

# Original Research Paper

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## Synthesis of Monodisperse Hydrophobic Silicone Particles in the Submicron Size

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### Abstract

In the electronics field, monodisperse hydrophobic particles in the submicron size are used as additives for adhesives. The purpose of this study is to develop a process to synthesize monodispersed poly(alkoxysilane) (silicone) particles that have strong hydrophobicity. Dimethyldimethoxysilane (DMDMS), methyltrimethoxysilane (MTMOS), and tetraalkoxysilanes, such as tetramethoxysilane (TMOS) and tetraethoxysilane (TEOS), were used as monomers for synthesizing the hydrophobic silicone particles. By optimizing the alkoxysilane concentration, the blending ratio of monomers, the amount of catalyst and the reaction temperature, it is possible to synthesize small monodispersed silicone particles.

**Key-words:** Silicone particle, Monodisperse, Dimethyldimethoxysilane, Methyltrimethoxysilane, Tetramethoxysilane

## 1. Introduction

Synthesis of monodisperse silica has attracted considerable attention because of their importance in technological applications in the fields of optical devices, catalysts, filler for polymers, and so on. The sol-gel process is an excellent process to synthesize hydrophilic monodisperse silica<sup>1-5</sup>. Especially the Stöber process of forming silica particles has been utilized in many investigations for preparation of narrowly dispersed silica particles<sup>6</sup>. However, the sol-gel process has rarely been exploited in more hydrophobic media. On the other hand, silica particles are used as additives for adhesive, which is a part of the electronic materials with high heat resistance and excellent reliability that matches the high requirements of the devices. Since monodispersed silica particles can be easily synthesized, surface treatment is the common way to control the surface hydrophobicity (wettability)<sup>7-11</sup>. However, scale-up production of polymer-grafted nanoparticles is still to be fully achieved. The production requires complicated procedures, such as centrifugation, filtration, and solvent extraction, which result in the consumption of a lot of spent solvent.

Poly(organosiloxane), i.e., silicone, particles are used for additives to impart lubricating property to paints, plastic, rubber, cosmetics, and paper, improving dispersibility and giving light-scattering function. However, the method to control the particle size can rarely be found in the published literature. For the electronic materials, controlling the particle

size is very important. S. H. Jung and his coworkers have reported that they could control the size of silicone particles made from organotrialkoxysilane as monomer<sup>11</sup>. In Japanese industry, the synthesis of monodisperse silicone particles was investigated by many researchers as a matter of necessity. Organotrialkoxysilane is used as monomer to synthesize monodispersed silicone particles. But silicone particles made by organotrialkoxysilane still have weak hydrophobicity<sup>12-17</sup>.

The purpose of this study is to develop a process to synthesize monodisperse silicone particles that have strong hydrophobicity. The methanol wettability was used to check the hydrophobicity of silicone particles. If the methanol wettability is less than 50 %, then the hydrophobicity of silicone particles is considered insufficient because of the residual silanol<sup>18</sup>.

We synthesized methyltrimethoxysilane (MTMOS)/tetramethoxysilane (TMOS) copolymerized polymers, MTMOS/tetraethoxysilane (TEOS) copolymerized polymers, and MTMOS/TMOS/dimethyldimethoxysilane (DMDMS) copolymerized polymers.

## 2. Experimental

### 2.1 Materials and reagents

DMDMS, MTMOS, TEOS and TMOS were obtained from Tokyo Chemical Industry Co., Ltd., Japan. Ammonia solution (28 %) and hydrochloric acid (HCl) from Wako Chemical were used as catalysts without further purification.