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Microwave Assisted Sol Gel Synthesis of Magnesium Oxide(Mgo)

*Anjali Maurya[1], Nanda Bhatia[2]
[1]*Dr.H.S. Gour Central University, Sagar, India*
[2]*B.L.P.Govt.P.G.College Mhow,D.A.V.V. Indore*

ABSTRACT: Magnesium oxide (MgO) nanoparticles have been synthesized by Microwave assisted Sol gel synthesis method by using the precursors citric acid ($C_2O_4H_2$) and magnesium chloride ($Mgcl_2.6H_2O$). It is a simple, novel and cost effective method. The structure, morphology and crystalline phase of the magnesium oxide nanocrystals have been investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD).Presence of functional groups and optical characters are analyzed by using FTIR and UV- visible techniques.

Keywords: Microwave assisted, Crystalline, Nanoparticles, X Ray Diffraction

I. INTRODUCTION

Magnesium oxide is one of the most representative materials among the oxides for its unique physical and chemical properties and for its technological applications. For example, this material is very stable thermally. In addition, MgO has high melting point (2850 ºC) and high boiling point (3600ºC)[1-5]. Magnesium oxide is one of the most useful ceramic materials. Because of its high melting temperature it is usually used in fire-resisting bricks or crucibles .In its bulk state, MgO is a highly ionic compound and a wide band gap (~ 7 eV) insulator. For small nanoparticles of MgO, a reduction in the band gap could be measured by using optical absorption techniques [6-7].

Magnesium oxide, or magnesia, is a white solid mineral that occurs naturally as Pericles. It exhibits a rock salt structure like oxides of other alkaline earth metals with space group Fm $\underline{3}$m, No. 225.It has an empirical formula of MgO. It is formed by an ionic bond between magnesium and oxygen atoms. Magnesium oxide is hygroscopic in nature and care must be taken to protect it from moisture. The magnesium oxide is very suitable material for insulation application due to their low heat capacity and high melting point. It is nonmagnetic insulator with a negligible electronic interaction with Fe, showing no significant oxidation-reduction reaction at the interface. MgO is soluble in water and ammonia, but it is insoluble in alcohols. MgO appears as white to grey powder. MgO plays a very prominent role based on surface properties. In the field of catalysis, magnesium oxide has promising applications as both catalyst and catalyst support in many organic reactions due to their adsorbent tendency with an enhanced surface area and with their intrinsically high surface reactivity [8-11] Magnesium oxide has wide applications in the detection and remediation of chemical waste and warfare agents [12],also used as an additive in heavy fuel oil[13],in paints, in refractory products and superconductor materials based on its versatile basic properties[14-16].

There are several methods for the synthesis of nano-sized MgO particles including the sol-gel method [17], chemical gas phase deposition [18], laser vaporization [19], hydrothermal synthesis [20], and combustion aerosol synthesis [21]. The morphology and properties of the resulting MgO differ and depend on the synthesis route and processing conditions. Microwave-assisted synthesis has attracted much attention because it has advantages of being faster, simpler, and more energy efficient [22-23]. In the microwave-assisted sol-gel method, the precursor solution is irradiated by a microwave source. The efficient energy transfer can result in a rapid heating process. Furthermore, microwave heating can result in the homogeneous heating of the precursor solution in a rather short time to achieve a uniform distribution of particle size.

II. EXPERIMENTAL

All chemicals are analytical grade and are used as received without further purification. For the synthesis of magnesium oxide 31.5g of citric acid ($C_2O_4H_2$) and 10.15 g of magnesium chloride ($Mgcl_2.6H_2O$) were dissolved in distilled water in a beaker. The pH of the resultant mixture was found to be 2. To obtained a homogeneous solution it was stirred for 30 minutes at a temperature of 60^oC on a magnetic stirrer .The solution was evaporated to dryness by exposing it to microwave for 8 minutes at power level 100%(800W).The material swells into a white colored gel which was dried. The product obtained was grinded and kept for calcination in a crucible at a temperature of 800^oC in a muffle furnace for 2 hrs. On calcinations an off white colored residue was obtained which was grinded in mortar and pestle to make a fine powder. The product obtained (fig 1) was kept for characterization.

Figure 1 Synthesized nanocrystalline Magnesium oxide

2.1Characterization of nanoparticles

UV-VIS absorption spectra of pure MgO were recorded using Vis-Cary 5E model spectrometer in the wavelength range 200 – 800 nm by dissolving the nanosamples in deionized water. FTIR spectra of magnesium oxide was recorded in KBr pellets using Shimadzu (model 8400S) spectrophotometer from 4000 cm^{-1} to 400 $cm^{-1.}$ **.** Raman spectrum was recorded On BRUKER RFS 27 Spectrometer. The phase and crystallinity were characterized by using a Bruker D8 Advance Cu Kα radiation (= 1.5406A˚,Rigaku Geiger Flex X-ray diffractometer).In order to analyze the structure and morphology of synthesized materials, scanning electron microscopy (SEM) measurement was carried out using JSM-7600FSEM instrument. TEM imaging was carried out with a Philips CM-200-Analytical transmission electron microscope working at 120kV.

III. RESULTS AND DISCUSSION

UV-VIS spectrum is shown in **figure 2.**The absorption spectra of pure MgO were recorded using Vis-Cary 5E model spectrometer in the wavelength range 200 – 800 nm by dissolving the nanosamples in deionized water. The spectra were recorded for IR, visible and UV region. A UV spectrum shows absorbance around 215 nm and 260 nm. From the spectrums it is observed that the sample shows very low absorption over entire IR and visible regions. This shows the optical transparency of the synthesized materials. Negligible absorption in the region between 300 to 900 nm is usual observation for oxide nanomaterials.

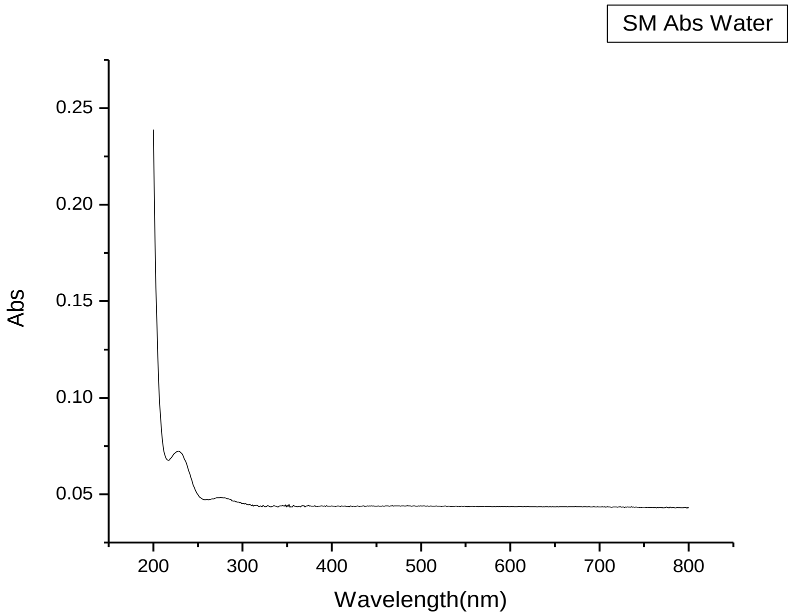

Figure 2 UV-VIS Spectrum of magnesium oxide

FTIR spectra of magnesium oxide was recorded in KBr pellets using Shimadzu (model 8400S) spectrophotometer from 4000 cm^{-1} to 400 cm^{-1}. IR spectrum of magnesium oxide is shown in figure **3.**The spectrum clearly shows one broad band with a splitting around 3000-3700 cm^{-1}. These peaks corresponds to the symmetric and asymmetric stretching of water molecules [24].The presence of these peaks occurs due to the special characteristics of MgO, which may absorb and adsorb moisture from the environment. Peaks localized at 1641,1629,1612 cm^{-1} and peaks at 1357,1384 cm^{-1} positions in table are assigned to asymmetrical and symmetrical stretching vibration of carboxylate (O-C=O).This may be due to the presence of minor impurity of the precursors used during the synthesis process. The absorption bands at, 860 875, 817 cm^{-1} are contributed to the characteristic absorption peaks of cubic MgO [25-26].

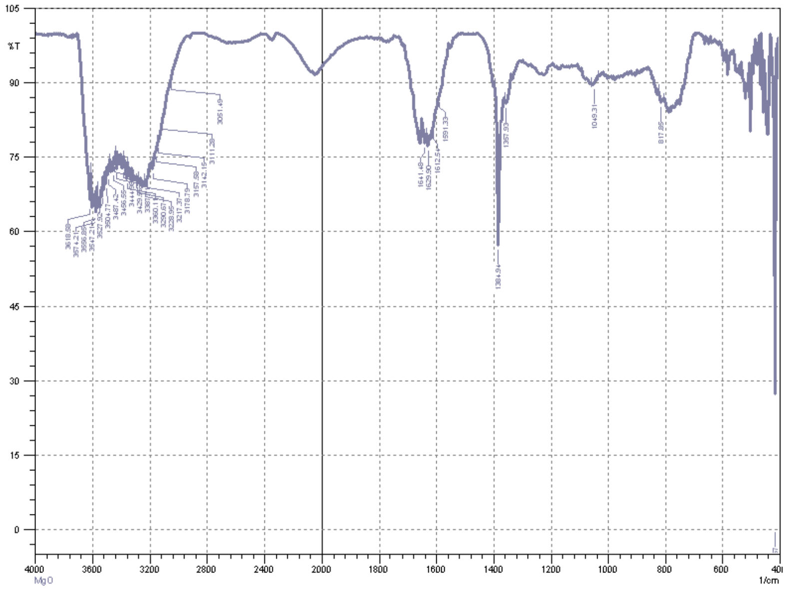

Figure 3 IR Spectrum of magnesium oxide

The Raman spectrum of magnesium oxide is shown in the figure **4.**Raman spectrum was recorded On BRUKER RFS 27 Spectrometer. A peak with splitting is observed around 2000 cm^{-1}. This may be due to symmetric stretching vibration of cubic MgO. Peaks around 1000 and 1100cm^{-1} have been reported by Beckerman and Schlecht [27]. Variations with respect to Raman band positions have been found in literature and also on the precursors or reactant used.

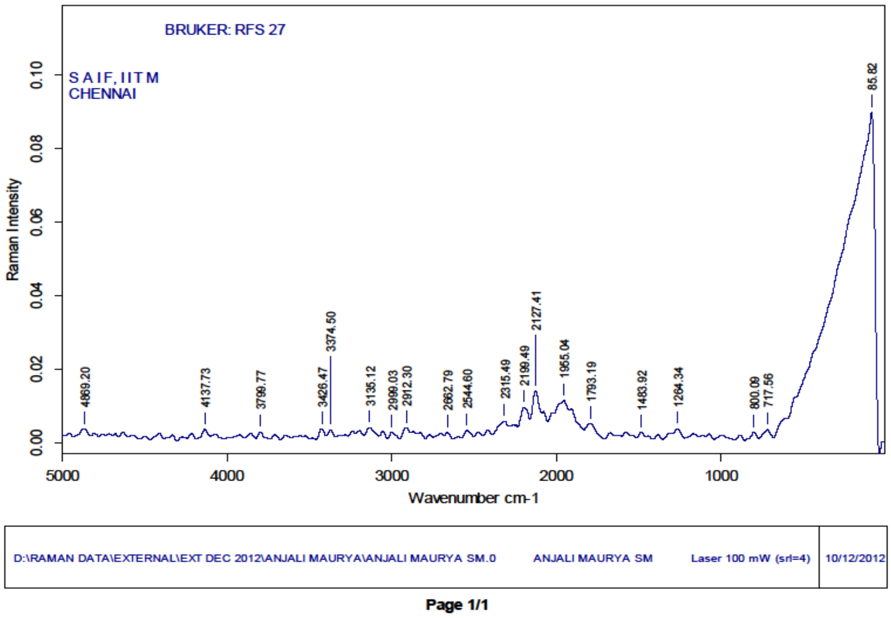

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Figure 4 Raman Spectrum of magnesium oxide

X-ray diffraction studies for the present investigation were performed using a Bruker D8 Advance using Cu Kα radiation (= 1.5406 A˚, Rigaku Geiger Flex X-ray diffractometer). X-ray powder diffraction studies were carried out to confirm the phase of the synthesized material and to determine the particle size by using Scherrer equation. Figure **5.**shows the XRD patterns of the as-synthesized magnesium oxide obtained by heat treatment of the precursors at 800°C for 2 hours. According to the (JCPDS card, No. 45-0946), the phase of the powders obtained from magnesium precursor can be indexed to the cubic MgO structure and the estimated cell constant.“ a” is (a=4.21 A˚) which is consistent with the results reported in the literatures [28-33] The intense peaks show that the powders are highly crystalline and in nano meter range. Average size which was calculated according to the Scherrer formula was found to be **24.6** nm.

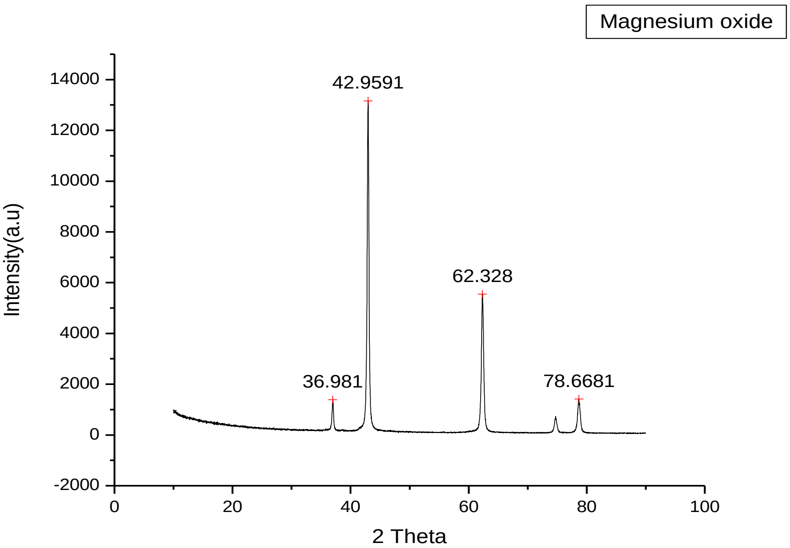

Figure 5 X-ray Diffraction Pattern of magnesium oxide

The scanning electron microscopy (SEM) measurement was carried out using JSM-7600FSEM instrument in order to analyze the structure and morphology of synthesized materials. The SEM micrographs of magnesium oxide calcined at 800^{o}C are shown in Figure **6.**The SEM images shows that the powder is porous and foamy with agglomerates of very fine particles of uniform diameter. In addition, it is noticeable that many pores and voids can also be seen in these SEM images. It is because during the synthesis process the reaction takes place in a very short period with the evolution of lot of gases that generally yields nanosized materials with pores and voids.

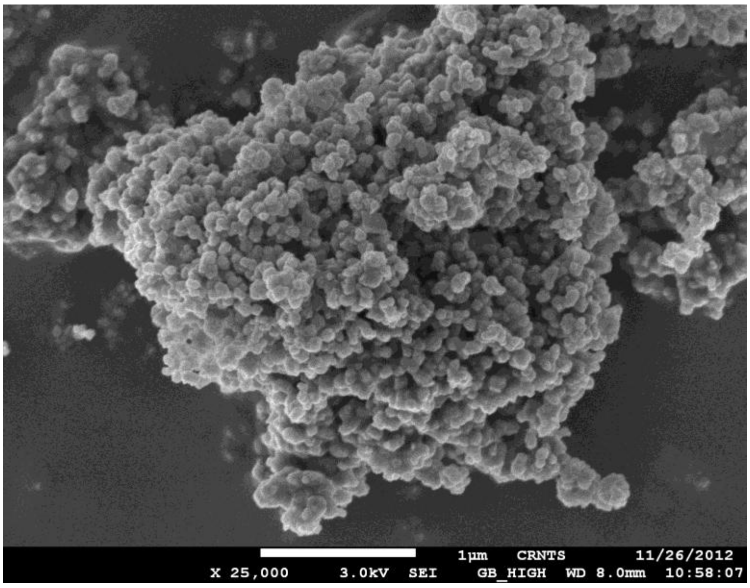

Figure 6 SEM micrographs for magnesium oxide

TEM imaging of the powder samples is the most direct and convenient method to see and analyze the structure of aggregates and to determine the size of particles. TEM imaging was carried out with a Philips CM-200-Analytical transmission electron microscope working at 120kV. The powder samples were supported on conventional carbon-coated film on copper grid. Particle size of the samples was directly found out and structure of aggregates was analyzed using TEM image, in the present study. The shape, size of the MgO nanopowder was confirmed by TEM images as shown in **Figure7A.** Hard agglomerates of MgO nanocrystalline were formed during the synthesis process. TEM micrograph of MgO, reveal that the particles are approximately spherical in shape of about diameter of **57.5 nm**. In the figure**7B** the well-defined selected area electron diffraction (SAED) pattern shows spotty rings characteristic of polycrystalline pattern, suggesting that the as prepared powder MgO is nanocrysta1line.Discontinuous rings with spots indicate that the particles are made of rather bigger crystallites. There is difference in the size of particles obtained from TEM compared to the crystallite size obtained by XRD. This can be attributed to differences of accuracy of measurements of the two different techniques. The XRD pattern cannot see minor differences that can be observed by TEM technique.

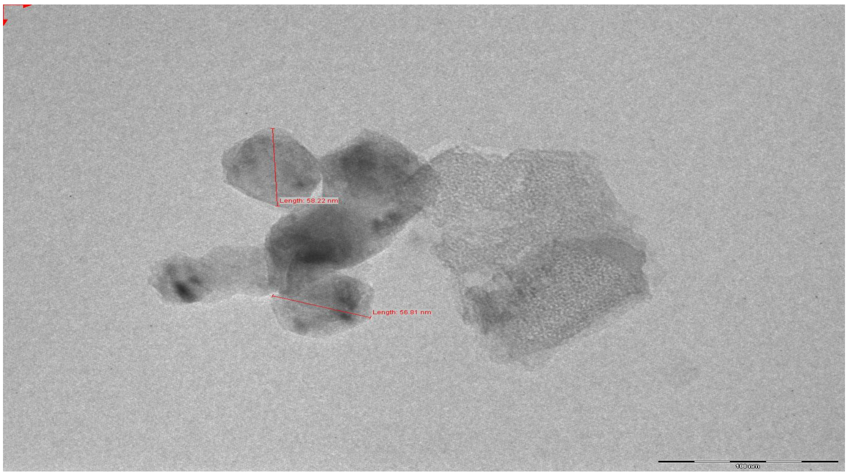

Figure7 A. TEM Images of magnesium oxide

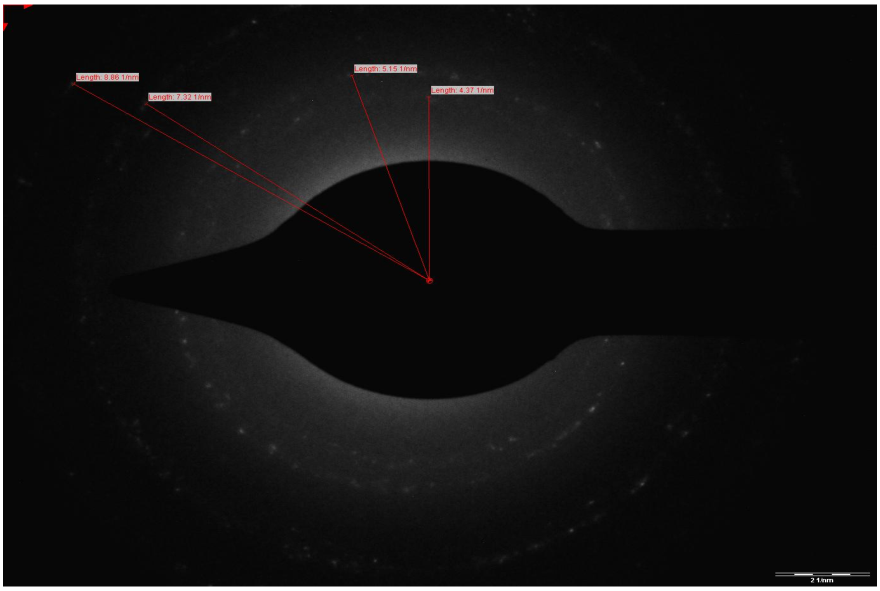

Figure 7 B. TEM (Selected area electron diffraction pattern) of magnesium oxide

CONCLUSIONS

Fine MgO powder was synthesized using citrate precursors derived from magnesium chloride and citric acid resulting to pure cubic MgO. The results obtained proved that nano MgO could successfully be prepared via microwave assisted Sol gel method. UV–Vis spectrum observed indicates absorption in UV region and no absorption in the visible region confirming optical transparency of the synthesized material. FTIR spectrum for the synthesized samples, were recorded and the vibrations of the synthesized samples were studied. The absorption bands at, 860 875, 817 cm^{-1} are attributed to the characteristic absorption peaks of cubicMgO.XRD characterization showed pure cubic crystallites of MgO nanoparticles with the average particle size of 24.6 nm. According to the (JCPDS card, No. 45-0946), the phase of the powders obtained from magnesium precursor can be indexed to the cubic MgO structure. The quality of the nanocrystals could be visualized by surface morphology using SEM studies. The SEM images shows that powder is porous and foamy with agglomerates of very fine particles of uniform diameter. The shape, size of the MgO nanopowder was confirmed by TEM images. TEM micrograph of MgO reveals that the particles are approximately spherical in shape of about diameter of 57.5 nm. The well-defined selected area electron diffraction (SAED) pattern shows spotty rings characteristic of polycrystalline pattern.

REFERENCES

[1]. P. Luches, S. Benedetti, M. Liberati, F Boscherini, I. I. Pronin, S. Valeri, Surf. Sci., 583, **(2005)** 191G. Bilalbegovic, Phys.Rev, B, 70 **(2004)** 45407

[2]. J. Q. Li, Y. J. Xu, Y. F. Zhang, Solid State Communication, 126, **(2003),** 107

[3]. E. Florez, F. Mondragon, T.N. Truong, and P. Fuentealba., Phys. Rev., B73, **(2006)** 115423

[4]. V. Kvachadze, G. Dekanozishvili, V.Vylet, M. G Galustashvili, Z. Akhvlediani, N Keratishvili, and D Zardiashvili, Radiat. Eff. & Defects in Solids, 162**(2007**) 17

[5]. Fernández-García, M.; Martínez-Arias, A.; Hanson, J.C.; Rodríguez, J.A. Chem. Rev. 104(**2004**) 4063.

[6]. A.M. Föller, Magnesium Oxide and its Applications; Vollhardt: Berlin, (**1978).**

[7]. H. Hideshi, Chem. Rev. 95**, (1995)** 537

[8]. B. Q. Xu, J. M. Wei, H. Y. Wang, K. Q. Sun, and Q. M. Zhu, Catal. Today 68**, (2001**)217

[9]. O. B. Koper, I. Lagadic, A. Volodin, and K. J. Klabunde, Chem. Mater. 9 **(1997).** 2468

[10]. K. P.McKenna, D. Koller, A. Sternig, N. Siedl, N. Govind, and P. V. Sushko, ACSNano5,**(2011**) 3003

[11]. Wagner G W, Bartram P W, Koper O, Klabunde K J. J Phys Chem B, 103(1999)3225

[12]. Davini P, Tartarelli R. Fuel,64**(1985)** 380

[13]. S. Rajagopalan, O. Koper, S. Decker, and K. J. Kiabunde, Eur. J. Chem.8, **(2002)**2602

[14]. C. M. Boyapati, R. S. Mulukutla, and K. J. Klabunde, J. Am. Chem. Soc.125, **(2003).** 2020

[15]. J. V. Stark and K. J. Klabunde, Chem. Mater. 8 **(1996)** 1913

[16]. B Q Xu, J M Wei, H Y Wang, K Q Sun, Q M Zhu. Catal Today,68**(2001**) 217

[17]. J S. Matthews, O Just, B Obi-Johnson, Rees W S. Chem Vapor Depos,6**(2000**), 129

[18]. M S El-Shall, W Slack, W Vann, D Kane, D Hanley. J Phys Chem,98**(1994)**3067

[19]. Y Ding, G T Zhang, H Wu, B Hai, L B Wang, Y T Qian. Chem Mater,13**(2001**)435

[20]. J JHelble. J Aerosol Sci,29**(1998)**721

[21]. T Krishnakumar, R Jayaprakash, M Parthibavarman, A R Phani, V N Singh, B R Mehta. Mater Lett, 63**(2009)**896

[22]. Davoodnia A, Rahimizadeh M, Atapour-Mashhad H, Tavakoli-Hoseini N. HeteroatChem, 20**(2009)**346

[23]. B. ManojGawande, S Paula. Branco, KalpeshParghi, J.JanhaviShrikhande, Rajesh Kumar Pandey, C. A. A. Ghumman, N.Bundaleski, O. M. N. D. Teodoro and V.RadhaJayaramCatal. Sci. Technol.1**(2011**) 1653

[24]. Pei et al. Materials Research. 13**(2010)** 339

[25]. Abdul Rahim Yacob, MohdKhairul Asyraf Amat Mustajab, and NurSyazeilaSamadi World Academy of Science, Engineering and Technology 56 **(2009**)

[26]. H.K. Bockelmann, R.G. Schlecht Physical review 10 **(1974)** 5225

[27]. K Chhor, JF Bocquet and C PommierMaterials Chemistry and Physics. 40**(1995)** 63

[28]. L Znaidi, K Chhor and C Pommier. Materials Research Bulletin. 31**(1996)** 1527

[29]. E Alvarado, LM Torres-Martinez, AF Fuentes and P Quintana. Polyhedron. 19**(2000)**2345

[30]. Y Ding, GT Zhang, H Wu, B Hai, LB Wang and YT Qian..Chemistry of Materials. 13**(2001)**435

[31]. Xubin Pan, Iliana Medina-Ramirez, Ray Mernaugh and Jingbo Liu, Colloids and Surfaces B: Biointerfaces.77 **(2010)** 82

[32]. A.I.Y Tok, F.Y.C, Boey X. L Zhao,.J.Mater.Process.Tech 178(**2006**) 270.

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