EFFECT OF HEAT-TREATMENT ON THE SURFACE AND PHYSICO-CHEMICAL PROPERTIES OF SOME PRECIPITATED PURE AND MIXED TRANSITION METAL OXIDES

BY

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CERTIFICATE

This is to certify that thesis entitled, "Effect of Heat Treatment on the Surface and Physico-Chemical Properties of Some Precipitated Pure and Mixed Transition Metal Oxides," being submitted by Miss Satish Nanda to the Indian Institute of Technology, Delhi, for the award of the degree of 'Doctor of Philosophy' in Chemistry, is a record of bonafide research work carried out by her. Miss SATISH NANDA has worked under my guidance and supervision and has fulfilled the requirements for the submission of her thesis.

The results contained in this thesis have not been submitted, in part or full, to any other University or Institute for the award of any degree or diploma.

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ABSTRACT

The thesis opens with an extensive literature survey of the surface and physico-chemical properties of the metal oxides of the first transition series. Although, the study of surface properties on individual metal oxides have been made in detail but the data on comparative study are very scanty. The investigations reported, contain the details of the experimental methods used for the preparation, activation and characterisation of the metal oxides. The different techniques used for studying the adsorption of ammonia, adsorption of pyridine and preferential adsorption from the binary mixture of isopropanol and carbon tetrachloride are given. The catalytic activity of the oxide samples have been compared by studying the kinetics of hydrogen peroxide decomposition.

The results of the various findings given in five different chapter show that, the samples obtained are microcrystalline in nature and are associated with sizable amount of water. The thermal methods of analysis and ATR infrared spectroscopy infer that this water is present in two different forms, i.e. bulk water and chemisorbed water. The effect of heat treatment at various temperatures, results in altering the surface area, surface acidity, surface morphology, adsorption behaviour and catalytic activity. The surface area increases with raising the temperature of activation upto 400^{o}C, thereafter a sharp decrease is noted,

which is due to the shrinkage of the samples as a result of sintering. The nature of surface active sites as revealed by the adsorption of ammonia and pyridine indicate the presence of both Lewis and Brönsted acid centres on the surface. Lewis acid sites, on heat treatment transform to Brönsted acid centres. The surface species observed in the case of ammonia adsorption are NH_4^+, NH_2 and free ammonia. In the case of pyridine, pyridinium ion have been detected along with hydrogen bonded and physically adsorbed pyridine.

The preferential adsorption studies show that the samples are alcophillic and the preference for alcohol changes with the change on heat treatment. The decomposition of H_2O_2 obeys first order kinetics and "Arrhenius equation." The investigations reported for the mixed oxides show that the samples activated at 200°C are the most active. The surface properties measured are not linearly related to the composition of the mixture but show a maximum activity when the composition of the mixture for copper and chromium is in the range of (1:1) and (1:2), showing thereby that surface and catalytic properties of mixed oxides are not additive but specific for the given composition. These investigations also suggest that similar studies on other binary and even ternary metal oxide mixtures will be extremely rewarding in preparing an active and selective catalyst.

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