ELECTROCHEMICAL STUDIES ON PROTON CONDUCTING PROPERTY OF BARIUM DIPROPYLMALONIC ACID METAL ORGANIC FRAMEWORK

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Abstract : Barium and Dipropyl malonic acid metal-organic complex has been synthesized and it was characterized by Fourier transformation spectroscopy and XRD methods. Proton conductivity studies normally carried out by AC Impedance evaluations. The coordinated water molecules and its hydrogen bonds give merits for the complex to its potential for the proton conduction. AC Impedance evaluations were obtained 10 min, 30 min, 1 hour, 3 hours, 6 hours, 12 hours, 24 hour and different temperatures from 35°c, 40°c, 45°c, 50°c, and 55°c. It observed that the impedance results proved that the proton conductivity increased even at 55°c.

***Index Terms*-Barium, Impedance, XRD, FTIR, SEM.**

1. INTRODUCTION

Porous coordination polymers are a type of metal-organic framework because a class of crystalline material [1] that is metal clusters (organic and inorganic) with organic bridging molecules. Metal-organic frameworks have a versatile structure [2] because of their chemical functionality, structure and thermal stability. Lithium-ion batteries have been widely used in our world due to its rechargeable property [3] and also several metal-organic frameworks were tested in the field of lithium-ion batteries [4-5]. Supercapacitors are also a type of metal-organic frameworks. Sometimes metal-organic frameworks and metal-organic framework derived nanomaterial offers the greatest application of supercapacitors due to their great potential and high surface area [6]. Electrolytes are used mainly for fuel cells, which constructed a metal-organic framework because of their increased efficiency and oxygen reduction reaction [7-9]. Functionalized graphene with a porphyrin metal-organic complex gives higher porosity of metal-organic frameworks [10]. To get high performance of oxygen reduction catalyst 'Wange et al' have to increase a precursor complex for non-precious metal electrolyte ion-based metal-organic frameworks used as precursors for fuel cell application cobalt metal-organic framework that gives non-porous structure and nitrogen doping carbon framework. Zeolites are a type of metal-organic framework because of their hydrothermal or solvothermal technic. Not only the metal-organic frame constructed by the bridging method [11] but zeolites also often made by the use of template method [12-13], some metal-organic frameworks demonstrated using microwaves [14]. Often metal-organic frameworks used in various fields due to their functionality, high surface area and pored size, thermal stability, separation, drug delivery, gas storage and proton conduction property [14]. In the present work barium- dipropyl malonic acid metal-organic frame woks have been synthesized and characterized by FTIR and PXRD methods. The characterized complexes have been examined to electrochemical study for evaluation of proton-conducting property under moist conditions with various temperatures in the timing range.

2. MATERIALS AND METHODS

2.1. Sample Preparation

The barium chloride compound purchased from NICE chemicals (M.W-208.23 g / mole) was used as metal cations. Dipropyl malonic acid used as organic ligands purchased from a seller (M.W-188 g / mole). 2.08 g metal ion dissolved with a minimum amount of water and then organic ligand 11.28 g were taken in a separate beaker and it was dissolved minimum amount of water. Then metal ion and organic ligand solutions were mixed thoroughly. After that, the mixed solution was warmed and filtered using normal filter paper. Then the solution kept under the atmosphere for a long time. After a month the solution was formed as crystallized in nature.

2.2 FTIR Spectra

Fourier transformation spectra of the chemically synthesized inorganic complex were recorded using KBR pellets in the range of 4000cm-1 – 400 cm-1 on a Perkin Elmer FTIR spectrometer (spectrum TWO).

2.3 X-RAY Diffraction method

The chemically synthesized sample was taken PXRD measurement. Model: SHIMADZU (XRD 6000). Scan range – 50000-80000 (deg), scan mode – continuous scan, scan speed – 10000(deg/min).

2.4. AC Impedance spectroscopy

The Impedance measurements were formed using a computer-controlled potentiostat (PARSTAT-2273). The impedance measurements were carried out in the frequency range 0.1KHZ-100KHZ.

2.5. Scanning electron microscopy

The surface morphology of the complex was studied by SEM monographs obtained by using Model: EVO-18, Make CAREL ZEISS.

3. RESULTS AND DISCUSSION

3.1 FTIR Analysis

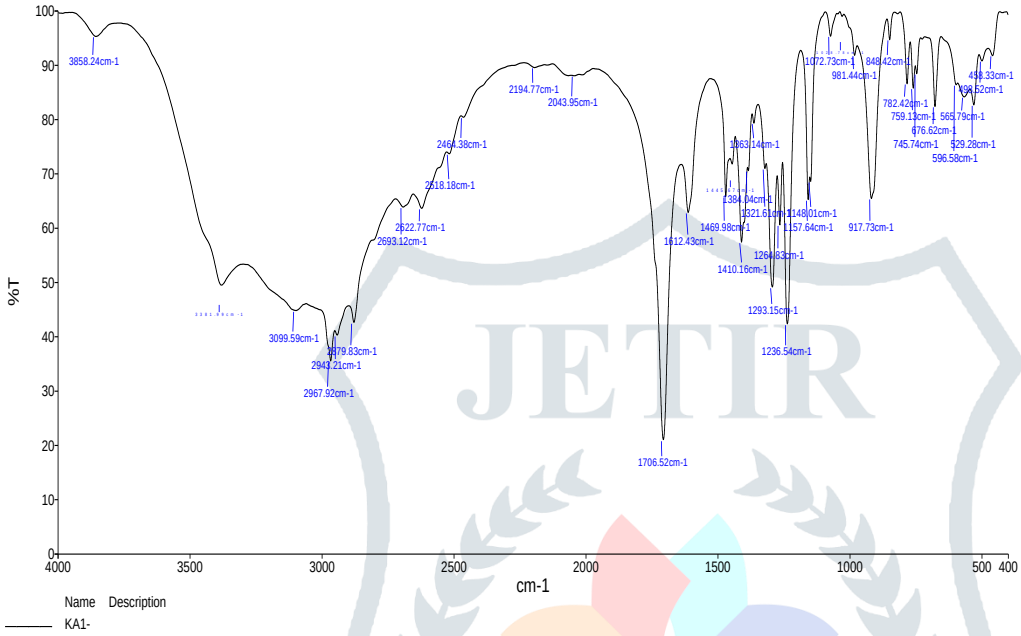

Fig: 3.1 FTIR spectrum of Barium and Diproprylmalonic acid MOF.

The vibrational bands at 1612.43cm-1 and 134.04 cm-1 are attributed to asymmetric and symmetric stretches of the carboxyl group respectively. 1706.52 cm-1 represents the protonated nature of the carboxylic acid groups. 3000-3500 cm-1 can be attributed to the O-H stretching frequency of hydrogen-bonded water molecules. 745.74 cm-1 and 676.62 cm-1 frequencies attributed to the co-ordinated and lattice water molecules. 1264.83 cm-1 attributed to aliphatic oxygen and barium stretching frequency. 1384cm-1 and 1445.57 cm-1 attributed to carbon-hydrogen stretching frequency. 596.58 cm-1 represents metal oxen stretching frequency.

3.2 P-XRD Analysis

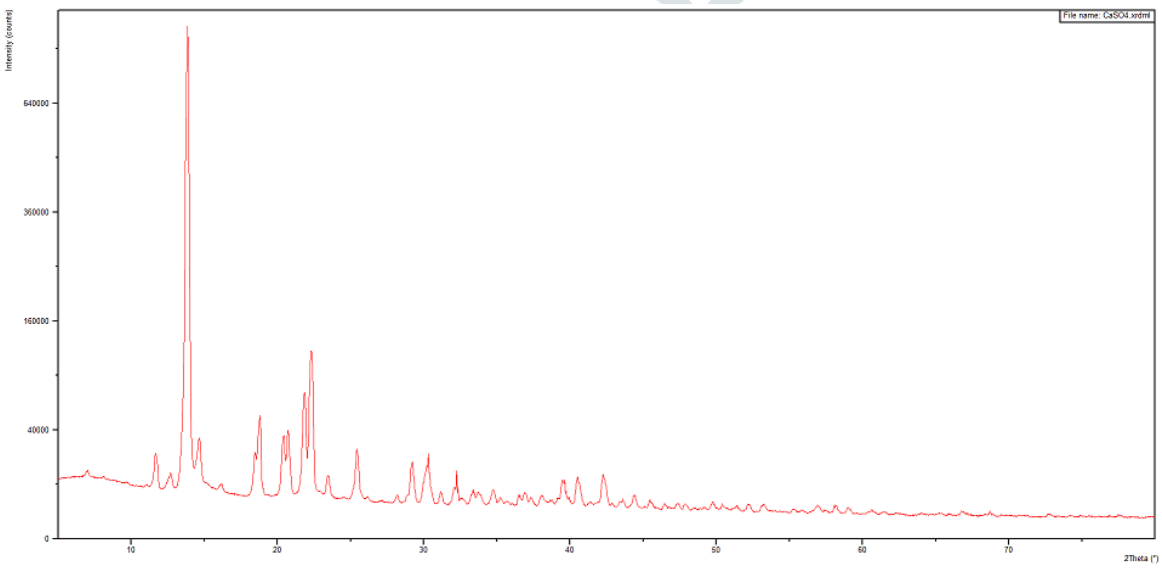

Fig: 3.2 Powder XRD of Barium and Diproprylmalonic acid MOF.

Fig 3.2 explains the original MOF of Ba-DPM in powdered form. The XRD image gives a very sharp and intense peak due to its crystalline nature. The XRD peak obtained at 18.1° and 24.2°. The dried powders under 100% atmospheric conditions at 25°c similar experiments were done. All the peaks were given very intense and sharp peaks. Each peak matches very closely as the synthesized phase moreover some slight differences appeared because some peaks merging at the lower angles in near reversibility of the situation.

3.3 AC Impedance studies.

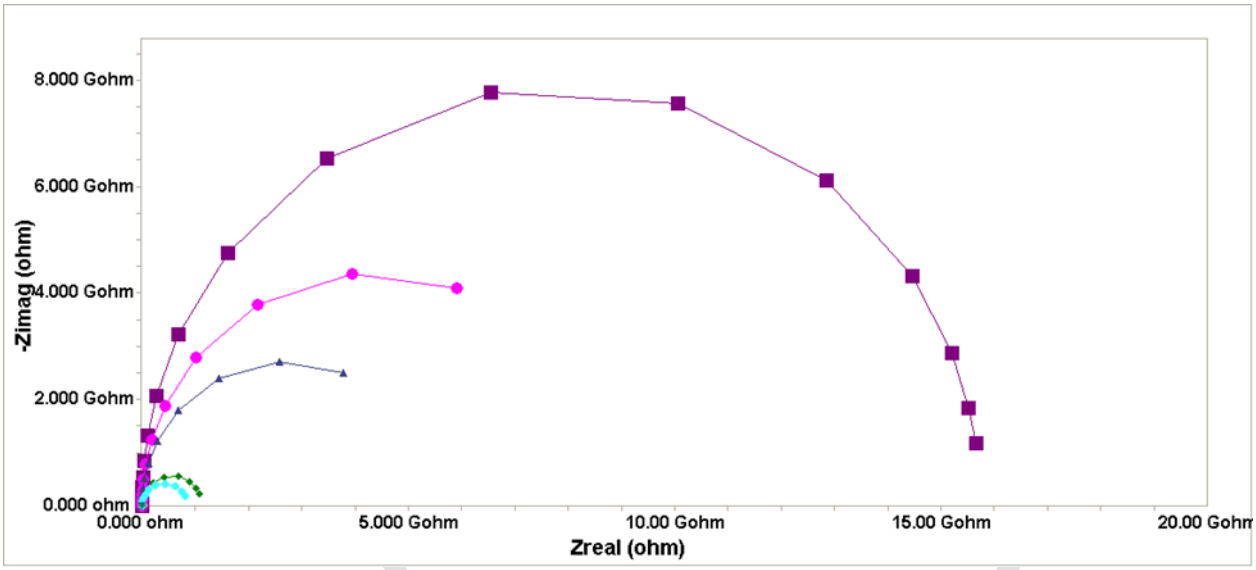

Fig. 3.3. The variation of proton conductivity with different temperatures.

The AC Impedance studies were done using electrochemical instruments under various humidity conditions at 25°c and higher temperatures which have been revealed that the ionic conductivities are visible. The Nyquist plots of the Barium and Dipropyl malonic acid sample which was obtained at 10 min,30 min, 1 hour, 3 hours, 6 hours, 12 hours, 24 hour and different temperatures from 35°c, 40°c, 45°c, 50°c, and 55°c. The increase in time and increase with temperatures to moist atmospheric conditions. Their by proton conductivity increases which are clearly shown with the help of Nyquist plots. The coordinated water molecules and its hydrogen bonds give merits for the complex to its potential for the proton conduction. Therefore the conductivity related to the transport of proton as $H3O^{+}$. It observed that the impedance results proved that the proton conductivity increased even at 55°c.

3.4 SCANNING ELECTRON MICROSCOPE

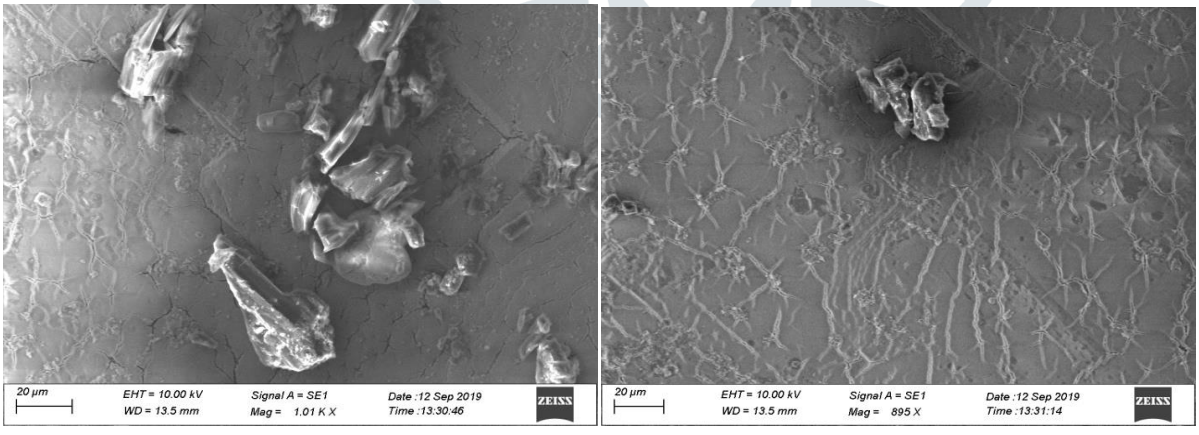

Fig. 3.3. The SEM image of the Ba-DPM of MOF.

The Ba-DPM complex morphology of synthesized material was investigated using scanning electron microscopy the image clearly showed that well-shaped and high-quality crystal appeared in the SEM micrograph of both MOF Ba-DPM.

4. CONCLUSION

1. Barium – Dipropyl malonic acid inorganic complex was synthesized.
2. Its good solid-state proton conductor.
3. The FTIR and PXRD experiments were confirmed the complex formation.
4. AC Impedance examinations were given good results under moist conditions.
5. The moisture-based proton conductance plays a crucial role in the conduction mechanism.

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