SUMMARY
Zeolite molecular sieves have been used commercially as sorbents and ion-exchangers and their recent use as catalysts has been established, and there have been several reports on the use of zeolites for selective organic transformations leading to the synthesis of fine-chemicals. Continuing studies on synthetic zeolites and their phosphatic derivatives called "SAPO" zeolites will lead to possible future applications besides those already reported. This thesis entitled "Synthesis and Studies of New Phosphatic Derivatives of Alumino-silicates" deals with the study of new phosphatic derivatives of synthetic zeolites provided by Indian-Petro Chemicals Limited, Baroda and their cation-exchanged derivatives using three phosphatic compounds of inorganic and organic origin namely, tricresyl phosphate, phosphoric acid and n-butyl phosphate.

Synthetic zeolites obtained as white fine powder have "A" and "X" zeolite type structure and sodium and calcium as cations. The aim of undertaking this work has been to prepare new phosphatic derivatives with different cations in the zeolite framework and to observe the structural changes in the zeolite after their interaction and their cation-exchanged derivatives with phosphatic compounds. The cations Li(I), Ca(II), Fe(III), and Th(IV) chosen for the ion-exchange were due to their importance in the field of fertilizer, catalysis and radio-active waste disposal. Interaction of these cation-exchanged derivatives of synthetic zeolites with phosphatic compounds tri-cresyl phosphate, phosphoric acid and n-butyl phosphate have been studied. They have been characterized with the help of elemental analysis, FT-IR, X-ray and thermal studies. The new phosphatic derivatives can find uses as catalysts in such processes which requires the special properties found in both zeolites and aluminophosphates. Analytical data have helped to establish the formulae of two synthetic zeolites provided by IPCL, Baroda:

Synthetic Zeolite "one": \(-\text{Na}_{11} \text{Ca}_{1} \text{Al}_{13} \text{Si}_{9.40} \text{O}_{43.24} \cdot 24 \text{H}_2\text{O}\)

Synthetic Zeolite "two": \(-\text{Na}_{17} \text{Ca}_{1} \text{Al}_{19} \text{Si}_{17.42} \text{O}_{73} \cdot 51 \text{H}_2\text{O}\)
This thesis is divided into four chapters.

Chapter I:-

In this chapter general information on the silico-alumino-phosphate i.e. SAPO zeolites and synthetic zeolites have been given. These include synthesis of SAPO zeolites, mechanism of phosphorus substitution, applications of SAPO zeolites, brief idea of structure of synthetic zeolites and theory of cation-exchange in zeolite. This chapter also includes the reports on many important and recent applications of zeolites including the SAPO variety.

Chapter II:-

This chapter is devoted to the experimental work done on sample preparation and data collection used in the present investigation. The instrumental and analytical methods used have been described from the point of view of their principle, technique and special uses in the studies of zeolites. FT-IR spectra of samples were recorded in KBr pellets between 4000 cm\(^{-1}\) and 400 cm\(^{-1}\) infra red region. The major structural groups present in zeolite samples have been detected from their IR pattern. Thermal studies (TG-DTA) in dynamic air upto 900\(^{\circ}\) C at a heating rate of 10 \(^{\circ}\)C per minute have helped to obtain weight losses due to various thermo-chemical changes like dehydration, dehydroxylisation, desorption and decomposition of adsorbate. X-ray diffractograms of the samples were recorded between 2\(\theta\) angle of 5\(^{\circ}\) to 70\(^{\circ}\) using Co \(\alpha\) radiation to elucidate their possible crystalline structure. Compositional studies of zeolite samples were carried out by simple gravimetric analysis and also using a flame photometer for Na(I) and Ca(II)
Chapter III :-

This chapter deals with the results obtained for the cation-exchanged derivatives of synthetic zeolite “one” and “two” provided by IPCL, Baroda. Instrumental methods have helped to determine the position of cations, degree of crystallinity of the cation-exchanged derivatives, thermal behavior of cation-exchanged derivatives and extent of cation-exchange. The extent of cation-exchange differs with four different cations i.e. Li (I), Ca (II), Fe(III), and Th (IV) used for ion-exchange.

Chapter IV :-

In this chapter the investigations about the interaction of three phosphatic compounds tri-cresyl phosphate [C₆H₄(CH₃)PO₄], phosphoric acid (H₃PO₄) and n-butyl phosphate [CH₃(CH₂)₃PO₄] with the cation-exchanged derivatives along with the original synthetic zeolite “one” and “two” have been discussed. FT-IR studies have shown the presence of adsorbed phosphorus compound and the changes occurring after interaction with phosphorus compounds. The IR bands which appear at 300-650 cm⁻¹, 900-1200 and 1200-1500 cm⁻¹ are assigned to δ (O-P-O), ν (P=O) and ν(P=O) vibrations respectively. X-ray diffractograms have shown the crystalline nature of new phosphatic derivatives. Cation-exchanged derivatives of synthetic zeolite “one” and “two” lost their crystalline nature after interaction with phosphoric acid as revealed by X-ray diffractograms confirming the collapse of crystalline framework structure. All the phosphatic derivatives, however, do not lose their crystallinity and some structures remain unchanged. Most of the samples show excellent thermal stabilities.

In conclusion, it can be said that the work presented in this thesis makes a comparative evaluation of phosphatic derivatives made with three phosphatic compounds tri-cresyl phosphate, phosphoric acid and n-butyl phosphate.