CHAPTER I

INTRODUCTION

1.1 The Interest of the Dispersion Polymerization

Many scientific and industrial applications require the use of polymeric particles of controlled size with the narrow particle size distribution. These particles can be used as coatings, inks, dry toners, chromatographic packings or supports for medical assays. Preparation of uniform polymer particles in the 1-10 μm size range has received great attention in recent years. Although it has been possible to prepare monodisperse sub micron particles for some time now, the preparation of larger particles has been elusive. Indeed, monodisperse micron-size polymer particles could not be prepared (in reasonable quantities) until Ugelstad et al. (1) developed a method of growing sub micron particles using a two-step swelling technique. There is sufficient interest in such particles that Vanderhoff and co-workers (2) have been studying the growth of polymer particles to the micron-size range aboard the space shuttle in microgravity. However, both approaches are rather tedious and time-consuming.

Dispersion polymerization was defined as the process for the preparation of polymer particles in the range between suspension and emulsion; namely, the approximate 10 μm particle size (3). In most cases the considerable amount of emulsion polymerization that takes place concurrently yields submicron particles and high molecular weights (4). Unlike the emulsion and suspension processes, in dispersion polymerization the monomer is completely miscible with the medium, the mixture starts as a homogeneous solution and the resulting polymer is insoluble under the same condition and then precipitates as spherical particles, stabilized by a steric barrier of dissolved polymer (5). The mechanism of these processes is of considerable interest, particularly with respect to the formation of monodisperse and polydisperse particle, and in the control of particle size.

Referring to the recent research work of Prof. N. Ogata et al. (6) at the Sophia University, synthesis of polystyrene (PS) super-fine particles by controlling the particle diameter through the solubilities of the matrix polymer in good and poor solvents based on the phenomenon that the free space changes depending entirely on the morphologies of matrix polymer solution. The smallest particle resulted from this research work was the polymer with a particle size of 0.64 μm. The process used in his research was the radical dispersion polymerization of styrene (St) in the solution of poly(methyl vinyl ether) (PMVE) as a matrix polymer, which was dissolved in 80:20 ethanol/n-hexane mixed solvent by weight%.

One of the wide applications of microsphere particle is the use in dry toner technology. Toner is one kind of inks (7), generally of low-melting temperature compounded with approximately 10% carbon black as colorant and other additives that is ground to a particle size of about 5-12 μm in diameter. The large toner particle sizes usually produce ragged lines and dots and thus degrade copy quality. As a result, smaller toner particle sizes have been found to be superior for noise reduction in general. However, for a given resin the smaller toner sizes require longer grinding times in manufacturing, thus, induce more expensive in production and more rapid developer degradation.

The objective of this research is, therefore, to produce the smaller particle-sized polymer. At the first stage, the binder resin which is the main component of toner was synthesized. The polymer, the target of this research, is the poly(styrene-co-methyl methacrylate).

Poly(styrene-co-methyl methacrylate) was synthesized by using the dispersion polymerization in the presence of PMVE as a matrix polymer. The reaction was carried out in the mixed solvent of 80:20 weight% ethanol/n-hexane. The copolymers produced will be further modified to be used as a dry toner for the other project.

1.2 Objectives of the Research Work

The objectives of this work fall into the following categories:

1) To synthesize the super-fine poly(styrene-co-methyl methacrylate) by dispersion polymerization.

2) To examine the effect of PMVE on particle size

3) To characterize the properties of the copolymers produced.

1.3 Scope of the Research Work

The effect of PMVE on particle formation of dispersion copolymer is the first parameter studied. This is done by comparing the copolymer composition of dispersion copolymerization with those of bulk and solution copolymerizations. The only difference of these three reactions is that the bulk and solution copolymerizations did not contain the PMVE matrix polymer. The effects of important parameters inclusive of comonomer feeds, mixed solvent contents, and reaction temperatures are studied. In summary, the topics investigated in this research work are as follows:

1) Effect of the matrix polymer on the particles formation.

2) Effect of the styrene feed on the particle size, size distribution, thermal and surface properties of the copolymer.

3) Effect of the solvent concentration of ethanol on the particle size, size distribution and average molecular weights.

4) Effect of the reaction temperature on the particle size, size distribution and average molecular weights.

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

1) Copolymer composition is elucidated by Fourier-Transform Infrared Spectroscopy (FT-IR), Nuclear Magnetic Resonance Spectroscopy (NMR) and Elemental Analysis (EA) methods.

2) Copolymer particle size is measured by Scanning Electron Microscopy (SEM) method.

3) Average molecular weights are studied by Gel Permeation Chromatography (GPC) procedure.

4) Thermal properties of the copolymer are done be DSC/DTA techniques.

In addition, X-ray Photoelectron Spectroscopy (XPS) or Electron Scanning Chemical Analysis (ESCA) is also utilized for investigatation of the surface property of the copolymers.