

## CHAPTER IV

### CONCLUSIONS

The “Oxide One Pot Synthesis (OOPS)” process can be used to synthesize organosilicon copolymers from silica, catechol, hydroquinone, and 4-tert-butylcatechol using TETA as catalyst in ethylene glycol solvent. The product is a pale brown powder, insoluble in most common solvents, but soluble in hot ethylene glycol, DMSO, and partially in methanol. Trace amounts of TETA are found when using high concentrations. The possibility of forming triethylenetetraminium tris(catecholato)silicate exists but it is difficult to isolate this product. The mole ratio of raw materials, the temperature, the reaction time, and the TETA catalyst concentration, influence the characteristics of the product.

FTIR, EI<sup>+</sup>-MS, and NMR data confirmed the presence of comonomer in the structure of synthesized products. EI<sup>+</sup>-MS, XRD spectrum obviously revealed crystalline pattern of the ladder structure component which mainly is bis(1,2-dioxyphenyl)silane polymer and partially obtained amorphous pattern of the comonomer molecules which reduced the remain ladder structure.

For thermal analysis, DSC profiles show endothermic processes at temperatures associated with the cleavage of bonds in product 280°C. TGA thermograms gave mass losses pattern under air atmosphere and more silicon contained when the mole ratio of hydroquinone to 4-tert-butylcatechol is higher.

The influence of TETA concentration is significant in the reaction. At lower amounts of TETA, the product will be found in methanol solution. The main product can be remained and precipitated itself in methanol solvent when using higher amount of TETA.

The initial rate method was used to study the reaction kinetics. The reaction order was first order with respect to silicon dioxide. Activation energy was 163.5 cal/mol. The small activation energy was obtained since this reaction used high amount of TETA catalyst. The dissolution rate of silica was slowly retarded as a function of reaction time because the active silanol groups on the surface of silica decreased.