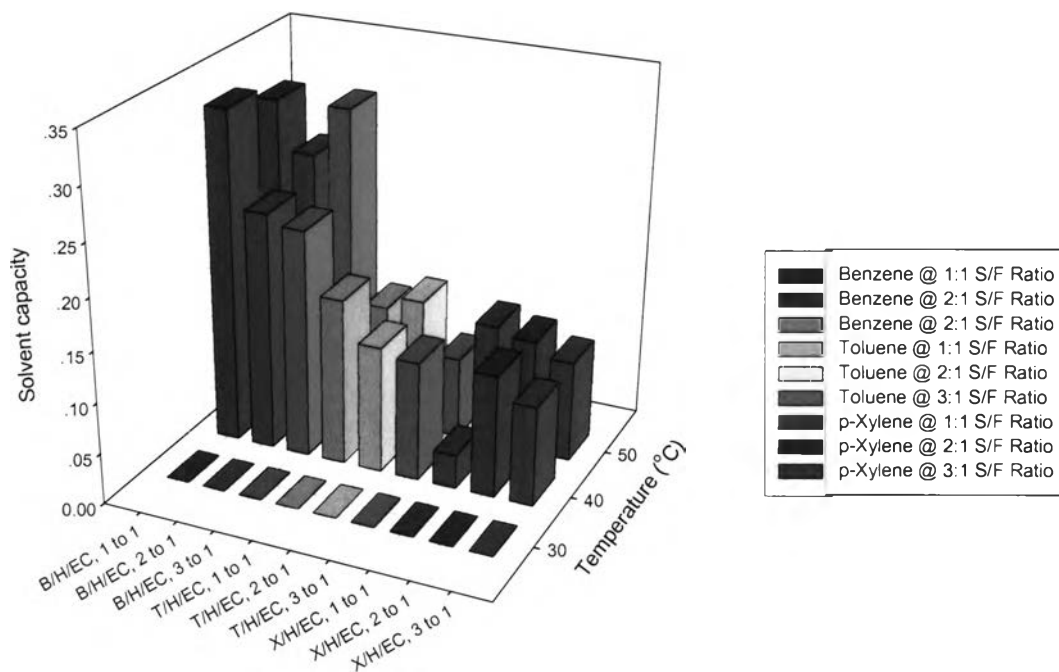
With using EC as a solvent for extraction, the extraction was not possible at 30°C because the melting point of EC is 36.2°C (Li and Balbuena, 1999). It was found that benzene was higher extracted than toluene and *p*-xylene. The mass fraction ranges of the extract phase were 0.122 to 0.229 for benzene, 0.065 to 0.115 for toluene, and 0.025 to 0.092 for *p*-xylene, respectively as shown in Figure 4.10. The similar behavior of lower aromatic-extracted amount at higher solvent-to-feed ratio was also found with EC systems. The selectivity of benzene was better than those of toluene and *p*-xylene, i.e. 33.57 to 57.73 for benzene, 13.86 to 25.91 for toluene, and 9.26 to 10.91 for *p*-xylene as shown in Figure 4.11. In solvent capacity, as shown in Figure 4.12, EC performed greater capacity with benzene than those of toluene and *p*-xylene; the ranges are 0.223 to 0.325 for benzene, 0.079 to 0.163 for toluene, and 0.033 to 0.120 for *p*-xylene. However, there was not much different in extracted amount and solvent capacity of toluene and *p*-xylene. In this system, the temperature also did not play significant effect in the range studied.



**Figure 4.10** Mass fraction of the aromatic in extract phase of aromatic/hexane/EC systems at the temperature of 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



**Figure 4.11** Selectivity of the extraction of aromatic/hexane/EC systems at the temperature of 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



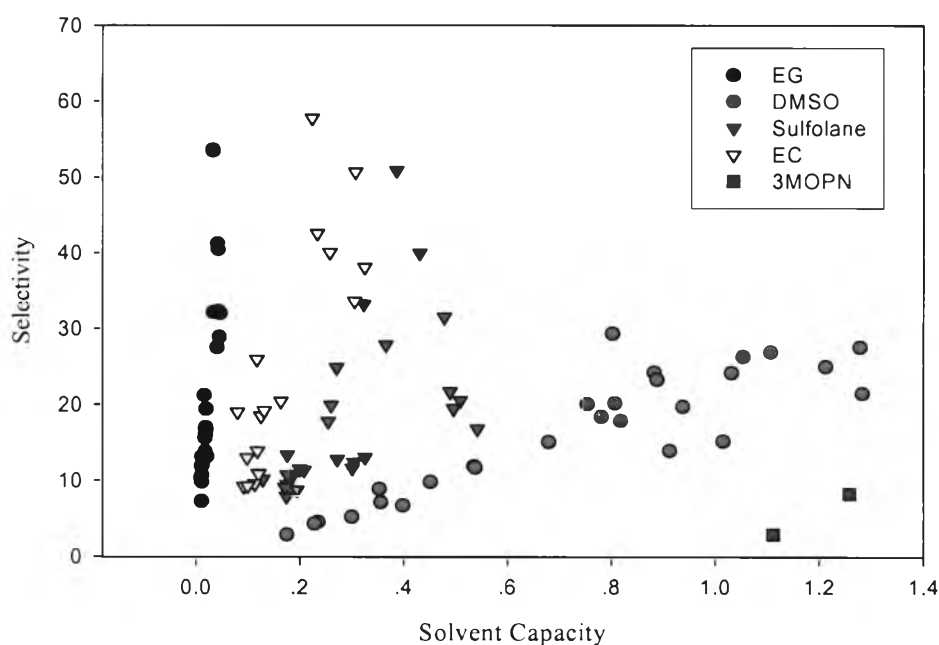
**Figure 4.12** Solvent capacity of the extraction of aromatic/hexane/EC systems at the temperature of 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.

#### 4.3.1.5 3-methoxy propionitrile (3MOPN)

The extraction by 3MOPN could be carried out at only 2 conditions because it was limited by the temperature that they formed homogeneous solutions as mentioned in the earlier section, critical solution temperature (CST), that are the CST of hexane/benzene/3MOPN system and hexane/toluene/3MOPN were found at 42°, 26°C, respectively, and for the system of hexane/*p*-xylene/3MOPN, the CST was not available at 25°C. However, these 2 batch-conditions were done to get the preliminary estimation of the relation between selectivity and solvent capacity of the solvent before using it as one of mixing components with other solvent as the mixed solvents. The benzene mass-fraction range was 0.320 to 0.336 and the selectivity and solvent capacity were in the range of 2.84 to 8.23 and 1.11 to 1.26 as shown in Figures 4.13, 4.14, and 4.15, respectively.

The single solvents studied were different in selectivity and solvent capacity for the extraction of the BTX aromatics. As shown in Figure 4.13, EG performed high capacity but poor in solvent capacity while EC was highly selective and had higher solvent capacity than EG. Sulfolane performed good in selectivity and solvent capacity. DMSO was quite high selective solvent but low in capacity. 3MOPN was very high capacity but very low in selectivity. Thus, ranking the selectivity from high to low are: EG>EC>sulfolane>DMSO>3MOPN. In solvent capacity ranking from high to low are: 3MOPN>DMSO>sulfolane>EC>EG.

From the relationship mentioned, two pairs of mixed solvent was selected; they are EC/DMSO and EG/3MOPN to get the best combination of properties, both in selectivity and solvent capacity.

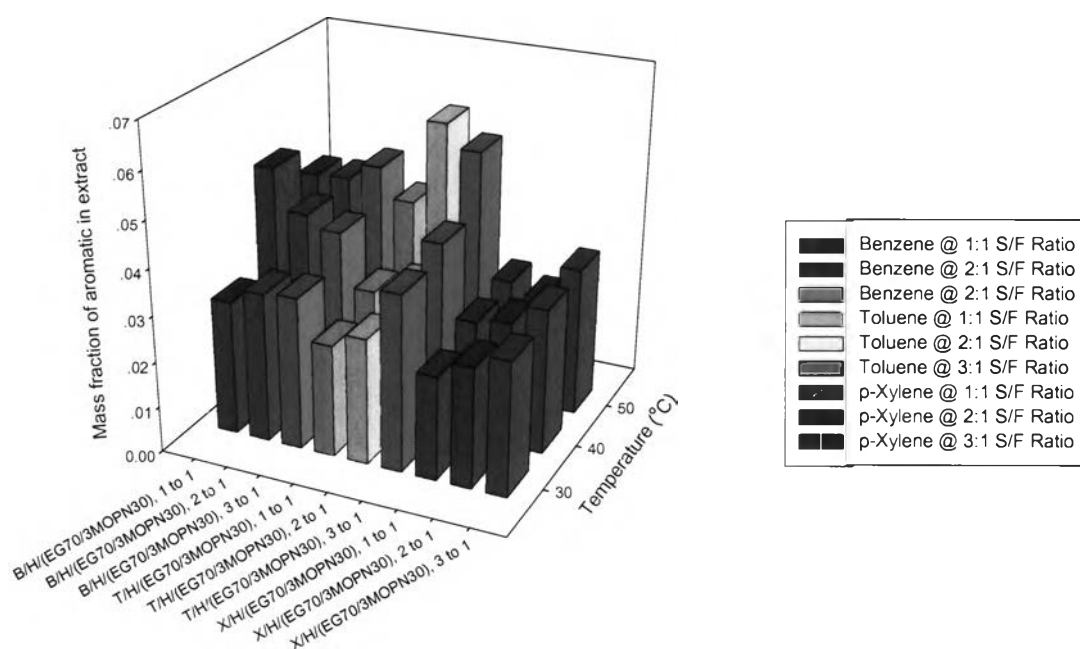


**Figure 4.13** Relationship between selectivity and solvent capacity of the single solvents studied.

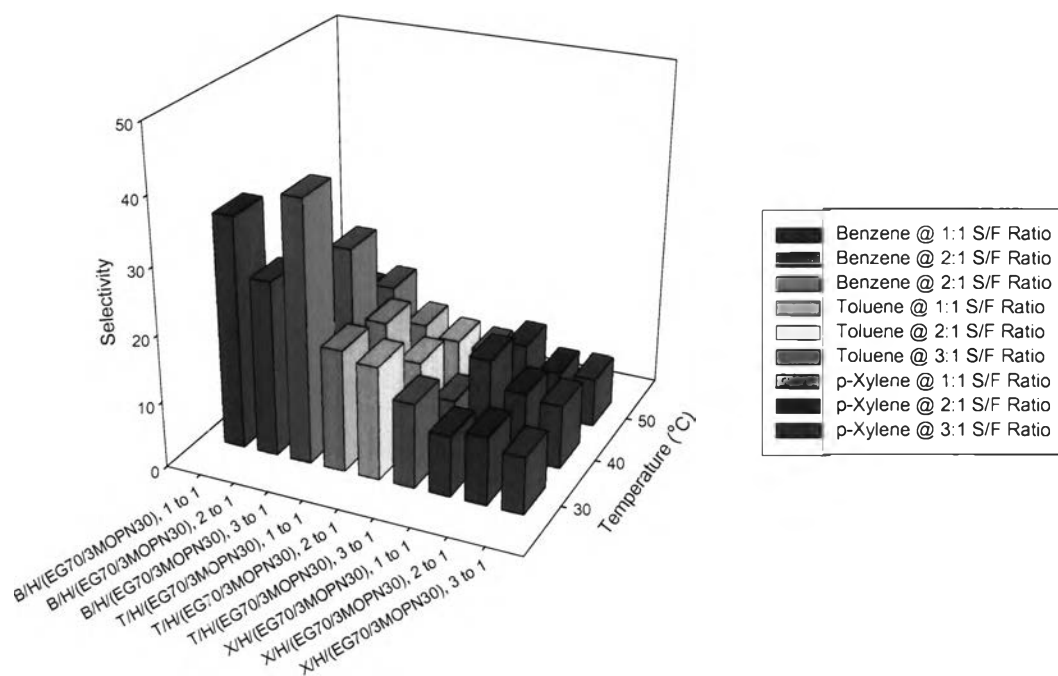
### 4.3.2 Mixed Solvent

#### 4.3.2.1 Ethylene Glycol/3-methoxy Propionitrile (EG/3MOPN) at 70/30 by Volume

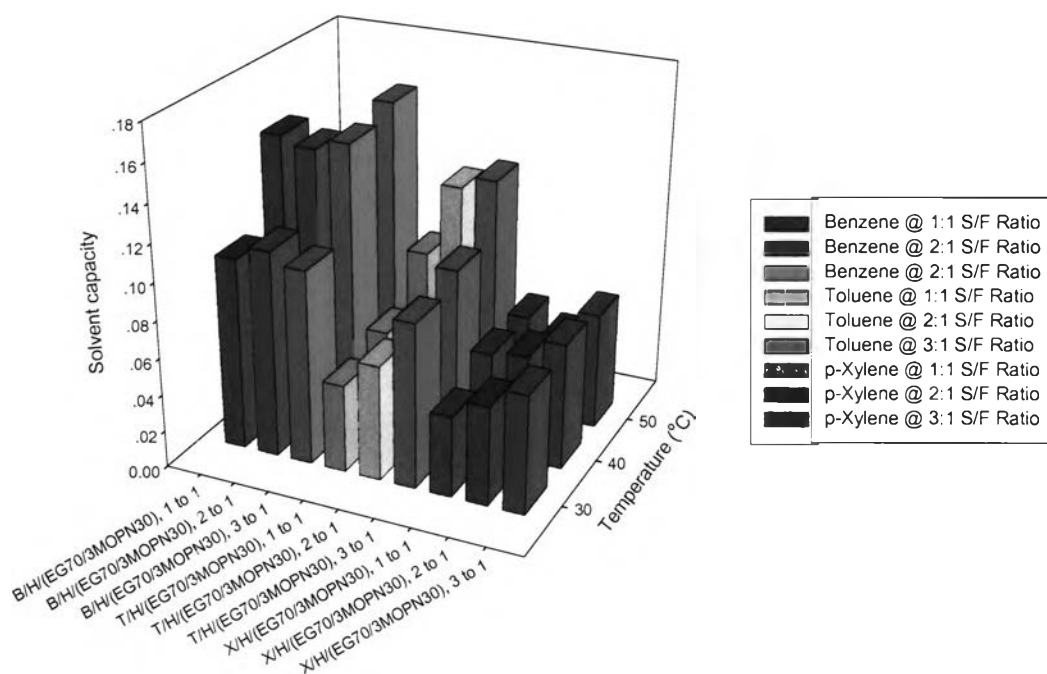
The mixed solvent of EG and 3MOPN at 70/30 by volume was used to extract the aromatic and performed slightly different in amount of aromatic extracted as they are benzene, toluene, and *p*-xylene. The ranges were 0.029 to 0.051 for benzene, 0.024 to 0.059 for toluene, and 0.022 to 0.032 for *p*-xylene as shown in Figure 4.14. The temperature affect the amount of aromatics extracted as the temperature increased, the aromatic extracted increased. For the selectivity, among the three aromatics, this mixed solvent had better selectivity to benzene than toluene and *p*-xylene, respectively, as shown in Figure 4.15. As the temperature increased the selectivity showed decreasing trend. Figure 4.16 shows the solvent capacity of the solvent to the three aromatics that they are higher for benzene than toluene and *p*-xylene, respectively. The capacity tends to increase with the increase of temperature. In this mixed ratio, the solvent performed well in capacity as the presence of EG but was not much upgraded in the solvent capacity.



**Figure 4.14** Mass fraction of the aromatic in extract phase of the systems of aromatic/hexane/mixed solvent of EG/3MOPN at 70/30 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



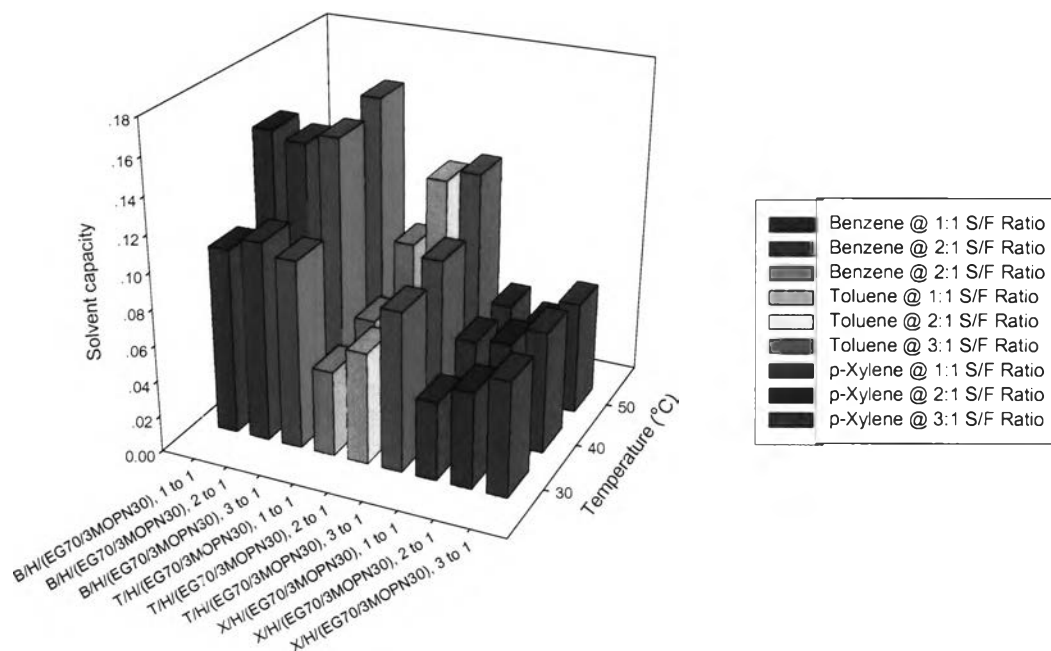
**Figure 4.15** Selectivity of the extraction of the systems of aromatic/hexane/mixed solvent of EG/3MOPN at 70/30 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



**Figure 4.16** Solvent capacity of the extraction of the system of aromatic/hexane/mixed solvent of EG/3MOPN at 70/30 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.

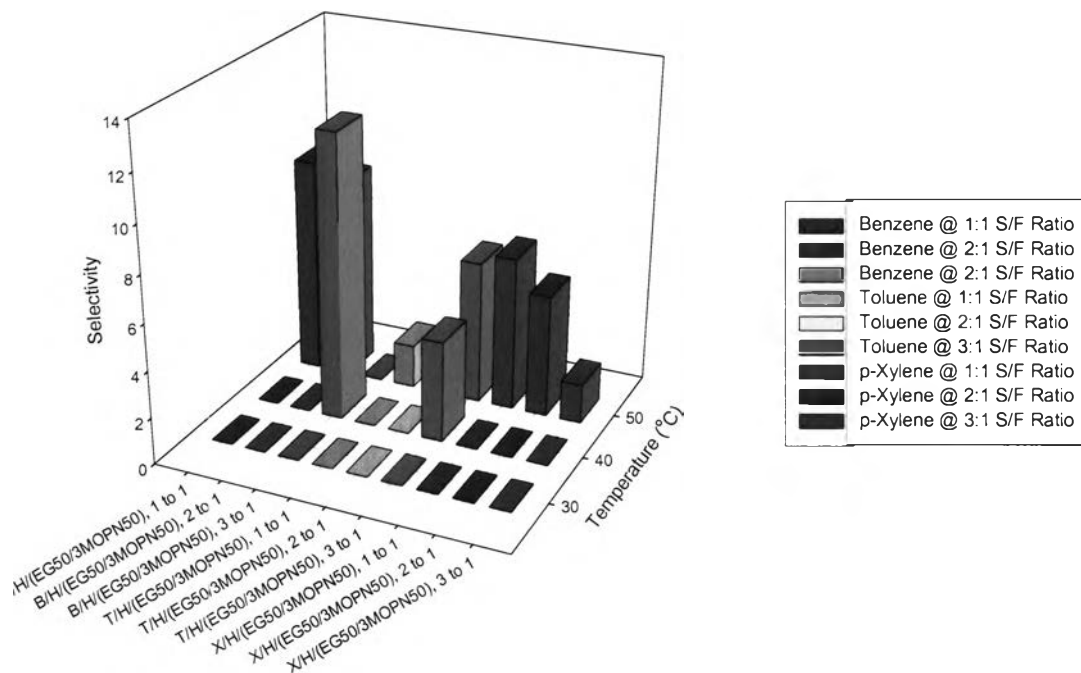
#### 4.3.2.2 Ethylene Glycol/3-methoxy Propionitrile (EG/3MOPN) at 50/50 by Volume

The extraction by the mixed solvent of EG and 3MOPN at 50/50 by volume was expected to get better capacity as the ratio of 3MOPN was increased but the systems can be done only in some conditions because they performed 3-phase solutions, especially at the low temperature, and one-phase solutions. The extraction run that were carried out to compare with other ration of the same mixed solvent and other solvents. The mass-fraction were in the range of 0.056 to 0.090 for benzene, 0.044 to 0.084 for toluene, and 0.041 to 0.056 for *p*-xylene as shown in Figure 4.17. The selectivity, in Figure 4.18, were in 0.20 to 0.36 range for benzene, and 0.14 to 0.30 for toluene, and 0.08 to 0.20 for *p*-xylene. Figure 4.19 indicated that toluene performed better solvent capacity with the solvent than benzene and *p*-xylene as they were in the ranges of 0.14 to 0.35, 0.20 to 0.36 and 0.08 to 0.20, respectively.

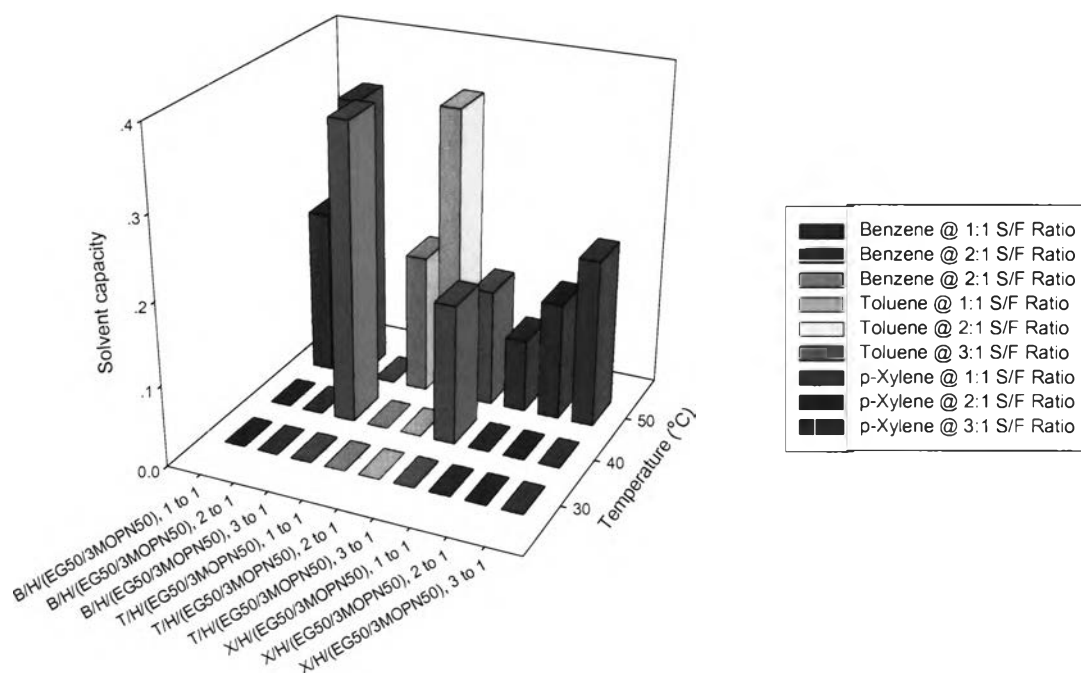


**Figure 4.17** Mass fraction of the aromatic in extract phase of the systems of aromatic/hexane/mixed solvent of EG/3MOPN at 50/50 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.





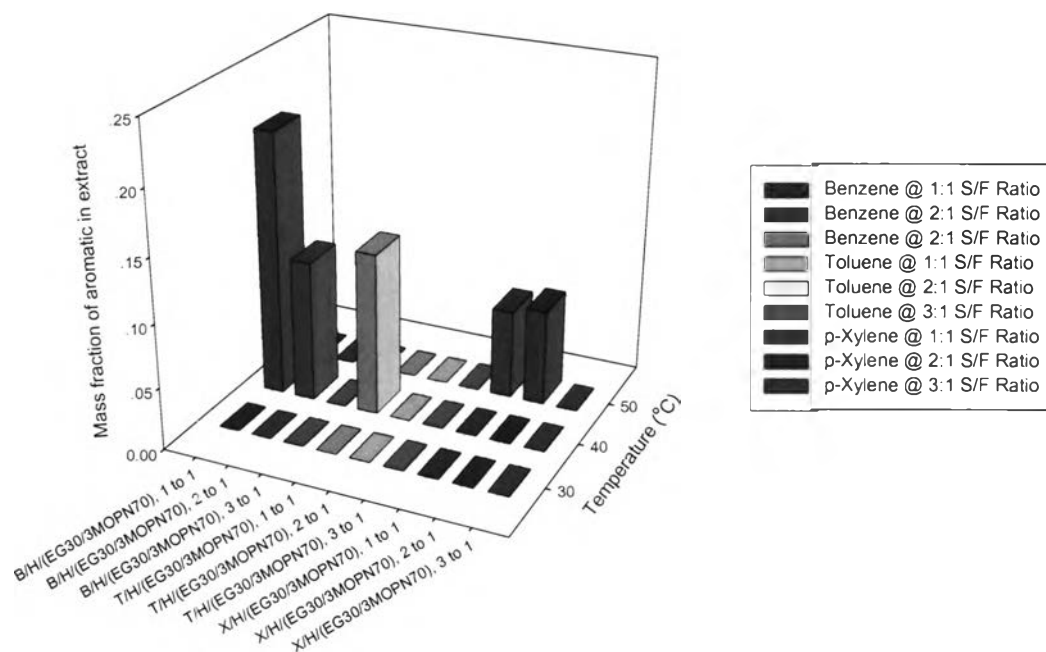
**Figure 4.18** Selectivity of the extraction of the systems of aromatic/hexane/ mixed solvent of EG/3MOPN at 50/50 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



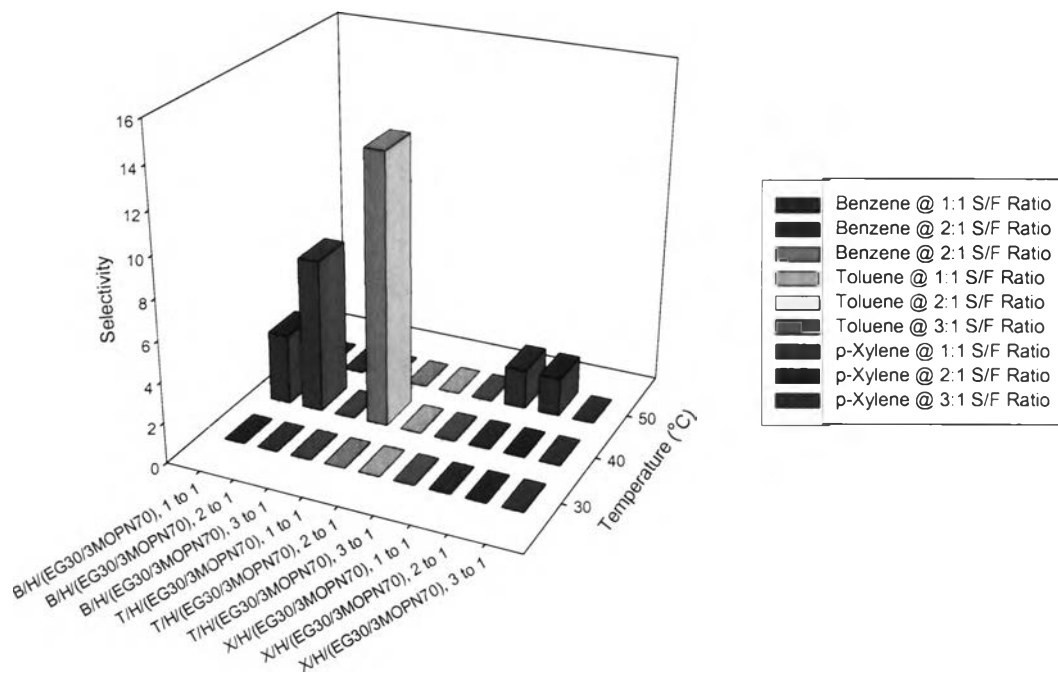
**Figure 4.19** Solvent capacity of the extraction of the system of aromatic/hexane/ mixed solvent of EG/3MOPN at 50/50 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.

#### 4.3.2.3 Ethylene Glycol/3-methoxy Propionitrile (EG/3MOPN) at 30/70 by Volume

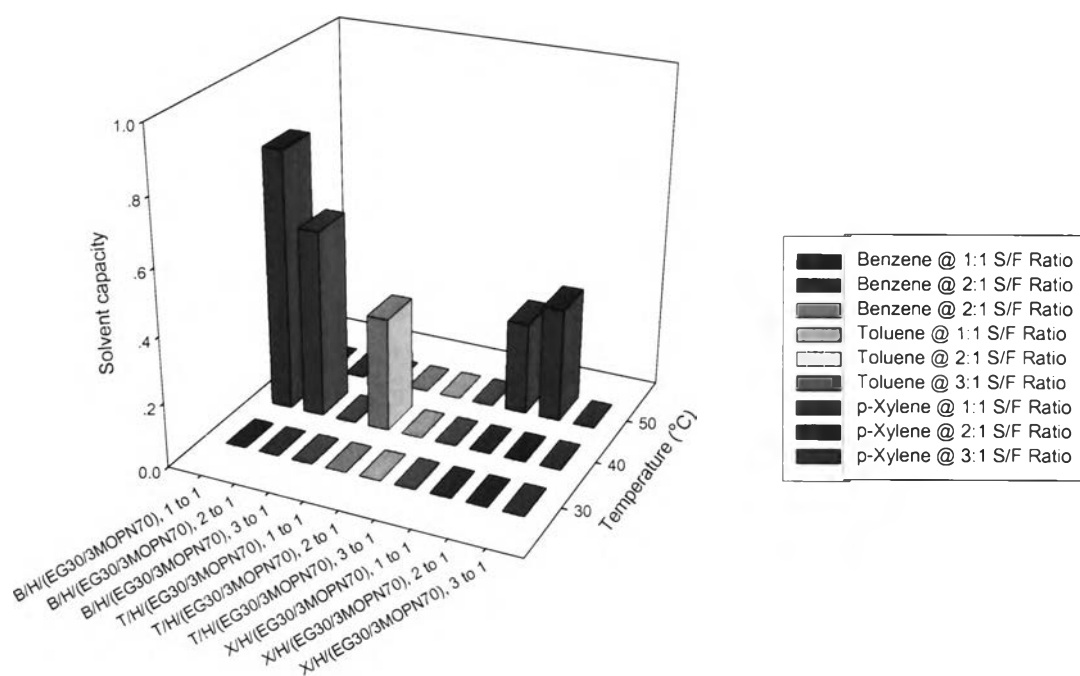
The more presence of 3MOPN in the mixed solvent of EG/3MOPN at 30/70 by volume was expected to obtain better capacity and to eliminate the 3-phase solution systems. However, the more presence of 3MOPN made the systems become homogenous in some conditions while the 3-phase solutions still performed in some conditions. As shown in Figure 4.20, the mass-fraction range of benzene was 0.11 to 0.20, for toluene was 0.12, and for *p*-xylene was 0.90 to 0.91. The selectivity of the extraction, in Figure 4.21, were in the ranges of 3.49 to 7.57 for benzene, 13.35 for toluene, and 1.88 to 1.94 for *p*-xylene. As shown in Figure 4.22, the solvent-capacity were in the ranges of 0.14 to 0.35, 0.20 to 0.36 and 0.08 to 0.20, respectively.



**Figure 4.20** Mass fraction of the aromatic in extract phase of the systems of aromatic/hexane/mixed solvent of EG/3MOPN at 30/70 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



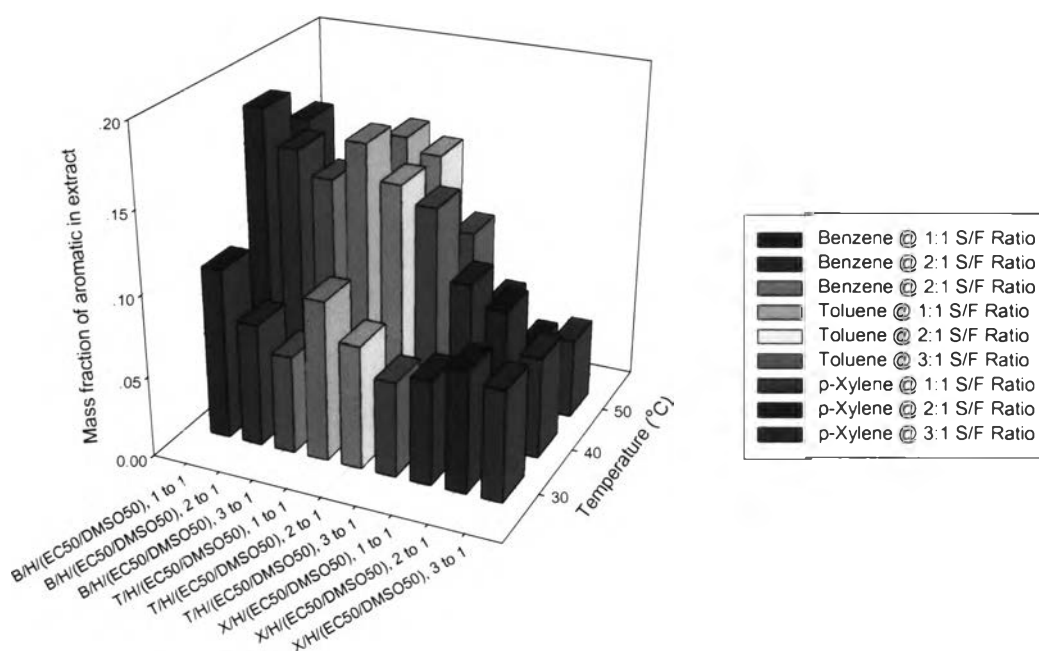
**Figure 4.21** Selectivity of the extraction of the systems of aromatic/hexane/mixed solvent of EG/3MOPN at 30/70 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



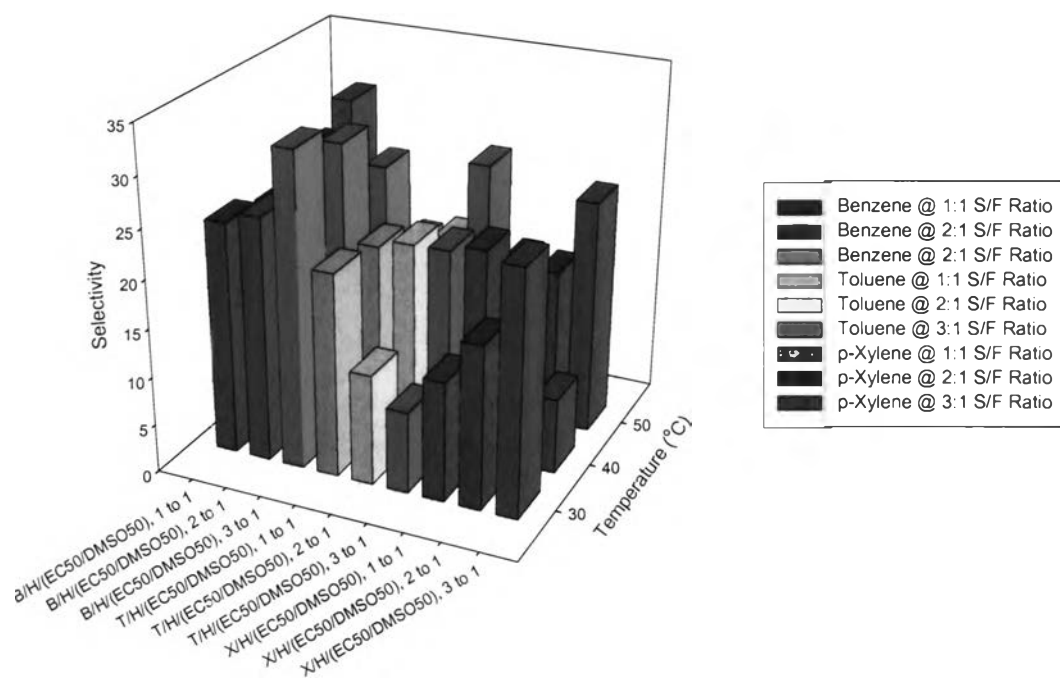
**Figure 4.22** Solvent capacity of the extraction of the system of aromatic/hexane/mixed solvent of EG/3MOPN at 30/70 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.

#### 4.3.2.4 Ethylene Carbonate/Dimethylsulfoxide (EC/DMSO) at 50/50 by Volume

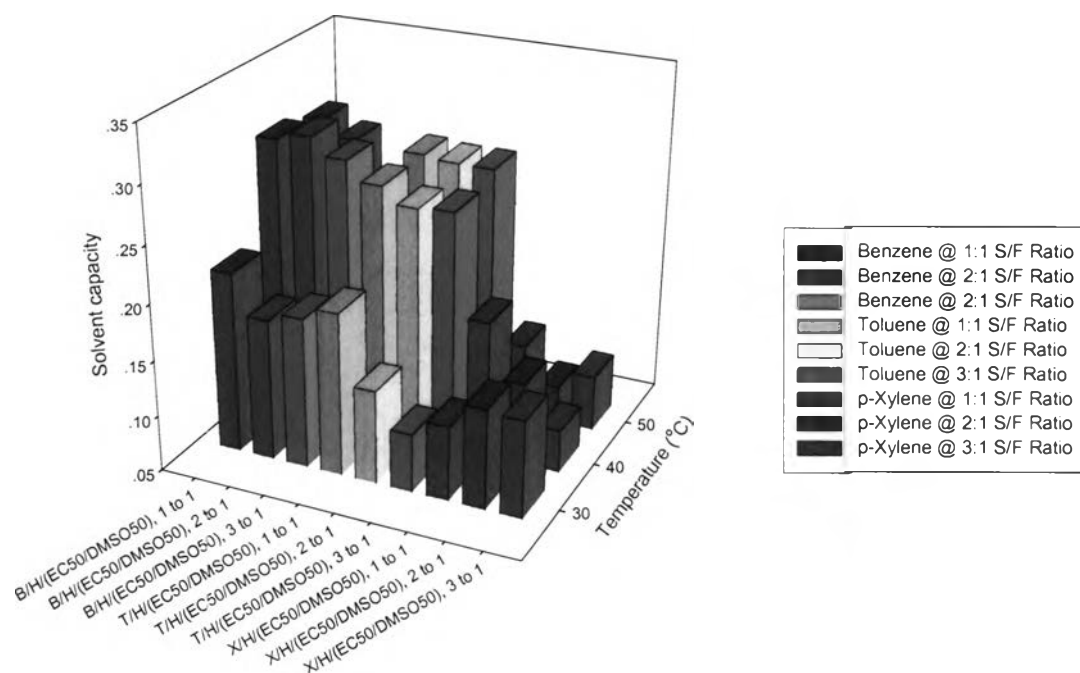
The extractions by the mixed solvent of EC/DMSO at 50/50 by volume was established to get half-way properties between EC and DMSO. The amount of extracted aromatic, as shown in Figure 4.23, was quite similar in the case of benzene and toluene and the ranges were 0.060 to 0.182 and 0.058 to 0.172 respectively while the range of *p*-xylene was 0.042 to 0.098. Figure 4.24 shows the range of the selectivity for benzene as 22.56 to 32.23, for toluene as 8.42 to 26.10, and for *p*-xylene as 7.79 to 25.10. The capacity were similar in the case of benzene and toluene which were higher than that of *p*-xylene as they are in the range of 0.176 to 0.305 for benzene, 0.102 to 0.274 for toluene, and 0.083 to 0.139 for *p*-xylene as shown in Figure 4.25. There was not much significant effect by the operating temperature. The combined properties of the mixed solvent were good in selectivity but not good in solvent capacity. The mixed solvent performance was closer to EC than DMSO as it was quite high selective solvent.



**Figure 4.23** Mass fraction of the aromatic in extract phase of the systems of aromatic/hexane/mixed solvent of EC/DMSO at 50/50 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.



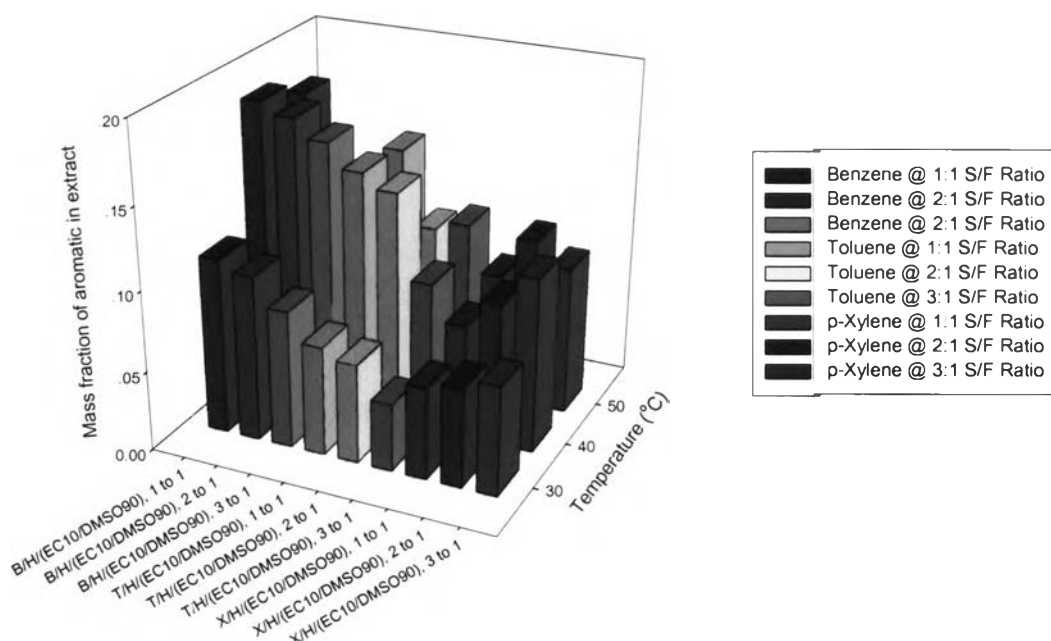
**Figure 4.24** Selectivity of the extraction of the systems of aromatic/hexane/mixed solvent of EC/DMSO at 50/50 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios



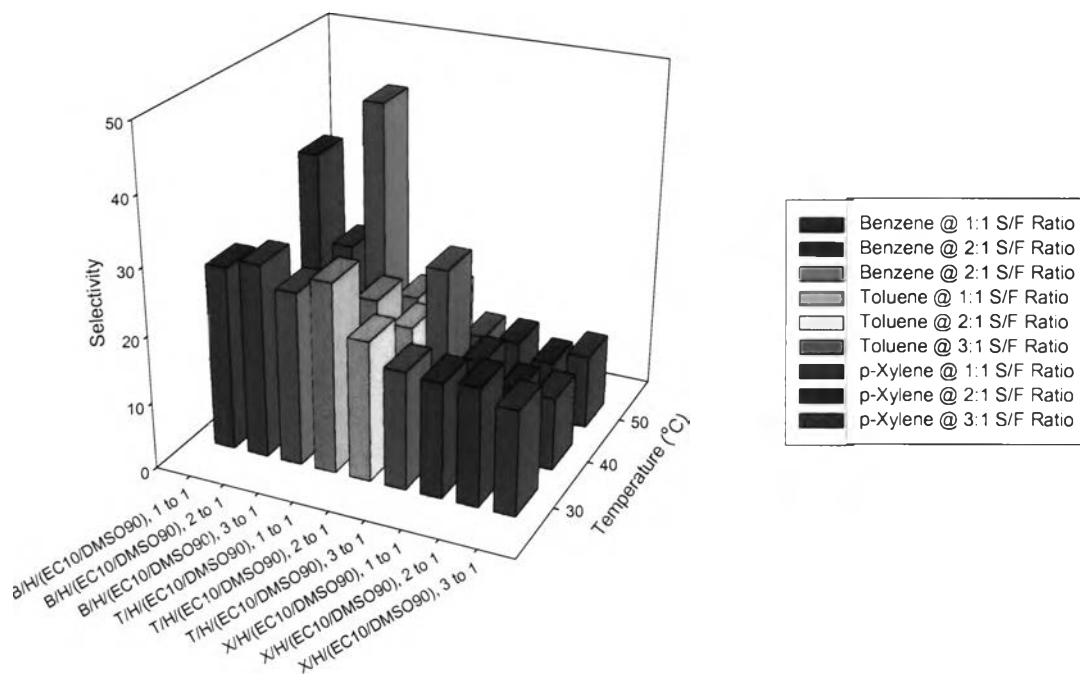
**Figure 4.25** Solvent capacity of the extraction of the system of aromatic/hexane/mixed solvent of EC/DMSO at 50/50 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios

#### 4.3.2.5 Ethylene Carbonate/Dimethylsulfoxide (EC/DMSO) at 10/90 by Volume

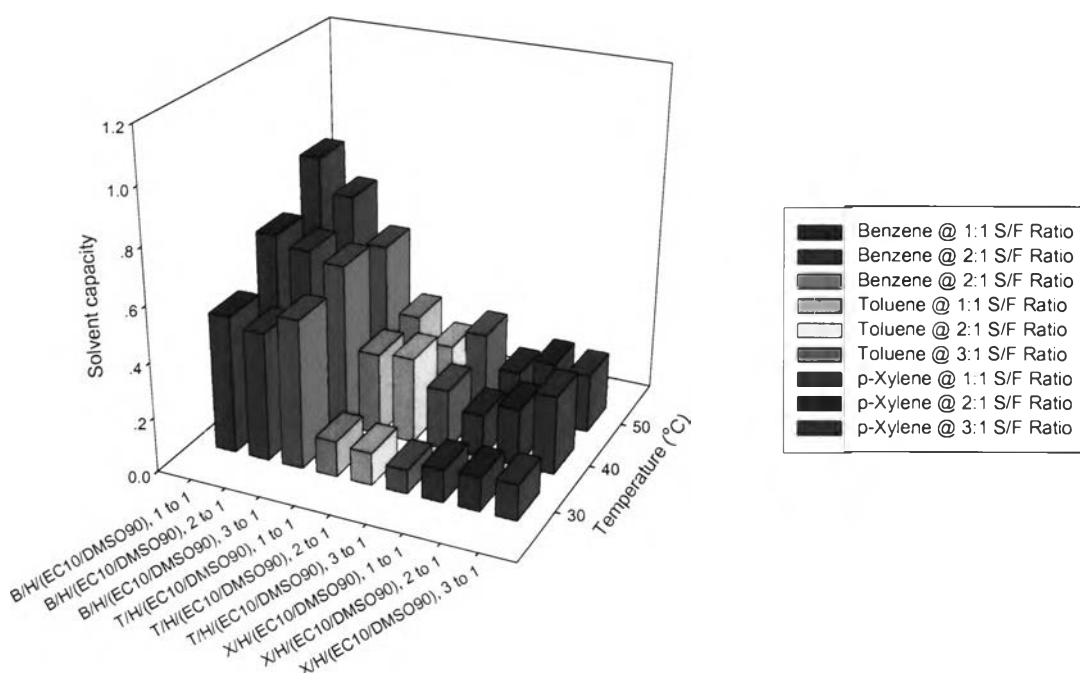
When using the mixed solvent of EC/DMSO at this ratio, it was expected to gain more capacitive properties at the mere presence of DMSO. Figure 4.26 indicates that benzene was better extracted than toluene and *p*-xylene, as the ranges of benzene, toluene, and *p*-xylene were 0.070 to 0.185, 0.041 to 0.152, and 0.056 to 0.108, respectively. The selectivity were in the ranges of 12.69 to 43.71 for benzene, 10.98 to 18.35 for toluene, and 8.97 to 17.72 for *p*-xylene as shown in Figure 4.27. The capacity was higher for benzene than toluene and *p*-xylene. The ranges were 0.46 to 0.82, 0.08 to 0.30, and 0.11 to 0.28 for benzene, toluene, and *p*-xylene, respectively, as shown in Figure 4.28. The effect of the operating temperature was no significant in the range studied. The overall properties of this mixed ratio of the dual solvent of EC/DMSO was better than that of 50/50 by volume while selectivity was quite good.



**Figure 4.26** Mass fraction of the aromatic in extract phase of the systems of aromatic/hexane/mixed solvent of EC/DMSO at 10/90 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios.

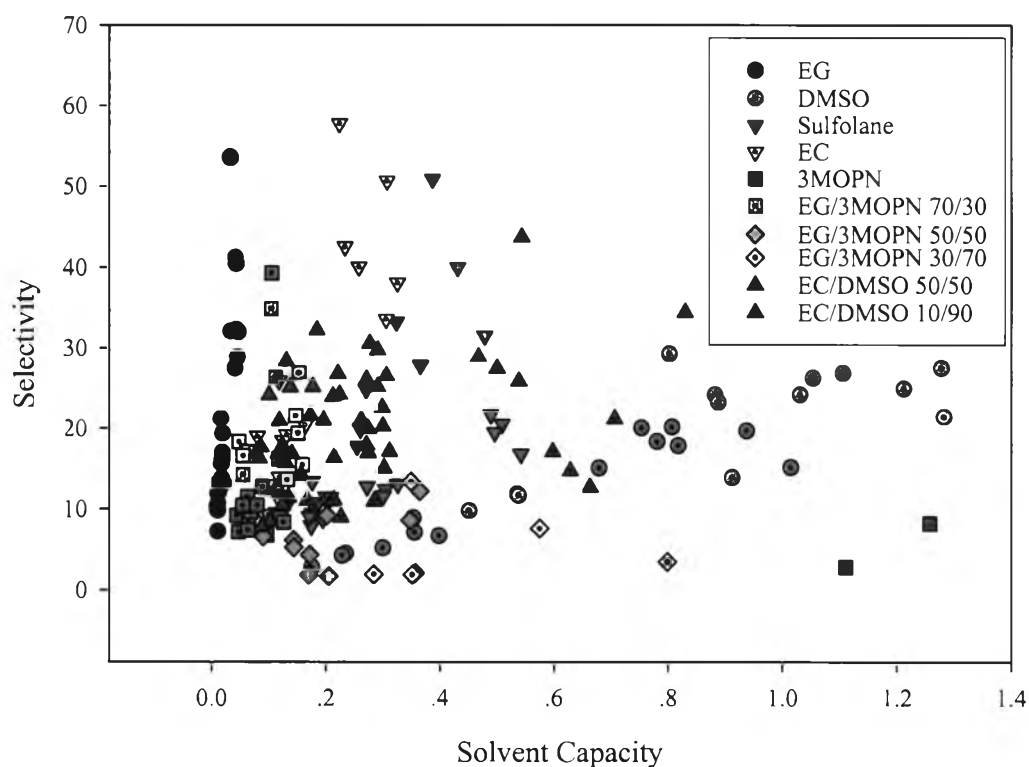


**Figure 4.27** Selectivity of the extraction of the systems of aromatic/hexane/mixed solvent of EC/DMSO at 10/90 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios



**Figure 4.28** Solvent capacity of the extraction of the system of aromatic/hexane/mixed solvent of EC/DMSO at 10/90 by volume at the temperature of 30°, 40°, and 50°C and 1:1, 2:1, and 3:1 of solvent-to-feed ratios

The mixed solvents showed combined properties of the single solvents that they are made of. As shown in Figure 4.29, the mixed solvent of EG/3MOPN at 70/30 by volume performed very close to EG, good selectivity but not good capacity, but the EG/3MOPN at 50/50 and 30/70 by volume perform quite poor in both selectivity and solvent capacity even though the 50/50 ratio performed better properties. Both ratios of the mixed solvent of EC/DMSO had better properties of selectivity and solvent capacity as compared to the mixed solvent of EG/3MOPN. The mixed solvent of EC/DMSO at 50/50 by volume showed good combined properties of the solvents, however, it was closer to EC, that means it was highly selective solvent. The mixed solvent of EC/DMSO at 10/90 by volume had higher capacity than the 50/50 ratio while performed quite well in selectivity. In overall, the combined properties of the mixed solvent of EC/DMSO at 10/90 by volume had good properties in selectivity and capacity and more similar to sulfolane than the other mixed solvents.



**Figure 4.29** Relationship between selectivity and solvent capacity of the single and the mixed solvents studied.