CHAPTER X
CONCLUSIONS AND RECOMMENDATIONS

Titanium glycolate and titanium triisopropanolamine are successfully synthesized using low cost starting materials, and a much simpler and milder reaction condition. The products show good property in moisture stability. The results from spectroscopy, namely, FT-IR, EA, Solid state NMR, and TGA, confirm the product structure. The transformation from anatase to rutile phases of calcined titanium glycolate indicates the anatase stability up to 900°C. The remarkable stability of the synthesized product provides researchers opportunities for use in many applications. Anatase TiO_2 nanoparticles were successfully prepared by the sol-gel technology, using moisture-stable titanium glycolate as precursor in 1M HCl solution. The calcination temperature and the $HCl:H_2O$ volume ratio have a substantial influence on the surface area, phase transformation and morphology of the products. Anatase titania is produced at calcination temperatures in the range of 600° to 800°C, above which transformation to rutile occurs. Increase of temperature results in anatase of higher crystallinity but lower specific surface area, and induces a morphological change from large irregular agglomerates to more homogeneous particles of spherical shape. From XRD measurements of average grain sizes, we deduce that nucleation rate dominates the kinetics at low temperatures, and growth rate becomes the controlling factor at high temperature and low $HCl:H_2O$ ratios. Increase of $HCl:H_2O$ ratio results in a small but significant decrease in porosity. The highest specific surface area 125 m^2/g is obtained at the lowest $HCl:H_2O$ ratio of 0.28 and the lowest calcination temperature (600°C). From rheological analysis, as evaluated by the Winter criteria, the gelation time increases with increase of $HCl:H_2O$ volume ratio. The fractal dimension determined from the frequency scaling exponent of the modulus at the gel point indicates a denses critical gel structure at low acid ratio. However, the complex viscosity and gel strength increase as a function of acid ratio. We interpret this behavior as indicative that, at low acidity, the gel is composed of poorly hydrated particles forming a dense but weak structure. Increase in acidity increases hydration and cross-link density leading to a more open and stronger gel

network. From the rheological study of different ceria gelling system using HCl:alkoxide molar ratio of 0.8, 0.9, 1.0 and 1.1, as evaluated by Winter et al., the gelation time increases as increasing HCl:alkoxide molar ratio. The gel strength increases as a function of acid ratio and the fractal dimension determined from the frequency scaling exponent of the modulus at the gel point indicate a tight structure at low acid ratio. The TS-1 with highly titanium incorporated in the zeolite framework was synthesized from moisture-stable precursors, silatrane and titanium glycolate, under microwave treatment. The effects of the compositions (TPA^+, NaOH and H_2O) and conditions (aging time, reaction time and reaction temperature) showed that the suitable condition for synthesizing TS-1 was Si:0.1Ti:0.4NaOH:0.3TPA:114H_2O at aging time 110h, 15h reaction time and reaction temperature of 150°C. As for the Si:Ti molar ratio, from XRD, FT-IR, SEM and DR-UV results, it is indicated that high crystallinity and the Ti atoms are occupying in the zeolite framework. Moreover, at higher titanium loading the higher reaction time was an important factor. It is also important to the photocatalytic decomposition of 4-NP. The samples showed high efficiency in PCD of 4-NP and the PCD increased with amount of titanium loading.

The titanium triisopropanolamine precursor was successfully prepared using the same method, and yields a TiO_2 catalyst with high surface area after calcinations of the crude precursor at 600°C for 2h. Polymeric membranes loaded with the as-prepared TiO_2 catalyst show an impressively high efficiency for the photocatalytic degradation of 4-NP. Examination of the properties of three different types of membrane (PAN, CA and PVAc) indicates that the highest stability and the lowest permeate flux are observed with the PAN membrane and the poor stability occurs with the PVAc membrane. The photocatalytic degradation of 4-NP increases with increasing percentage of TiO_2 loaded in the PAN membrane.

From this work, it is recommended that the prepared catalysts should have their catalytic efficiencies tested using waste water generated by the petrochemical industry.