

CHAPTER IV

CONCLUSIONS

An inorganic/organic copolymer was synthesized from silica, catechol and hydroquinone via the “oxide one pot synthesis (OOPS)” process using TETA as catalyst in ethylene glycol solvent. The product is a yellowish fine powder. Although this copolymer is not very soluble in common organic solvents it readily dissolves in both DMSO and hot ethylene glycol. The characteristics of the copolymer depend on the proportion of catechol to hydroquinone in the product and the reaction time.

FT-IR and NMR data confirm the presence of hydroquinone in the structure of synthesized products. For thermal analysis, DSC profiles show endotherms in the temperature range of 280°C to 350°C. TGA characterization exclusively attained under air atmosphere (N₂ and O₂) gave approximately 20 % ceramic yield. The XRD spectra obviously reveals a crystalline pattern in the product which is a ladder structure component. EI⁺-MS spectra also indicate $m/z = 244$ as the base peak which is a fragment of this product.

A short reaction time, 3 h, could be used to synthesize this copolymer at the high ratio of hydroquinone to catechol, however the excess hydroquinone destroys the ladder structure and decreases crystallinity. Furthermore, the influence of TETA catalyst is considerably significant in the synthesis. The product will not form if the amount of TETA used in the reaction is less than 17 %mol silica. Higher amounts of TETA make product isolation more difficult because TETA can form a stable salt complex, triethylenetetraminium tris(catecholato)silicate [TETA-H₂-SiO₆(C₆H₄)₃].

From all the data obtained, the optimal conditions for synthesizing this novel inorganic copolymer can be stated, as follow ; When the amount of silica:catechol:hydroquinone was defined as 1:2-X:X, where the X value should be in the range of 0.2-0.4 mole at 5 h reaction time and the amount of TETA should be in the range of 67% mol Si -100% mol Si.