CHAPTER II

MATERIALS AND EQUIPMENT

2.1 Materials

All chemicals and solvents used in these experiments are listed in Table 2.1.

Table 2.1 List of chemicals and solvents

Name	MW	bp(or mp), °C	Source
1. Natural rubber latex concentrated	~10^6	-	Local
2. Teric 16A16	-	-	ICI
3. Phenylhydrazine	212.25	314	MERCK
4. 2-Ethoxyethanol	90.12	134-137	FLUKA AG
5. Methacrylic acid	86.09	163	FLUKA AG
6. Acetic acid	60.05	118	FLUKA AG
7. Acetone	58.08	56.3	Dist.
8. Benzene	78.12	80.1	Dist.

Table 2.1 (continued)

Name	MW	bp(or mp), °C	Source
9. Benzophenone	182.22	46-49	FLUKA AG
10. Carbon tetrachloride	153.82	76.8	Dist
11. Chloroform	119.38	61.2	Dist
12. Copper acetate monohydrate	199.63	115	FLUKA AG
13. Copper acetylacetonate	261.76	>300 (F)	FLUKA AG
14. Copper bromide	223.37	498	FLUKA AG
15. Anhydrous copper chloride	134.45	630	FLUKA AG
16. Anhydrous copper sulphate	159.61	3560 (decomp.)	FLUKA AG
17. 2,6-di-*tert*-butyl-p-cresol	220.36	183-186(F)	FLUKA AG
18. N,N-dimethyl aniline	121.18	192-194	FLUKA AG
19. Hexane	86.17	68.7	Dist
20. Methanol	32.04	64.7	Dist
21. Phenothiazine	199.28	69-71(F)	FLUKA AG
22. Phosphorus pentoxide	141.96	340	FLUKA AG

Table 2.1 (continued)

Name	MW	bp(or mp), °C	Source
23. Sodium bicarbonate	84.0	Lost CO_2 at 50°C	BDH
24. Sodium hydroxide	40.01	318.4	EKA
25. Anhydrous sodium sulphate	142.04	884.0	CARLOERBA
26. Sodium wire	22.99	881.4	FLUKA AG
27. Sulfuric acid conc	98.08	10.5	BDH
28. Tetrahydrofuran	72.10	66.0	Dist
29. Toluene	92.15	110.6	Dist

2.2 Equipment

All equipment used in these experiments is listed in Table 2.2.

Table 2.2 List of equipment

Equipment	Company
1. ^{1}H NMR spectrometer EM-360, 60 MHz.	VARIAN, U.S.A.
2. IR spectrophotometer A-302	JASCO, JAPAN
3. Vacuum oven	TOWNSON & MERCER LTD., ENGLAND
4. Vacuum pump	EDWARDS HIGH VACUUM, ENGLAND
5. Rotary evaporator	BUCHI, SWITZERLAND
6. Cooler, eyela cool ace	TOKYO RIKAKIKAI CO. LTD., JAPAN
7. Aspirator A-25, eyela	TOKYO RIKAKIKAI CO. LTD., JAPAN
8. Mechanical stirrer	COMTEX, U.S.A.

Table 2.2 (continued)

Equipment	Company
9. Air compressor	HITACHI CO.LTD.,JAPAN
10. Electronic precision balance Model 2462	SANTORIUS, W. GERMANY
11. Top loading balance Model P 1200	METTLE, SWITZERLAND
12. Centrifuge	MSE, ENGLAND
13. Water bath	B. BRAUN, W. GERMANY
14. Digital tachometer DT-240	DIGICON, U.S.A.
15. Viscometer	FISHER, U.S.A.
16. Other laboratory glasswares	

2.3 Purification of monomers and solvents [18]

2.3.1 Monomers

(a) Methacrylic acid

Methacrylic acid was dried overnight with anhydrous sodium sulphate in the presence of phenothiazine and 2,6-di-*tert*-butyl-p-cresol as inhibitors, then filtered and fractionally distilled under reduced pressure to give a colorless liquid (80-84 °C / 20 mm Hg). Methacrylic acid was stored under nitrogen atmosphere with some inhibitors in a refrigerator and redistilled before used.

(b) 2-Ethoxyethanol

2-Ethoxyethanol was dried with anhydrous sodium sulphate, filtered and then fractionally distilled.

2.3.2 Solvents

(a) Benzene

Benzene was purified by consecutive shaking with conc H_2SO_4, water, and diluted NaOH until the solution was basic to litmus; then it was washed again with water, dried with anhydrous sodium sulphate, filtered, and then fractionally distilled.

(b) Toluene

Purification of toluene involved shaking twice with cold conc H_2SO_4 (100 mL of acid per liter) followed by shaking with a water rinse; then the solvent was shaken again with 5 % $NaHCO_3$ or NaOH, and again with water. The solvent was dried with anhydrous sodium sulphate, filtered and dried again with P_2O_5, and then fractionally distilled from P_2O_5.

(c) Tetrahydrofuran

Tetrahydrofuran was purified by refluxing with sodium wire and benzophenone until the blue color was established, then fractionally distilled. This solvent was stored above sodium wire.