Summary and Conclusion

In recent years there have been a growing interest to find out a new and better ways to prepare oxides of high surface area and thus high catalytic activity.

The objective of this work is the Preparation and characterization of microporous TiO_2 and V_2O_5:TiO_2 materials that having shape selective properties like zeolites.

This thesis consists of three chapters:

Chapter 1 deals with a general introduction of the subject and literatures survey.

Chapter 2 includes the materials, preparation of the samples by conventional and novel methods by using micelle-template Techniques, in which different types of surfactants were utilized such as:

1- Cationic surfactants: CTAB, TB and CPB.

2- Anionic surfactants: SDS.

3- Poly -ol group's compounds: xylitol, sorbitol and glucose.

All of these compounds are used in the preparation processes at different conditions, pH, varying surfactant concentrations and calcination temperatures.

Also, this chapter includes experimental procedures and techniques that are used for samples characterization.

Chapter 3 deals with the obtained results and discussions; this chapter consists of six parts that are reported as follows:

Part (A): Surface properties that were determined by measuring the adsorption of nitrogen at 77 K using conventional volumetric apparatus. The general trend of the surface characteristics indicates the followings:-

a) All the TiO, and $V_2$05/TiO2 samples prepared with different methods show high surface areas.

b) The prepared catalysts show type II, with hysteresis loop of type F1. 4 that indicates the presence of micro-and mesoporsity, which was also ascertained by V_l-t plot. The percent of microporosity changes with the conditions of the preparations, type of surfactants, pH and calcination temperatures.

Part (B) Structural characterization of the catalysts and the phase changes were determined using thermal analysis and X-ray diffraction techniques. The results obtained from both techniques supported each other and indicate:

a) The formation of a rutile phase at low temperature (623 K) when using cationic surfactants and in the presence of vanadium. Where the sample prepared by using CPB in the presence of vanadium showed an anatase phase. This indicates the role of both surfactant type and the presence of vanadium in the formation of the phase formed.

b) The formation of anatase phase was depicted when using anionic surfactant at different preparation conditions, except the sample prepared at lower pH (pH=2.0). From thermal analysis data, this phase showed a high thermal stability.

c) The formation of anatase phase was also shown with poly-ol groups at different conditions, except the sample prepared by using glucose showing the formation of mixed phases (rutile and anatase). This is ascertained by thermal analysis data which indicates the presence of peaks related to the phase transformation and rather indicates the stability of the phase formed up to higher temperature (1073 K).

d) the absence of any peaks related to V,O; in X-ray patterns confirms. the well dispersion of vanadium oxide in TiO2.

e) From X-ray results, it can be seen that all the samples are in the nanoparticle size range, and the cn.stallinity of the formed phases differs according to varying the preparation conditions.

Part (C): Includes the results obtained by the scanning electron microscope and showed the micrograph of the surface of the prepared samples which indicate the variation of the surface morphology and the particles size distribution as a result of varying the preparation conditions

Part (D): Deals with the results obtained from studying the FTIR-spectra of the prepared samples in high wavenumbers region (3500-3800 cm^{-1}) as well as in low wavenumbers region (400-1000 cm), that were characteristic for the phase formed.

a) The results obtained supported those derived from X-ray data.

b) The shift of the bands and the variation of its intensity depend on the preparation conditions.

c) The results also confirm that there are no peaks characteristic for the $V_2$05 phase, as revealed from X-ray results.

Part (E): Deals with the results obtained from the surface acidity measurements of the prepared samples by adsorption of pyridine at 300 K and traced by FTIR spectroscopy. The results obtained revealed the presence of Bronsted and Lewis acid sites and their concentrations varied with varying the preparation conditions.

Part (F): Includes the results obtained from the studying the photocatalytic activity of the samples toward the reduction of Hg(II) from the waste water. The data indicates that:

a) The samples that show higher photoactivity are the samples prepared by using SDS at pH=7.0 (T sDs ti,D·7·()) and that prepared by glucose.

b) The photocataMic activity was studied at different conditions for the $T_s ps$ sample: pH of the Hg(II) solution. initial concentration of Hg(II) solution, amount of catalyst, addition of surfactant and source of radiation. From this data, it was found that the best conditions for having the maximum reduction efficiency were when using 0.1 g of catalyst and pH=5.5 of solution without surfactant and with 50 ppm as the initial concentration.

c) It was found that the sample prepared with glucose show high durability since the performance of the catalyst was almost same in three runs while using fresh Hg(II) solution in each run achieving complete photoreduction of Hg (II) in about 80 mm. illumination time.