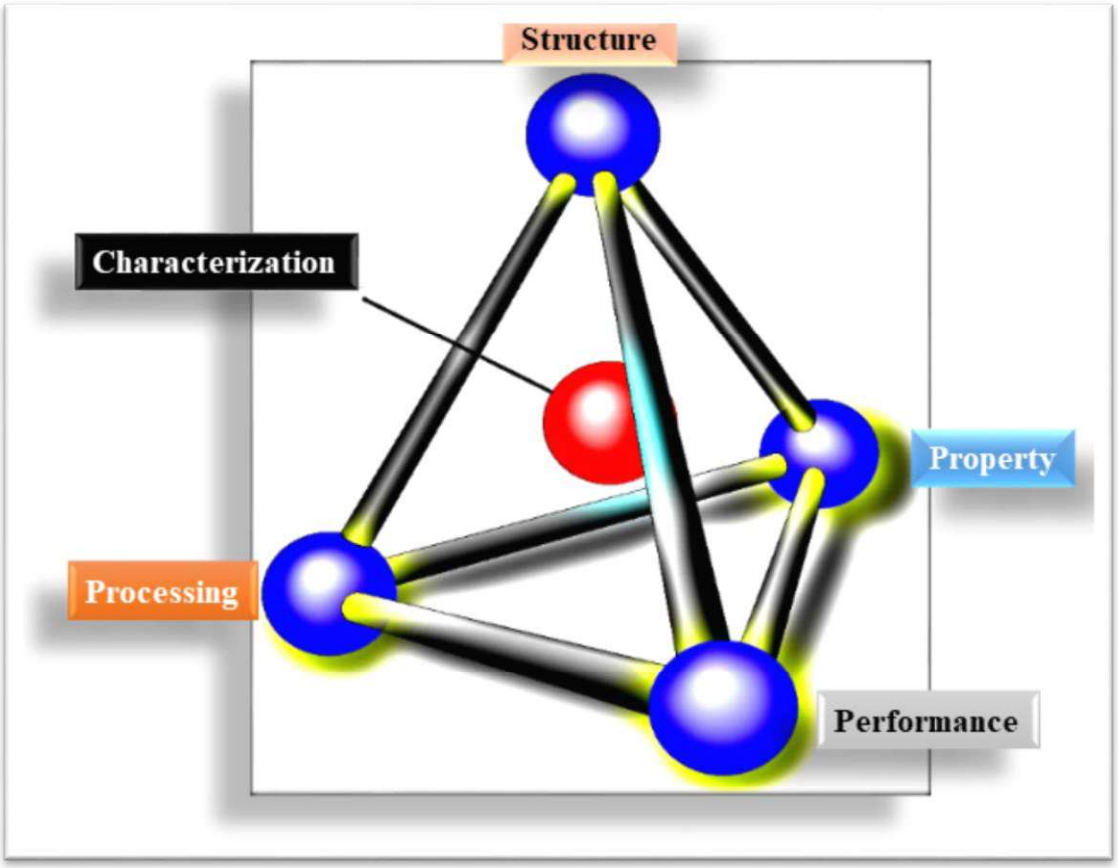
Chapter 2

Experimental and Characterization techniques



Chapter 2: Experimental and characterization techniques

2.1 Introduction:

In this chapter, we discussed the experimental process for the development of Potassium oxide K_2O (weight by weight percentage wt%) added Lithium Niobate i.e. $LiNbO_3$ + K_2O(wt%), Samarium modified Lithium Niobate i.e.($Li_{(1-x)}Sm_{x/3}NbO_3$), and Lithium Niobate Dysprosium Titanate based Solid Solutions i.e. (1-x)$LiNbO_3$-x($Li_{0.5}Dy_{0.5}$)TiO_3. They were characterized using a variety of physiochemical approaches. According to our research, the conceptualized Ceramics materials would be synthesized and characterized as (1) Synthesis of various systems with varying compositions, (2) Heat treatment at high temperatures, (3) Crystal phase and structural studies, and (4) Microstructural characteristics (5) Oxidation state and elemental analysis (6) Crystal phase Dielectric behavior investigation w.r.t. frequency and temperature (7) Polarization-Electric Field Investigation (8) Absorption and Band Gap Analysis (9) Photoluminescence analysis (10) Transmittance and functional group analysis (11) Thermogravimetric analysis.

In the present research work (a) $LiNbO_3$ (Solid state reaction), (b) $LiNbO_3$ + x(wt%) K_2O (Solid state reaction), (c) ($Li_{(1-x)}Sm_{x/3}NbO_3$) (Solid state reaction), (d) (1-x)$LiNbO_3$-x($Li_{0.5}Dy_{0.5}$)TiO_3 (Solid state reaction). All ceramics were synthesized using a high-energy ball mill solid-state reaction method. To be used in the synthesis of all ceramic high-purity raw materials, namely Li_2CO_3, Nb_2O_5, K_2CO_3, Sm_2O_3, Dy_2O_3, TiO_2, MgO, and all other chemicals with the specifications listed in the table below.

2.2 List of Precursor/Chemical

We used different chemical/precursors as raw materials for the synthesis wok, in the following table all used materials has been listed with details of their chemical formula, purity and the manufacture.

Table 2.1: List of used precursor and chemicals for the synthesis of the samples.

Raw Material	**Minimum Assay/Purity**	**Manufacturer**
Lithium Carbonate (Li_2CO_3)	98.5%	Sarabhai M. Chemicals/ Sigma Aldrich product
Niobium Pentoxide (Nb_2O_5)	99.9%	Sigma Aldrich product
Potassium Carbonate (K_2CO_3)	99%	Himedia Chemie Pvt. Ltd
Dysprosium Oxide (Dy_2O_3)	99.9%	Sigma Aldrich product
Titanium Dioxide (TiO_2)	99%	Loba Chemie
Magnesium Oxide (MgO)	96 %	Loba Chemie
Samarium Oxide (Sm_2O_3)	99.9%	Sigma Aldrich product
Acetone	99.5%	Sigma Aldrich product
Isopropyl Alcohol	99%	Sisco Research Laboratories Pvt. Ltd.
Ethanol	99.9%	Shree Chemicals Industries Pvt. Ltd.
Polyvinyl Alcohol	2 wt%	Sigma Aldrich product

2.3 Synthesis of materials

The synthesis of the material has been completed by Ball milling of reactants, Calcination of mixed powder, Binder mixing in calcined powder, pellet formation of PVA mixed powder and the Sintering of green pellets. All these processes are discussed briefly as following.

2.3.1 Solid state reaction method via High energy ball mill (Mechanochemical process):

A variety of materials, including ceramics, metals, and alloys, can be synthesized using the solid-state reaction process. The solid-state reaction method, also known as the powder method, is one of the most frequently used synthesis processes for the bulk production of ceramic polycrystalline materials for technological advancements. In this method, easily available oxides, carbonates, nitrites etc, and other forms of desired salts are used as reactants. This method is very simple and less costly for bulk production. It's a Top-to-bottom materials synthesis process where a High-energy ball mill is used for the mixing of materials, where materials are kept in a jar with zirconia balls, and the collision of balls to reactants is carried in the presence of a liquid wet medium at a constant rpm. When the jar rotates, the balls are raised and subsequently dumped into the powder mixture, which grinds and breaks the particles. The process of high-energy ball milling must be conducted in an inert environment to prevent oxidation or in a reactive atmosphere to encourage the reaction. The ball mill's solid-state reaction process is separated into two stages: the initial stage and the final stage. The reactants are first combined and grind to form a homogeneous mixture. The grinding balls strike and smash with each other with the reactants during this process, so the formation of small particles with a large surface area is obtained. Therefore, increased surface area facilitates the reaction by increasing the number of reactive sites.

The reaction happens in the last step as a result of the high temperature and pressure created during ball milling. The mechanical energy input and friction between the balls and the powder mixture create heat during the high-energy ball milling process. This heat might induce the reactants to achieve their activation energy, which makes sure the reaction to occur.

In comparison to other methods, the solid-state reaction method from high-energy ball milling offers the following advantages:

High degree of homogenization: The high-energy ball milling process completely mixes and homogenizes the reactants in result uniform reaction achieved.

(i) **Decreased reaction time:** By expanding the surface area of the reactants, the high-energy ball milling technique may drastically reduce reaction time.

(ii) **Adjustable reaction:** The reaction may be adjusted by changing milling parameters such as milling duration, rotation speed, and ball-to-powder ratio.

(iii) **Scalable**: The high-energy ball milling solid-state reaction process is scalable and may be utilized to create huge quantities of bulk materials.

Nevertheless, the high-energy ball milling solid-state reaction process has certain drawbacks, including:

(i) **High energy consumption:** The high-energy ball milling method necessitates a significant quantity of energy, which might raise manufacturing costs than few other methods.

(ii) **Agglomeration:** The high-energy ball milling process can generate agglomeration within powder; it may be a reason for a non-uniform un desired response.

(iii) C**ontamination:** Use of different materials balls during grinding, the wet medium might contaminate and change the characteristics of final product.

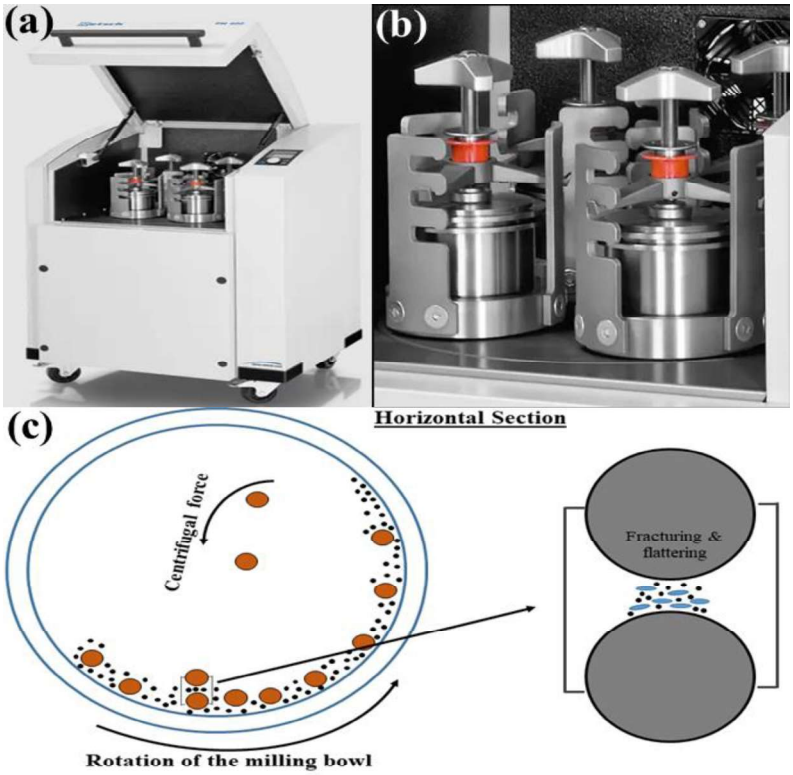


Figure 2.1 (a-c): (a) High energy planetary ball mill Retsch PM400, (b) Horizontal section of High energy ball mill, (c) Schematic diagram of the mechanism inside the high energy ball mill.

2.3.2 Calcination Process:

Calcination is a thermal treatment procedure at high temperature which applied to solid materials to induce thermal breakdown, phase change, elimination of volatile part or elimination of undesired impurity of synthesis material. Typically, the calcination process occurs at temperatures below the melting points and sintering temperature of the pellet of the final product.

2.3.3 Pellets formation (Pressing) and Sintering of Samples:

The calcined powder was kept in an agate mortar pestle and once ground for uniformity within the materials. A few drops of polyvinyl alcohol of 2% by weight (we tried to maintain the constant ratio between material weight and PVA for all the samples) were added and well mixed into the powder till it dried as we needed. The weighed powder was put

in desired shape dye after that dye put in hydraulic press, a tangential pressure of about 5 ton applied for 90 seconds and after that pressure were release very smoothly, the pressed powder takes out from the dye which is circled form with about 1 mm thickness and 10 mm diameter. The formed pellets were sealed in an Alumina crucible with magnesium oxide and held at 550 °C for around 6 hours to completely burn out the binder (PVA).

Sintering is the process of densifying the porosity of a compact pellet by heating the materials at the appropriate temperature. In this process, pellets were kept at their respective optimized sintering temperatures for an optimized period of time. The temperature was then raised to the necessary sintering temperature, at which point solid-state reactions between various components took place, and also sintering was completed. In this study, these pellets were sintered at various temperatures with their respective components for 6 hours. The following picture depicts a schematic chart depicting the many steps for preparing these materials using this method.

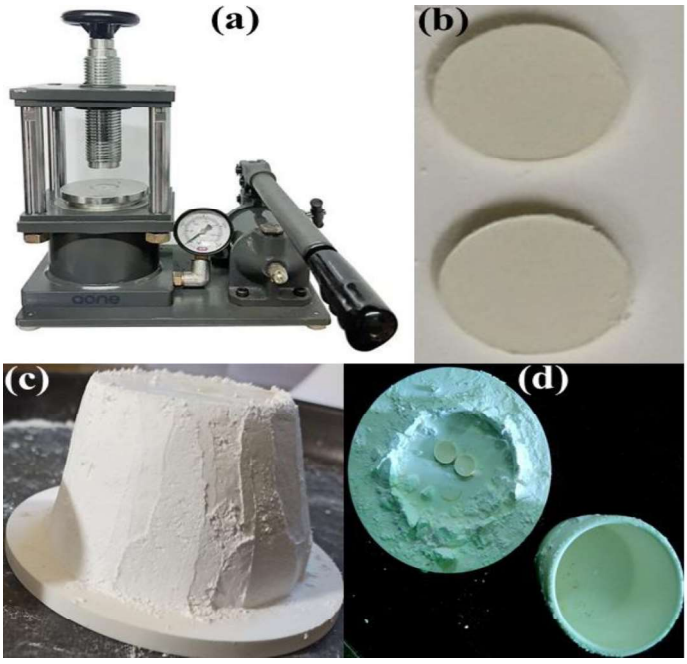


Figure 2.2 (a-d): (a) Hydraulic Press Pellet Maker, (b)Green Pellet, (c) MgO Sealed Alumina Crucible, (d) Finale sintered Pellets.

2.3.4 Schematic diagram for the Synthesis and Sintering of materials

There in the Figure 2.3, we have shown the Schematic diagram for the whole synthesis process of synthesized materials of present research work and it also shown how sintered pellets is how further prepared for the desired characterization. Given schematic diagram included ball milling process, calcination process, binder mixing, palletization, sealing of pellet to provide close environment, binder burning, electroplating and grinded pellet annealing.

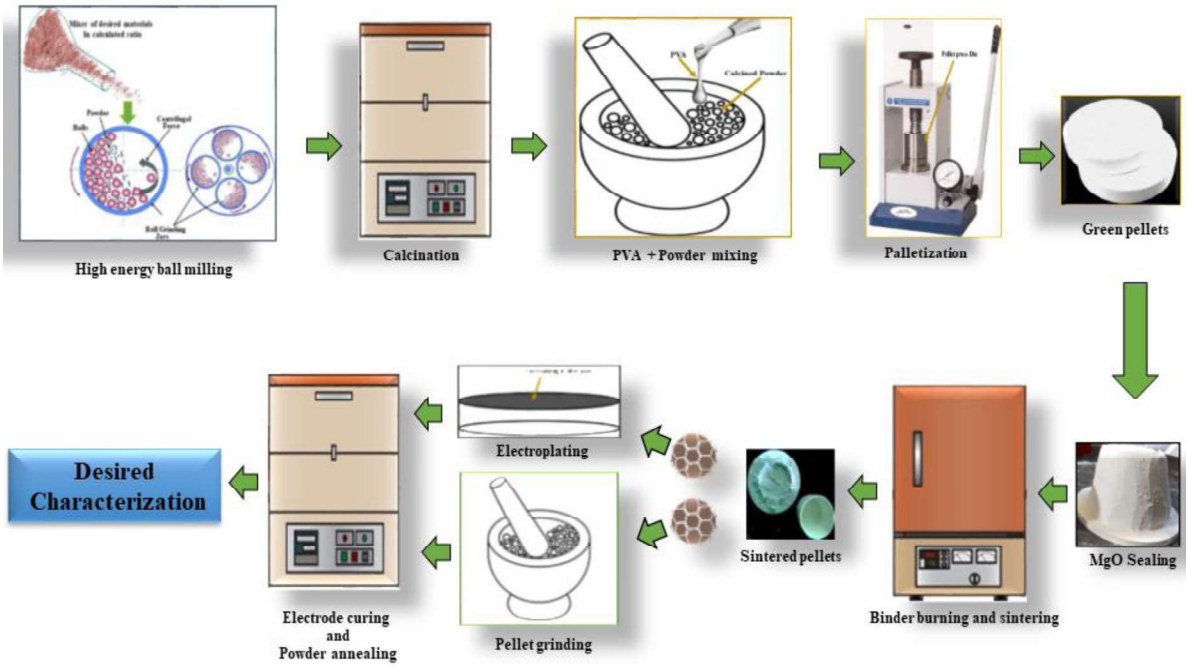


Figure 2.3: Schematic diagram of solid state reaction method for the materials sysntehsis.

2.4 Characterization Techniques

Different characterization techniques has been used for the present research work like Powder X-ray diffraction (XRD), Scanning Electron microscope (SEM), XPS, PE, Impedance spectroscopy, FTIR, UV Visible spectroscopy, TGA to get the various kind of physical, chemical and thermal characteristics, and these are discussed as follows.

2.4.1 Powder X-ray diffraction (XRD)

X-ray diffraction (XRD) characterization can be used to investigate phase formation and other structural parameters of materials and also average crystallinity and grain size. It is a non-destructive and versatile analytical technique along with varying condition like temperature and pressure. X-ray diffraction peaks are formed by the constructive interference of a monochromatic X-ray beam, which diffracted at particular angles from each set of lattice planes in a material. When the path lengths of x-rays diffracted from different planes which differ by multiple wavelengths, so the constructive interference occurs. The x-ray source's radiation strikes the material and is diffracted in all directions, the interference of x-rays diffracted from different crystalline planes produces a regular pattern for the atomic arrangements in a crystalline material. The peak intensities and their positioning at x-axis gives the desired information through analysis.

The X-ray diffraction measuring principle is based on the scattering of electromagnetic waves within the 1 angstrom range as shown in the Figure 2.4(b). Which obey the equation $2d\sin\theta = n\lambda$, which is known as Bragg's law, and this equation is utilize for X-ray diffraction (XRD) method, through this equation determination of various crystalline or polycrystalline properties is possible. Where θ represents the scattering angle, λ represents the wavelength of the X-ray, d represents the distance between the structural planes, and n represents the order of the diffraction pattern. Since Bragg's law is satisfied for a discrete value of θ, the diffracted beam only originates for that value, fulfilling the geometry of a reflection. Since, $(\sin\theta)_{max} = 1$. Using Bragg's law ($2d\sin\theta = n\lambda$) equation.

$$\frac{n\lambda}{2d} \leq 1 \qquad \text{.....2.1}$$

This equation indicates that the value of λ must be less than or equal to twice the interplanar distance; otherwise, there will be no diffraction.

During the characterization the X-ray source is stationary while the detector and sample are rotated by angles of 2θ and θ, respectively. In contrast, the sample remains stationary in most of the cases even when the source and detector are both rotating at the same time by an angle of θ. The goniometer is located in the middle of the diffractometer, and it is used to make sure the rotation with very high precision. In most cases, the sample is mounted on a rotational axis, and both the detector and the X-ray source move around the periphery of the rotating axis. Generally, sufficiently cover the most valuable region of the diffraction pattern, 2θ degrees, or 10° to 80° which is needed. For a room temperature operational diffractometer system Cu-K_α radiation (a=1.540598 Å) uses. For the XRD characterization slow scanning rate around 2 degrees/minute with a step size of 0.02 degree. An applied voltage of 40 kV and a current of 15 mA maintain during the whole process. In the present research work, the synthesized material was studied using a Bench Top X-ray diffractometer (Rigaku Miniflex, Japan) Shown in the **Figure 2.4(a).**

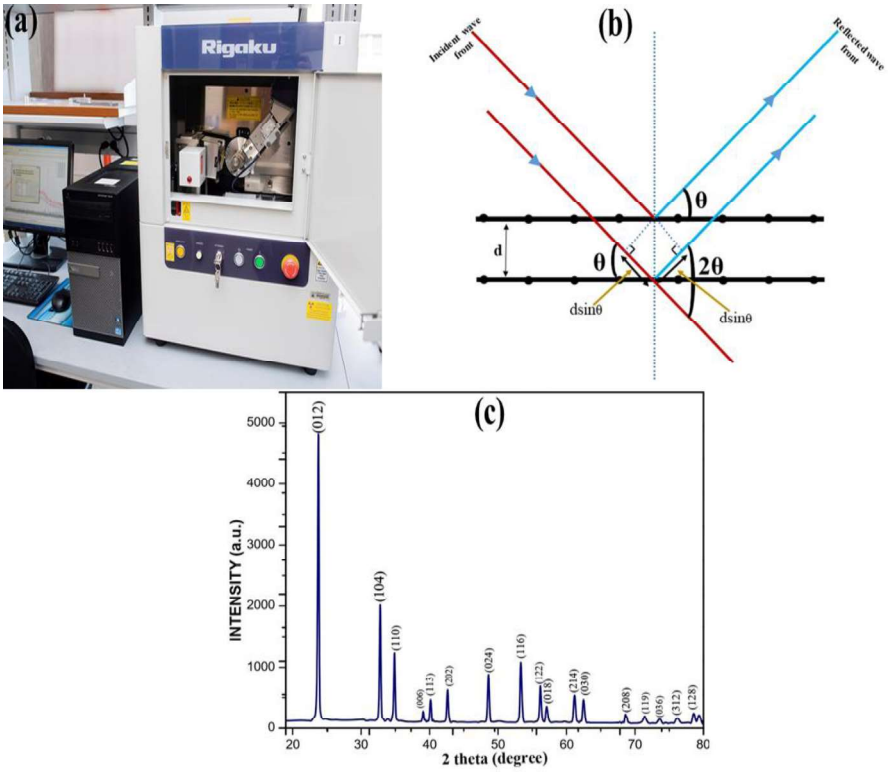


Figure 2.4 (a-c): (a) Rigaku Miniflex Benchtop XRD Machine, (b) XRD Phenomenon based on Braggs Law, (c) XRD Pattern of a ceramic material.

2.4.2 X-ray photoelectron spectroscopy (XPS)

X-ray photoelectron spectroscopy used to analyze the surface chemical composition in the range 1 to 10 nm, electronic state, and oxidation state of a material. Additionally, we can use photoelectron spectroscopy to determine which elements are present in the sample and in what oxidation state, concentration. During this characterization procedure, a sample is subjected to soft X-ray radiation in an ultra-high vacuum (UHV = 10^{-8} millibars to 10^{-9} millibar) chamber. Direct energy transmission from photon to core level electron takes place when photoelectrons are released from surface atoms. In an XPS spectrum Some of the photo-ejected electrons passes through the material and experiences inelastic scattering before reaching the surface. On the other hand, some electrons go through an accelerated emission process and then escape both the surface and the vacuum that surrounds them without any loss

of energy. Electron analyzers are used to measure the kinetic energy of the electrons after these photons have been ejected. That Electron analyzer produces an energy spectrum that plots the binding energy of electrons (the energy they have just before they leave the atom) vs the intensity of the electrons (number of photo-ejected electrons versus time). X-ray photoelectron spectroscopy is capable of identifying every element that is present in the sample because of the its principle on which its works. Practically it is able to identify elements with an atomic number Z greater than or equal to 3 (lithium). The atomic numbers Z=1 and Z=2 of atoms like hydrogen and helium are difficult or impossible for it to detect.

This method can be used to investigate a wide variety of materials, including ceramics, glasses, papers, metal alloys, inorganic compounds, polymers, semiconductors, catalysts, bio-materials etc.

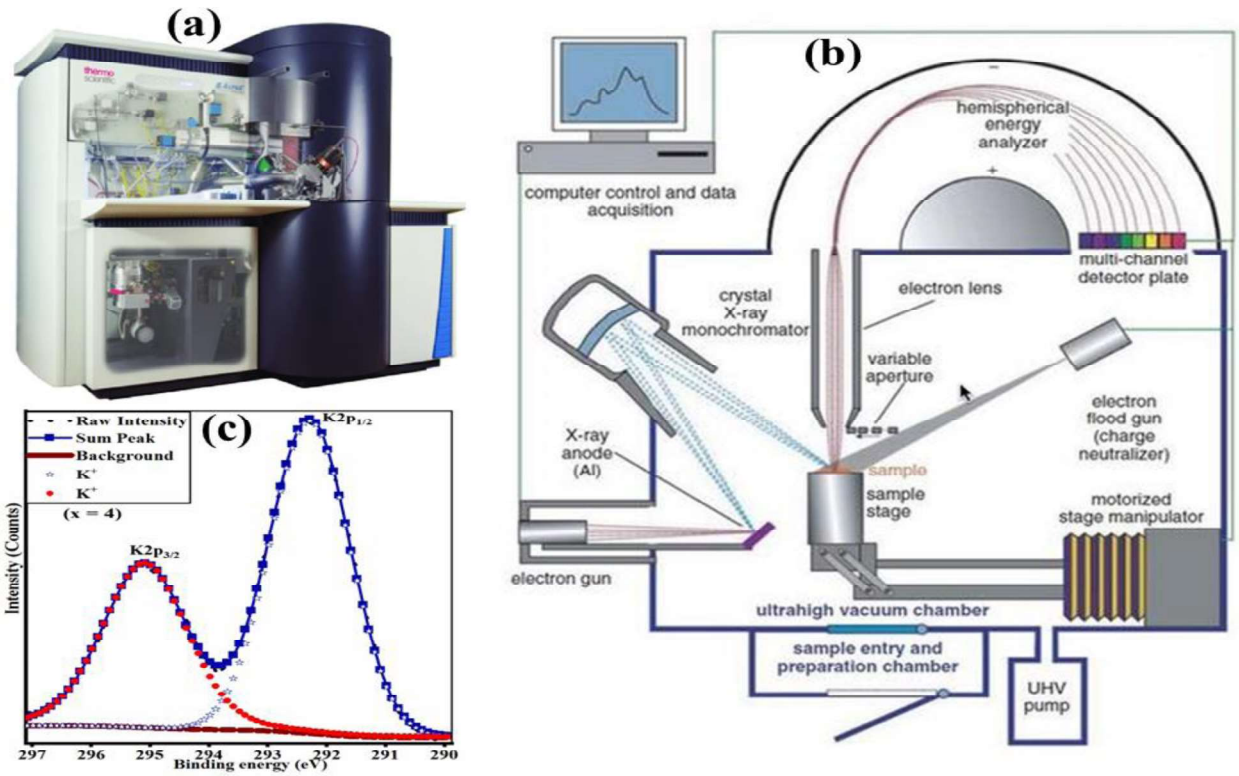


Figure 2.5 (a-c): (a) XPS Instrument, (b) Schematic diagram of mechanism of XPS, (c) XPS Graph of a ceramic material.

2.4.3 Scanning Electron Microscopy

The field emission scanning electron microscope (FESEM) is an instrument that can observe morphology of the surface of a material that can be small as 1 nanometer (nm) in size. It is possible to adjust the magnification of the observation according to the requirements. The morphology of ceramics, alloys, metals, inorganic compounds, polymers, etc., can be investigated using the FESEM. However, this type of microscope can also be used to analyze the percentage of elements as well as distribution of the elements in the system and these characterizations are known as EDS and elemental mapping. The FESEM microscopy utilizes electrons with a negative charge beam, instead of using light beam, a field emission source which is known as electron gun is used to produce these electrons. When Electrons emit from a source which known as electron gun, then accelerated in an electrical field gradient with a high potential difference. These produced primary electrons are focused and deflected by electromagnetic lenses within the high vacuum chamber to produce a narrow scan beam that is bombards to the target. This process takes place within the chamber itself. Secondary electrons are hence released from every single spot on the target. The angle and velocity of these secondary electrons are related to the surface structure of the target. A detector acts as a collector of secondary electrons and generates an electrical signal as a result. This signal is then amplified and turned into a monitor for digital image which further save and analyze. In the FESEM a cylindrical chamber which is mounted on a desk and within this chamber the electron gun contained. During the characterization the direction and strength of the electron beam can be control by using the control knobs which is also present with system control panel. The material target must be conductive for the current so that a coating of gold or gold palladium is apply for the coating which as thin as approximately 300 nm.

Furthermore, the target must be able to sustain the strong vacuum force and not changes the vacuum's condition in any condition, such as it should be not releasing gasses or other molecules. The schematic diagram of FESEM is shown in the **Figure 2.6(b).**

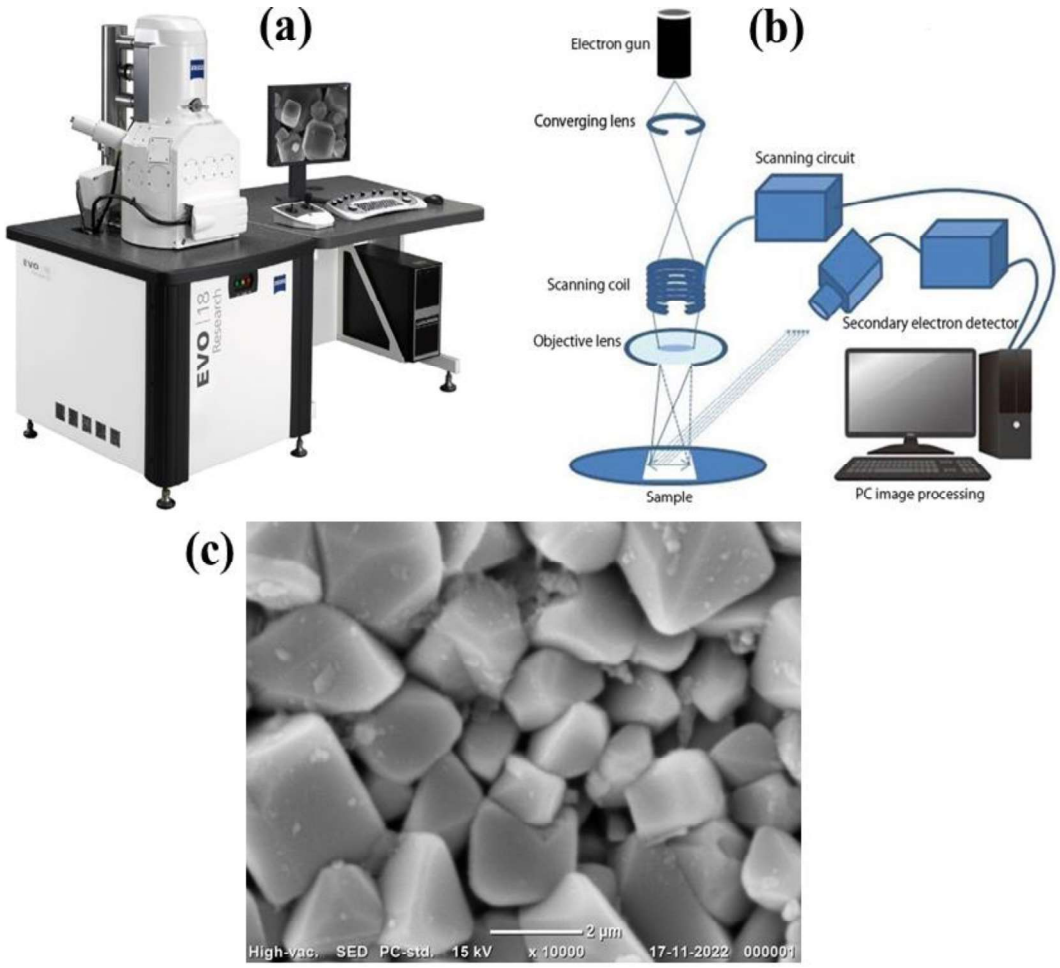


Figure 2.6(a-c): (a) FESEM Instrument (EVO - Scanning Electron Microscope MA15 / 18 CARL ZEISS Microscopy ltd), (b) Ray diagram for SEM, (c) The FESEM image of a Ceramic Material.

2.4.4 Ultra Violet- Visible spectroscopy

UV-Vis spectrometers have become the most essential analytical tools for the examination of the optical characteristics of materials because of their ease of use, speed, diversity, precision, and cost-effectiveness, Using UV-Vis spectroscopy, we observe the absorption and reflection of samples in the ultraviolet in the visible spectrum range. Moreover,

the UV-Vis data and by applying Touc plot equation we can calculate the bandgaps of the materials (bulk powders, liquids, and thin films).

Touc plot equation is

$$(h\nu)^2 = A(h-Eg) \qquad \text{.....2.2}$$

α is the absorption coefficient,

A is a constant related to the probability of absorption,

$h\nu$ is the photon energy

E_g is the bandgap energy (the energy difference between the valence band and the conduction band),

Beer-law Lambert's is the fundamental concept of the functionality of UV/Vis/NIR spectroscopy. There is a linear relation between the concentration of an absorbing material and its absorbance, there for the definition is as follows:

Where A, E, b, and c represent, respectively, the absorbance, the wavelength-dependent absorptive coefficient, the path length, and the material concentration. In addition, I_0 and I represent the intensity of the light that is incident on the object and the intensity of the light that is transmitted, respectively. There are four primary components that made the spectrophotometer. The light source comes in at number one, the sample holder comes in at number two, the diffraction grating of the monochromator comes in at number three, and the detector rounds come at number four. In terms of visible wavelengths, the radiation source functioned in a mode known as continuous operation. As a detector, we can use either a charge-coupled device (CCD) or a photodiode, as well as a photomultiplier tube. For the

purpose of scanning the monochromatic, photomultiplier tubes and single photodiode detectors are utilized. It eliminates all undesired wavelengths by using a filter that allows only one wavelength at a time to pass through to the detector. The diffraction grating makes it possible for each wavelength to be "step-through" transferred by scanning the monochromatic. This enables the intensity of the light to be measured and analyzed as a function of the wavelength at which it was emitted.

where E_g, α, A, and hν represent, respectively, the energy absorption coefficient, the optical band gap, an energy independent constant, and the incident photon energy. "n" is the power factor of transition modes, and the values for "n" are "1/2" for direct allowed transitions, "2" for indirect allowed transitions, "3/2" for direct forbidden transitions, and "3" for indirect forbidden transitions. The schematic instrument diagram of a UV Visible spectrometer is shown in the **Figure 2.7(b)**.

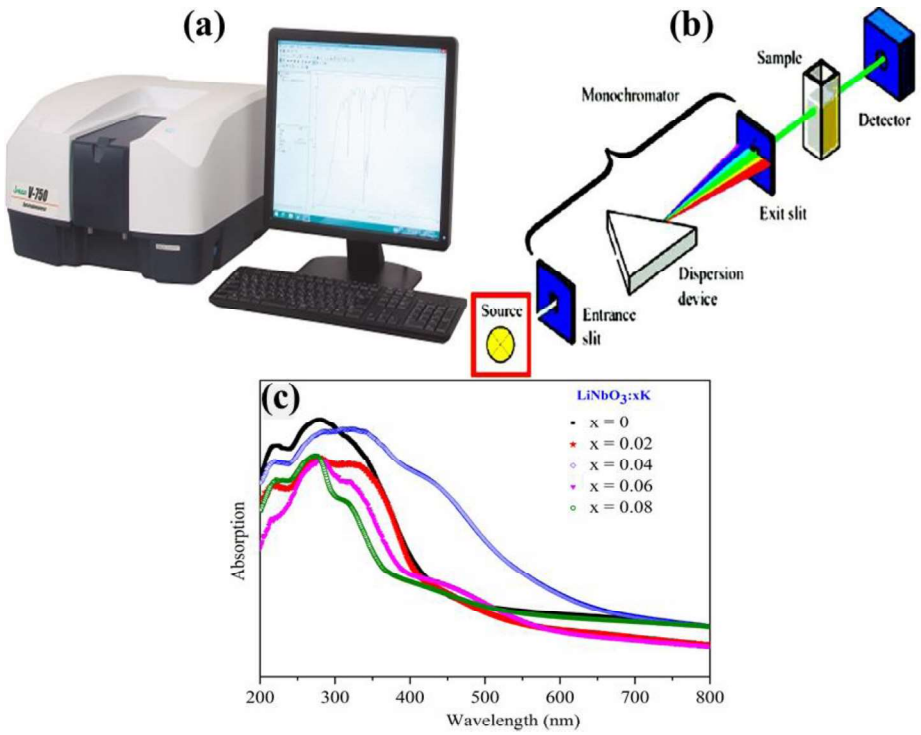


Figure 2.7(a-c): (a) UV Instrument (Jasco v-650), (b) Schematic diagram of UV-Visible spectroscopy, (c) UV Spectrum of Ceramic Materials.

2.4.5 Fourier transform infrared spectroscopy

Fourier transform infrared (FTIR) spectroscopy is a powerful analytical technique that examine the transmission or absorption of infrared radiation by a material. In chemistry, materials science, and biochemistry, it is frequently used to identify and quantify chemical compounds, analyze molecular structures, and monitor chemical reactions. FTIR spectroscopy operates on the basis of interaction between an infrared beam and the sample being studied. The wavelength range of infrared radiation on the electromagnetic spectrum is 0.78 to 1000 microns (m), which lies between visible light and microwaves. Molecules vibrate and rotate due to the absorption of infrared radiation by their chemical bonding. The energy of the absorbed radiation is equal to the vibrational and rotational energies of the molecule, which are characteristics of its chemical structure. In an FTIR spectrometer, the infrared light comes from a source like a laser or a globar, and then it goes through an interferometer. The interferometer is made up of a beam splitter that splits the radiation into two beams. The beams are then reflected by mirrors and brought back together at the beam splitter. When the beams are put back together, they interfere with each other, making a pattern called an interferogram. The interferogram provides information about the spectrum composition of the sample, but it is not interpretable directly.

To get the spectrum of the sample, a mathematical algorithm is used which known as "Fourier transform" to the interferogram. The Fourier transform changes the interferogram from the time domain to the frequency domain. This creates a spectrum that shows how the sample absorbs or transmits light based on frequency. The result is called an FTIR spectrum, and it has peaks that match the different chemical bonds in the sample.

The FTIR spectrum can be analyzed in two ways: by comparing it to reference spectra in a spectral database or by using spectral interpretation software to match peaks to specific chemical bonds. Spectral databases have the spectra of thousands of known compounds. The spectra of unknown samples can be used to find out what they are by comparing them to the spectra of known compounds. Spectra interpretation software uses Mathematical Algorithms to identify peaks in the spectrum and correlate them with type of the bonding and functional groups. FTIR spectroscopy is better than other ways of analyzing samples in many number of ways. It doesn't destroy or change samples properties, so they can be examining without being destroyed or changed. It is also very sensitive and able to find out the compounds in a sample those are present at a very small quantity. FTIR spectroscopy is also very flexible. It can be used to analyze solids, liquids, and gases, so it can be used in many different situations. The schematic diagram of FTIR shown in **Figure 2.8(a)**.

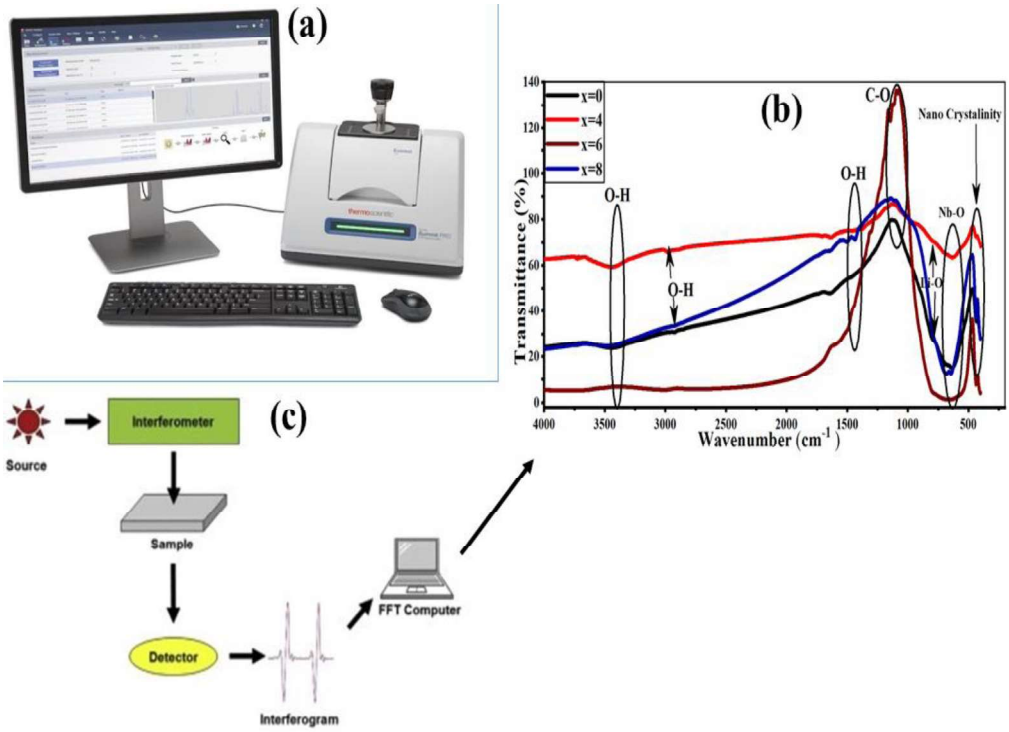


Figure. 2.8(a-c): (a) FTIR Instrument (ThermoScientific Nicolet summit), (b) FTIR Pattern of ceramic materials, (c) Schematic Diagram of FTIR Instrument.

2.4.6 Electric and Dielectric Measurement

The sintered samples pellets were polished with emery paper of different grade to get a flat smooth surface and the flat surfaces painted with Silver (Ag) paint which know as electroplating for the electrode making purpose, electroplated pellets cured at 550°C for 15 minutes. The Capacitance (C) of the particular sample as well as the dielectric loss (tanδ) were measured as a function of frequency (100 Hz -1 MHz) in the temperature range 30°C – 500°C.

The dielectric constant (ε_r) of each ceramic pellet was determined using the equation shown below.

$$\varepsilon r = \frac{C \times d}{\varepsilon 0 \, d} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 2.3$$

where ε_0 is the dielectric constant of empty space ($8.854\ 10^{-12}$ F/m), C is capacitance (in farad), A is electrical conductor area (in sq. m), and d is dielectric layer thickness (in mm).

Dielectric permittivity and tangent loss investigated by varying the frequencies and the temperature for all the ceramic pellets. Impedance spectroscopy was used to examine the contributions of the materials' grains and grain boundaries for the resistance and capacitance of the samples. The equivalent electrical circuit known as Schering bridge is shown in the Figure 2.9(b), where the Schering bridge is a basically a four arm AC bridge, which provide the measurement on the balancing of the loads on its arm.

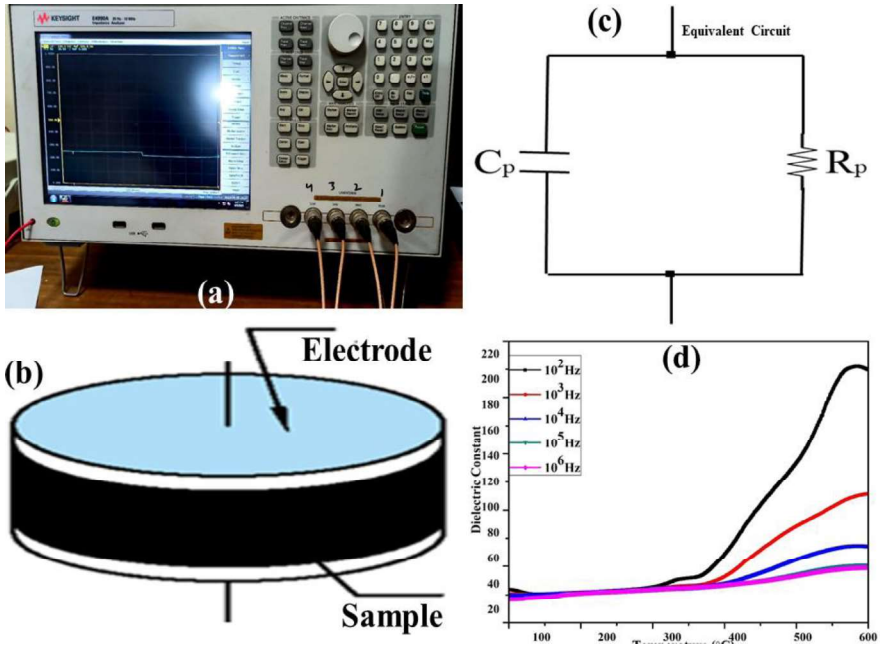


Figure 2.9(a-d): (a) Impedance Analyzer (Keysight), (b) Ceramic sample with electroding, (c)Equivalent Circuit, (d) Dielectric Constant of ceramic materials.

2.4.7 Polarization-Electric field hysteresis loop characterization

Ferroelectric materials shows spontaneous polarization. The P-E hysteresis loop measures ferroelectric behavior. **Figure 2.20** depicts a P-E hysteresis loop based on the Sawyer-Tower circuit. During P-E loop measurements, voltage is cycled using signal generators. A ferroelectric capacitor (sample) is linked to a reference capacitor in Sawyer-Tower. The polarization 'P' is calculated using the formula P= Q/A = (C*V/A), where 'Q' is the capacitor charge, which is the same for both capacitors because they are connected in series, 'A' is the sample electrode area, and 'V' is the voltage across the reference capacitor, which is measured with an oscilloscope. Coercive field and residual polarization are provided via the P-E loop. The Precision Premier-II ferroelectric tester from Radiant Technologies was used in this experiment to examine ferroelectric polarization as a function of the external field.

At various temperatures as well as at room temperature, P-E hysteresis loops were captured. Using 1Hz to 1kHz sinusoidal AC-voltage, PE loops are monitored.

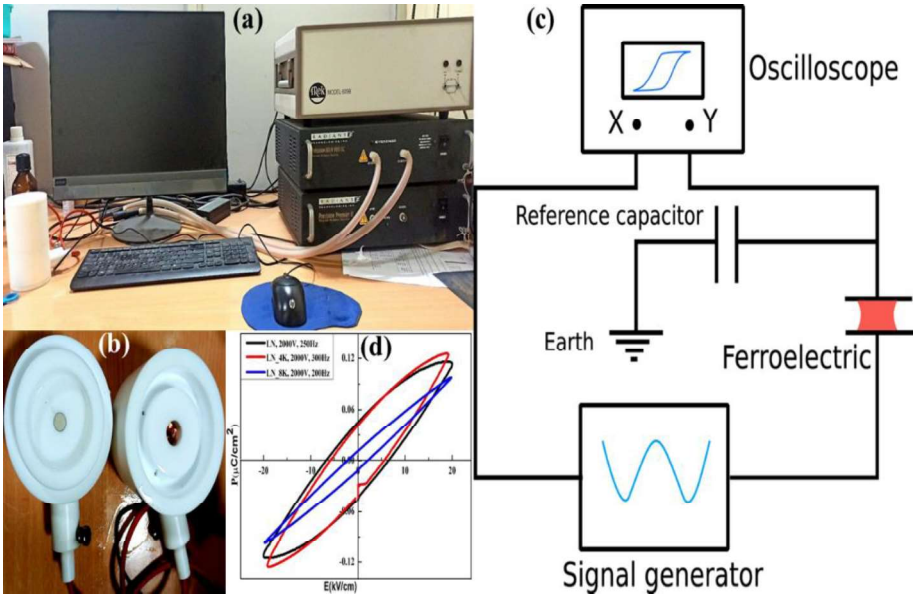


Figure 2.10 (a-d): (a) PE Analyzer Instrument (Radiant Technology), (b) Sample Holder with Si oil, (c) Schematic diagram of PE Analyzer, (d) PE Loop of ceramic materials.

2.4.8 Photoluminescence spectroscopy

Photoluminescence is a nondestructive, contactless optical method to examine electronic structure of materials. Photoexcitation is a process which occurs when light is bombarded on a sample; light is absorbed and transfers more energy into a material. The sample has two options for releasing this extra energy like as luminescence or light emission. Through this light can be examined spectrally, spatially and temporally. When a material is exposed to light, immediately a transitions occur to a higher electronic state, followed by the release of energy (photons) as the materials relaxes back to its lower energy state. The light or luminescence that results from such a process is known as photoluminescence (PL). The schematic diagram of the apparatus, a photoluminescence spectroscopy picture, and an output data plot are all displayed in **Figure 2.11 (b).**

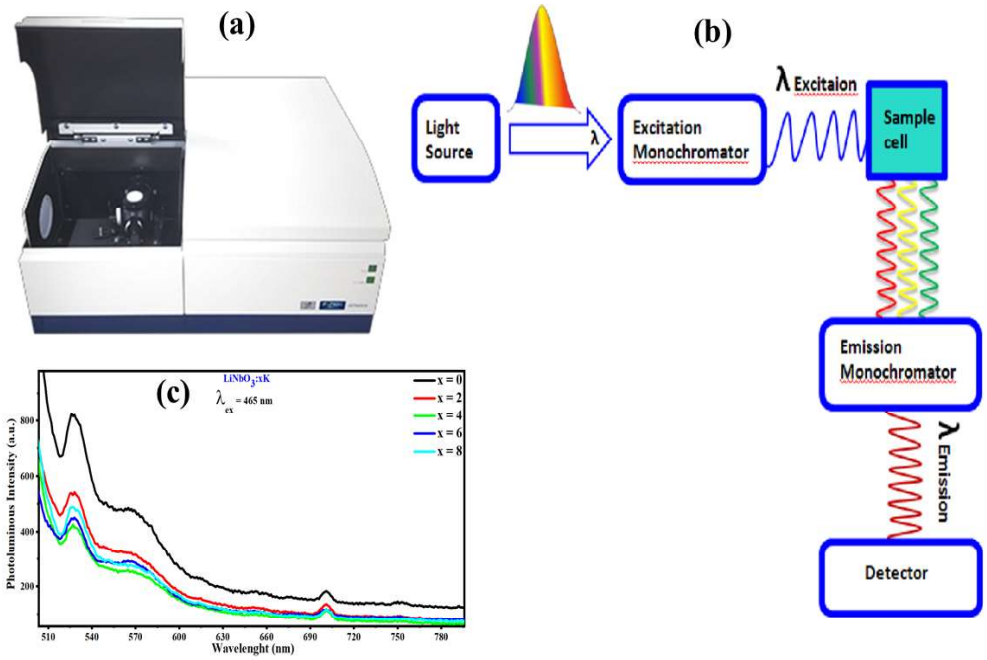


Figure 2.11 (a-c): (a)PL Instrument, (b)Schematic diagram of Photoluminscence spectroscopy, (c) PL Spectra of Ceramic materials.

2.4.9 Thermogravimetric analyzer:

TGA (Thermogravimetric analysis) is a method used to investigate the thermal characteristics of materials. It determines the weight change of a sample while it is heated or cooled in a controlled setting. TGA analyzers are widely utilized in R&D, quality control, and industrial applications in a variety of sectors including polymers, pharmaceuticals, ceramics, and food. TGA is an assembled unit of A sample holder, a furnace, a boiler, a thermocouple, and a data gathering system. The sample holder, which can be formed of ceramic, metal, or quartz, can contain a small amount of sample (usually a few milligrams). The balance is used to determine the sample's weight, which is usually accurate to a few micrograms.

In the TGA analyzer a heating furnace is uses which heats the sample at desired constant heating rate. As design capabilities of the furnace the sample can be heated from room temperature to 1500°C. The sample and furnace temperatures are both detected by the

thermocouple. Temperature and weight data are recorded by the data collection system and plots at the monitor. During the TGA experiment, either the sample was heated or cooled, it was maintained at a steady state, and the change in weight was continually recorded. Thermal phenomena such as evaporation, breakdown, oxidation, and reduction generate the weight change. The temperature at which these processes take place can provide important details about the material's thermal stability as well as the composition, and degradation kinetics. Several kinetic models, such as the Kissinger process, Ozawa-Flynn-Wall technique, and Coats-Redfern method, can be used to the TGA data to compute the activation energy of the decomposition reaction. TGA can also be used with other analytical methods, such as mass spectrometry (MS) and Fourier transform infrared spectroscopy (FTIR), to identify the gases generated during thermal processes. This can also provide vital information on the chemical composition and reaction process of the materials. The schematic TGA diagram is shown in the **Figure 2.12 (b)**.

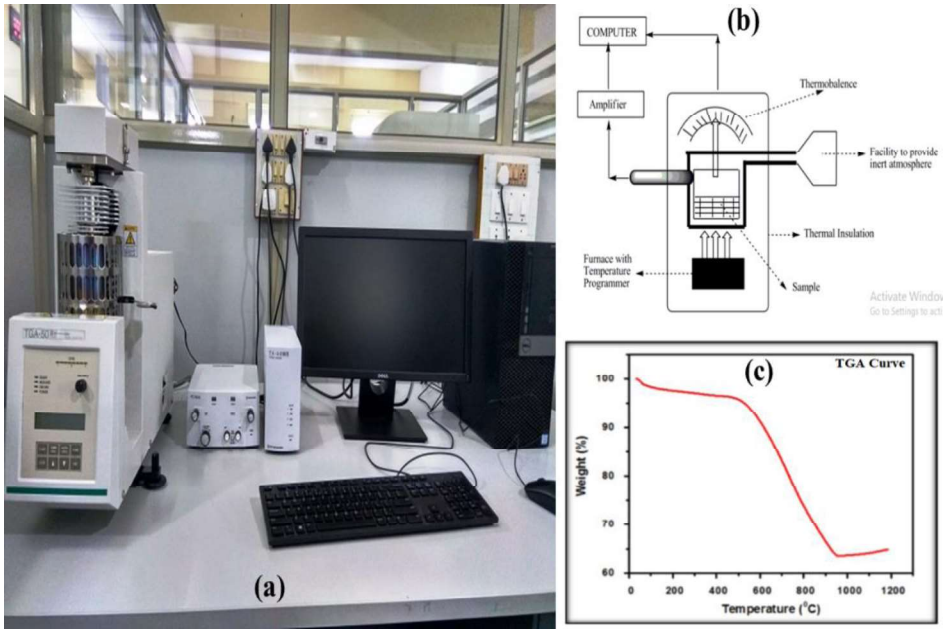


Figure 2.12 (a-c): (a)TGA Instrument, (b) Schematic diagram of TGA analyzer, (c) TGA Curve of a ceramic material.

2.5 Conclusion:

In this chapter we tried to briefly describe the materials synthesis process and the adopted characterization techniques. We have prepared all the materials composition by using solid state reaction method. Different characterization methods and updated latest corresponding software has been used to analyze the data to get the characteristics of the prepared materials. The subsequent chapters will discuss the all the characterization results and analyze data of all prepared samples.