PROCESS STUDY OF XEROGEL AND AEROGEL FROM SILICON GLYCOLATE BY SUPERCRITICAL FLUID REACTOR

Ph.D. DISSERTATION

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ABSTRACT

This study concerns the preparation of silica aerogel via alcogel by the use of supercritical fluid in order to maintain the inherent microconfiguration of the aerogel.

The alcogel was achieved from the synthetically prepared silicon glycolate by the sol-gel method. The synthesis of silicon glycolate was made possible by the direct reaction of amorphous silica and the organic ligand ethylene glycol together with the strong organic base triethylamine which was used as the promoter catalyst. The operational conditions acting on the yield of the reaction product are the mole amounts of silica, mole amounts of triethylamine and time factor of reaction. In the reaction system ethylene glycol acts as a labile ligand and also as a solvent. Experimental runs were made on one pot synthesis at near boiling point of ethylene glycol.

The optimal yield of silicon glycolate (40.23 %) corresponds to (0.66 mol) of silica and (28.9 m mol) of triethylamine. The time factor for complete reaction to form silicon glycolate was 4 hours. The silicon glycolate served as precursor to produce xerogels and aerogels by the use of phosphoric acid.

Physicochemical properties for silicon glycolates measured were density, boiling point, refractive index, viscosity, solubility and also by UV analysis.

Xerogel with a yield of 85.40% was prepared by the slow evaporation of the alcogel, whereas aerogel with a yield of 82.50% from alcogel was prepared by using supercritical fluid (carbon dioxide) in a fabricated supercritical fluid reactor at 31 °C and with the pressure of 750 psi.

In the case of dry aerogel, the pore size distribution with the pore diameter of 26 nm which may be considered to possess mesopores and relatively few microspores was determined by nitrogen adsorption / desorption analysis. The prepared xerogel and aerogel were characterized by conventional and modern technique such as FT – ir, XRD, SEM and thermal analysis (TG / DTA)