

CHAPTER I

INTRODUCTION

1.1 The Interest in Monosized Polymeric Microbeads

Monosized polymeric microbeads are used in a wide variety of application including instrument calibration standards, standards for the determination of pore size and the efficiency of filters, column packing material for chromatographic separation, inks, coatings, ion-exchangers, support materials for biochemicals, dry toners, medical diagnostic tests, microelectronics, and so on (1). The level of success in each of these applications ultimately depends upon the particle size and size distribution, the morphology of the particles and surface properties. For ultrafine toner particles, control of the glass transition temperature and surface charging of the final copolymer beads may be particularly important (2). Different surface groups of monosized polymeric microbeads is an importance of success in diverse biomedical applications.

Control of particle size and its uniformity has been a major area of interest. It proves to be particularly challenging to prepare monodisperse polymeric particles in the micronized range (1-10 μm). Several techniques for the preparation of monosized polymeric particles have been developed. Traditionally, micronized particles have been prepared by suspension polymerization. This technique is usually used for the production of spherical polymeric particles between about 50-1000 μm . A wide particle size distribution is usually observed because of the inherent size distribution in the mechanical homogenization step. The "two-stage swelling method" developed by

Ugelstad et al. (3) used the first successive seeding method to obtain monodisperse polymeric particles of micronsize usually in the range of 0.2-1.5 μm . Micron-sized particles as large as 50 μm may be obtained by a multistep process. But this process is very tedious and typically requires several reaction steps.

Monosized polymeric microbeads have also been prepared by dispersion polymerization technique (4). A basic difference between dispersion polymerization and emulsion polymerization is that a hydrocarbon or alcohol medium, instead of water, is normally used in dispersion polymerization, and more characteristically, the monomers, initiators, and matrix polymer have to be soluble in a medium that does not dissolve the resultant polymer. The first studies of this technique were carried out in non-polar organic hydrocarbon solvents. This technique has recently received much attention for producing monosized polymeric particles in the micron range, because of the simplicity of the process and the wide variety of monomers which can be polymerized into polymer particles by this process.

The interest in monosized polymeric microbeads for use as dry toners has led to detailed investigation of the dispersion copolymerization of styrene and methyl methacrylate in polar solvents as a method for the formation of polymeric microbeads. Dry xerographic toners consist of a colorant in a binder resin (5). Beyond these essential ingredients, a particular toner design may contain charge control additives to control the charge level, surface additives to control flow and cleaning properties, magnetic additives to aid in toner control, and waxes to promote toner release from the fuser roll. Several different families of resins have found frequent application in xerography depending on the fixing technique selected. The role of the resin in a toner

is to bind the pigment to the paper or a transparency material to form a permanent image. This is typically done by selecting a polymer that will melt at a reasonable temperature when heat is applied in any of a number of ways or one that can be used into the paper fibers at high pressure without additional heat. The development of a toner involves the choice of the components described above, starting with the choice of polymer and proceeding through the selection of each component to fit the particular application. Toner particle sizes are generally in the range of about 7 to 12 μm in diameter. Particle sizes significantly larger than this usually produce ragged lines and dots and thus degrade copy quality. As a result, smaller sizes have been found to be superior for color reproduction and for noise reduction in general. However, for a given resin the smaller sizes require longer grinding times in manufacturing, hence are more expensive to produce. Even if the average size is reasonable, a broad particle size distribution will introduce significant amounts of the small and large toner particles that cause dirt and copy quality problems. As a result, toner processing strives for the narrowest particle size distribution. This is typically done with well-designed micronization equipment followed by air classification and possibly sieving, however narrow particle size distribution of resin is also of importance.

As mentioned above, the objective of this research work is therefore to produce poly(styrene-*co*-methyl methacrylate) particles ranging from submicron to micron size with narrow size distribution by dispersion polymerization in polar solvents.

1.2 Objectives of the Research Work

The objectives of this research work are as follows:

a) To synthesize the super-fine poly(styrene-*co*-methyl methacrylate) particles by dispersion polymerization in an organic polar media.

b) To investigate various polymerization parameters, which affect the particle size and size distribution, average molecular weights, copolymer composition, and thermal properties of poly(styrene-*co*-methyl methacrylate) particles.

c) To prepare poly(styrene-*co*-methyl methacrylate) microbeads with a narrow size distribution.

d) To characterize poly(styrene-*co*-methyl methacrylate) particles prepared by dispersion polymerization by conventional techniques.

1.3 Scope and Workplan of the Research Work

This research work involves the synthesis and characterization of superfine particles of poly(styrene-*co*-methyl methacrylate) based on the free volume in polymer solution and solubility parameter concepts.

The work planned to be carried out for this research is focused on the topics investigated as follows:

a) The influence of the matrix polymer concentration on the particle size, size distribution and average molecular weights.

b) The influence of the ethanol/water ratio on the particle size, size distribution and average molecular weights.

c) The influence of the reaction temperature on the particle size, size distribution, and average molecular weights.

d) The influence of the reaction time on the particle size, size distribution, average molecular weights and conversion.

e) The influence of the agitation rate on the particle size, size distribution and average molecular weights.

f) The influence of the styrene feed on the particle size and size distribution, average molecular weights, copolymer composition.

g) Determination of the conversion.

h) Investigation of the particle growth.

The resulting poly(styrene-*co*-methyl methacrylate) particles are characterized for the following properties.

a) The copolymer compositions are studied by Nuclear Magnetic Resonance Spectroscopy (NMR), Elemental Analysis (EA), and Fourier-Transform Infrared Spectroscopy (FT-IR).

b) The average particle size and size distribution of poly(styrene-*co*-methyl methacrylate) microbeads are evaluated by Scanning Electron Microscopy (SEM).

c) The average molecular weights and molecular weight distribution are measured by Gel Permeation Chromatography (GPC).

d) Thermal properties of poly(styrene-*co*-methyl methacrylate) are studied by Differential Scanning Calorimetry (DSC).