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Synthesis and Characterization of ɣ-Fe_2O_3 Nanoparticles Photo anode by Novel Method for Dye Sensitized Solar cell

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Abstract- Pure ɣ-Fe_2O_3nanoparticles are prepared by photo irradiation method using complexes solution of iron and successfully used as a photo anode for dye-sensitized solar cells (DSSCs). The samples were characterized by using powder XRD, SEM, TEM, UV-Visible and Current-Voltage curve. Results show that nanoparticles have uniform cubic shape with average size of 17 to 26 nm and it is applied to fabricate novel Dye solar cell which has energy conversion efficiency about 4.13 % by (ITO/ Ɣ-$Fe_2O_3Np_s$/ Dye (Rhodamine 6 G) /iodine / Ag film / ITO).

I. Introduction

Energy is one of the most challenging needs of mankind, and is highest on the list of priorities and requisites for human welfare. According to the International Energy Agency (IEA), World's primary energy demand will increase by 36% between 2008 and 2035. Electricity demand is expected to grow by 2.2% per year between 2008 and 2035. Taking in account the carbon dioxide emissions and the global climate change impact on life and the health of the planet. Renewable energy sources will have to play a central role in moving the world onto a more secure, reliable, and sustainable energy path(1,2). Solar energy is the most inexhaustible, abundant and clean of all the renewable energy resources till date. Dye solar cell has been attracted much attention because it low cost, possible fabrications of flexible devices, and relatively efficient devices for the conversion of solar energy(3). In order to develop Dye solar cell with excellent photovoltaic properties, researchers have tried to control the nanostructure and morphology of metal oxides photo-electrodes(4). Iron oxide nanoparticles is one of the important oxides, which has many of applications (5-13). There are many forms of iron oxide crystalline phases, which include: Hematite α-Fe_2O_3,β-Fe_2O_3, Maghemiteγ-Fe_2O_3, Epsilon Fe_2O_3andMagnetite (Fe_3O_4). γ-Fe_2O_3 is a red-brown (14). compound and is the second most stable polymorph of iron (III) oxide. The band gap of bulk γ-Fe_2O_3 is 2.22 eV and 2.43 eV for the nano crystalline form. Of the known Fe_2O_3 polymorphs, γ-Fe_2O_3 is regarded as the most practically important and useful due to its simple synthesis and the interesting magnetic characteristics of its nano sized objects (15). γ-Fe_2O_3 occurs naturally in soils as a weathering product of Fe_3O_4, to which it is structurally related (16).

II. EXPERIMENTAL

All chemical were used without any purification, Irradiation cell, fig (2-1) was used to irradiate complexes of iron as sources of iron oxide nanoparticles. Immersed UV source (125 W mercury medium pressure lamp) was used with maximum light intensity at 365 nm. The cell contains a quartz tube as a jacket for immersion UV source in the complex solution of iron. Pyrex tube was used as a reactor. The reactor was cooled by ice bath to avoid the rising in temperature as a result of the UV irradiation.

A **B**

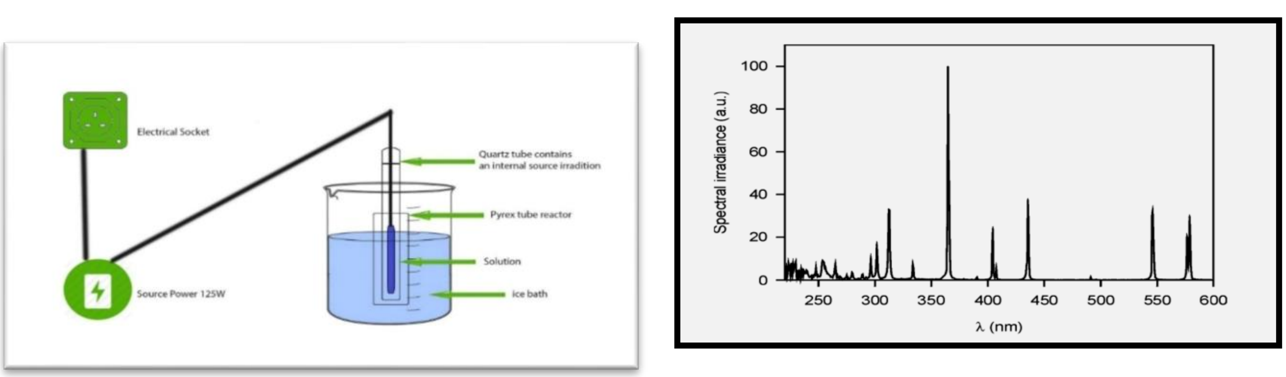


Fig (2-1) **A:** photo irradiation cell, **B:**uv.visble spectrum of medium pressure mercury

Synthesis of ɣ -fe$_2$O$_3$ nanoparticles

1-using ethylene glycol with iron (III) nitrate (F1)

0.6 g of $Fe(NO_3)_3$ is dissolved in 20 ml ethylene glycol and is stirred 30 min at 40 C°. then, the solution is irradiated by photo cell for 30 min Using with chilling under 5 C° . A yellow – brown precipitate ofiron (II) complex is obtained and the product is filtered and washed several times with acetone and deionizes water. The precipitate is dried in an oven at 60°C for 4 h and calcined at 450°C for 3 h. A red-brown magnetic precipitate is obtained.

2-using Salicylic *acid* with iron (III) nitrate (F2).

1.38 g of Salicylic acid is dissolved in 10 ml methanol and 1.21 g of $Fe(NO_3)_3$ is dissolved in 10 ml methanol, The two solutions were mixed and stirred for 30 min. then, it has been irradiated by photo cell for 30 min with chilling under 5 C° . A red precipitate of iron (II) complex has obtained and the product has filtered and washed several time with acetone and deionize water. The precipitate has been dried in an oven at 60°C for 4 h and calcined at 450°C for 3 h. A red-brown magnetic precipitate has been obtained.

Fabrication of dye sensitizer solar cells[17,18]

Indium doped tin oxide, (ITO) coated class (resistant 8 ohm, transmission 83%) washed with acetone, ethanol, and distilled water several times in ultrasonic bath to remove impurities then dried using air blower. A dye-sensitized solar cell (2 X 1 x 0.1 cm) was fabricated according to the following process:-

A colloidal solutionofɣ-Fe_2O_3 nanoparticles was prepared by mixingiron oxide nano powder with ethanol. The photo anode was obtained using dropper to cover colloid solution on the conductive side of the glass and then annealed at 200 °C for 30 min in air. After being cooled, ɣ-Fe_2O_3nanoparticles electrode was immersed into 0.5 mMrhodamine 6G Dye for 24 hours at room temperature. The counter electrode was silver nano film coated on ITO glass by Vacuum thermal evaporation technique. Sensitized iron oxide nanoparticles (photo anode) and the sliver nano film coated (counter electrode) were assembled the liquid electrolyte (I^- /I^{-3})solution drop penetrated into the working space via capillary action, the produced cell was held with a binder clip as shown in Fig (2-2)

A B

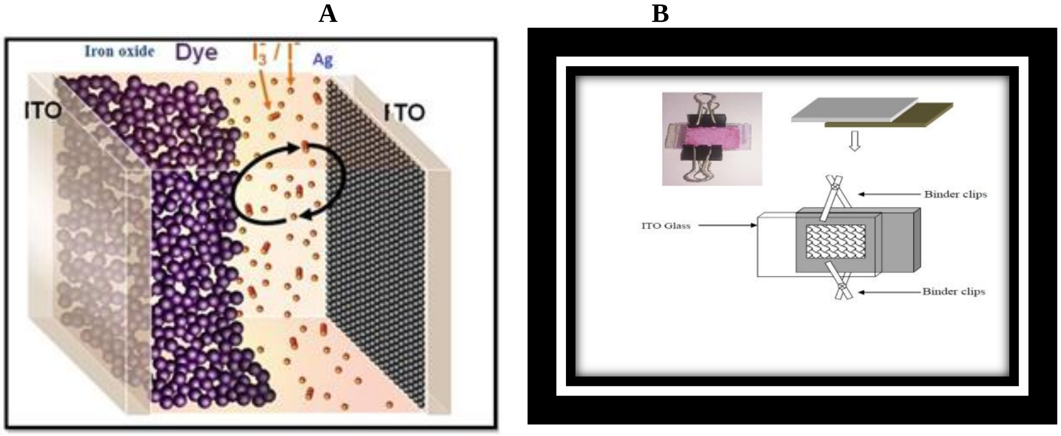


Fig (2-2) A:Schematic of typical dye sensitizer solar cell,B: design of dye sensitizer solar cell.

Photocurrent –voltage behaviors of the DSSCs

The photocurrent–voltage of the DSSC with ɣ-Fe_2O_3 nanoparticles are characterized by the open circuit voltage (Voc) and the short-circuit current (Isc) of each sample measured at I=0 mA and V=0 V. In addition the power conversion efficiency of the solar cell was determined by:-

$$\eta = \frac{Pmax}{Pin} = \frac{Vos*Jsc*FF}{Pin} * 100 \qquad (2\text{-}1)$$

where V_{oc}, J_{sc}, P_{in} represent the open-circuit photo voltage, the short-circuit density photocurrent and the incident light power respectively. Furthermore the fill factor is given by :-

$$FF = \frac{Vmax*Jmax}{Voc*Jsc} \qquad (2\text{-}2)$$

Where V_{max} and J_{max} represents the voltage and the current density at the maximum output power point respectively[17,18].

III. RESULTS AND DISCUSSION

Fig (3-1) shows the XRD patterns of the samples calcined in air at 450 °C.ɣ-Fe_2O_3 can be assigned to cubic crystal phase according to the standard XRD patterns (JCPDS 39-1346)

F1 F2

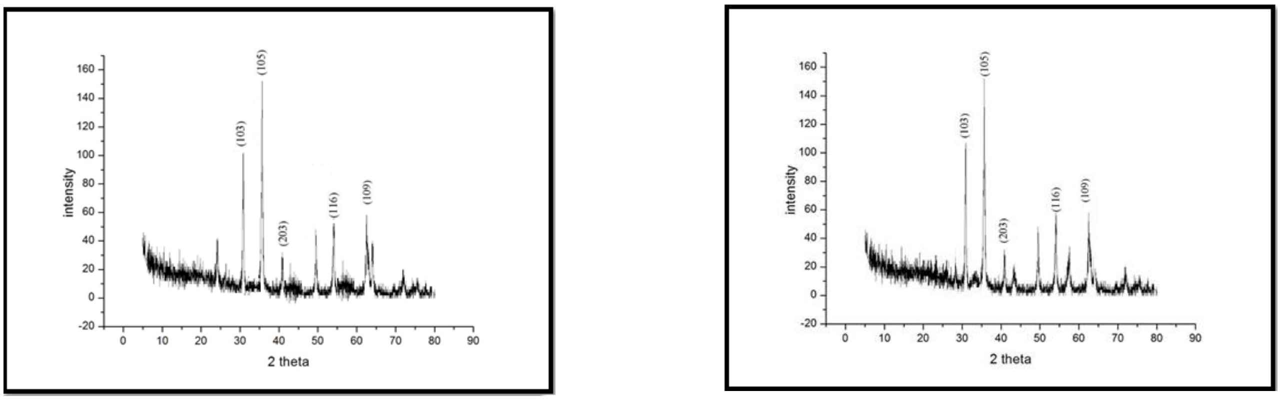


Fig (3-1) XRD pattern of Ɣ-$Fe_2O_3Np_s$ prepared by F1 and F2

There are significant amounts of broadening lines which are characteristic of nanoparticles. The crystal size can be calculated according to Debye-Scherrer formula [19]

$$D = \frac{k\ \lambda}{\beta COS\ \theta} \qquad (3\text{-}1)$$

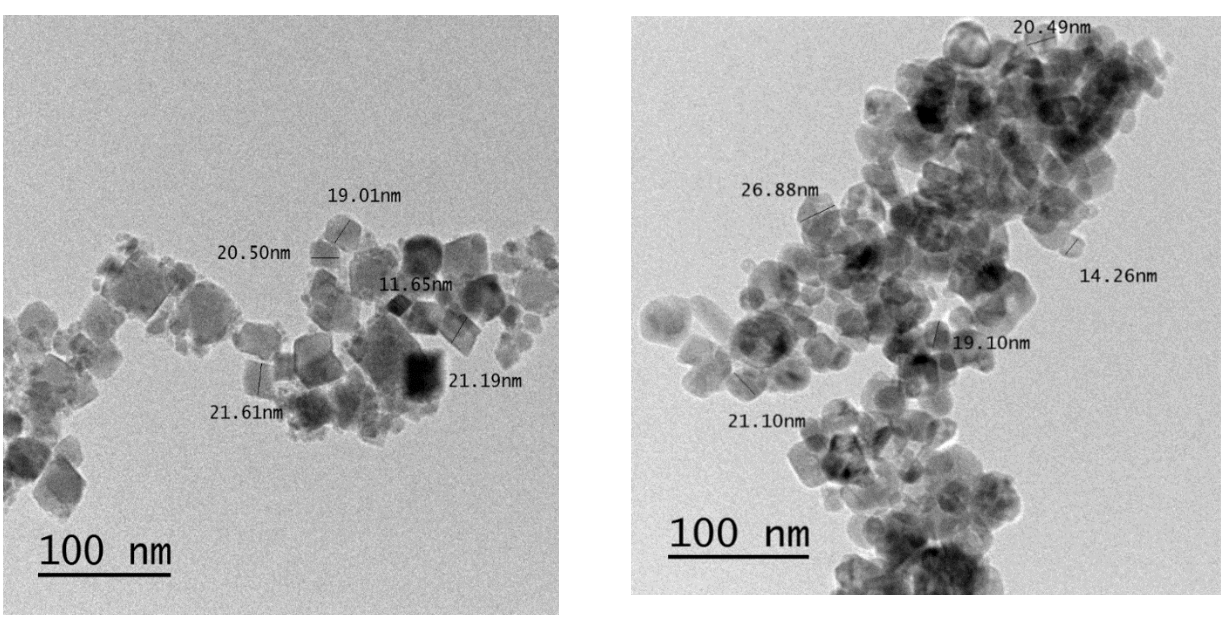
Where k=0. 9 scherrer constant, λ is the wavelength of the Cu-Kα radiations, β is the full width at half maximum and θ is the angle obtained from 2θ values corresponding to maximum intensity peak in XRD pattern. The mean crystal sizes of nanoparticles are listed in table (3-1).

Table (3-1) crystal size and crystal system of Ɣ-$Fe_2O_3Np_s$as calculated from Scherrer formula

sample	phase	Crystal system	2θ (deg)	crystal size (nm)
F1	Ɣ-Fe_2O_3	cubic	35.51	24.4
F2	Ɣ-Fe_2O_3	cubic	35.57	26.2

The diffraction peaks corresponding to (210),(211),(220),(221),(310),(311) are quite identical to characteristic peaks of theƔ-Fe_2O_3 crystal with the cubic spinal structure.The application of Scherrer's formula to the (311) reflection peak indicated the formation ofcubic Ɣ-Fe_2O_3 .The morphology and particles size were determined by TEM. The mean particle size and distribution were determined randomly on the TEM image, fig (3-2) shows TEM image of the samples.

F1 F2



(3-2) TEM images of γ-Fe_2O_3NPs prepared by F1 and F2

The mean particles sizes were obtained from TEM images listed in table (3-2).

Table (3-2) average particle size from TEM images

sample	phase	ave. Particles size (nm)
F1	γ-Fe_2O_3	18.8
F2	γ-Fe_2O_3	20.5

SEM images, fig (3-3) show The morphology and size distribution of γ-Fe_2O_3nanoparticles.The surface of nanoparticles is smooth with good crystallinity. The average particle size and distribution were determined randomly on the SEM images.

F1 F2

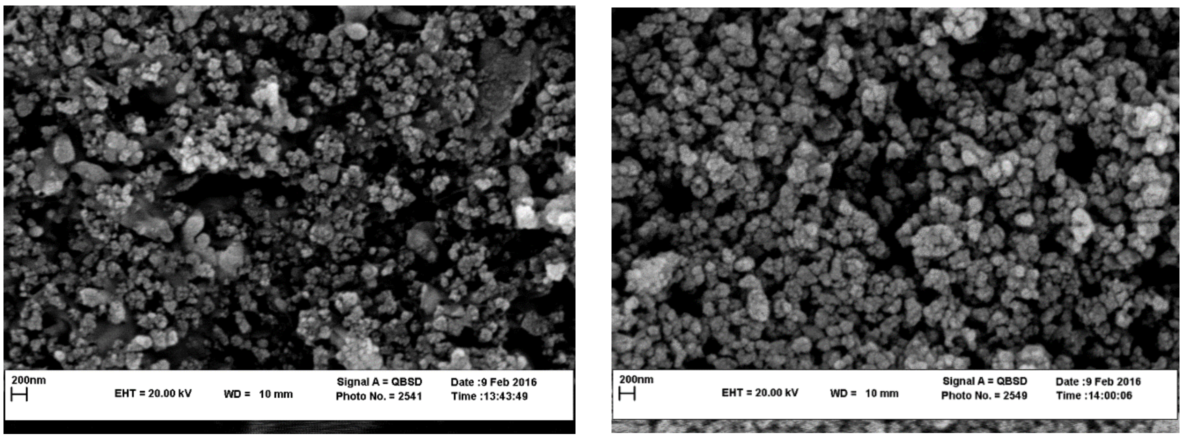


Fig (3-3) SEM images of γ-Fe_2O_3 NPs prepared by F1 and F2

The mean particles size were obtained from SEM images listed in table (3-3).

Table (3-3) Average particle size from SEM images

sample	phase	ave. Particles size (nm)
F1	α-Fe_2O_3	16.6
F2	α-Fe_2O_3	17.2

The UV–vis spectra of γ-Fe_2O_3Nano film was used to determined energy gap (Eg) by edge of absorption of Nano film. The energy gap (Eg) is determined by the formula,

$$Eg = \frac{1240}{\lambda} \qquad (3\text{-}2)$$

Where λ(nm) is the wavelength of the absorption edge in the spectrum. The energy gaps of samples, show mostly blue shift and exhibit distinct synergistic effects.

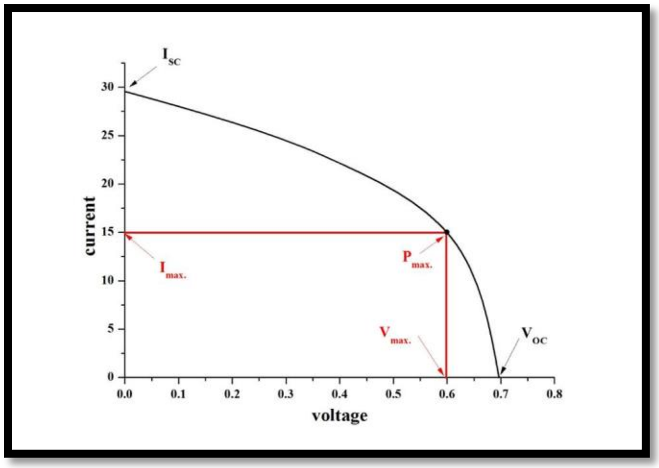
Table (3-5) shows the values of energy gab using eq. (3-2).From these result we conclude that there are increasing in the band gap of the nanoparticles with the decreasing in particle size and it may be due to a quantum confinement effect[20].

Table (3-5) Band gap and absorption edges of γ-Fe_2O_3 NPs.

sample	phase	Absorption edge (nm)	gap energy (eV)
F1	γ-Fe_2O_3	481	2.58
F2	γ-Fe_2O_3	494	2.51

Photovoltaic properties of dye-sensitized solar cells

Fig (3-4) shows the photovoltaic properties of DSSCs based on Ɣ-Fe_2O_3NPs as a working electrode. Table (3-6) summarizes the photovoltaic parameters of sample has 4.13 % energy conversion efficiency. From the I-V characteristics it is noticed that our DSSC has high the short circuit current and high open circuit voltage. This is because of the molecular structure (favorable for the separation of electron/hole pairs)and the diffusion rate of redox electrolyte.



(3-4) photovoltaic properties of the DSSC prepared by Ɣ-Fe_2O_3NPs

Table (3-6) The parameters of dye sensitizer solar cell
(ITO/ Ɣ-Fe_2O_3Nps/ Dye (Rhodamine 6 G) /iodine / Ag film/ ITO)

phase	**V_{oc} (V)**	**J_{SC} (A\cm^2)**	**V_{max} (V)**	**J_{max} (A\cm^2)**	**P_{max} (W\cm^2)**	**FF**	**η %**
Ɣ-Fe_2O_3	0.702	0.014	0.59	0.007	0.0041	0.420	4.13 %

IV. Conclusion

1- Photo Irradiation process is a suitable method for preparing Ɣ-Fe_2O_3 nanoparticles and it is a novel method for this propose.

2-The systematic analysis for the evaluation the structural properties of synthesized nanoparticles was studied by XRD, SEM, TEM, and UV visible , these techniques prove that the nanoparticles size was less than 29 nm.

3- the fabricated solar cell was suitable for :
(ITO/ Ɣ-$Fe_2O_3Np_s$/ Dye (Rhodamine 6 G) /iodine / Ag film/ ITO).

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