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Synthesis and Characterization of Materials

This chapter describes about the synthesis method and characterization techniques which are adopted during the thesis work. The chapter is composed of five sections and sixteen sub-sections. The section 2.1 describes introduction of the chapter. The procedure of materials synthesis is elaborated in the section 2.2. The physical characterization techniques, their basic principle, magnetic characterization and transmission reflection loss measurements are expressed in the section 2.3. The electrochemical characterization, working electrode preparation method, are presented in the section 2.4 and the section 2.5 covers closing remarks.

2.1 INTRODUCTION

The synthesis method has crucial role in determining chemical and physical properties of material. The sub-section 2.2.1 contains the details about analytical grade chemical utilized to synthesize materials of interest. Several synthesis methods were attempted including sol-gel and hydrothermal. The hydrothermal method is described with schematic flow chart in Figure 2.1. The physical properties are evaluated by employing physical characterization techniques. Electrochemical characterization techniques are executed to evaluate photo electrochemical properties. Magnetic measurements and transmission-reflection loss measurements were performed to assess magnetic and microwave absorption properties respectively.

2.1.1   Analytical Grade Chemicals

The Fluorinated hematite was synthesized via hydrothermal method. The material synthesis was performed using analytical grade chemicals. The details about reagents are tabulated in Table 2.1.

Table 2.1: Analytical grade chemical, formula and supplier

Chemical Description	Chemical Formula	Physical State	Lot/Batch No.	Supplier Product No.
Potassium hexacyanoferrate(III)	$K_3[Fe(CN)_6]$	Solid	1667/1 7108-1	HiMedia Laboratories Pvt. GRM627
Selectfluor (> 95% F^+ active)	$C_7H_{14}B_2ClF_9N_2$	Solid	STBF0638V	Sigma-Aldrich, St. Louis, USA
Uranyl nitrate	$UO_2(NO_3)_2.6H_2O$	Solid	2220710	BDH Chemicals Limited, Poole, England 30553
Iron(III) nitrate nonahydrate	$Fe(NO_3)_3.9H_2O$	Solid	29527111-1	Fisher Scientific 23655
Ferric chloride, anhydrous	$FeCl_3$	Solid	3097-7308-7	Fisher Scientific 23585

Ferrous sulphate	$FeSO_4.7H_2O$	Solid	1167/1 7108-1	Fisher Scientific 15285
Oxalic acid	$(COOH)_2,2H_2O$	Solid	1195-7206-7	Fisher Scientific 19175
Ferrocene	$C_{10}H_{10}Fe$	Solid	T-8341626	SRL Pvt. Ltd. 0648135
N-Cetyl-N,N,N-tri-methyl-ammoniumbromide	$C_{19}H_{42}BrN$	Solid	K93365042 307	Merck 1.2342.0100
Urea	NH_2CONH_2	Solid	G08Z/0807/0205/3 1	S D Fine Chemical Ltd. 20290
Ammonium fluoride	NH_4F	Solid	10199924	Alfa Aesar A16279.22
Hydrofluoric acid 48% GR	HF	Liquid	DK8D48265/B	Merck 334
Sodium fluoride	NaF	Solid	DOA-22770700-31	S D Fine Chemical Ltd. 20246
Hydrogen peroxide	H_2O_2	Liquid	309	Ases Chemical Works
Sodium hexametaphosphate	$(NaPO_3)_6$	Solid	MKBH8729V	Sigma-Aldrich 1001279769
Sodium acetate	$C_2H_3NaO_23H_2O$	Solid	H03Y/0203/3107/31	S D Fine Chemical Ltd 20235
Polyvinylpyrrolidone	$(C_6H_9NO)_n$	Solid	BCBH3938V	Sigma Aldrich 101205545
Deionized water, 18.2 MΩ cm	H_2O	Liquid	Model No: ELIX 10, Synergy UV-R	Merck Millipore
Acetic acid, glacial 100% GR	CH_3COOH	Liquid	SK9SS49283/2	Merck 1/17843
Terpineol 98%	$C_{10}H_{18}O$	Liquid	81301738	Alfa Aesar
Nitric acid	HNO_3	Liquid	2081050617	Fisher Scientific 29757
Hydrochloric acid, 35.4% AR	HCl	Liquid	K08A/0108/1711/31	S D Fine Chemical Ltd 20125
Sodium hydroxide	NaOH	Solid	B02Y/0102/0402/31	S D Fine Chemical Ltd 20252

2.2 SYNTHESIS OF SURFACE FLUORINATED HEMATITE

The successful synthesis of surface fluorinated hematite was achieved by hydrothermal method. However, before attempting hydrothermal method, several other synthesis methodologies have also been tried. The material used, reaction conditions and outcome of the reactions are summarized below.

Table 2.2: Summary of chemical reactions

SNo	Materials used	Reaction condition	Outcome
1.	$FeCl_3 + FeSO_4 + H_2O_2$ (1:2: 5%)	Addition at 0 °C and reaction for 24 hrs. at RT	Fe_2O_3 precipitate and unreacted precursor
2.	Iron Nitrate + NaOH and SHMP, pH ~ 2 by HCl	1 hr. Heating at 100 °C at 500 rpm (Temp., Time and RPM varied) :20 Reaction	Hematite synthesized, However, difficult to control morphology
3.	Iron Nitrate + NaOH and SHMP, pH ~ 2 by HCl	Heating in Teflon vessel	No Change in XRD, FTIR of samples

	and fluorination using HF and NaF		
4.	Oxalic acid and Ferrocene	Crushed and placed in pre heated furnace at 800 °C	Powder form fumes in the furnace and it became red colored
5	Ferric chloride +Sodium acetate + PVP in DI water	HT vessel at 180 °C and 18 hrs.	Hematite formed with contribution of amorphous phase also
6.	Iron Nitrate+ CTAB (CA)+ Urea	HT vessel at 80 °C and 36 hrs.	
7.	Hematite + NH_4F, or HF or NaF or SF	Mixed /heated / Pressed	No sign of Fluorination
8.	$K_3Fe(CN)_6$ and water	0.1 M to 0. 01 M placed in HT vessel at 120 -180 °C for 12 Hrs. to 72 Hrs.	Successful synthesis of hematite. Reproducible morphology and XRD pattern at optimized condition
9.	$K_3Fe(CN)_6$ and water and Fluorinating agents like HF, NaF and NH_4F	HT vessel at 140°C for 48 Hrs.	Very small change in XRD pattern
10.	Selectfluor and $K_3Fe(CN)_6$	HT vessel at 140°C for 48 Hrs.	Changes in XRD peak intensity and XPS Successful synthesis of Surface fluorinated hematite

2.2.1 Hydrothermal Synthesis of Fluorinated Hematite

α-Fe_2O_3 and Surface fluorinated Fe_2O_3 (F-Fe_2O_3) were synthesized using hydrothermal method. The synthesis methodology is displayed in Figure 2.1. Aqueous solution of (15 mM) of $K_3[Fe(CN)_6]$ was heated in Teflon lined hydrothermal vessel at 120 °C -140 °C for 12-72 hrs. However, the reaction was optimized for 140 °C and 24 hrs. The obtained precipitate was washed with de-ionized (DI) water followed by ethyl alcohol wash to get rid of the by-products and un-reacted materials. It was further centrifuged and dried in hot air oven. In-situ fluorination was carried out using Selectfluor™ (N-Chloromethyl-N'-fluorotriethylenediammoniumbis(tetrafluoroborate), F-TEDA, Sigma Aldrich) as the fluorinating agent. F-TEDA was mixed in different weight percent with respect to $K_3[Fe(CN)_6]$, the principal reactant, before the start of reaction. F-TEDA is a commercially available and chemically stable fluorinating reagent that acts as a source of electrophilic fluorine in organic synthesis. In this study, F-TEDA was used for fluorination of α-Fe_2O_3, an in-organic material, owing to its ease of handling high and reactivity as compared to the other sources of fluorine such as HF, XeF_2 and F_2. Synthesis is scaled up from 400 mg to 4-5 g in different-sized Teflon vessels of 100, 200 and 800 mL capacity as shown in Figure 2.2.
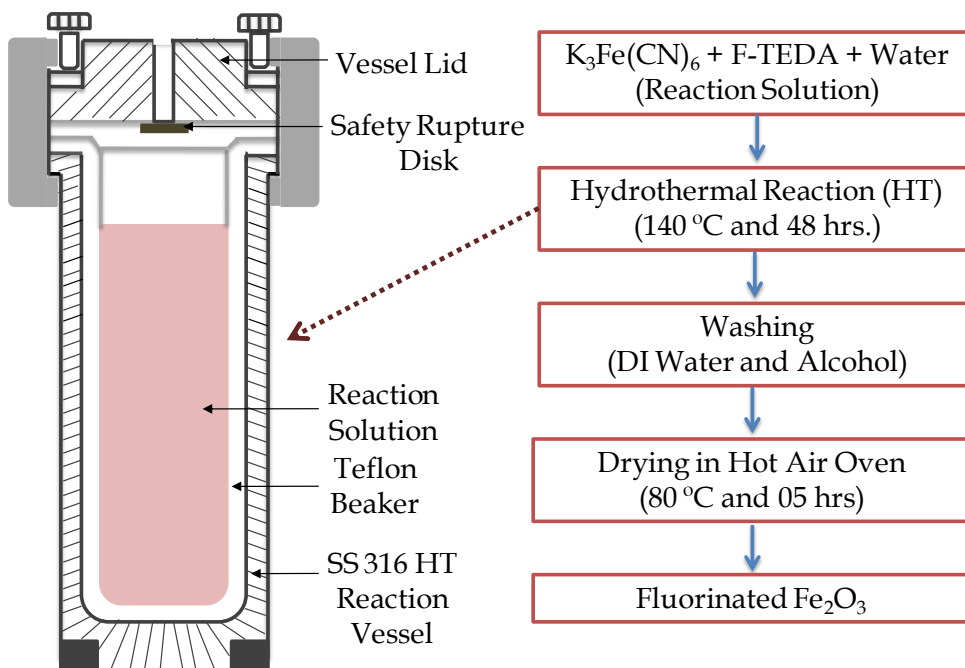
Figure 2.1: Hydrothermal reaction methodology

2.2.2 Chemical Reaction

Fluorinated hematite forms in three steps. Under hydrothermal condition, Firstly $[Fe(CN)_6]^{3-}$ ions resolve slowly into Fe^{3+} ions. Subsequently, Fe^{3+} ions hydrolyzes to form FeOOH or $Fe(OH)_3$. F-TEDA releases fluoronium ion, F^+ in the reaction mixture which replaces surface hydroxides to form fluorinated iron hydroxide. Finally, the resulting $FeO(OH)_{1-x}F_x$ or $Fe(OH)_{3-x}F_x$ decomposes into fluorinated-α-Fe_2O_3.

$$[Fe(CN)_6]^{3-} \rightarrow Fe^{3+} \rightarrow FeOOH/Fe(OH)_3 + \text{F-TEDA} \rightarrow FeO(OH)_{1-x}F_x/ Fe(OH)_{3-x}F_x \rightarrow \text{F-}\alpha\text{-}Fe_2O_3 \quad (2.1)$$
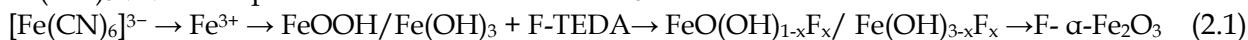
Figure 2.2: Hydrothermal reaction vessel of 100 mL, 250 mL and 800 mL capacity

The term hydrothermal (HT) usually refers to any heterogeneous reaction in the presence of aqueous solvents or mineralizes under high-temperature and pressure (HTHP) conditions to dissolve and recrystallize materials that are comparatively insoluble under normal conditions. The HT technique is one of the popular methods for the synthesis of nanomaterials. Hydrothermal name refers to geological origin. In hydrothermal, '*hydro*' means water and

'thermal' means heat. The hydrothermal method has several advantages over other existing methods of synthesis. The biggest advantage is use of water as a reaction medium under hydrothermal condition. Water as a solvent is present abundantly in nature, it is nonflammable, nontoxic and thermodynamically stable. Water can be easily removed after the reaction is complete. The polarity of water can be controlled by controlling temperature and pressure. Increasing the dielectric constant increases polarity and hence increases solubility of polar compounds.

2.3 PHYSICAL CHARACTERIZATION TECHNIQUES

After the synthesis of materials, it becomes essential to analyze their physical properties. The following physical characterization techniques were used to characterize the material

2.3.1 X-ray Spectrophotometer

The rhombohedra structure of hematite with hexagonal crystal system was studied by powder X-ray diffraction (XRD). Bruker D8 advance diffractometer having Cu-Kα radiation of 1.54 Å wavelength was used for XRD. The diffraction pattern was analyzed for phase, crystallinity and orientation of α-Fe_2O_3, before and after fluorination. The calcined and grinded powder sample was filled in circular cavity holder (diameter = 50 mm and depth = 2 mm) and upper surface was flattened using glass slide. Spectrum of diffraction intensity versus angle between diffraction and incident beam is recorded by continuously changing the incident angle from 10° to 80° with scanned speed ~1° per minute in locked coupled mode of the X-ray beam.

The path difference between incident x-rays (1 and 2) and diffracted x-rays (1' and 2') for constructive interference is given by Bragg's condition as equation (2.2)

$$2d\ sin\theta = n\lambda \tag{2.2}$$

Where:

d - Spacing between two atomic layers (Å)

θ - Angle of x-ray incident beam with normal to plan (in degree)

n - Order of diffraction

λ - X-ray wavelength (here, Cu (k_α) = 1.5418 Å)
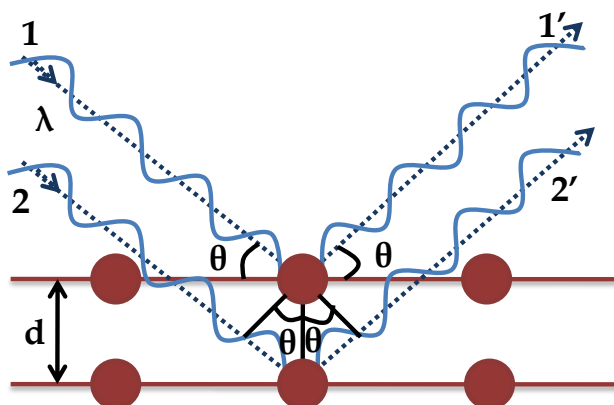

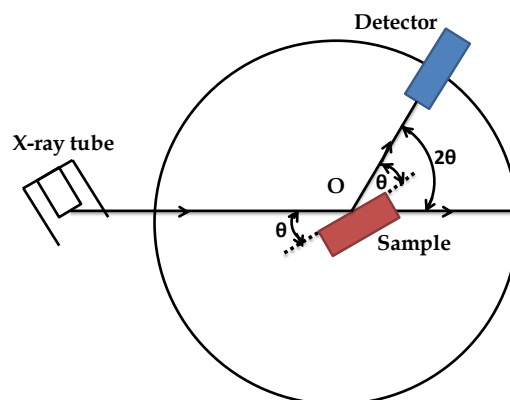**Figure 2.3:** X-ray spectrometer principle

Figure 2.4: Essential features of X-ray

The essential features of an x-ray spectrometer are shown in Figure 2.4. X-rays from the X-ray tube are incident on a sample which may be set at any desired angle to the incident beam by rotation about an axis through O, the center of the spectrometer circle. Detector is an ionization chamber or some form of counter which measures the intensity of the diffracted x-rays; it can also be rotated about and set at any desired angular position. In use, the crystal is positioned so that its reflecting planes make some particular angle θ with the incident beam, and detector is set at the corresponding angle 2θ. The intensity of the diffracted beam is then

measured and its wavelength calculated from the Bragg's law, this procedure being repeated for various angles θ.

Hematite possess hexagonal structure and it is very similar to the tetragonal structure; among the three sides, two of them are equal (a = b ≠ c). Two of the inter axial angles are 90° while the third is 120° (α = β = 90° ; γ = 120°). The d-spacing and the miller indices (h k l) are related by the following equation.

$$\frac{1}{d^2} = \frac{4}{3}\left[\frac{h^2+hk+k^2}{a^2}\right] + \frac{l^2}{c^2} \tag{2.3}$$

2.3.2 Scanning Electron Microscopy and Elemental Detection

A scanning electron microscope (SEM) is able to zoom in on a sample more than 500,000 times and is primarily used for determining morphological and topographical assessment. The surface morphology and elemental analysis are carried out on SEM EVO special edition, Bruker instrument. For recording the SEM image, the powder sample was spread over silver adhesive tape and it was pasted on sample holder (stub). Subsequently, the sample was sputtered with gold-palladium target for making it conducting. Mini sputter (SC7620) coater, manufactured by Quorum Technology, United Kingdom was used for coating the sample. The sputtering parameter was as follows:

Time: 180 sec., plasma current: 8-10 mA and chamber pressure: below 0.06-0.09 mbar.

The SEM works by focusing high energy electron beam generated from a field emission gun onto the sample using electromagnetic apertures and lenses. The SEM images were recorded with high energy (EHT: 20 kV) focused electron beam incident on sample surface, of current, 80 pA. The electrons beam interact with surface atoms and emit secondary electrons, backscattered electrons, bremsstrahlung radiation, augur electrons and characteristic x- ray photons. All of these emissions are the result of inelastic interactions except backscattered electron emission, which is elastic, denoting that energy is conserved. The secondary electrons (SE) and backscattered electrons are detected with Everhart-Thornley detector and 4Q-BSD/Scintillate detector respectively. The surface morphology of sample was produced by scanning small surface area with focused electron beam. The electron intensity was used to produce image by automated in built software [EVO, 2008]. The secondary electron image is a property of the surface structure (topography) of the sample rather than any underlying structure. Therefore, the image is said to display topographical contrast. However, the back scattered electron provides information about compositional contrast.
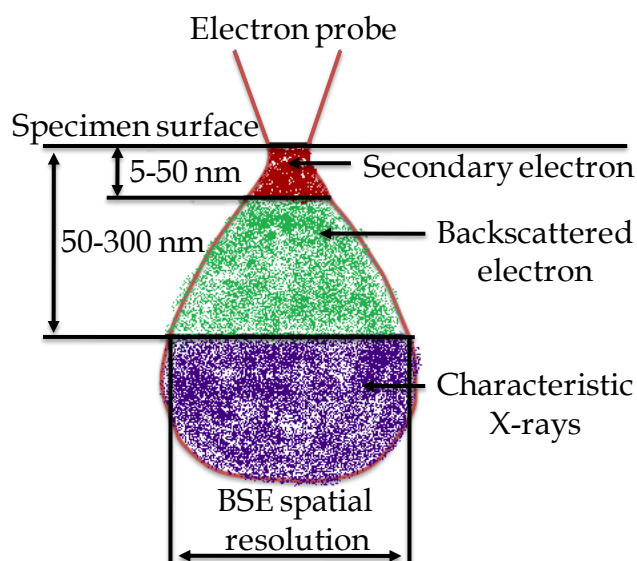

Figure 2.5: SEM instrument   **Figure 2.6:** Interaction of electron and sample atom

The characteristic x-ray of different elements was analyzed on EDX detector, Oxford Instruments, attached as additional assembly with SEM EVO special edition. The recorded information was converted into an energy spectrum by automated in-built software (INCA). The EDX system's INCA software was used to analyze the energy spectrum in order to determine the abundance of specific element. The EDX has limitation on detection of low atomic number (Z< 14) [EVO, 2008, INCA, 2006].

2.3.3 Transmission Electron Microscopy Technique

The high resolution transmission electron microscopy (HRTEM) measurements were performed on FEI Technai G2 T20 ST with an accelerating voltage of 200 kV. It was equipped with energy-dispersive X-ray spectroscopy (Burker X-100). For imaging, 10-15 mg of sample was dispersed in ethanol and drop casted on a copper grid and allowed to dry before imaging. HRTEM images are analyzed using Image J and Gatan digital micrograph software. TEM is a form of microscopy that can resolve images on atomic scale. However, it can only work with ultra-thin samples. It works by transmitting a high energy electron beam through an ultra-thin sample and images the interaction that occurs between the beam and the sample.

The selected area electron diffraction pattern (SAED) is also recorded for selected samples. Each spot of SAED corresponds to a satisfied diffraction condition of the sample's crystal structure. SAED diffraction patterns are either ring patterns corresponding to powder diffraction from multiple crystals with variable orientation or simple spot patterns corresponding to single-crystal diffraction. SAED is commonly used for determination of structural intergrowth, phase identification, determination of growth directions etc.

2.3.4 Raman Spectrum

The selected samples are characterized for iron-oxygen-fluorine bonding information. Raman analysis is carried out by AIRIX Corporation, Japan STR 500 confocal Raman spectrometer. The excitation source was an air cooled diode laser source, generating 532nm green laser of 1.5mW power. The measurements were performed on powder sample at 50x zoom and 10 sec acquisitions.

2.3.5 BET Specific Surface Area Technique

The specific surface area analysis was performed using AutoSorb (iQ3), Quantunchrome Instrument, USA. Initially 300 mg sample was degassed in N_2 atmosphere at 300 °C for 3 hr. Further, 40 points data were recorded in each adsorption and desorption process under N_2 atmosphere [Autosorb, 2016].

2.3.6 ^{19}F-NMR Spectra

^{19}F-NMR spectra were collected from the reaction mixture before and after reaction (supernatant) in D_2O using Bruker 500 spectrometer operating at 500 MHz.

2.3.7 Fourier Transform Infrared Spectrum

Fourier-transform infrared spectra (FTIR) were recorded on a Vertex 70v spectrometer (Bruker). KBr palette was used to record spectrum. It was prepared using ~1 mg of sample and ~200 mg of spectroscopic grade KBr. They were thoroughly mixed, powdered and pressed at 10 Ton to form a pellet which is inserted into a holder in the spectrometer. Infra-red (IR) spectroscopy deals with the interaction between radiation from the IR region of the EM spectrum (IR region = 4000 - 400 cm^{-1}) and a molecule. IR radiation causes vibrations of bonds within molecule. The frequency of vibration mode (ν), which depends on stiffness of the bond and the masses of the atoms at each end of the bond, is as follows:

$$\nu = \frac{1}{2\pi}\left(\frac{k}{\mu}\right)$$ (2.4)

Where,

    k – force constant

    µ – reduced mass $\left(\frac{m_1 m_2}{m_1 + m_2}\right)$, where, m_1 and m_2 are the masses of the atoms at the ends of the bond.

2.3.8 UV-Visible Spectrum

The Diffuse reflectance (DR) spectra were recorded in solid state using UV–vis spectrophotometer, Varian Cary 4000 with polytetrafluoroethylene (PTFE) as the reference material. The DR spectra were captured in order to determine the bandgap of material. The obtained reflectance data were converted to modified Kubelka–Munk (K-M) function using following equation:

$$F = \frac{(1-R)^2}{2R} \tag{2.5}$$

The K-M function was plotted using Tauc equation, as mentioned below, to get the bandgap of material.

$$Fh\nu = A(h\nu - E_g)^n \tag{2.6}$$

Where, A is a constant, $h\nu$ is photon energy, E_g is the allowed energy gap, n = ½ for allowed direct transition and n = 2 for allowed indirect transition. The hematite has allowed direct transition, therefore n = ½ and the equation becomes:

$$(Fh\nu)^2 = A(h\nu - E_g) \tag{2.7}$$

The Tauc's region was extrapolated to $(Fh\nu)^2 = 0$ to obtain the bandgap.

2.3.9 X-ray Photoelectron Spectrum

X-ray photoelectron spectroscopy (XPS) analysis was carried out using a commercial Omicron EA 125 source with Al-Kα radiation (1486.7 eV). Palette of 13 mm x 1 mm size was prepared to record the XPS. It is a chemical analysis technique that provides quantitative information about surface chemical constituents such as elemental composition, chemical states, electronic state and empirical formula. In XPS, material is placed under high vacuum and spectra are obtained by irradiating it with a beam of X-rays while concurrently measuring the kinetic energy and number of electrons that escape from the top 1 - 12 nm of the sample being analyzed.
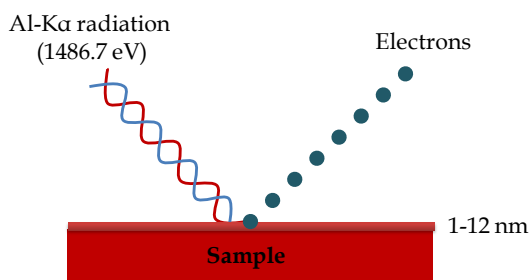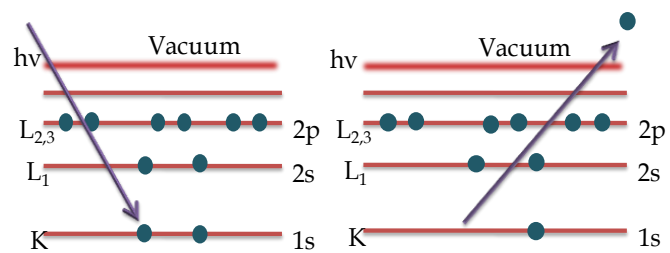

Figure 2.7: Sample irradiation in XPS

Figure 2.8: Electron escaping from surface

2.3.10 Magnetic Characterization Technique

Magnetic measurements were performed using Superconducting Quantum Interference Device (SQUID) magnetometer, Ever Cool MPMS XL, Quantum Design, 2002. SQUID magnetometer is very sensitive as it is configured to detect small magnetic field. It consists of two superconductors separated by thin insulating layers to form two parallel Josephson junction. An alternating magnetic flux in the pick-up coil produced when the sample moves up and down. The magnetic signal of the sample is obtained via a superconducting pick-up coil together with a SQUID antenna. The device acts as a magnetic flux-to-voltage converter. The voltage is then amplified and read out by the magnetometer's electronics.

2.3.11 Transmission and Reflection Loss Measurement

Network analyzer, HP8510C, was used to carry out transmission and reflection loss measurements in the C (6-8 GHz), X (8-12 GHz) and Ku (12-18 GHz) bands in the microwave frequency. Magnetic loss tangent ($tan\,\delta_\mu = \mu_r''/\mu_r'$) of the materials were calculated from measured permeability values, over 5.85–18 GHz frequency range, using the Nicolson–Ross–Weir (NRW) algorithm

2.3.12 Trace Elemental Analysis

Inductively Coupled Plasma Optical Emission Spectrophotometer (ICP-OES), Prodigy7, by Teledyne Leeman Labs was used for detection of trace elemental analysis. The instrument was used for detection of uranium in contaminated water for finding the uranium removal efficiency of fluorinated hematite. Samples were introduced in ICP through peristaltic pump and further nebulized in cyclonic spray chamber using Ar gas at 25 psi pressure. The produced aerosol was directed into argon plasma. The plasma was viewed in axial mode and the photons were sensed at CMOS detector. The flow of auxiliary gas (Ar) and coolant gas (Ar) was kept at 0.5 L/min and 15 L/min respectively.
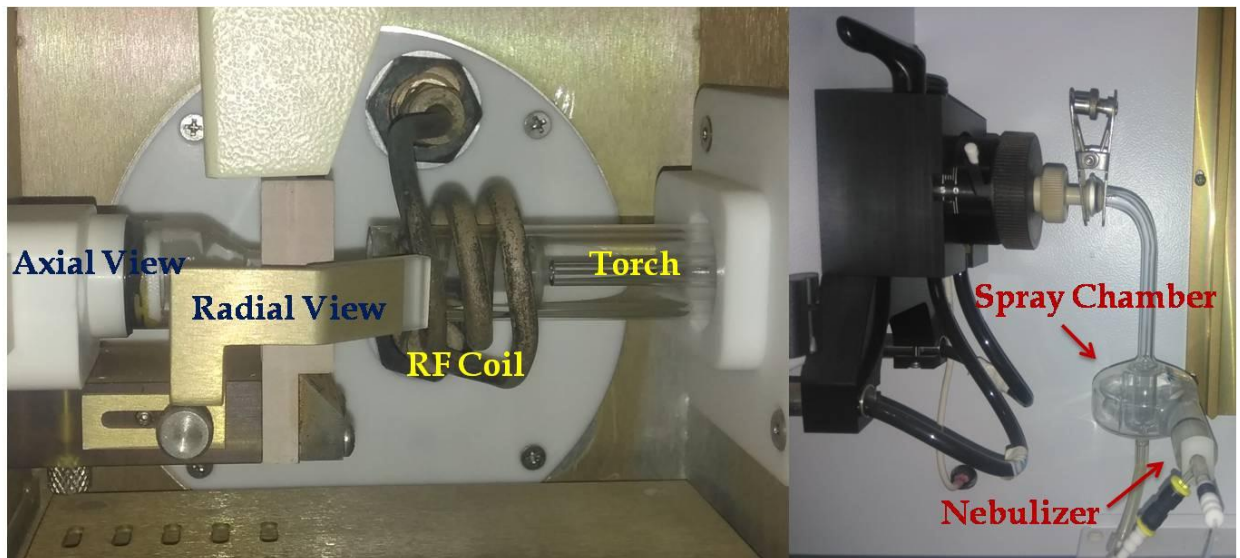

Figure 2.9: ICP OES Torch, Spray Chamber and Nebulizer

ICP-OES is an elemental analysis technique that uses the emission spectra of the sample for qualitative and quantitative analysis. Here, samples are introduced into the plasma through cyclonic spray chamber and torch. Plasma de-solvates, ionizes, and excites the component elements (atoms). When excited atoms return to low energy level, emission rays or spectrum rays are released which corresponds to the photon wavelength. Identification of the constituent elements are made by their characteristic emission lines and the intensity of the same lines quantifies them.
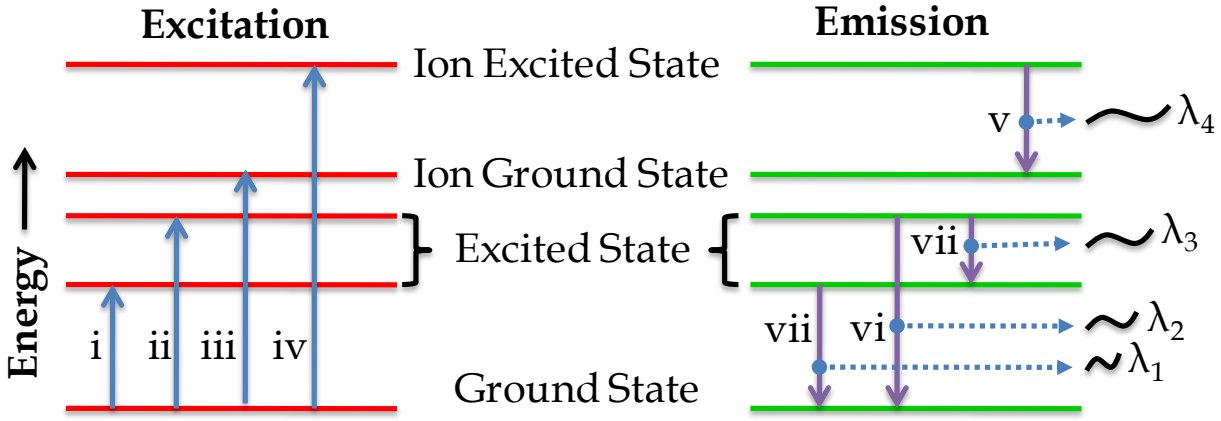
Figure 2.10: Excitation and emission diagram (Excitation and emission diagram showing energy transitions. I & II is excitation, iii is ionisation, iv is ionisation/excitation, v is ion emission and vi is atom emission)

2.4 ELECTROCHEMICAL CHARACTERIZATION TECHNIQUES

Electrochemical workstation (CHI6600) was used for I-V measurements, chronoamperometry experiments and impedance spectroscopy (EIS) analysis. Three-electrode system set-up was made using screen printed thin film sample on fluorine doped tin oxide (FTO) as working electrode, Pt wire as counter electrode and Ag/AgCl (3 M KCl) as reference electrode. 1 M KOH solution (pH=13.6) was used as electrolyte. The measured potential was converted to the reversible hydrogen electrode (RHE) scale using following standard Nernst equation.

$$E_{RHE} = E_{Ag/AgCl} + 0.059pH + E^0_{Ag/AgCl} \qquad (2.8)$$

Where, E_{RHE} is the potential *vs.* RHE in *V vs.* RHE (V_{RHE}), $E_{Ag/AgCl}$ is the experimental potential measured against the Ag/AgCl reference electrode in *V vs.* Ag/AgCl ($V_{Ag/AgCl}$), and $E^0_{Ag/AgCl}$ is the standard potential (0.1976 V) of Ag/AgCl (saturated KCl) at 25 °C.

I-V curves were recorded in the form of linear sweep voltammetry at scan rate of 20 mv/sec and -0.4 *V* initial voltages and 0.6 *V* final voltages of Ag/AgCl.

The Mott–Schottky curves of pristine and fluorinated hematite were collected in 1 M KOH aqueous solution. The samples were scanned from initial -1 volt to final 0.6 volt (*vs* Ag/AgCl electrode) with 50 mV increment potential at 1 kHz frequency. The flat band potential and donor densities of films were calculated from the Mott–Schottky equation:

$$\frac{1}{C_{sc}^2} = \frac{2}{\varepsilon \varepsilon_0 e N_d}\left(E - E_{fb} - \frac{kT}{e}\right) \qquad (2.9)$$

where C_{sc} is the capacitance of the space charge layer, ε is the dielectric constant of hematite (ε = 80), ε_0 is the permittivity of vacuum (8.854 × 10^14 F/cm), *e* is the electronic charge (1.602 × 10^19 C), N_d is the donor density, E is the applied bias, E_{fb} is the flat band potential, k_b is the Boltzmann constant, and T is the temperature in K. EIS graphs were plotted from a frequency range of 1–10^5 Hz.

Chronoamperometry experiments were performed at 0.6 V (1.6 V versus RHE) vs. Ag/AgCl. The measurements were carried out under illumination of 100 W/m² (one-tenth of 1 Sun) while the light source was switched on and off after every 5 seconds. Solar Simulator (model number SS50AA, Photoemission Tech) was used to illuminate the samples. The photocurrent value at the end of the chronoamperometry measurement was taken as the steady-state photocurrent and was used to compare different samples.

Figure 2.11: I-V measurement under 100 W/m^2 solar simulator radiation

2.4.1. Sample Preparation for Photo-Electrochemical Characterization

The films of α-Fe$_2$O$_3$ and F-Fe$_2$O$_3$ for photo-electrochemical measurements were prepared by three layers screen printing on conducting FTO glass surface. The paste of appropriate viscosity was made by mixing 0.3 mL of α-terpinol, 0.05 g of ethyl cellulose and 0.02 mL glacial acetic acid in a mortar and pestle for 30-40 min. After the complete dissolution of ethyl cellulose, 0.1 g of hematite was added and mixed for 10-15 min. Ethanol was used for viscosity adjustment. The prepared paste was layer by layer coated on FTO glass while using 100 mesh screen and squeegee. Every layer was dried using hot air gun before coating next layer. The prepared films were annealed at 500 °C for 5 h for electrochemical measurements.

2.5 CLOSING REMARKS

In this chapter, detailed synthetic processes using hydrothermal method are discussed. The different physical characterization techniques, such as XRD, SEM, TEM, XPS, FTIR, UV Vis spectrophotometer, Raman spectroscopy, ICP-OES and it's basic principles have been mentioned. Magnetic characterization and transmission & reflection loss measurement techniques and methods are also summarized. The electrochemical characterization techniques such as I-V measurements, chronoamperometry experiments and impedance spectroscopy (EIS) analysis are presented. The working electrode preparation methods have also been discussed.