CHAPTER 1

INTRODUCTION



1.1 The interest of the membrane emulsification

The requirement of using polymeric microspheres of controlled size with a narrow size distribution has grown steadily since it can be used in scientific and industrial applications as chromatographic packing materials, dry toner in xerography, support for medicine and so on. Aimed at xerographic technology, dry xerographic toners consist of a coloring material and other additives as charge control agent supported on a polymeric material. The polymeric microsphere is an important component for the process of electrophotography. For realizing fine images and high resolution in processes, the polymeric materials are being improved. In particular, a technology for employing fine toner particle in terms of particle size is singled out as a powerful method for enhanced image quality. The size of currently used toner particles generally ranges from 7 to 10 µm [1]. Usually, these particles were produced by melt-mixing and crushing method, it is difficult to synthesize particles having a narrow particle size distribution and the incorporating charging agent and other additives to be contained in each particle. The large particles can produce ragged line, dot, and may give incomplete melt that simply splits apart from an image. As a result, a smaller size has been found to be superior for color reproduction, and for noise reduction, but requires a longer grinding time in manufacturing by crushing method, hence giving an expensive product. Also, smaller sizes tend to produce more dirt corresponding to rapidly degradation. Thus, uniformity on the particle size of dry toner is one of the attractive features.

The rheology of toner is the other consideration, concerned with the temperature in which the image is fixed to paper. Commercially, toner has the glass transition temperature (Tg) between 50 to 60°C [2]. Varying the component of polymer and additives can modify control of the fusion properties. Furthermore, the other aspect is its triboelectric properties, i.e., electrostatic capacity. It is considered as the particle surface that an amount of functional groups or additives are concerned. Therefore, the performance depends on the selections of the polymerization method.

Nakashima et al. [3,4] fabricated a microporous glass membrane called an SPG (Shirasu Porous Glass) membrane, with high uniformly controlled pores with a narrow size distribution. The tendency of using SPG membrane emulsification is increasing. Such a membrane emulsification technique, which can be used with a wide range of polymers, is an important alternative. A simple and easy procedure using two immiscible liquids, one termed as a disperse phase was permeated through the membrane into the other liquid termed as a continuous phase. The stabilization of emulsion droplets was controlled by using surfactants and stabilizers. The SPG emulsification was followed by suspension polymerization; the uniform microspheres with sizes ranging from 1 µm up to 100 µm were obtained.

Referred to the previous work in SPG emulsification, Omi et al. [5] synthesized polystyrene and crosslinked polystyrene particles by varying the inert solvent. The formation of micropores in polystyrene particles was adjustable through the solubility of solvent based on the phenomena that voids inside particles can be controlled entirely by good or poor solvents. Poly(methyl methacrylate) particles were also prepared. Applications of crosslinked microspheres for gel permeation chromatography and as carriers for enzyme immobilizations were demonstrated [5,6,7,8].

The objective of this research work is the production of uniform microspheres with a narrow size distribution of poly(styrene-co-methyl methacrylate); poly(styrene-co-methyl methacrylate-co-n-butyl methacrylate), poly(styrene-co-methyl methacrylate), poly(styrene-co-n-butyl methacrylate), and poly(styrene-co-2-ethylhexyl methacrylate). The copolymers and terpolymers, which can be a main component of dry toner were synthesized. The polymers produced will be further modified to be use as a dry toner for other applications.

1.2 Objectives of the research work

The objectives of this work fall into the following categories:

1.) To synthesize the super-fine particles of narrow size distribution of poly(styrene-co-methyl methacrylate) by an SPG emulsification.

- 2.) To study reaction parameters affecting the particle size and size distribution of poly(styrene-co-methyl methacrylate).
- 3.) To characterize the properties of the copolymer produced.

1.3 Scope of the research work

Particle morphology affected by the structure of polymer particles was studied. This is done by varying the copolymer composition, crosslinking agent and hydrophobic additives. In summary, the factors investigated in this research work are as follows:

- (1) Effect of crosslinking agent on the structure of copolymer
- 2) Effect of hydrophobic additives on particle morphology, particle size, size distribution and average molecular weights.
- 3) Effect of different pore sizes of SPG membrane on particle morphology, particle size, size distribution and average molecular weights.
- 4) Effect of initiator on particle morphology, particle size, size distribution, average molecular weights and kinetic of polymer conformation.
- 5) Effect of monomer on glass transition temperature of polymer on particle morphology, particle size, size distribution and average molecular weights.

Instrumental techniques employed to characterize the copolymer obtained from each experiment are the following:

- 1) Monomer droplets is observed and the diameter of which calculated by an Optical microscope (OM)
- 2) External morphologies and the diameter of particles are measured by Scanning Electron Microscopy (SEM)
- 3) Average molecular weights are studied by Gel Permeation Chromatography (GPC) procedure
- 4) Copolymer composition is determined by Nuclear Magnetic Resonance Spectroscopy (NMR) method
- 5) Thermal properties of the copolymer are done by Differential Scanning Calorimetry (DSC) method
 - 6) Functional group in copolymer is investigated by Fourier Transform Infrared Spectroscopy (FT-IR)