https://dx.doi.org/10.22161/ijaers.5.6.12 ISSN: 2349-6495(P) | 2456-1908(O)

Photocatalyical and Thermal Properties Consideration of nanocomposites preparation of $La_2Ti_2O_7$-Zeolite-MCM-41

Nasim Mahdian

Department of chemistry, Tehran North Branch, Islamic Azad University, Tehran, Iran
nasim_mahdiyan@yahoo.com

***Abstract*— *In this paper, nanocomposite $La_2Ti_2O_7$-Zeolite-MCM-41is synthesized by optimization of physical properties of MCM-41Zeolite and Nano powder $La_2Ti_2O_7$ sol-gel method in stearic acid media. In the first step, the $La_2Ti_2O_7$Nano power- was prepared by sol-gel process and then nanocomposites of $La_2Ti_2O_7$-Zeolite-MCM-41 with (5,10,15 and 20) percent weight. Then we have prepared of sol-gel at 700-900 and 1000 and for further confirmation of structure used of x-ray and XRD.*

Result of SEM, the size of the $La_2Ti_2O_7$Nano powder (29nm) and nanoparticles in nanocomposite $La_2Ti_2O_7$-Zeolite-MCM-41 20%, (29nm) were shown EDX the purification of nanoparticles and nanocomposite are convenient.

The BET method was showed the differential porosity value in comparison of zeolite and nanocomposite $La_2Ti_2O_7$-ZMCM-41 the calcination condition, weight percent nanoparticles, the type of reagents PH and indicator concentration are influenced on nanocomposite.

Properties by optimization of mentioned parameters, the best results for 10% and 20% nanocomposite in 900 temperature calcination with (29 nm) particle size are yield. The UV methylene blue in 80 and 100% are out come respectively. All results were confirmed by spectroscopies data.

Keyword*— *Composition- Synthesized- Nanocomposite-Calcination- Zeolite.

I. INTRODUCTION

The composite is a multicomponent material that its properties are better than each component, while the different components improve each other performance (1). Generally, a composite material is defined as the physical mixture made of two or more different materials in the macroscopic scale that these materials kept its physical and chemical properties and form the specific boundary together (2). The composites have distinguished mechanical properties and possess the suitable flexibility in the design and their fabricate procedures are rather easy. The composites are light, corrosion and impact resistance materials with excellent fatigue resistant, strength and permanency and they are capable of transforming into a product with the part.

The composites are divided to 5 groups according to the material shapes in the composite:

1. Fiber composites
2. Laminar composites
3. Particle composites
4. Flake composites
5. Filler composites

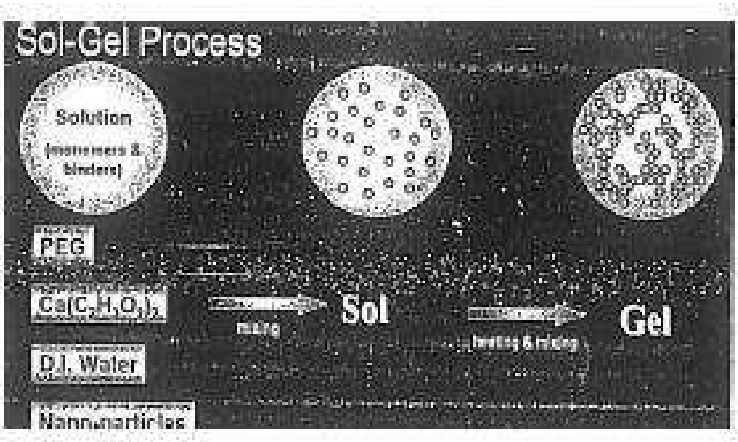
The nanocomposite which is synthesized in this study is a $La_2Ti_2O_7$-ZMCM-14 composites type in which zeolite plays a matrix role and $La_2Ti_2O_7$-ZMCM-14 nanoparticles have a reinforcement role. These nanoparticles have been added to improve the physical and chemical properties of zeolite. We blend or composite them to change and optimize the physical and chemical properties of materials. In fact, the purpose of composite creation is to obtain the combinational material with expected properties (1). The nanocomposites components have the better properties due to the surface interaction between the base material and filler materials. The kind and amount of interactions have an important role in their different properties of nanocomposites such as solubility, optical properties, electrical and mechanical properties (3, 4).

The particular properties of nanocomposite are explained as follow:

1. The composite Nanopowders have a suitable area-volume ratio.
2. Most of atoms are on the surface of the composite Nano powders and the microstructure nanocomposite grain boundaries (3, 4).

https://dx.doi.org/10.22161/ijaers.5.6.12 ISSN: 2349-6495(P) | 2456-1908(O)

The Fabrication method of Nanocomposite



1. The Sol- gel method
2. In situ polymerization using the applicative finding in the receiver- accepter system chemistry
3. The composite of emulsion polymerization for replacing the mineral components in the organic matrix

The sols are dispersed colloidal particles with dimension of 1 to 100 nanometers in solution which remain suspended form since these particles are small in the solution.

Elman produced the silica gel for the first in 1864 and Kasa could produce alumina gel in 1870. Also, Pichini presented a method to modification in 1967. There are the different methods to fabricate a mineral composition such as SiO_2 which can be based on melting of the primary mineral compositions and quenching.

Zeolites have general formula $(AlO_2)_X$ $(SiO_2)_y$ Mh_2O Mx/n. Although, the zeolites chemically were being confined to aluminum silicates until 1982, but recently the range of these compounds has extended so that about 10 other elements Ti, As, P, Zn, Co, Mn, LI, Ng, and B are also included in the range besides previous known elements i.e. Fe, Ge, Ga, AI, Si (7).

According to Demoor theory, one of the valuable properties of zeolites is the reversible quenching.

Although, all of zeolites have 10 to 20 % water in their composition. This amount of water can be lost at 350 ° C.

The meso-pore structure and its kind control by choosing a suitable frame (surfactant) with adjustment of temperature situation, pH, and synthetic solution composition.

The molecular structure of zeolites is a tetrahedral that the oxygen atoms are around a silicon atom (SiO_4) (6, 8).

The MCM- 41 synthesis zeolite has the general chemical formula $SiO_2(_{0/9875}(Al_2O_6)_{0/0125}$. × H_2O) and the hexagonal symmetry.

Lanthanum titanate is a white oxide of mineral collection with $La_2Ti_2O_7$ chemical formula whose two chemical compounds TiO_2 and La_2O_3 dissolve difficultly in acids and they are in the perovskite category (9).

II. EXPERIMENTAL

STEP 1- experimental method

The solid stearic acid was poured in the crystallizer and it began to melt in 70˚C and a transparent liquid was obtained after fully melting of stearic acid, then lanthanum acetate was slowly added to it in stoichiometric content and was stirred by a stirrer.

The metallic salt was fully melted in 85˚C. Then, titanium butoxide was added drop by drop in the stoichiometric content and was stirred severely, so that a transparent and homogeneous gray-white sol was obtained. The resulting sol was transferred to porcelain crucible to be cold in the ambient temperature and was dried at 120˚C in 12 hours. Subsequently, the resulting gel was put in a furnace and was calcinated duration of 4 steps.

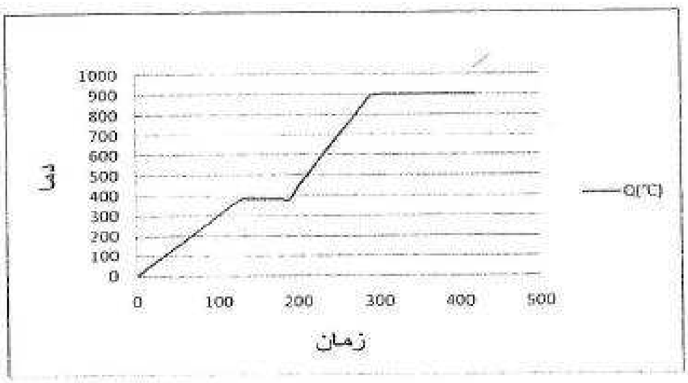


Diagram 1.2: the temperature control of nanoparticles fabrication process

https://dx.doi.org/10.22161/ijaers.5.6.12 ISSN: 2349-6495(P) | 2456-1908(O)

STEP 2- Lanthanum titanate MCM-41 zeolite Nanocomposite synthesis using the sol-gel method

For 4 kinds of composites, all of the steps were done same as the first method with the different weight percentages of 5, 10, 15, and 20 and at the temperatures of 700, 900, and 1000 °C.

STEP 3 - Test with methylene blue color

The 0.1 g methylene blue was diluted with deionized water. A 1000 ppm concentration solution of methylene blue color was obtained.

250 ml of 1 ppm methylene blue with 0.1 mg Nanocomposite were weighted and was added to methylene blue color solution. The beaker is put on the magnetic stirrer and under the UV source.

In this test, temperature and UV visible light intensity factors were kept stable and pH factor was investigated.

Investigation of pH factor:

For this work, at first, pH of methylene blue solution was measured by pH meter. This pH was neutral and around 7. For this purpose, the pH factor was investigated in both acidic and alkaline range.

Test with H_2O_2:

1 g methylene blue color dilute with the deionized water. The solution with 1000 ppm concentration obtains from methylene blue color. For this work, 100 ml of main solution with 1000 ppm concentration was diluted with the deionized water, then was picked up 2.5 ml of 100 ppm methylene blue solution by scaled cylinder and was transferred to a volumetric flask of 250 ml and then was diluted with the deionized water so that a 1 ppm concentration solution of this color was obtained.

The respective flask was closed and the UV light was radiated. The sampling was done every 15 min until 60 min.

Then, the samples were centrifuge at 3000 rpm until 15 min to powder was separated from the sample and then, its UV visible spectrum was lost.

The following table illustrates the phase index compatibility of MCM- 41 and $La_2Ti_2O_7$ zeolite nanoparticles with XRD standard carts study in 900˚C.

III. RESULT AND DISCUTION

Intensity, standard carts, sample, lanthanum titanate, zeolite

(Severity %)	Standard Carts	Sample	MCM-41
100%	00-045-0406	23/2904	$2\theta 1$
99/18%	00-045-0406	23/0169	$2\theta 2$
97/78%	00-045-0406	20/9495	$2\theta 3$
97/51%	00-045-0406	22/7482	$2\theta 4$

SPECTROSCOPY of (XRD) La2Ti2O7 9000C

$La_2Ti_2O_7$	Sample	Standard Cards	(%) Severity
$2\theta_1$	38/4250	00-027-1182	100%
$2\theta_2$	27/6670	00-027-1182	96/36%
$2\theta_3$	33/5239	00-027-1182	94/31%
$2\theta_1$	38/4250	00-027-1182	100%

SPECTROSCOPY of (XRD)

Zeolite MCM-41	La2Ti2O7 $900°C$	La2Ti2O7-MCM-41 20 % $700°C$	La2Ti2O7-MCM-41 20% $900°C$	La2Ti2O7-MCM-41 20% $1000°C$	La2Ti2O7-MCM-41 15% $900°C$	La2Ti2O7-MCM-41 10% $900°C$	La2Ti2O7-MCM-41 5% $900°C$
$2\theta_1 = 23/29$	$2\theta_1 = 38/42$	$2\theta_1 = 25/62$	$2\theta_1 = 32/98$	$2\theta_1 = 28/91$	2θ=44/1n.m	$2\theta_1 = 29/86$	$2\theta_1 = 27/67$
$2\theta_2 = 23/01$	$2\theta_2 = 27/66$	$2\theta_2 = 3/16$	$2\theta_2 = 32/99$	$2\theta_2 = 32/71$		$2\theta_2 = 32/25$	$2\theta_2 = 21/04$

https://dx.doi.org/10.22161/ijaers.5.6.12 ISSN: 2349-6495(P) | 2456-1908(O)

$2\theta 3 = 20/94$	$2\theta_3 = 33/52$	$2\theta_3 = 27/79$	$2\theta_3 = 29/80$	$2\theta_3 = 33/49$		$2\theta_3 = 33/02$	$2\theta_3 = 29/82$
$2\theta_4 = 22/74$	$2\theta_4 = 78/04$						

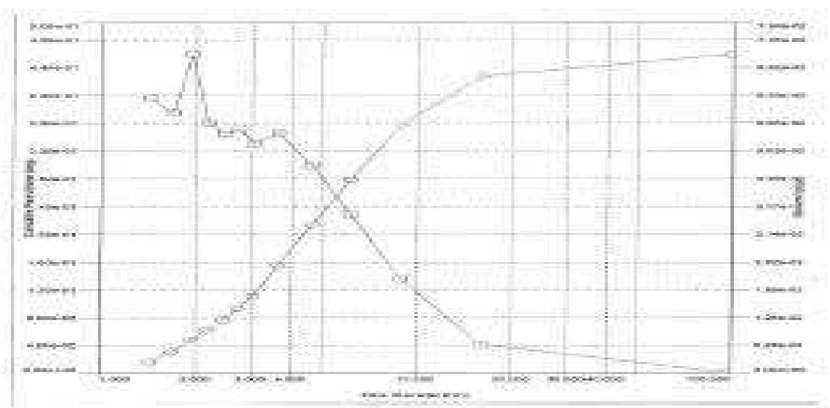
SPECTROSCOPY OF (BET)

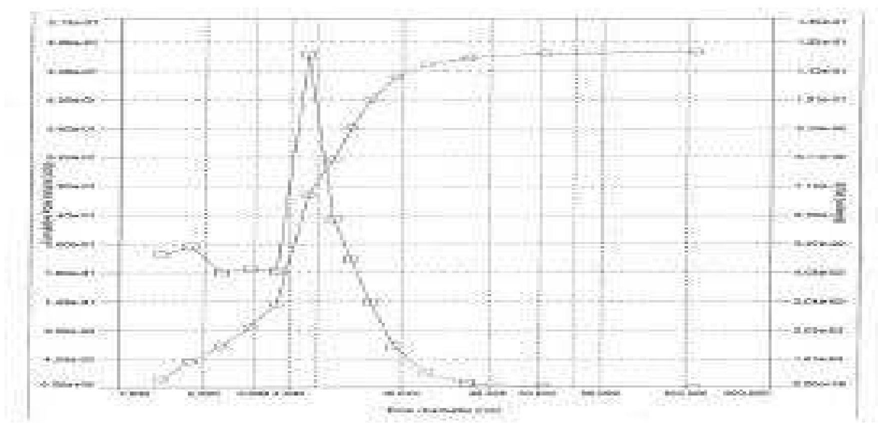
Sample	MCM-41	La2Ti2O7-MCM-41 20% $900°C$	Unit
V_m	300	52	$[cm^3 g^{-1}]$
$a_{s,BET}$	3/836	5/80	$[m^2 g^{-1}]$
Total pore volume	4/618	8/002	$[cm^3 g^{-1}]$
Average pore Diameter	4/815	5/511	$[nm]$

Unit, sample

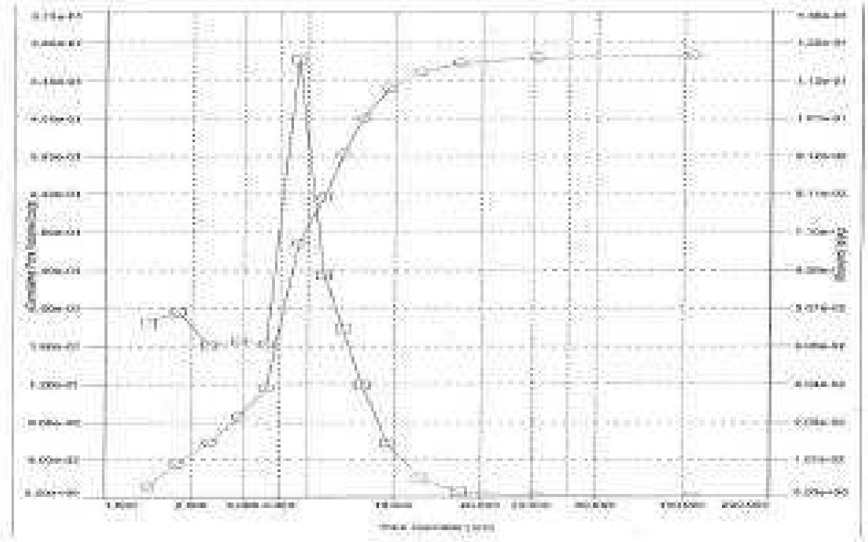
The BJH analyze diagram of MCM-41 zeolite

V_m	300	$[cm^3 g^{-1}]$
$a_{s,BET}$	3.836	$[m^2 g^{-1}]$
Total pore volume	4.418	$[cm^3 g^{-1}]$
Average pore Diameter	4.815	$[nm]$





Isothermal absorption and de-absorption analyze diagram of MCM- 41zeolite



The BLH analyze diagram of MCM-41 zeolite

SPECTROSCOPY of BJH

MCM-41		La2Ti2O7-MCM-41 20% $900°C$	
Plot data	Adsorption branch	Plot data	Adsorption branch
V_p	$0/459\ [cm^3 g^{-1}]$	V_p	$0/084\ [cm^3 g^{-1}]$
$r_{p,peak}(Area)$	$1/938\ [nm]$	$r_{p,peak}(Area)$	$1/188[nm]$
a_p	$408/914\ [m^2 g^{-1}]$	a_p	$76/214\ [m^2 g^{-1}]$

Volume table, radius, the surface of pore, the diagram peak

SPECTROSCOPY of SEM $La_2Ti_2O_7$

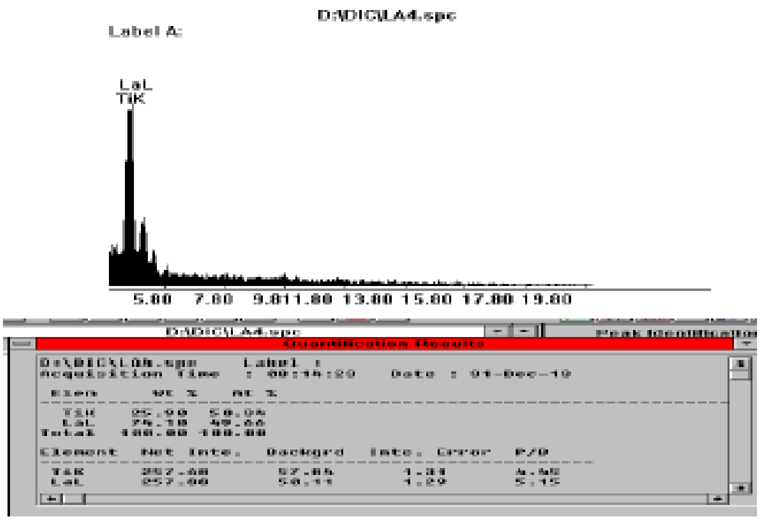
In the obtained images, the light points show the eminence surface and the darker areas illustrate the pores and concavity of surface.

SEM image of $La_2Ti_2O_7$ (24.8 nm lanthanum titanate nanoparticles) in the calcination temperature of 900˚C

The following image illustrates the lanthanum nanoparticles [24.8 nm] EDX in the calcination temperature of 900˚C.

SPECTROSCOY OF EDX

https://dx.doi.org/10.22161/ijaers.5.6.12 ISSN: 2349-6495(P) | 2456-1908(O)

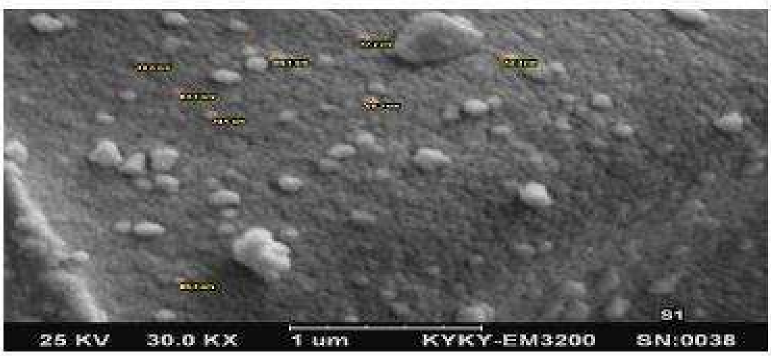


SEM image of $La_2Ti_2O_7$ – MCM-4 samples in the calcination temperature of 900˚C

In the above SEM image, the lanthanum titanate nanoparticles with the size of 44.1 nm are observable.

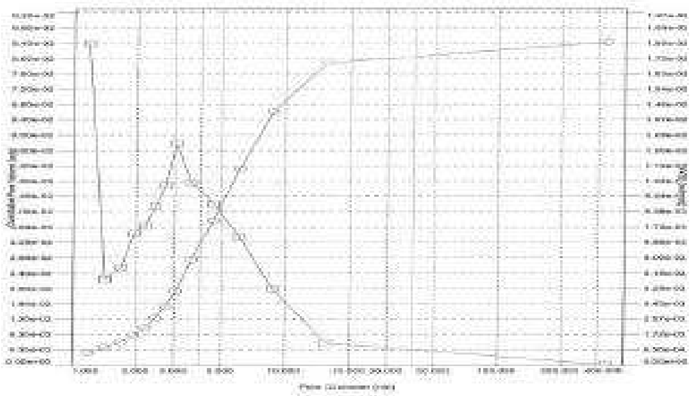
SPECTROSCOPY of SEM Zeolite- MCM-41:

In this image, MCM-41 zeolite particles with size of 40.6 nm are observable.

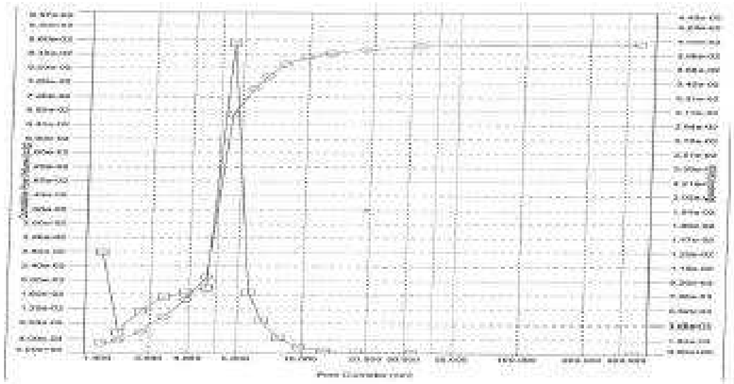


SEM image of MCM-41 zeolite has illustrated the isotherm absorption and de-absorption diagram.

https://dx.doi.org/10.22161/ijaers.5.6.12 ISSN: 2349-6495(P) | 2456-1908(O)



Isothermal absorption and de-absorption diagram of La2Ti2O7 Zeolite- MCM-41 nanocomposite



The BJH analyze diagram of MCM-41 zeolite

Plot data	Adsorption branch
V_p	$0/084\,[cm^3 g^{-1}]$
$r_{p,peak}(Area)$	$1/188[nm]$
a_p	$76/214\,[m^2 g^{-1}]$

Volume table, radius, the surface of pore, the diagram peak

FT-IR

SPECTROSCOPY of (FTIR)

FTIR cm $^{-1}$						
MCM-41	La2Ti2O7 900°C	La2Ti2O7-MCM-41 20% 900°C	La2Ti2O7 1000°C	La2Ti2O7-MCM-4120% 1000°C	La2Ti2O7 700°C	La2Ti2O7-MCM-4120% 700°C
3441.68 cm $^{-1}$	3439.89 cm $^{-1}$	1640.04 cm- ^{1}	1419.16 cm $^{-1}$	1104.33 cm $^{-1}$	3434.23 cm $^{-1}$	3445.86 cm $^{-1}$
1636.04 cm $^{-1}$	1629.38 cm $^{-1}$	1047.15 cm $^{-1}$	1116.90 cm $^{-1}$	788.20 cm $^{-1}$	1628.42 cm $^{-1}$	1636.17 cm $^{-1}$
1085.83 cm $^{-1}$	1386.18 cm $^{-1}$	803.01 cm $^{-1}$	872.77 cm $^{-1}$	553.03 cm $^{-1}$	1485.04 cm $^{-1}$	1078.15 cm $^{-1}$
926.97 cm $^{-1}$	1139.11 cm $^{-1}$	643.38 cm $^{-1}$	539.50 cm $^{-1}$	458.21 cm $^{-1}$	1384.28 cm $^{-1}$	818.51 cm $^{-1}$
799.10 cm $^{-1}$	664.29 cm $^{-1}$	465.88 cm $^{-1}$	458.13 cm $^{-1}$		775.89 cm $^{-1}$	465.88 cm $^{-1}$
465.87 cm $^{-1}$	540.76 cm $^{-1}$				663.51 cm $^{-1}$	
	463.55 cm $^{-1}$					

It can be seen from the above table that the FT-IR spectrum of the 20% $La_2Ti_2O_7$-ZMCM-41nanocomposite in the three temperatures of 700, 900 and 1000˚C is differ from $La_2Ti_2O_7$ Nano powder structure in these temperatures and MCM-41 zeolite due to the essential variations in the crystal lattice. Consequently, this led to change the lattice formation energy and the unit crystalline energy and finally change the tensile and transfer frequency of each bond in the structure.

https://dx.doi.org/10.22161/ijaers.5.6.12 ISSN: 2349-6495(P) | 2456-1908(O)

FUNCTIONAL GROUP	FRENQUENCY(cm^{-1})	INTENSITY
Water OH stretch	3700-3100	Strong
Alcohol OH stretch	3600-3200	Strong
Carboxylic acid OH Stretch	3600-2500	Strong
N-H stretch	3500-3350	Strong
≡C−H stretch	~3300	Strong
=C−H stretch	3100-3000	Weak
−C−H Stretch	2950-2840	Weak
−C−H aldehydic	2900-2800	Variable
Stretch	~2250	strong
C≡C stretch	2260-2100	variable
C=O aldehyde	1740-1720	strong
C=O anhydride	1840-1800, 1780-1740	weak, strong
C=O ester	1750-1720	strong
C=O ketone	1745-1715	strong
C=O amide	1700-1500	strong
C=C alkene	1680-1600	weak
C=C aromatic	1600-1400	weak
CH_2 bend	1480-1440	medium
CH_3 bend	1465-1440, 1390-1365	medium
C−O−C stretch	1250-1050 several	strong
C−OH stretch	1200-1020	strong
NO_2 stretch	1600-1500 and 1400-1300	strong
C−F	1400-1000	strong
C−Cl	800-600	strong
C−Br	750-500	strong
C−I	~500	strong

REFERENCES

[1] Juang L, wangC, Lee C. (2006).ChemesPhere. 64:1920-1928.

[2] LiJl, (2011). powders prepared by stearic acid gel method, Boston. USA.

[3] lFisc-her.H.(2002) .MaIer.Sci.F.ng.C23.7r,3-772.

[4] LcBnronP.C,(1999).Applied Clay Science. 15: 11-29.

[5] Kirck o, (1997). Encyclopedia of chemical technology :,21,104-109

[6] BrcckDW (1974).Zeolite molecular sieves , structure , chemistry and use John wiley sons,10:127-1327-RamonF,

[7] Rodrigacs E, Deana L, (1984). Zeolite science and technology natoasi series, 80:1-35.

[8] DZA,(2010). Annual Meeting Dulch Zeolite, eliminate and arsenate removal from water polluted usingraw and modified A,X.Y and clinopliolite zeolites. Nclherland.

[9] Dwycr J. Dycr A, (1984). chemistry and Industry, 2: 237-240 .

[10] TrasnkiO,OhsnnaT,ctnl. (1993).chcmmater.5:452-458.

[11] Onozukak, Kawakamiy, lmaiH, yokoiT ,TatsumiJ.n.Kando, (2012).Jornalsalid state chemisteri, 192: 8-92.

[12] AraújoR.S, Costa F.S, Maia D.A.S,Sant`H.B,Ana and C.L.Cavalcante Jr. (2007). Brazilian Journal of Chemical Engineering. 24: 135 – 141.

[13] Miguel A.H, Gabriela I.H, Roberto P, Martha A.S, Fernando R,Vitalii P. (2017). Journal of Nanomaterials, 10.

[14] Mehdi R,Saeid M, Kourosh A. (2017). Journal of Springer Science+Business Media New York .

[15] Haifengshi, Xiukai Li, Defawang, Yupeng Yuan, Zhigang Zou, Jinhua ye,(Springer) 16-July 2009, (205-212).

[16] DhineshR, Kumar R. (2014). Jayavel. Springer. 27:3953-3961.

[17] Hairui L, Haifa Z, chunjie H, Jien Y, Zhiyung L. (2017). Nanoscale Research Letters, 10: 1-10.

[18] Ina U, Shaukat A, Mohammad A, Ali Sh. (2012). International Jouarnal of chemical and Biochemical sciences 15: 60-77.