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Aqueous chemical route synthesis of Mn_2O_3 and investigation on the effect of calcination temperature

T. Kamali[1], Amal George[1], A. Dhayal Raj[1], A. Albert Irudayaraj[1]*

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Abstract

The novel reflux method has been exploited to synthesize manganese oxide nanoparticles. The prepared material is calcinated at two different temperatures (300°C and 500°C) and it is subjected to X-ray diffraction (XRD), UV-vis techniques (UV-vis), Fourier transform infrared spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). The XRD pattern reveals an increase in crystallite size of the prepared samples with the increasing calcination temperature. An increase in calcination temperature resulted in the increase of bandgap values. The different chemical bonds on these samples were identified from their FTIR spectra. Reduction of agglomeration were also noticed while increasing the calcination temperature which is confirmed from their respective SEM analysis.

Keywords: Mn_2O_3, reflux method, optical properties, calcination temperature.

1 Introduction

Manganese oxides are having more applications in wide fields such as rechargeable batteries, catalysis, ion-sieves, microelectronics and chemical sensing devices, waste water treatment, supercapacitors [1,2]. The magnetic properties of manganese oxide attract research interest to prepare manganese oxide nanoparticle due to its various magnetic alignment and higher in the intrinsic high atomic moment of Mn [3]. Especially MnO and MnO_2 are used as an anode material in lithium-ion batteries for their high theoretical capacity, environmental benignity, inexpensive and superior properties [4]. Electrode materials are plays predominant role in the lithium-ion batteries. Manganese oxide nanoparticles have electrochemical properties for energy storage due to their various phases [5].

Manganese belongs to transition metals group and it has different oxidation sates. Such Oxides can be used in wide range of technology applications. Yeow Hong Yap (6) et.al., have been prepared copper manganese oxide nanoparticles and they have reported that a catalyst copper manganese oxide is used to remove unstable organic compounds and such greenhouse gases for example carbon monoxide. Vineet Kumar (7) et.al., have reported the manganese oxide nanoparticles with ~4nm size are used as an electrochemical sensor for para-nitrophenol. They have studied from the results that manganese oxide nanoparticles are used as a sensor to detect very harmful chemicals and also useful in many biological applications. They also noted that in targeted drug delivery, size is reduced to a nanometre are very good delivery of the drug on a regular basis. S.Ganeshan (8) et.al.,. have been reported that manganese dioxide nanoparticles are having great capacity to eradicate all the pollutants. They stated that the adsorption mainly depends on the effects of pH and contact time parameters. They have also described that manganese dioxide nanoparticles are having great capacity to eradicate all the pollutants. Y. F. Shen (9) have synthesized manganese oxide octahedral molecular sieve which is thermally stable belongs to natural todorokite prepared under such alkaline conditions with reactions between Mn^{2+} and MnO_4^-.

In this work, manganese oxide is prepared from novel reflux method. The aim of this study is to reveal the structural, morphological vibrational and optical properties of the material. The chemical analysis was performed with XRD (X-Ray Diffraction), FTIR (Fourier Transform Infrared Spectrometer), SEM (Scanning Electron Microscope) and UV-Visible analysis.

2 Experimental

2.1 Materials

The materials were used to synthesis Manganese oxide nanoparticles are Manganese acetate tetrahydrate, Potassium permanganate, Cetyl Trimethyl Ammonium Bromide, Ethanol and double distilled water were used to prepare the solutions.

2.2 Experimental Method:

The manganese oxide nanoparticles were synthesized by reflux method. The 50ml of ethanol dissolved in the

*Corresponding author: e-mail dhayalraj@gmail.com,

[1]Department of Physics, Sacred Heart College (Autonomous), Tirupattur District, Tamil Nadu, India
[1]Department of physics, Sacred Heart College (Autonomous), Tirupattur District, Tamil Nadu, India.

50ml of distilled water. The required amount of manganese acetate tetrahydrate was taken and added into the dissolved ethanol, stirred for 10 minutes. The required amount of $KMno_4$ and Cetyl Trimethyl Ammonium Bromide were added and stirred for 20 minutes. The prepared solution was kept at reflux setup with constant temperature 100°C and continuously stirred for 6 hours. The solution was washed with distilled water for several times. The solution was dried and the black colored precipitate was obtained. The obtained precipitate was calcinated at different temperatures of 500°C and 300°C for 1 hour. The precipitate was ground well using mortar to make a fine powder.

3 Results and Discussion

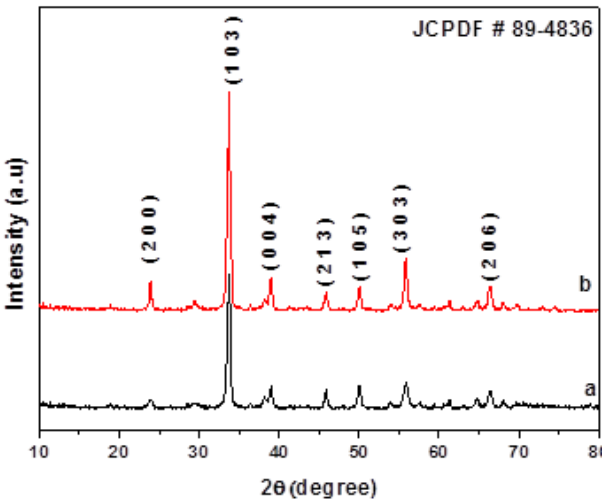

Fig. 1. XRD pattern of Mn_2O_3 samples prepared with a)300℃ b)500℃

X-Ray Diffraction technique is used to determine the crystallinity, phase and purity of the prepared samples [10]. The diffraction peaks at 2θ values matching with the hkl values indicated the formation of Mn_2O_3 excellent crystallinity. The structure and crystallinity of the grown nanoparticles were investigated by XRD. Fig. 1 (a and b) shows the XRD patterns of the samples calcinated at 300ºC and 500ºC respectively. The XRD patterns shows the sharpening of the peaks with increasing calcinations temperature which indicates the increase in crystallite size [2]. The peak assigned to diffractions from various planes correspond to cubic structure of Mn_2O_3. The peaks match with JCPDS Card 89-4836 with good increase in peak intensity which might be due to the increasing calcination temperature. The crystallite sizes were calculated using Scherrer's equatio n given below.

$$D=k\lambda/\beta\cos\theta$$

Where, d is particle size in manometer, λ is the wavelength of the radiation,

k is constant equal to 0.94, β is the full width at maximum intensity and θ is peak position[11]. The size of the crystallites obtained from XRD patter are 19.23nm for the sample calcinated at 300ºC and 23.44 nm for the sample calcinated at 500ºC. Thus, it is concluded that the increase in calcinations temperature increases crystalline nature of the sample along with increase in crystallite size.

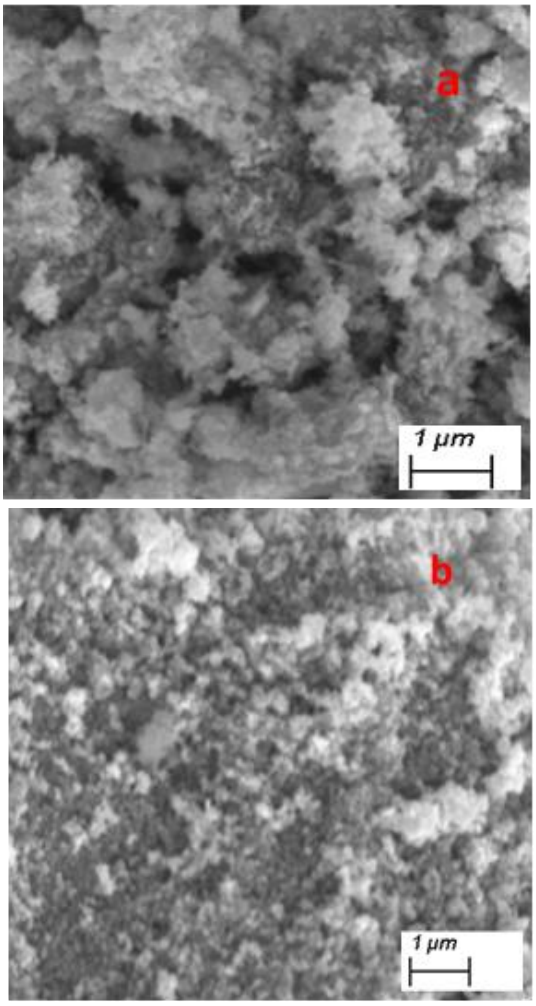

Fig 2: SEM images of Mn_2O_3 samples prepared with a)300℃ b)500℃

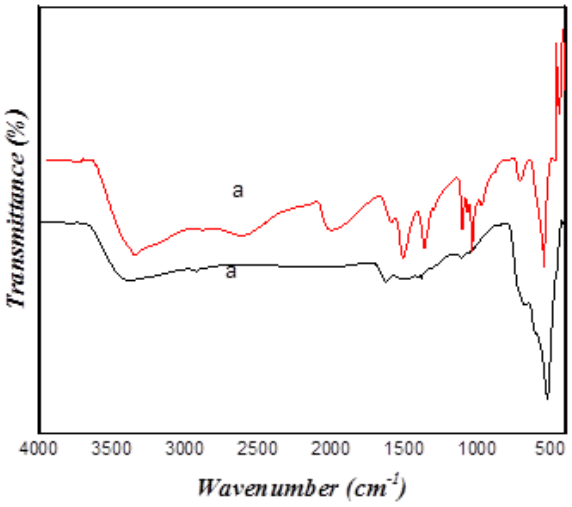

Fig 3. FTIR spectra of Mn_2O_3 nanoparticles calcinated at (a)300ºC and (b) 500ºC

The SEM images of Mn_2O_3 samples prepared with a)300°C b)500°C are depicted in Fig. 2 (a and b) respec-

tively. It is highly noted from the pattern that while increasing the calcination temperature of the synthesized material there is reduction in agglomeration as can be seen from fig. 2. The increase in calcination temperature is expected to have removed the surfactant traces left behind in the sample. Fig. 3 show FTIR spectrum of prepared sample Mn_2O_3 nanoparticles. Both the FTIR spectra show vibrational peaks around 623.21 cm^{-1} and 498.21 cm^{-1}confirm the formation of Mn_2O_3 nanoparticles [12]. The band at 623.21 $cm^{-1}$is characteristic of Mn-O vibration while the band at 498.21 cm^{-1}can be attributable to Mn-O-Mn vibration in cubic structured manganese oxide [13, 14]. Fig. 3. shows a fewer intensive band at 1640cm^{-1} and 3400 cm^{-1} due to O–H stretching vibrations. When the sample is calcinated at higher temperature, there is no possibility of the traces O-H. However, we witness weak traces of these peaks even in the sample calcinated at 500[15]. This clearly depicts that some moisture would have adhered to the KBr during sample preparation [16]. However, the other peaks that may have raised from the surfactants seem to vanish on increasing the calcination temperature as can be seen from fig.3b.

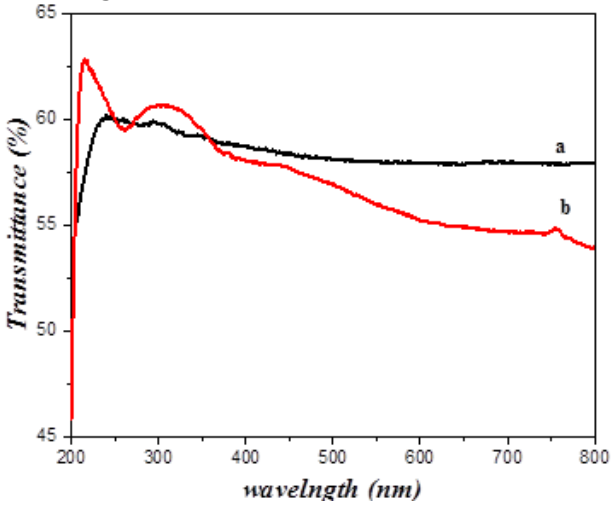

Fig. 4 Optical spectra of Mn_2O_3 nanoparticles calcinated at (a) 300ºC and (b) 500ºC

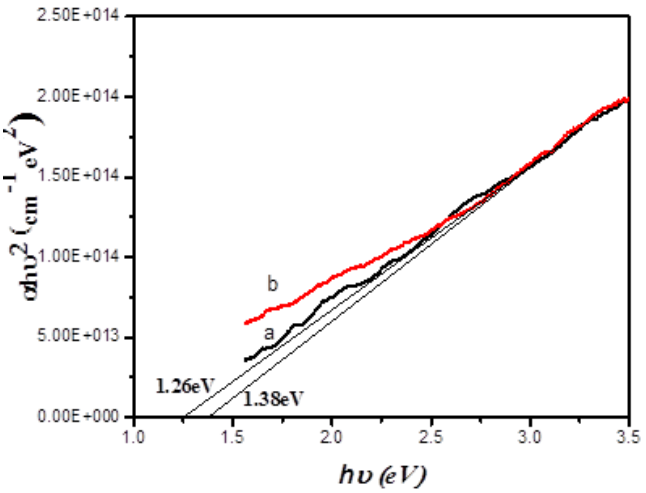

Fig.5 tauc plot of Mn_2O_3 nanoparticles calcinated at (a)300ºC and (b) 500ºC

The transmittance spectrum of the prepared Mn_2O_3 nanoparticles calcinated at (a) 300ºC and (b) 500ºC are shown in fig 4 (a and b). It is understood from the graph that the sample calcinated at 300°C shows more transmittance in IR region when compared to the sample calcinated at a higher temperature of 500°C. A tauc graph is plotted to find out the energy of the prepared manganese dioxide nanoparticles and it is represented in fig.5 [17]. The sample prepared in the presence of CTAB and calcinated at 300°C shows a band gap of 1.26eV and 1.38eV for the sample prepared at 500ºC

4 Conclusion

The Mn_2O_3 nanoparticles were prepared by reflux method by changing calcination temperature. This reflux method was chosen among other preparation methods, since uniform heating and fast reaction rate is achieved in this. This will result in good morphology growth of the material. The morphology of the prepared sample was studied using SEM images. It was found that the pure Mn_2O_3nanoparticles samples showed a reduction in agglomeration when increasing the calcination temperature. The structure of the prepared sample was concluded from XRD pattern as cubic crystal system and a considerable amount of peak shift was observed which may be correlated with increasing calcination temperature. The prepared Mn_2O_3nanoparticles samples showed a good absorption in the entire visible region. This was observed from the UV-Vis transmission spectra, it was concluded that the material exhibits good transmission nature.

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