LABORATORY SYNTHESIS AND CHARACTERISATION OF NANOPOROUS ZEOLITE USING FLY ASH

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Abstract:

Zeolites were synthesised using fly ash obtained from a thermal power plant in Chennai and characterized using IR spectrum, SEM and EDAX. The IR spectrum confirms the presence of zeolite and EDAX confirms the structure and chemical composition.

Key Words: zeolites, porosity

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1. INTRODUCTION

With the development of technology specific crystals with predetermined properties are in demand in industry, especially in electronic chips, antennas, and biosensors. Such demand for theses materials have ushered in the need for research in crystal growth.

Zeolites are millennium minerals and nature's gift. They occur naturally also. Hence synthesis and characterization of zeolites will have applications in environment ecology and energy conservation which are topics concerning humanity.

Zeolites have nanoporous structures. These act as adsorbents, molecular sieves and can be synthesised with different porosities. The emission of NO_2 from stationary and automotive sources such as power plants is a major environmental pollution. N_2O is a greenhouse gas. The catalytic reduction of nitrogen oxides to N_2 using selective catalytic reduction with zeolite has been tested. Moreover nanoporous photo catalytic zeolites oxidise methanol, trichloroethylene and reduce pollution in enclosed aircrafts etc. Hence it was decided to synthesise zeolite at laboratory scale and study its various properties.

Zeolites have unique atomic structure consisting of an open three dimensional network of channels and cavities which are interconnected. Micro porous crystalline alumino silicates with pore dimensions less than 15A, allow water ions to flow through these pores. When zeolites are heated water molecules in it escape and heat energy is stored in it. When water molecules are absorbed heat energy is released. Different type of zeolites can be prepared with different porosities and their properties analysed. Ammonia and Potassium can be exchanged from zeolite sites. Hence lot of research is going on in synthesis and characterization of this nanoporous material.

A historical perspective of zeolite in science and technology has been given by Milton [1]. Michael etal [2] at Minnesota have unlocked the secrets of crystal formation using sophisticated microscope. The ability to prepare zeolites with different pore sizes have been described in data base of zeolites[3].Many experiments in zeolites have been described by Blatter etal. and Copperthwaite etal.[4]. Zeolite synthesis has been described by Rollmann etal [5].The GSA [6] resources have classified zeolites into three functional classifications of gas adsorption, water

adsorption and ion exchange. Perzes R. etal [7]. have synthesised PbS nano-particles in zeolites Na-X by means of Pb exchange .They studied the structure by X-ray diffraction and the optical properties by diffuse reflective spectroscopy. Z.K Tang etal [8] have reported the electric transport properties of nano sized particles fabricated in the channels AlPO-5 Zeolite crystals. G.R.Olhoeft [9] have modelled the chemically surface reactive materials like zeolites and studied electrical properties. Sarah Larsen [10] in University of Iowa has developed nanometer sized zeolites as environment catalysts.

Lot of studies are going on in zeolite structure in India. Byrappa etal [12] have described how solvents work under temperature and pressure and recrystallize normally insoluble materials with necessary equipment. Lakshmi Kantan etal [13] have synthesised nano crystalline zeolite beta in supercritical fluids , by two stage varying temperature synthesis and super critical fluid aided crystallisation. M.S. Joshi etal [14] have described hydrothermal growth and characterization of VSZ-4 zeolite crystal.

P.S Alegaon etal.[15] have found evidence for density dependent field emission characteristics in carbon nano tubes in AlPO -5zeolite[4]. M.S.Joshi etal [16] have synthesised zeolites using natural crystals scolecite[5] hydrothermally at 180.

M.S.Joshi etal [17] have conducted scanning electron microscope studies on hydrothermally grown VSZ-3 and VSZ-4. S.S Raylu [18] has estimated crystallinity in fly ash based zeolite A using XRD and IR spectroscopy.

2. MATERIALS AND METHODS

2.1 Experimental procedure:

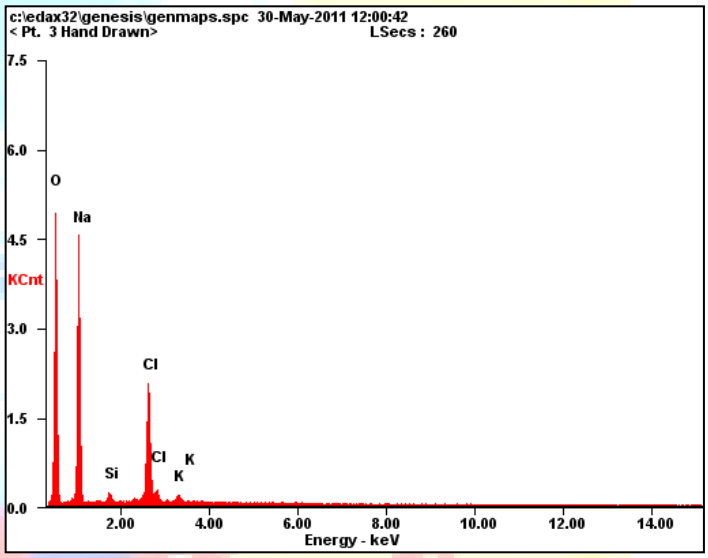
8gms of fly ash was mixed with 10 gms of NAOH and ground in a mixer .Later it was heated for 3 hrs in a furnace at 550 degrees .The molten mass was powdered and mixed with water and stirred and kept in a constant temperature bath of 30 degrees with continuous stirring for 10 days. After that it was left at room temperature for 15 days at the end of which microcrystals were obtained.

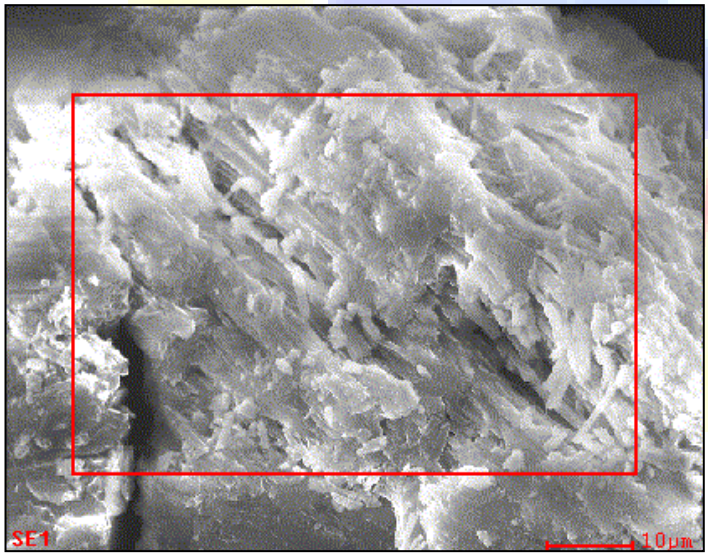
ANALYSIS

SEM Analysis.

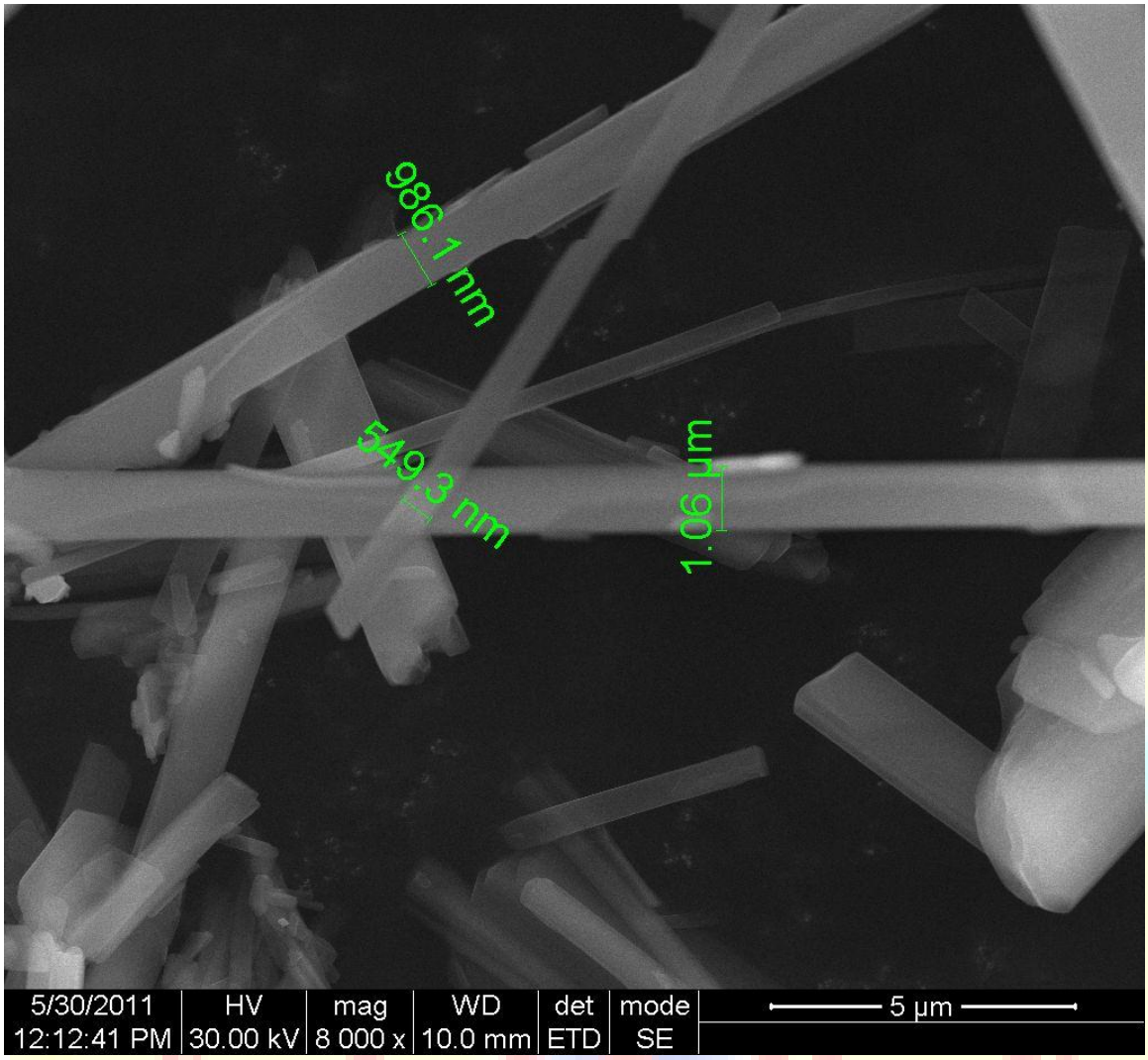
The SEM analysis was done at SAIF in IIT Chennai.

Element	*Wt%*	*At%*
OK	52.13	62.71
NaK	38.03	31.83
SiK	00.9 5	00.65
ClK	08.39	04.55
KK	00.50	00.25
Matrix	Correction	ZAF

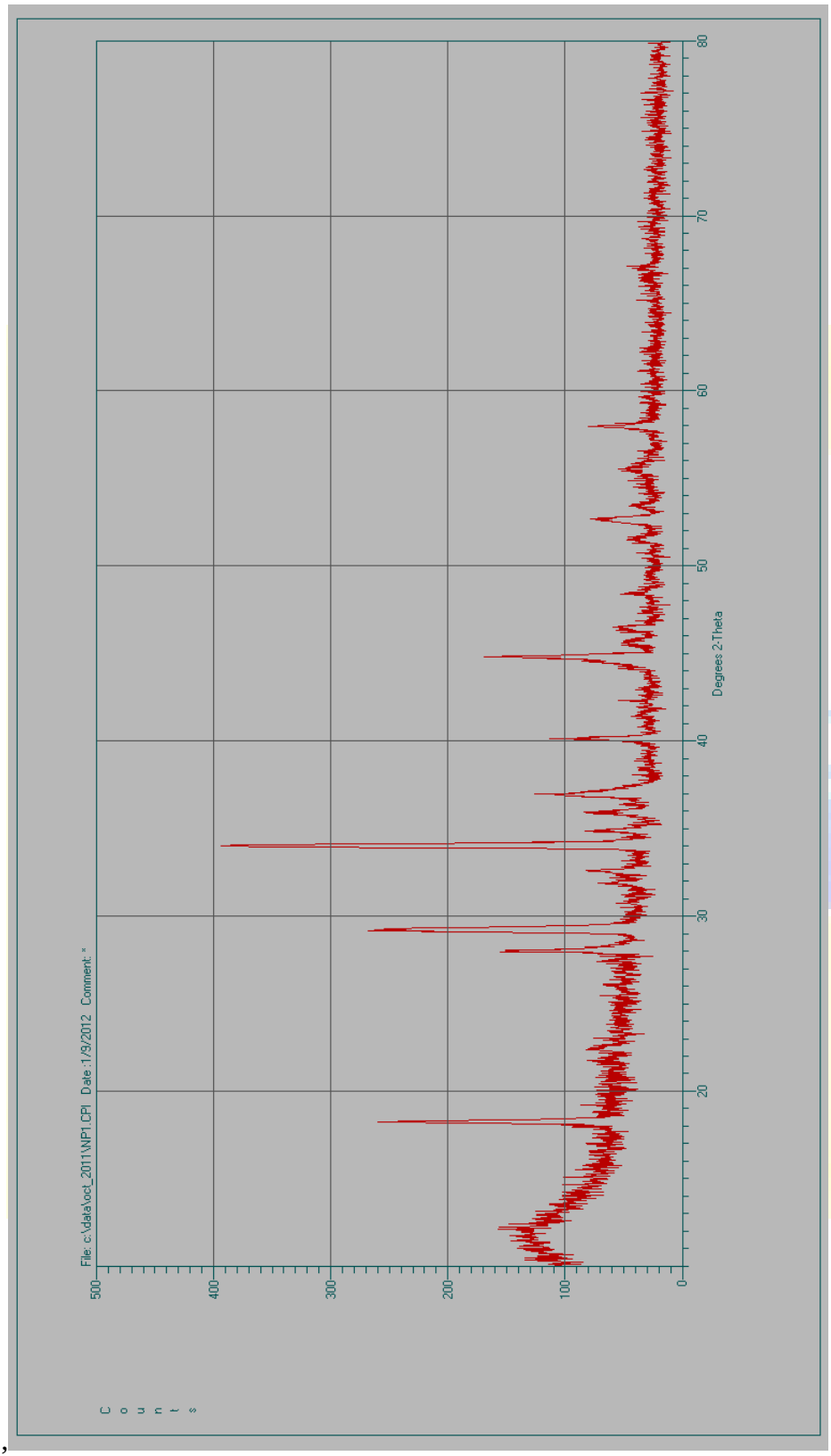




SEM analysis result is illustrated below.



XRD analysis was done at IISC Bangalore and results are illustrated below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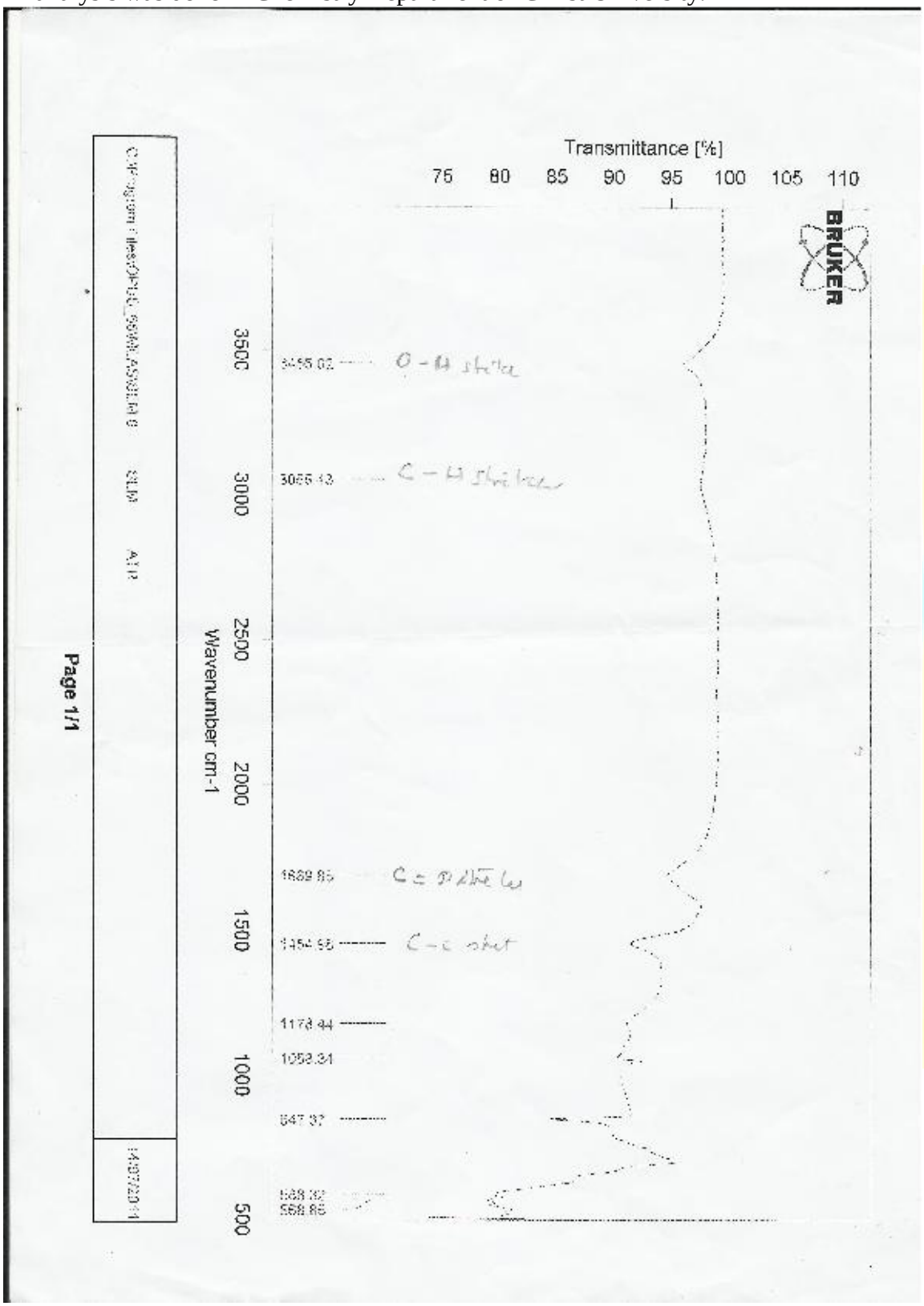
IR analysis was done in Chemistry Department of Christ University.



3. CONCLUSIONS

SEM analysis when compared with the previous authors shows rods type structure. In XRD spectrum the peak positions and intensities especially for 2θ =16, 30 indicate the crystalline components. Additional peaks indicate impurities in the sample. IR spectrum of zeolites gives typical peaks in mid infra red pore opening vibrations 1150 - 1050 cm^{-1}. Extra frame species such as aluminium entities is also detected at about 1450 cm^{-1}. Adsorption of pyridine on cations of zeolite structure produces bands in 1438-1452 cm^{-1} range.

The SEM analysis and IR spectrum when compared with the analysis by previous authors confirm that the product is a zeolite.

Results indicate that zeolite porous crystals can be synthesised at low temperature economically and has wide range applications.

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