CHAPTER IV

EXPERIMENTAL

4.1 Apparatus

Standard tank configuration is used. It's diameter is 25 cm. The vessel is equipped with four symmetrically located baffle of width T/10. The impeller used is standard six-blade turbine positioned in the vessel axis, at the center between bottom and top of the still liquid, see Figure 4.1

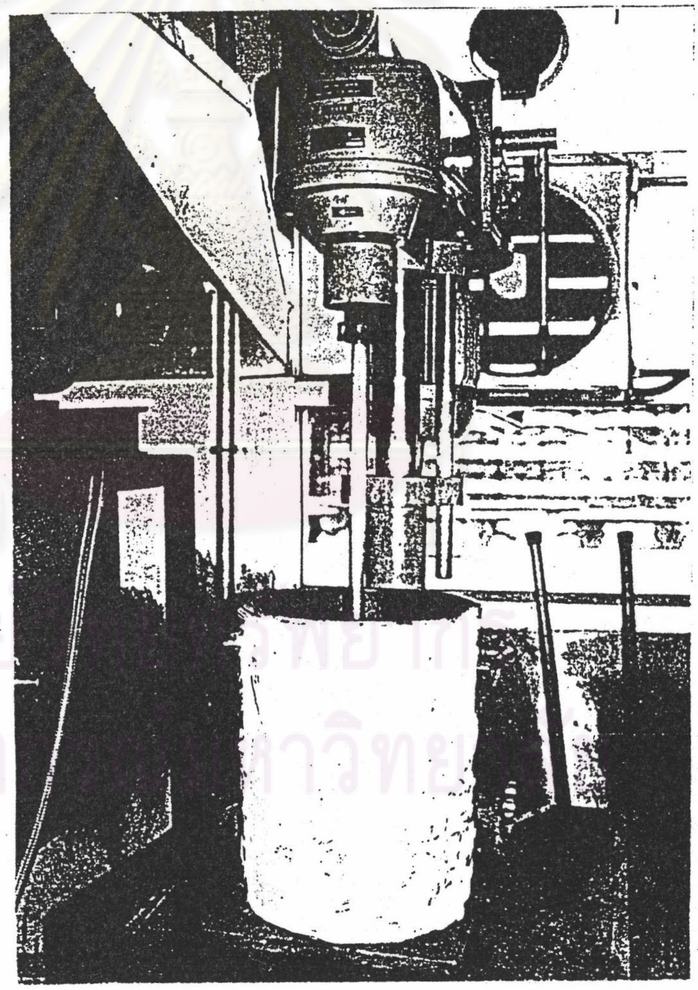


Figure 4.1 :Standard 6-bladed turbine and vessel.

4.2 Instruments

The following instruments were used to determine various parameters that influence on solid dissolution of styrene-butadiene in mineral oil.

- FT-IR spectrophotometer
- Viscosity bath
- Viscometer
- Thermometer
- Weight Scale
- Pyconometer

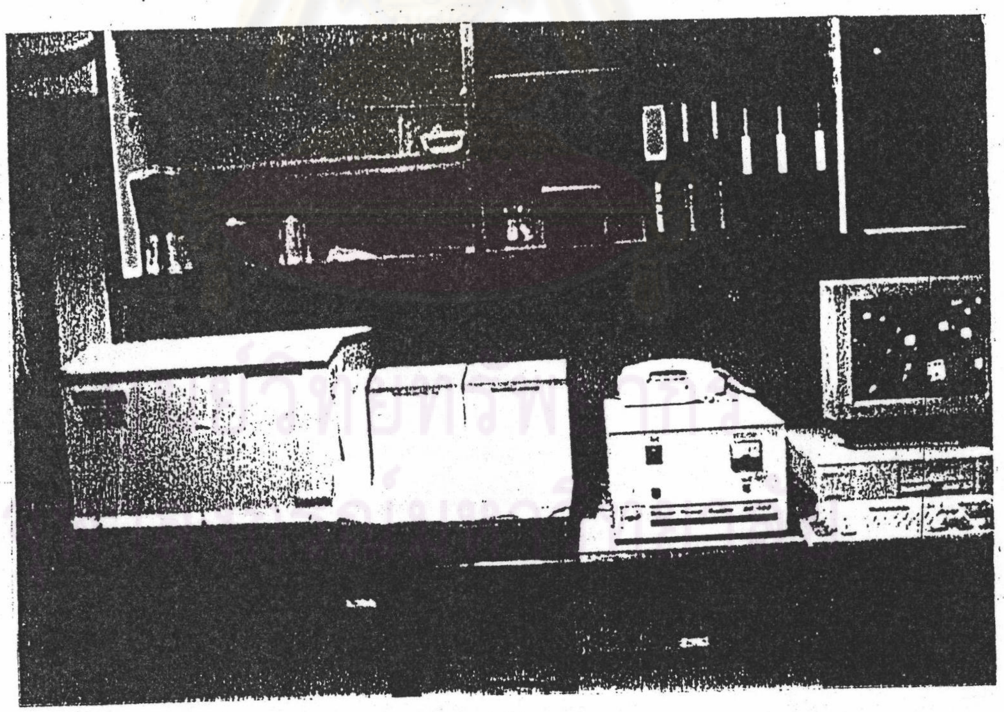
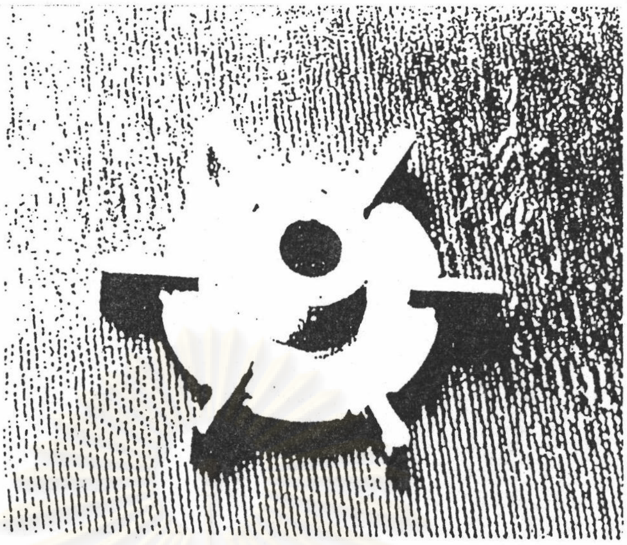
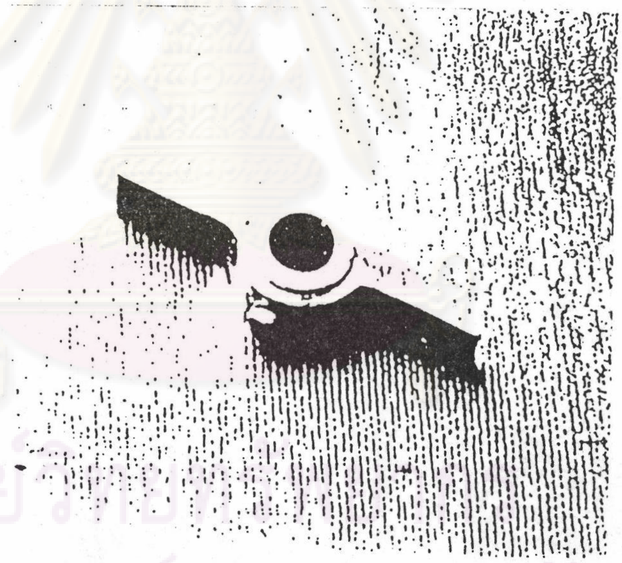


Figure 4.2 : FT-IR spectrophotometer



Standard 6-blade Turbine



Paddle

Figure 4.3 : Impeller Types

4.3 Materials

The following materials were used in the experiments.
-Mineral Oil .... Shell Co. (Thailand) Ltd.
-Styrene-Butadiene copolymer............ Shell Co. (Thailand) Ltd.

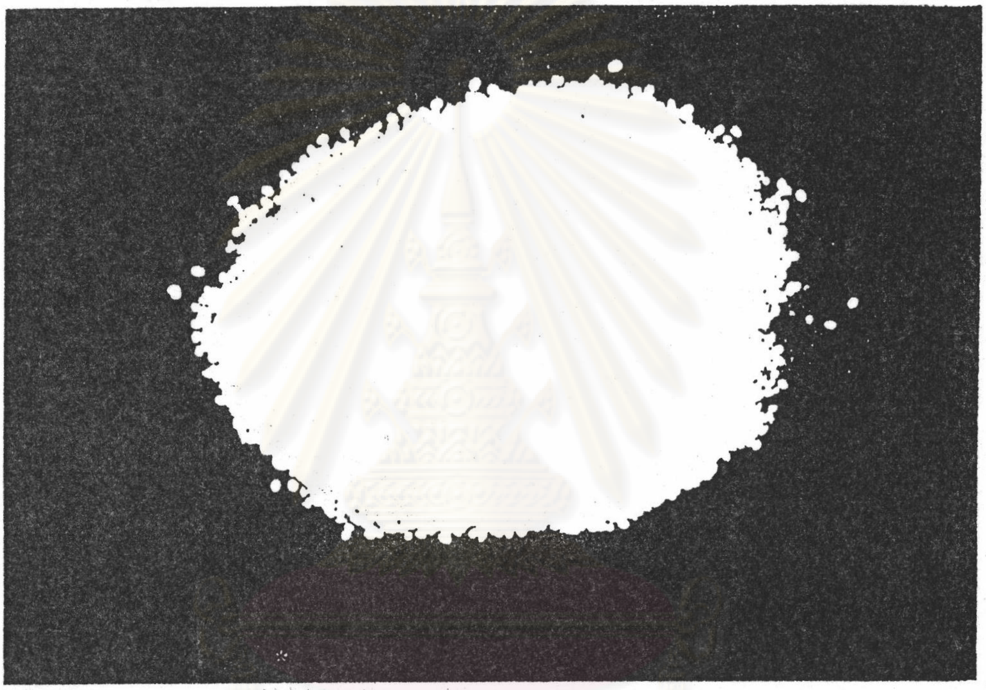


Figure 4.4 Polystyrene-Butadiene solid particles.

4.4 FT-IR spectrophotometer Analysis of Styrene-Butadiene Concentration

FT-IR Model System2000 is used to detect styrene-butadiene concentration at a wave length of 700 cm^{-1} The saturated concentration, (C_s) of definite temperature is checked by spectrophotometer before starting each experiment At the end of each experiment, the concentration (C_f) solution in the lower compartment is again examined by the same device. The calibrated data of various concentrations versus percent transmittance is shown in Figure B1. The concentration at different temperatures ranging between 95°C and 140°C can be read out from the area under two curves (shown in Appendix B).

4.5 Determination of Solid Dissolution Rate Coefficient [21]

The determination of dissolution rate coefficient concerns the measuring of concentrations before and after diffusion.

Rate of solution of solid particles in this study is calculated by using Hixson and Crowell Eq. (2.2)

The particle diameter d_p is defined by the following sphere diameter of equivalent volume.

$$d_p = [6/\pi(W/n\rho_s)]^{2/3} \qquad \text{......................(4.2)}$$

Also we shall use the shape factor defined by the following equation(4.3) which is equal to π for sheres.

$$\alpha_w = A/\sum nd_p^2 \qquad \text{.....................(4.3)}$$

$$A = n\pi d_p^2 \qquad \text{.....................(4.4)}$$

4.6 Determination of Diffusion Coefficient in Liquid Phase.[11]

The determination of Diffusion Coeffieient will be estimated by methods of Stokes-Einstein's equation.

The assumtion of this determination, the particle diameter, d_p, is defined by the following sphere diameter of equivalent volume. In the Stokes-Einstein's equation for spherical molecule and solvent viscosity

$$D_{AB} = \kappa T/6\pi r_A\mu_B \qquad \text{.................................(4.5)}$$

$$dp = \sqrt[3]{6/\pi(W/\rho_s n)} \qquad \text{.................................(4.6)}$$

$$r_A = 1/2 \sqrt[3]{6/\pi(W/n\rho_s)} \text{.............................(4.7)}$$

Then,

$$D_{AB} = \frac{(\kappa T/\mu_B)}{3\pi \sqrt[3]{6/\pi(W/n\rho_s)}} \quad \text{.................................(4.8)}$$

where :

κ	=	Boltzmamn Constant = 1.38 x 10^{-16} ergs/K.
r_A	=	Solute particle radius [cm]
μ_B	=	Solvent viscosity [cSt]
T	=	Temperature [K]
W	=	Mass of solid particles [g]
n	=	Total number of solid particles
ρ_S	=	Density of solid particless [g/cm^3]

4.7 Experimental Procedure

The following of experimental procedure was carried out for each run. The vessel was filled with mineral oil to a level equal to its diameter. The temperature was measured and adjusted to the value required. The temperature never varied more than 1°C during any one of the experiments performed. Set the rotational speed of the impeller to the proper setting and measure the speed using the digital tachn
ometer mounted at the top of the drive shaft, see fig. 4.4 Fluctuations in rotational speed was no greater than 1 to 2 rpm. at any set speed. The lower limit of impeller speed corresponded to solid suspension, i.e. no particles were weighed and introduced into the vessel and the stop watch was started simultaneously. Samples were collected at five minutes interval and the percent weight of solid were determined using FT-IR spectrophotometer. The range of rotational speed studied was from 350 to 550 rpm., and the temperature range from 115°C to 135°C. At least 180 such runs were performed.

The densities of solid particles were determined, see Appendix A. Solubilities of styrene-butaidene in mineral oil vs temperature are shown in Appendix B. Viscosity and density of mineral oil vs temperature are shown in Appendix C and D respectively. Viscosity of 2wt%, 3wt% and 4wt% of styrene-butadiene in mineral oil vs temperature are shown in Appendix E. Density of 2wt%, 3wt% and 4wt% of styrene-butadiene at various temperatures were determined by a using pyconometer and the values are shown in Appendix F. The diffusion coefficient(D_v) of styrene-butadiene in mineral oil was estimated by methods of Stokes-Einstein's equation (4.8) are shown in Appendix G.

The solid dissolution coefficient *(K)* was determined by using method presented by Hixson and Crowell [21] equation (4.1). For a dissolution process, the driving potential is $C = C_s\text{-}C$. The mass needed to satuarate the liquid,W_s is a function of the temperature only, see Appendix B, the concentration, C was calculated from the mass dissolved, m (measured by FT-IR spectrophometer and converted by calibration curve) and the liquid volume. Diffusivity of samples vs temperature were obtained from calculation, see Appendix G. To calculate the dimensionless number, Sh_T, Re_a and Sc, see Appendix M. Appendix O was curve fitting for relationship between Sherwood number and Reynolds number or Schmidt number. And Appendix P was the determination of the constant r in the correlations.

Weighings were made with a satorius automatic balance having an accuracy of $\pm$ 0.0001 g.

4.8 Determination of Solubility

Accurate values of satuaration concentration (solubility) are essentail for a proper calculation of mass diffusion. No previous work has been reported for solubilities of styrene-butadiene in mineral oil at any other temperatures. Therefore solubilities at different temperatures between 95°C and 140°C were determined in this work. The experiment procedure is described below

100 ml of mineral oil was added into the agitated vessel which was installed with agitator and magnetic hot plate. The magnetic hot plate is switched on an set to the desire temperature. A few solid-particles of styrene-butadiene of the known weight were added into the agitated vessel. The temperature of the solution in the vessel was double-check by a thermometer. Styrene-butadiene grains were added into the solution till saturation was attained and checked by FT-IR spectrophotometer until the concentration of the solution is constant.

Styrene-butadiene weight at saturation concentration at that certain temperature was measured by FT-IR spectrophotometer (percent trasmittance and converted to percent weight by calibration curve) Three data were recommended for each single temperature point. The same procedure was repeated for other temperatures ranging between 95°C and 140°C

The solubility data and curve of styrene-butadiene in mineral oil at temperatures between 95°C and 140°C were presented in Appendix B

4.9 Dimensional Analysis of Dissolution Rate Correlation

In solid liquid agitation the entire mass transfer relation can be expressed by five independent variables as

$$K = f(D_i, \rho, \mu, D_v, w)$$

In agitated solid liquid systems, the appropriate equation is,

$$KT/D_v = f(D_i^2 w\rho/\mu, \ \mu/\rho D_v)$$
$$Sh_T = f(Re_a, Sc)$$

where

Sh_T is the Sherwood number referred to agitated tank ,KT/D_v

Re_a is the Reynolds number referred to agitator $D_i^2 w\rho/\mu$

Sc is the Schmidt number, $\mu/\rho D_v$

In this work the result will be correlated in terms of tank Sherwood number as a function of agitator Reynolds number and Schmidt number.

Table 4.1 Dimension of Apparatus

Types of agitator	Standard 6-blade disc turbine	Paddle
Impeller diameter (cm)	8.33	8.33
Impeller height from the tank bottom (cm)	8.33	8.33
Impeller blade width (cm)	1.67	1.67
Impeller blade length (cm)	2.08	2.08
Number of blades	6	2
Angle of blade	90°	90°
Tank diameter (cm)	25	25
Liquid height (cm)	25	25
Number of baffles	4	4