

CHAPTER IV
RESULTS AND DISCUSSION

4.1 Catalyst characterization

4.1.1 FTIR spectroscopy

Silica Hi-Sil[®] 233 was treated to form the chemical bonded monolayer with octadecyltrichlorosilane (ODS). The changes in the absorption band due to the treatment of ODS are listed in Table 4.1. The absorption at 1463, 2855 and 2925 cm⁻¹ due to the alkyl group attributed to the octadecyl group appeared. The absorption at 1000-1100 due to the stretching vibration of Si-O were increased slightly whereas the absorption band at 3300-3600 decreased because of the loss of silanol group. These data qualitatively indicated that the ODS group was fixed on the silica surface.

Table 4.1 Changes of IR spectra of silica by the treatment of ODS

Band (cm ⁻¹)	
Appearance or increase in intensity	Decrease in intensity
ν(CH ₃) 2855,2925 ν(Si-O) 1000-1100 δ(CH ₃) 1463	ν(Si-OH) 3300-3600

4.1.2 Elemental analysis

The elemental analysis results are listed in Table 4.2. This result can be applied to indicate the amount of ODS presented in the treated silica. The silica ODS/0.5 indicated the silica reacted with ODS 0.5 ml.

Table 4.2 Percentage of carbon in the treated silica

Silica Type	%C	%C from ODS
Untreated	0.330	0.000
ODS silica/ 0.5	3.605	3.280
ODS silica/ 1	5.720	5.390
ODS silica/ 3	7.645	7.320
ODS silica/ 4	8.28	7.950
ODS silica/ 5	9.355	9.030
ODS silica/ 6	9.207	8.880

4.2 Adsorption of bonded ODS on silica Hi-Sil[®] 233

The ODS adsorption isotherm and the amount of bonded ODS on Silica Hi-Sil[®] 233 is shown in Figure 4.1. In general, the adsorption isotherm of surfactants on the charged solid surface can be divided into 4 regions. But, the adsorption isotherm of bonded ODS onto silica is not clear. The maximum adsorption of bonded monolayer of ODS on silica surface is 429.22 $\mu\text{mole/g}$ of treated silica.

The result showed that the adsorbed ODS increased rapidly in the first range and then gradually increased until it leveled off due to the bulkiness of the bonded ODS. This is because of the decreasing of the unoccupied silanol groups on the silica surface and the shielding effect of the bulkiness bonded ODS monolayer to the silanol groups.

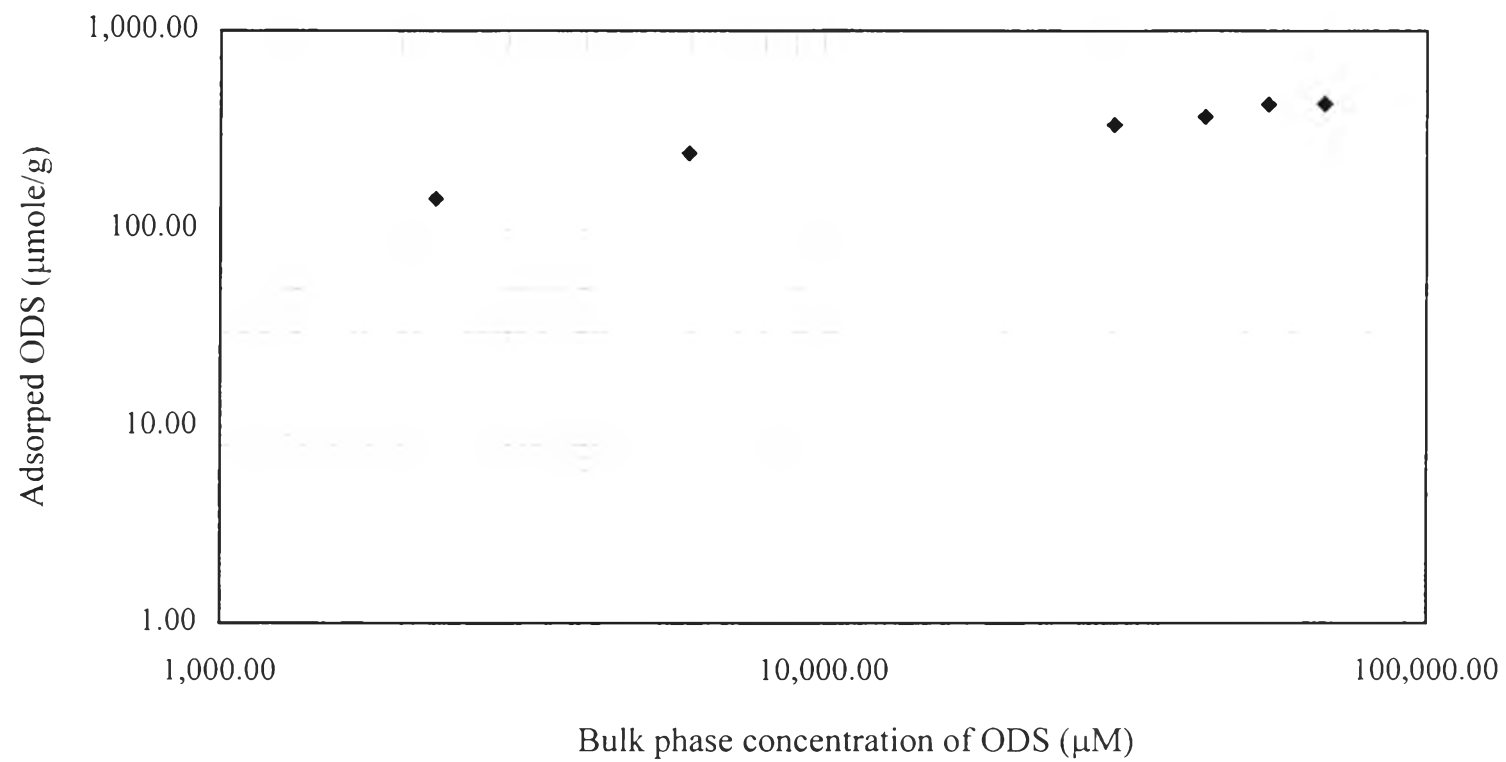


Figure 4.1 ODS adsorption isotherm on silica III Sil[®]233.

4.3 Adsolubilization of trichloroethylene (TCE) into ODS monolayer

The relationship between the amount of equilibrium TCE in aqueous solution and TCE in admicelles is shown in Figure 4.2. The result showed that the relationship was straight line. The shape did not have the plateau region. This implied that there is not the attraction or the repulsion between TCE in bonded monolayer and the oncoming TCE.

4.4 Effectiveness of catalyst

Comparison of fractional conversion between with and without catalyst is shown in Figure 4.3. The result showed that the catalyst could greatly improve the ozonation process. This was caused presumably by the increase of the interaction between the adsolubilized TCE on the catalyst and ozone gas.

4.5 Reaction Studies

4.5.1 External film resistance

The presence of external film resistance was treated by measuring the reaction rate when varying stirring speed of the mechanical stirrer at the constant ozone concentration, flow rate, temperature and TCE concentration. As the stirring speed was increased, the boundary layer thickness decreased until the mass transfer across the boundary layer is no longer limited the rate of reaction.

Figure 4.4 showed the relationship between TCE concentration and reaction time at various stirring speeds. Figure 4.5 also showed the external

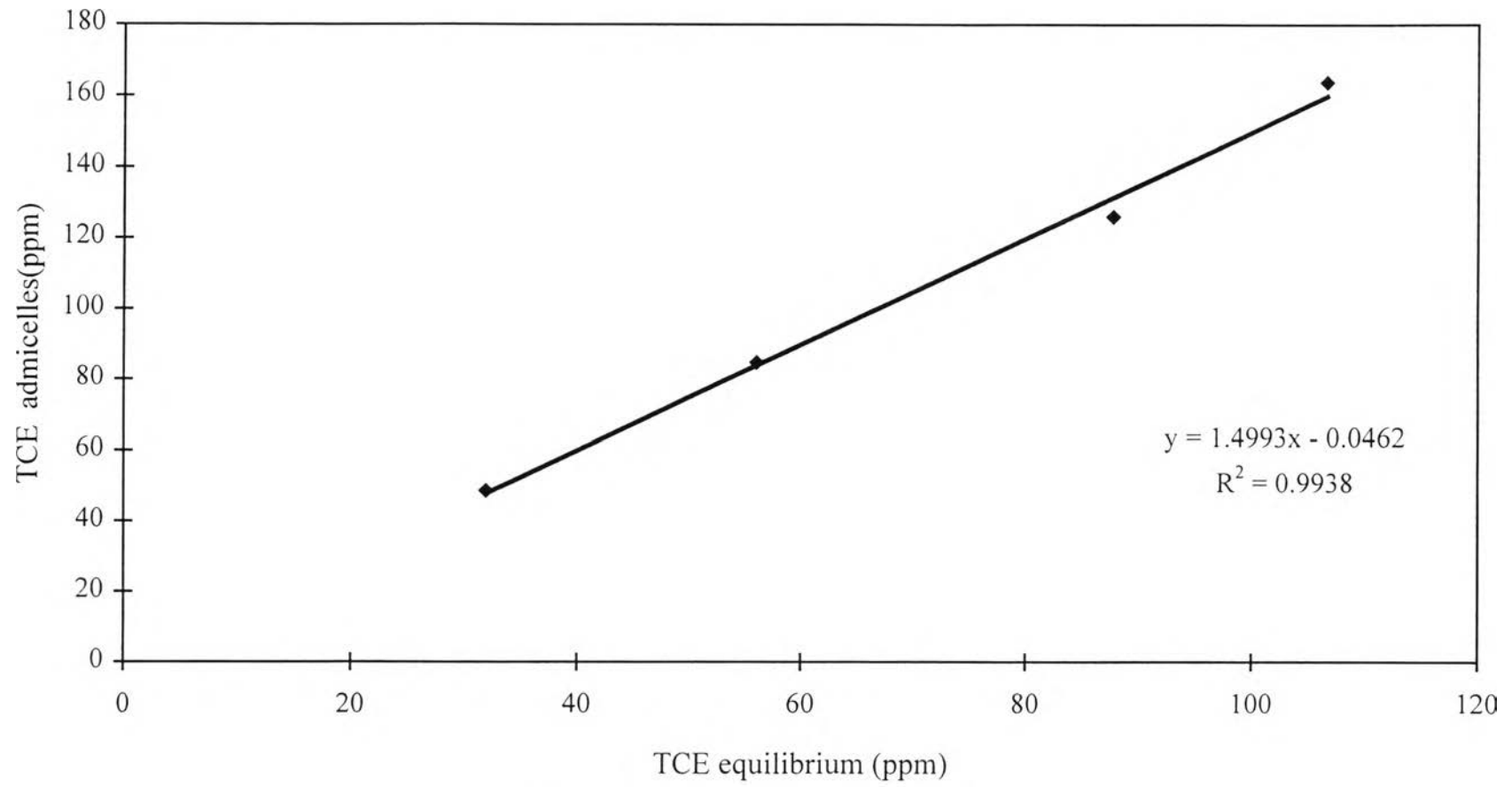


Figure 4.2 Adsolubilization curve.

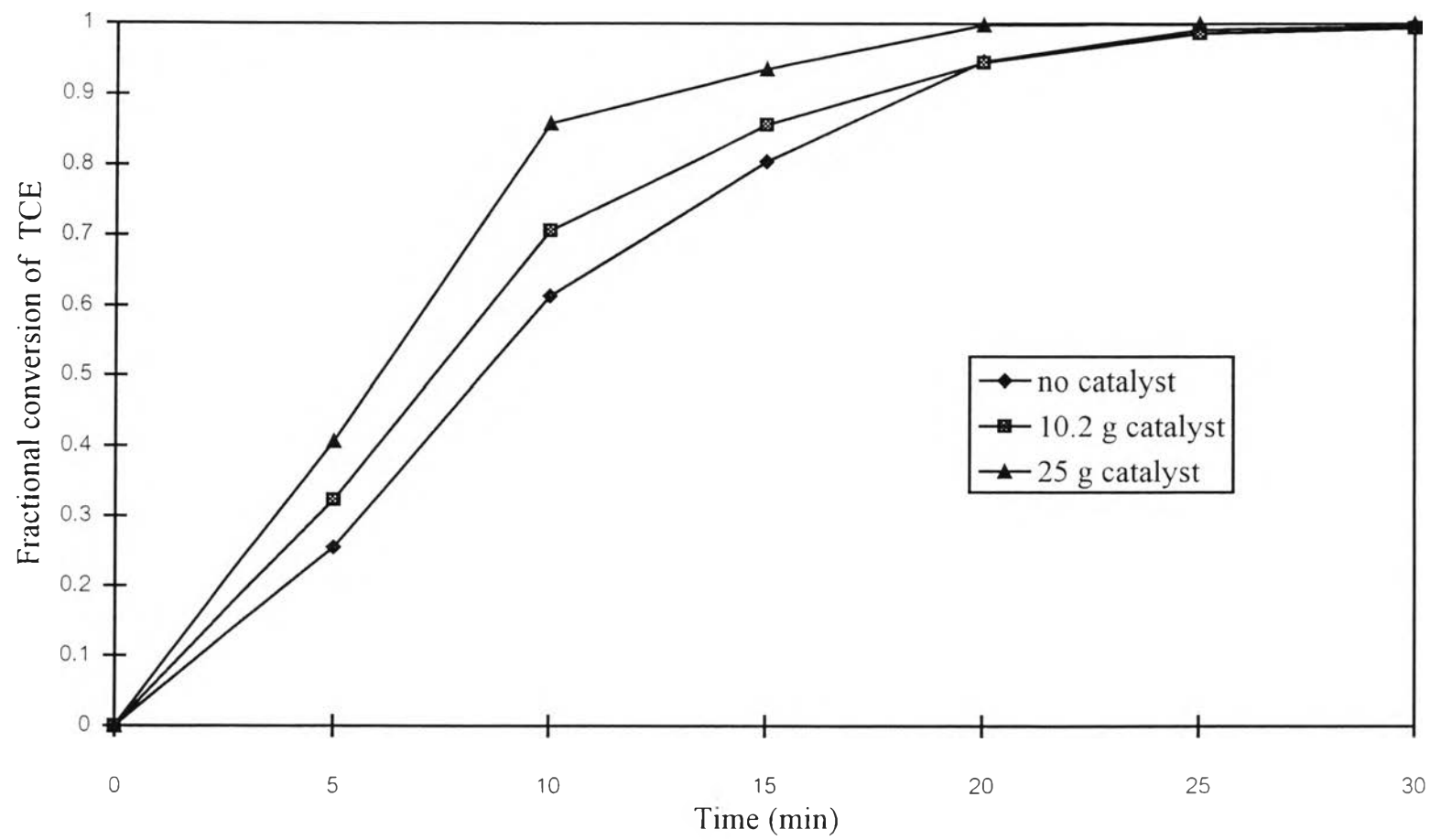


Figure 4.3 Effectiveness of catalyst.

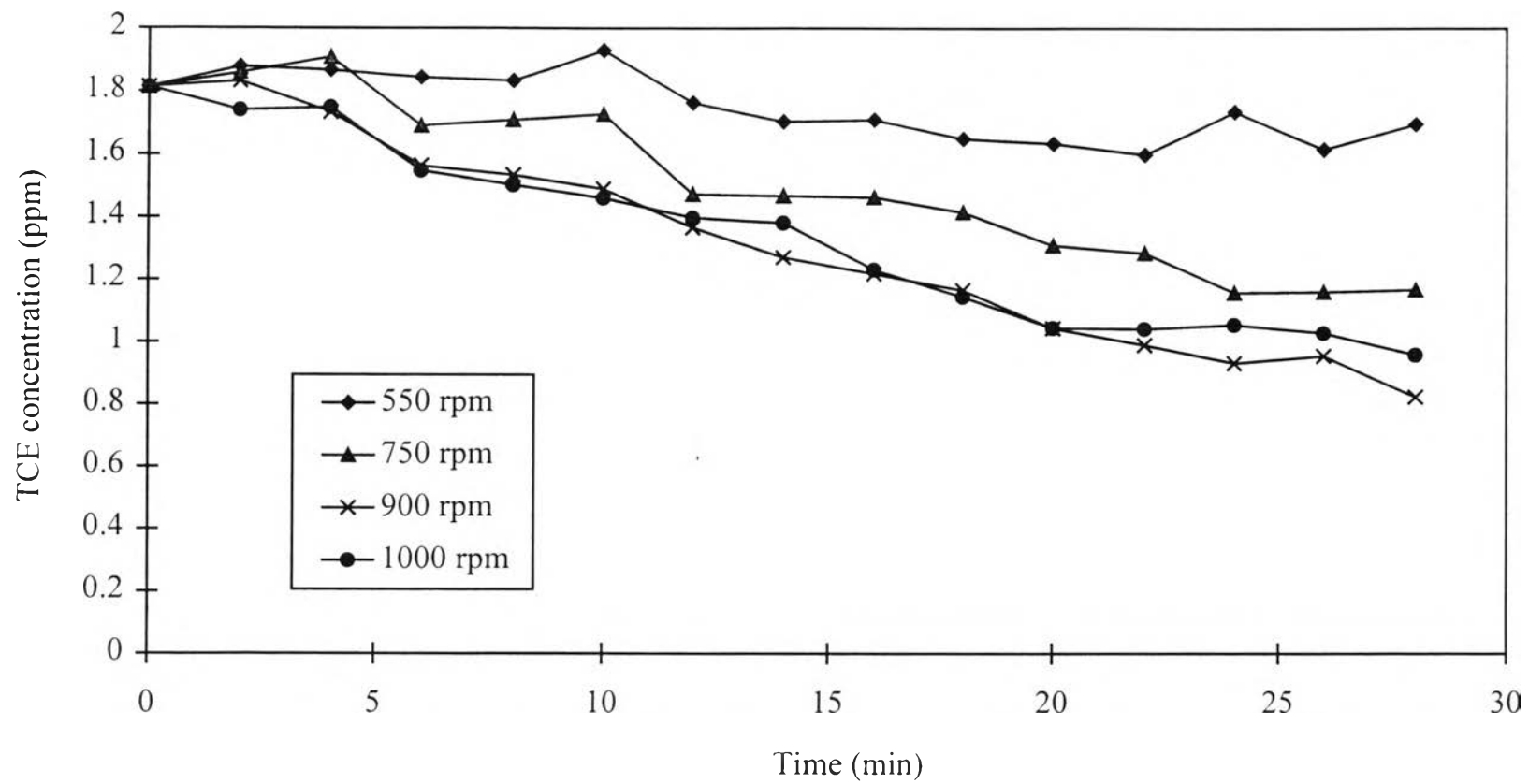


Figure 4.4 Effect of stirring speed on TCE concentration.

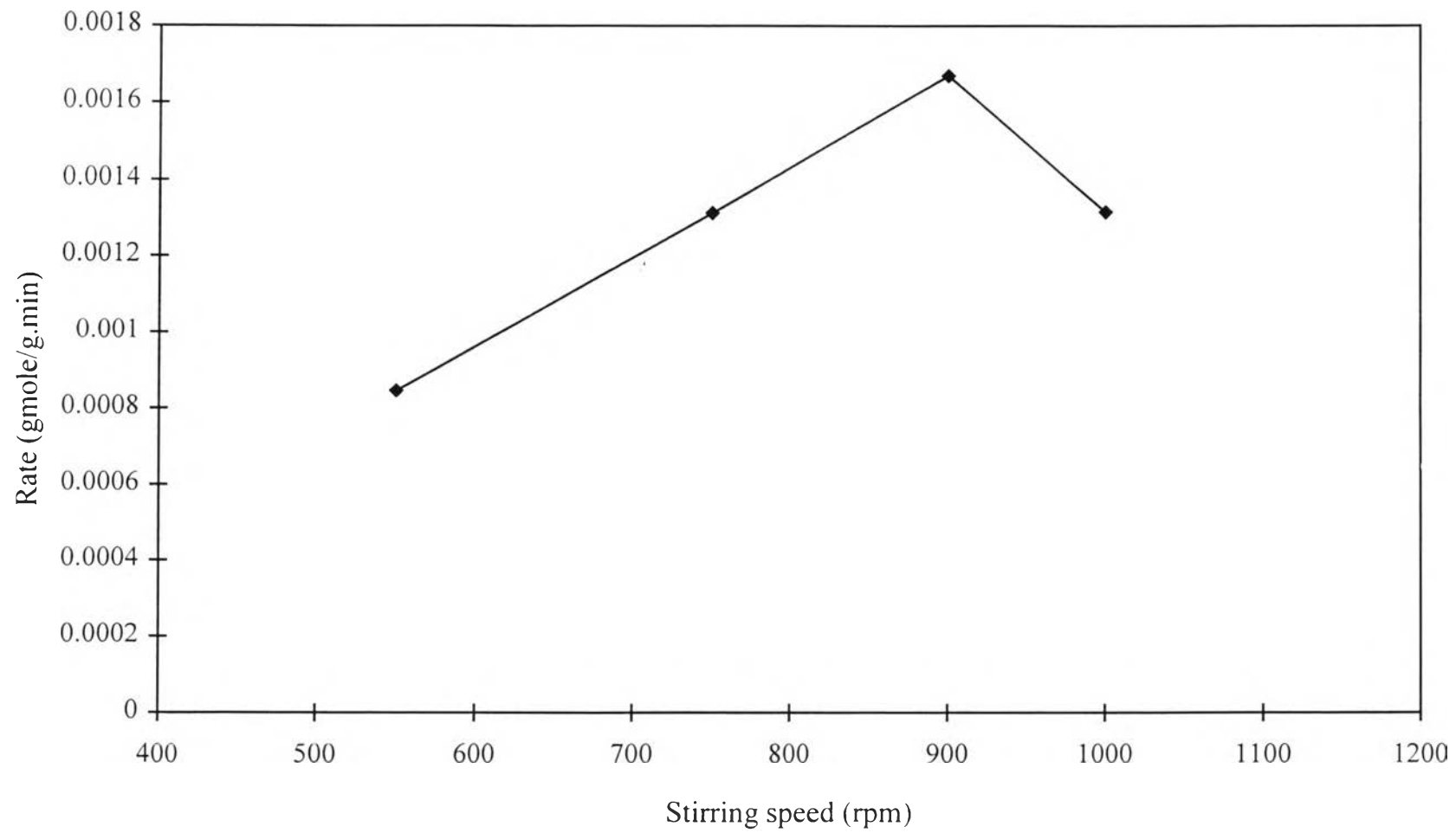


Figure 4.5 External limitation tests.

limitation by plotting the relationship between the reaction rate versus stirring speed. The result showed that the external resistance was negligible at stirring speed greater than 900 rpm. From these results, a stirring speed at 900 rpm was chosen for all kinetic data runs.

4.5.2 Reaction order

The reaction order of ozone oxidation in the presence of catalyst can be described by using the equation 2.7 as already described in chapter II.

$$\frac{1}{W} * \ln\left(\frac{[M]}{[M]_0}\right) = -k * t$$

The rate constant can be determined by the slope of the relationship between $(1/w)*\ln [M/M_0]$ and time. It was found that the reaction rate equation was the first order with respect to the TCE concentration over the entire condition studied. The overall apparent pseudo first order rate constant was $0.002 \text{ (g.min)}^{-1}$. This is in agreement with Bellamy et al. (1995) and Glaze et al. (1995), their results also showed the same reaction order with respect to TCE. However, the overall apparent pseudo first order rate constant was different. The problem may arise from the different experimental system used. Their works used the $\text{H}_2\text{O}_2/\text{O}_3$ to destroy TCE.

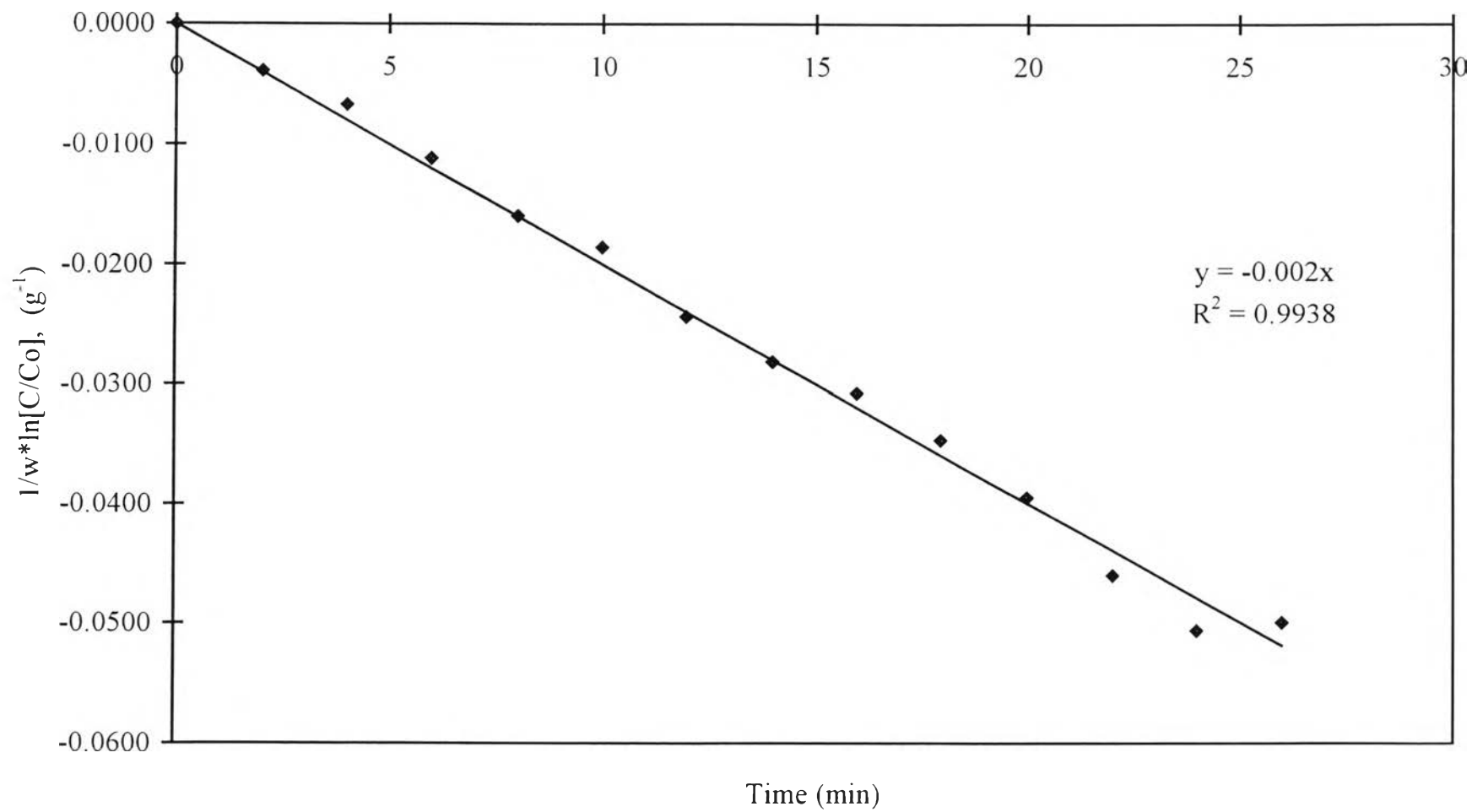


Figure 4.6 TCE reaction order.