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Fabrication and Catalytic Applications of Iron Oxide Thin Films on FTO using Ferrocene as Source of Iron

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Abstract

This research is based on fabrication of iron oxide thin films on fluorine doped tin oxide (FTO) substrate having (dimensions 40 x 40 x 2mm) using ferrocene precursor by applying pyrolysis method. We employed ferrocene precursor and prepared a sample slide denoted as S-1, To ensure even dispersion of iron oxide thin films with clear morphology and controlled particle size distribution, which effectively increases the surface area of the FTO substrates and thus catalytic ability. Thin film was fabricated using accurate masses of ferrocene (0.125 g) in small crucible, finally ferrocene was thermally decomposed at 500 °C for 30 minutes in an electric furnace. The Scanning Electron Microscopy (SEM), was brought into action to analyze surface morphology of thin films, which confirmed a successful homogeneous distribution of iron oxide thin film on FTO. Energy Dispersive X-ray Spectroscopy (EDS) was used to investigate elemental analysis, which authenticated existence of iron (Fe) and oxygen (O) as principal elements, verifying enhanced purity and productive adsorption of iron oxide onto the FTO substrate. Finally, catalytic activity of thin film (S-1) was investigated through Methylene Blue (MB) dye degradation in the presence of sunlight, exhibiting their significant potential for environmental decontamination and catalytic activity. Sample slide S-1 showed catalytic ability with 40% effective degradation.

Keywords: Iron Oxide Thin Films; Ferrocene; Photocatalyst

Introduction

The nanotechnology sometimes also called nanoscience, is an advanced science which deals with the study of synthesis, characterization and applications of nanoparticles, nanowires, nanocomposites, nano films and nano-rods. The particles owing size between 1 nm to 100 nm are considered as nanoparticles or nano-materials. The word Nano refers to extremely small, nearly billionth part (Bohlom, 2016, Mdkour, 2019). On structural grounds, the nano materials are classified into four types, zero dimensional including quantum dots, nanospheres, nanoclusters, frequently used in bio imaging, drug delivery, digital displays. One dimensional including nano-rods, nano-tubes, nano-fibers, nano-wires have emerging applications in photovoltaic devices, sensors, photvoltaic devices. Two-dimensional nano materials include nnao sheets, graphene, hexagonal boron nitride (BN) consumed in

membranes, flexible electronic electronics and transparent electrodes. Three dimensional nanomaterials includes aerogels, nano porous materials, nanocomposites are employed in catalysis, filtration, energy storage (Nersisyan et al., 2017, Singh et al., 2020).
Among variety of diverse properties of nanomaterials because of their, large surface area, excellent conductivity, ability to distribute uniformly on transparent conducting glass substrates, as photocatalyst for organic wastes, ability to vary their electric conductivity by doping and changing band gaps, their tendency to easily be customize make them distinct and can be utilized in desired applications using modern innovation methods and experimental setups (Zhang et al., 2018, Zhu et al., 2013). The nano-materials can show their potential properties in number of ways including nano-particles, nano-rods, nanofilms. The chemical stability, inertness and decomposing abilities of certain elements vary when they are at nanoscale or at macroscale, copper is opaque in bulk while transparent in nano form. Similarly, platinum shows extraordinary catalytic ability at nano scale and almost inert at macroscale (Mahar et al., 2021).
In recent past, metal oxide nano particles are finest semiconducting materials regarding the ease with which they can be fabricated and their utilization in various fields (Campbell et al., 2020). Iron oxide nano-materials can exhibit their potential application in the form of nano-particles, nano-tubes, nano-rods or thin films, our research is based on iron oxide thin films over fluorine doped tin oxide (FTO). From the beginning of 20^{th} century, Iron oxide thin films are effectively used for the excellent magnetic, catalytic and semiconducting characters (Aronniemi et al, 2024). These films have attained noticeable attention in recent past because of their diverse roles, like sensors, photocatalysis and energy accumulation devices (AKL et al, 2004). These films are also highly interesting due to their distinctive and excellent physical and chemical properties also promising scientific and technological applications (Kumar et al, 2014). The number of methods are used to fabricate iron oxide thin film hydrothermal process, electron beam evaporation, pulse laser deposition, solvothermal process, wet chemical process (Kukli et al, 2012). We used most simple, pyrolysis method using organic precursor ferrocene, because of easily availability, its simplicity, cost-effectiveness and tendency to produce thin films with ambitious properties. Various conducting glass substrates are available in market such as Indium Tin Oxide (ITO), Aluminum doped zinc oxide (AZO), Tin oxide (TO), Zinc oxide (ZO), Gallium doped zinc oxide (GZO) but Fluorine doped tin oxide (FTO) semiconducting glass is an emerging substrate for assembling Iron Oxide thin films, owing to excellent electrical conductivity, chemical stability and excellent optical transparency. These properties make FTO transparent glass as an ideal and distinctive platform for increasing performance of iron oxide thin films in various applications. This research aiming at, facile fabrication of iron oxide thin films over FTO transparent glass substrate using simple pyrolysis method. This research emphasizes on the synthesis of the transparent glass substrate, the fabrication process followed annealing processing to improve crystallinity and performance of fabricated film. The ferrocene precursor choice was simply based on the fact that, it organic compound and easily decompose over 400^{O}C in an electric furnace, producing high quality iron oxide fabricated films and with controlled morphology and excellent photocatalytic properties. Thin films made from iron oxide showed good photocatalytic ability in decomposing organic contaminates.

Materials and Method

Materials

Chemicals used in this research were ferrocene (purchased from Sigma Aldrich), ethyl alcohol, glycerin, ethylene glycol, ammonium hydroxide, sodium chlorate, sodium hydroxide, distilled water. For experimentation, extra pure water and only laboratory grade chemicals were utilized.

Method

In entire research work extra, pure water was used. Prior to fabrication of iron-oxide thin films on FTO, the glassware to be utilize were dehydrated in microwave oven at 95 ^{0}C. For deep cleaning, FTO glass before fabrication, the FTO glass was sonicated above 40 kHz frequency using 2-proanol and deionized water solvents to eliminate mineral and organic residue. The synthesis of iron-oxide thin can be achieved by simple pyrolysis of ferrocene by heating it above 400 ^{0}C in an electric furnace.

Fabrication of Iron oxide nanofilms over FTO

After sonication, washing and drying, 0.125g of active Ferrocene precursor was taken in crucible and its surface was occupied by FTO glass having insulating side in downward and conducting side in downward direction. The crucible was placed in an electric furnace and heated at 500 ^{0}C for 30 minutes as a result, reddish brown colored this film of iron oxide was deposited over FTO. After depositing iron oxide film on FTO glass, the film was annealed to enhance its crystallinity and remove any residual stress. Finally annealing of thin iron oxide film was performed at 750°C for 10 minutes using electric furnace, which results a uniform dark brown thin layer deposition of iron oxide.

Table 1. Iron Oxide Thin Film Formation Using Ferrocene Precursor

Composites	**Specimen codes**	**Original slides**
Formation of Iron-oxide thin film by decomposing 0.125g of $Fe(C_5H_5)_2$	S-1	

Synthesis scheme of Iron Oxide thin films by pyrolysis of ferrocene

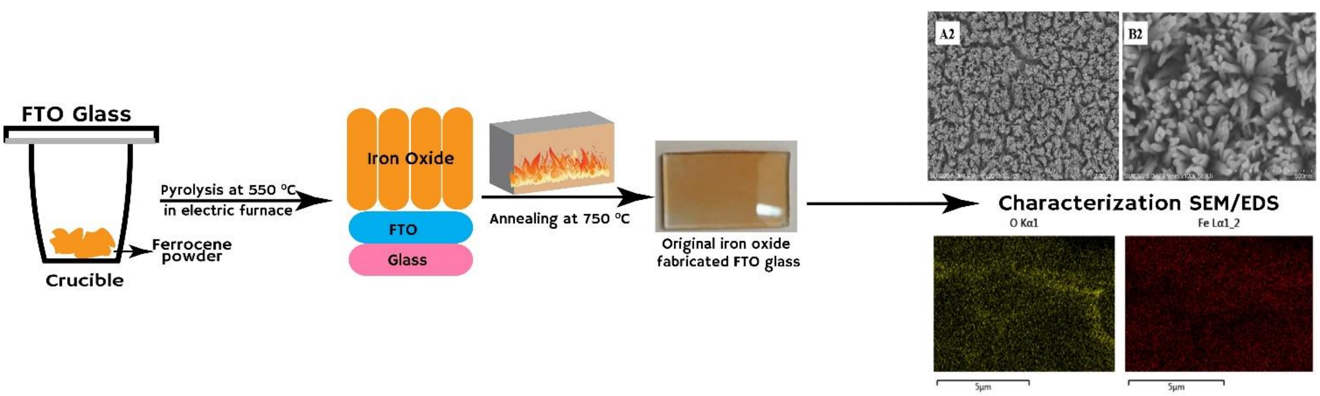

Figure 2. Scheme of formation of iron oxide nano-film on FTO substrates

Dye Degradation

To observe catalytic efficiency of iron oxide thin film, the breakage of commercial Methylene Blue (MB) dye was investigated in sunlight. In this experiment the iron oxide thin film deposited over (FTO) was introduced in a beaker containing methylene blue (MB) dye at the bottom with fabricated surface in upward direction. Magnetic stirrer was used for maximum performance of catalyst and to increase the contact between methylene blue and iron oxide photo catalyst. The UV-Visible absorption peak for methylene blue was observed to 665 nm.

Characterization

Characterization of iron oxide by FTIR

The Fourier-transform infrared (FTIR) spectrum of iron-oxide sample is given in figure 4. The examination revealed absorptions bands in the 3421 and 542 cm^{-1} regions. The absorption range 3600-3000 cm^{-1} is typically concerned with –OH unsymmetrical and symmetrical stretching vibrations. Addition to to 3421 cm^{-1}, the vibrational band observed at 542 cm^{-1} is a result of stretching vibrations of iron-oxygen (Fe-O) bond. This comprehensive examination of FTIR spectrum gives insight into the chemical bonding and structural features of synthesized iron oxide thin films

Morphology of surface

An advanced characterization instrument, the Scanning-Electron-Microscope SE(M) was effectively utilized to analyze the topography of bare Fluorine doped tin oxide (FTO) glass prior to fabrication of iron oxide thin film. SE(M) shows that FTO glass consists of network of interconnected crystals (grains) of poly crystalline fluorine doped tin oxide.

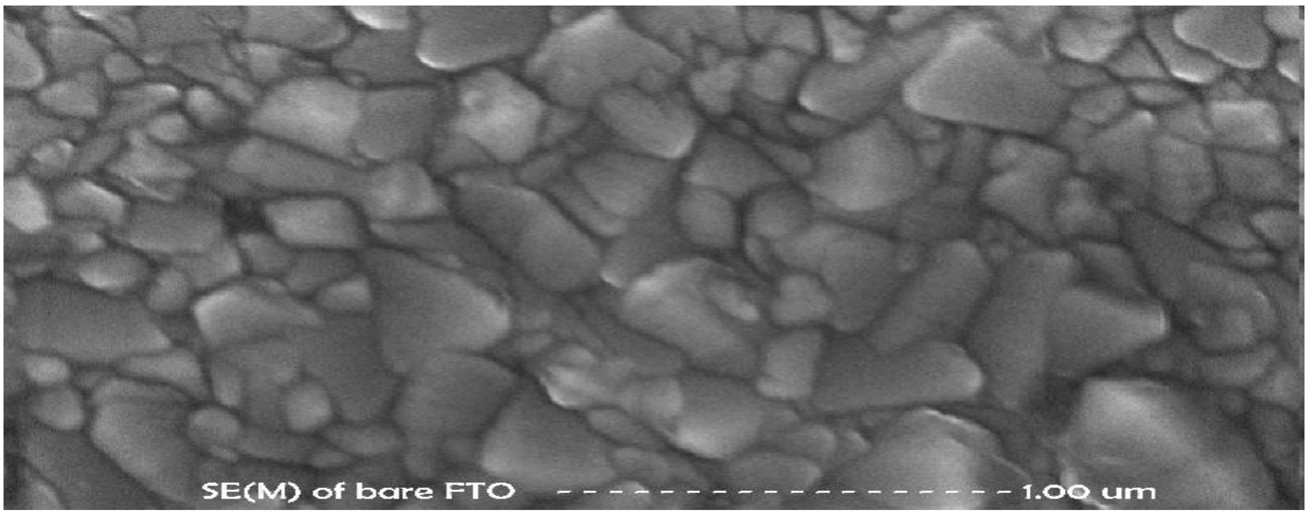

Figure 3. SEM image of bare FTO

Fig. 4, A1 and B1 represents SEM images of iron-oxide thin film fabricated from 0.125 g of ferrocene, on FTO substrates. Two different magnifications were used to capture different images one at low (500 µm) and other at high (1 µm), images clearly justify the presence of porous structure of thin film evnly covering the FTO surface. The micrographs depict scattering and uniform distribution of nanoscale platelets over FTO.

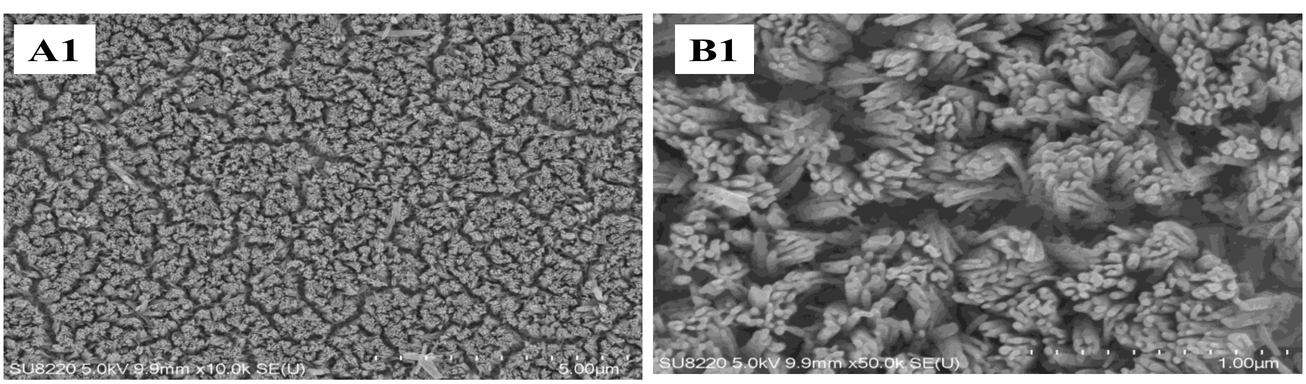

Figure 4. Sample S-1 SEM Images-(A1) Low, (B1) High Resolution

Elemental Examination

Figures 5 and 6 show distribution of Iron (Fe) and Oxygen (O) was investigated by EDS spectrum mapping. The red colour in Fe (L□1) mapping represents the uniform distribution of iron. It is also evenly distributed over iron oxide thin film like O. The green colour in O (K□1) mapping indicates the uniform distribution of oxygen in iron oxide thin film. The uniformity of green colour reveals that oxygen is evenly spread over iron oxide thin film. The mapping of O and Fe is evident of formation of iron oxide over FTO. It also indicates that the fabrication of thin film is almost perfect because of absence of areas of depletion or aggregation.

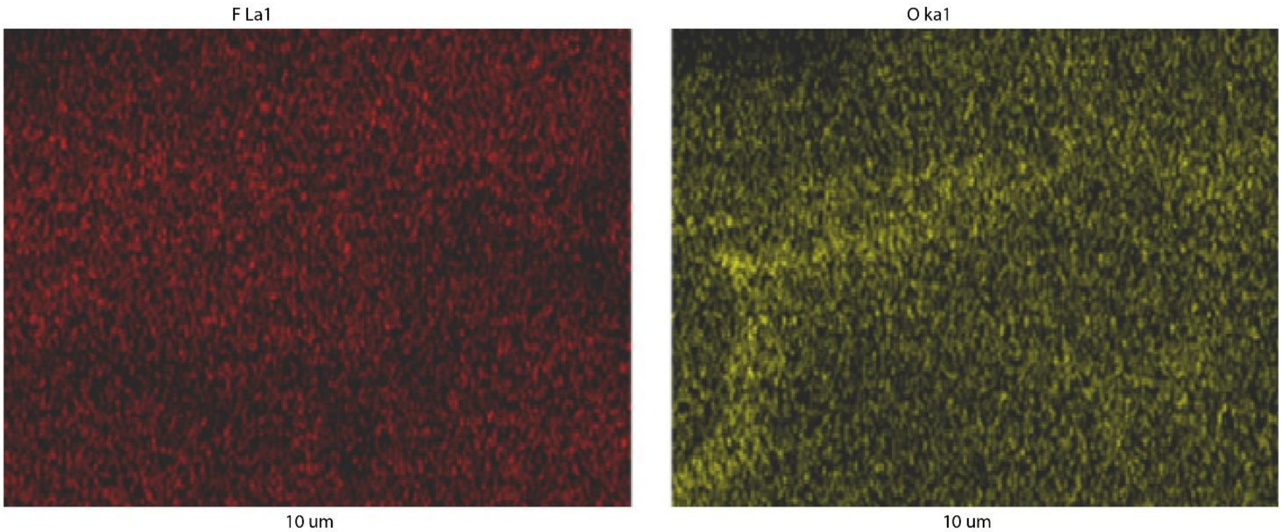

Figure 5 and 6 Elemental Profiling of Thin Film Catalysts Using EDX

The catalytic degradation of Methylene Blue dye was examined using iron-oxide thin film fabricated over FTO substrate in the presence of sunlight as a source of energy. The distinctive peak of the MB dye identified at 665 nm which functioned as an indicator for assessing the degradation process applying UV Visible Spectroscopy. With the lapse of time, color of Methylene Blue (MB) dye diminished gradually pointing out degradation of the dye initiated by iron oxide thin film. Substational changes were seen in the MB dye solution when it was exposed to sunlight for a time period between 0 to 30 minutes.

Figure 7. Photodegradation of Methylene Blue: 30-Minute Sunlight Exposure

Conclusion

In conclusion, the study demonstrates a facile strategy for fabrication of premium iron oxide thin films over FTO glass via simple pyrolysis. The resulting films exhibit uniform morphology and elemental purity, as confirmed by SEM and EDX analysis. Moreover, annealing enhances the films crystallinity and functional properties. This research underscores the potential of these thin films for various applications, highlighting the effectiveness of the pyrolysis approach in fabricating functional materials for diverse technological advancements. The iron oxide thin films were successfully used as photocatalyst in solar decomposition of Methylene Blue (MB) dye.

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