Chapter 5
Conclusion

5.0 Conclusion

In this work, four families of Mo based compound have been obtained through several changes made on the experimental parameters for the precipitation reaction namely $Mo_{36}O_{112}^{4-}$, hexagonal molybdenum oxide, trimolybdate and orthorombic MoO_3. Among the experimental parameters found to have effect on the precipitate crystal structure consists of (i) the type of cation, (ii) solution temperature and (iii) the concentration of acid and molybdenum in the solution. Lithium, being the smallest ion, was found to be insufficient in causing spontaneous precipitation in comparison to the other cations tested.

Temperature was found to have dominant effect in determining the outcome of the crystal structure as compared to the acid and Mo concentration in solution. For ammonium cation, supramolecular phase structure was yielded at 30 °C in all cases independent of acid and molybdenum concentration. Supramolecular and hexagonal phase yielded randomly at 50 °C independent of acid and molybdenum concentrations. At 70 °C, hexagonal phase was observed for all cases.

When potassium is used as counter ion, 3 different structures have been observed, hexagonal potassium molybdenum oxide, supramolecular-like phase and trimolybdate. Supramolecular-like phase was obtained at low temperature independent of temperature. At higher temperature, more concentrated Mo solution needed to precipitate this phase. While a mixture of phases comprising trimolybdate as the major phase at higher temperature.

Typically, at lower concentration and higher temperature, hexagonal potassium molybdenum oxide was the main yield whereas trimolybdate is the predominant phase at higher temperature and Mo concentration.

No spontaneous precipitation occurred when lithium and sodium were used as counter ions. Further heating to 80 °C was needed for precipitation to occur. After heating, the product yielded for lithium counter cation is molybdenum trioxide orthorhombic (PDF-file 5-508). While for sodium counter cation, hexagonal molybdenum oxide was detected in all three samples with very similar diffraction patterns.

This study on nano-preparation of molybdenum oxide model catalysts has utilized a multitude of characterization techniques. It has been demonstrated that the development of different molybdenum oxides phases can be followed *in-situ* and *ex-situ* by using Raman Spectroscopy and Electron Microscopy as complementary technique to Powder XRD.