

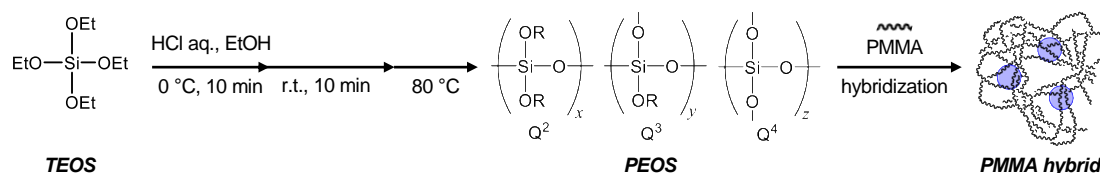
## Preparation of polyethoxysiloxane and their transformation to organic-inorganic hybrid

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The preparation of organic-inorganic hybrids is widely studied which is based on the sol-gel method.<sup>1</sup> The hybrid materials have, however, disadvantages such as low homogeneity and complicated procedure. Recently, we succeeded in the preparation of solid polyethoxysiloxanes (PEOS) with high molecular weight starting from tetraethoxysilane (TEOS). Solid PEOS is soluble to organic solvents and has a melting point at approximately 100 °C. Since solid PEOS is expected to contribute for easy and good dispersion method to prepare organic-inorganic hybrids, we will report that preparation of solid PEOS and their transformation to organic-inorganic hybrids as shown in the scheme.

Solid PEOS was synthesized by the controlled sol-gel reaction of TEOS as a solid with  $M_w = 10,000$  and high  $\text{SiO}_2$  content for 65%. Solid PEOS was soluble to benzene, ethanol, methanol, and THF and insoluble to hexane. Based on the result of  $^{29}\text{Si}\{^1\text{H}\}$  NMR spectrum, PEOS was found to be mainly composed of  $\text{Q}^3$  structure,  $\text{Si}(\text{OSi})_3(\text{OR})$ . The organic-inorganic hybrids composed of poly(methyl methacrylate) (PMMA) and solid PEOS or silica gel (WG) were prepared by simple two methods: (i) the liquid-melt blending method (LMB) and (ii) the solid-melt blending method (SMB). The hybrids by LMB method were obtained as films while those by SMB method were solids or powders. The flatness images on SEM for PMMA-PEOS suggested that homogeneous dispersion state of solid PEOS should be fulfilled in hybrids on a microscale compared to those for PMMA-WG. The thermal stability of the hybrids, excluded PMMA-WG by SMB method, were thermally stable than that of pristine PMMA. Notably, TGA traces of PMMA-PEOS by SMB were varied from these of the hybrids by LMB, specifically,  $T_{d20}$  of PMMA-PEOS-1 and 5wt% by SMB were higher for 10–20 °C compared to that of the hybrids by LMB. Since the PMMA in the hybrids, except for PMMA-PEOS by SMB, were extracted with THF, the hybrids by LMB are classified as the dispersion type and the hybrids by SMB are classified as local interpenetrating type. Well dispersion for organic matrix and easy preparation of the hybrids was achieved using solid PEOS as an inorganic polymer.



1) T. Ogoshi, Y. Chujo, *Composite Interfaces*, **11**, 539–566 (2005).