3.1 Introduction:

In the present study is to Prepare and structural characterisation of MgO nanoparticles by using sol-gel method and liquid phase method with large surface area in short reaction time at room temperature and this method is the simplest, cost effective, ecofriendly method.

3.2 Materials and methods:

3.2.1 Prepartionof MgOnanoparticles by using sol-gel method:

3.2.1.1. Chemicals:

Magnesium Nitrate Hexahydrade [$Mg(NO_3)_2(H_2O)_6$], and Ammonium solution NH_4OH, and lemon juise.

3.2.1.2. Method:

Initially Extract from lemon juice prepared and filtered with filter paper. 20ML of lemon juice extract was taken and placed in a 100ML beaker, then 5.0g of magnesium nitrate hexahydrade was added to it.

After that, mixture kept under magnetic stirring until the magnesium nitrate is will dissolved. Drops of ammonia were added to the solution until PH= 7, then the solution kept in magnetic stirring at 80 ^oC until gel liquid was formed.

After 1h, the temperature was raised to 120 ^oC until xerogel was formed after ignition process.

Finally the product is calcinated at 500 ^oC for 150 min. the fine powder was collected which is MgO with white color for testing.

3.2.2. Prepartionof MgOnanoparticles by using liquid phase method:

The synthesis of MgO nanoparticles is divided into various steps, such as mixing, stirring, filtering, drying and calcination. Finally by the calcinating the powder at 500^{o} C for 2.5 h, the MgO is obtained in the nanoparticles form.

3.2.2.1. Chemicals:

Magnesium Nitrate Hexahydrade [$Mg(NO_3)_2(H_2O)_6$], and Sodium Hydroxide (NaOH) powder, The distilled water, and Ethanol used as a solvent and washing reagent in the chemical reaction respectively.

3.2.2.2. Method:

Initially the Magnesium Nitrate Hexahydrade of wt. 5.21 gm (0.2 M) and dissolved in 100 ML of distilled water. The 0.8 gm (0.2 M) of NaOH in 100 ML distilled water. Then 100 ML of NaOH solution is added in solution of [$Mg(NO_3)_2(H_2O)_6$] drop-wise by using glass rod. After that, solution kept under magnetic stirring for 2 h, after stirring the solution was kept on table at rest for 2 h so that, the precipitation is formed at the bottom of beaker. This precipitation was filtered and washed several times by using distilled water and Ethanol so as to get the final products.

The final product is kept in vacuum oven at 80°C for 4 h for drying product and removing the moisture. This dried powder is then crush and make it very fine powder by using mortal pestle. Finally the fine powder of MgO is calcinated at 500^{o}C for 2.5 h for the removal of impurities present in the powder. So that we will get synthesized MgO possessed high crystallinity with the particle size in nanosized range.

3.2.2.3. Reaction:

$Mg(NO_3)_2 + 2NaOH \rightarrow Mg(OH)_2 + 2Na(NO_3)$

$Mg(OH)_2 \xrightarrow{500\,^{c}} MgO + H_2O$

3.3 Instrumentations:

3.3.1 Infra-Red spectroscopy:

Infra-Red Spectroscopy (IR) was recorded on (FTIR). 8400, instrument (shimadsu , Japan).

Molecules and crystals can be thought of as systems of balls (atoms or ions)connected by springs (chemical bonds). These systems can be set into vibration, and theyvibrate with frequencies determined by the mass of the balls (atomic weight) and by the stiffness of the springs (bond strengths). The molecular and crystal vibrations are at very highfrequencies ranging from 10^{12} – 10^{14} Hz (3-300 μm wavelength), which are in the infrared (IR) region of the electromagnetic spectrum. The oscillations induced by certain vibrationalfrequencies provide a means for matter to couple with an impinging beam of infraredelectromagnetic radiation and to exchange energy with it when the frequencies are inresonance. These absorption frequencies represent excitation of vibration of the chemicalbonds and are thus specific to the type of bonds and group of atoms involved in the vibration.In the infrared spectroscopy, the intensity of a beam of infrared radiation is measured beforeand after it interacts with the sample as a function of light frequency. A plot of relativeintensity versus frequency is the infrared spectrum. The term ‘FTIR’ refers to FourierTransform Infrared Spectroscopy, when the intensity-time output of the interferometer issubjected to a Fourier Transformation to convert it into the familiar infrared spectrum(intensity

versus frequency). The identities, surrounding environments and atomicarrangements, and concentration of chemical bonds that are present in the sample can bedetermined.

3.3.2 Scanning Electron Microscope (SEM):

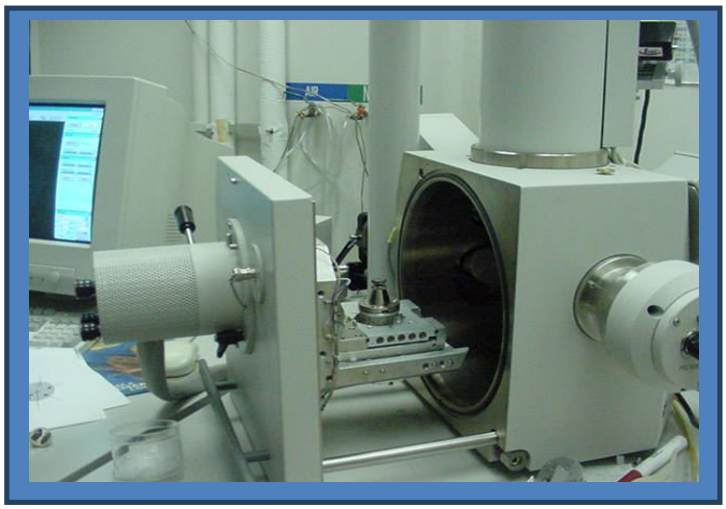

Fig. Scanning electron microscope device

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. The electron beam is scanned in a raster scan pattern, and the position of the beam is combined with the intensity of the detected signal to produce an image. In the most common SEM mode, secondary electrons emitted by atoms excited by the electron beam are detected using a secondary electron detector (Everhart-Thornley detector). The number of secondary electrons that can be detected, and thus the signal intensity, depends, among other things, on specimen topography. Some SEMs can achieve resolutions better than 1 nanometer.

Specimens are observed in high vacuum in a conventional SEM, or in low vacuum or wet conditions in a variable pressure or environmental SEM, and at a wide range of cryogenic or elevated temperatures with specialized instruments[18].

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD). The design and function of the SEM is very similar to the EPMA and considerable overlap in capabilities exists between the two instruments.

3.3.3 Ultraviolet–visible spectroscopy (UV):

Ultraviolet–visible spectroscopy(UV–Vis) refers to absorption spectroscopy or reflectance spectroscopy in part of the ultraviolet and the full, adjacent visible spectral regions. This means it uses light in the

visible and adjacent ranges. The absorption or reflectance in the visible range directly affects the perceived color of the chemicals involved.

In this region of the electromagnetic spectrum atoms and molecules undergo electronic transitions. Absorption spectroscopy is complementary to fluorescence spectroscopy, in that fluorescence deals with transitions from the excited state to the ground state, while absorption measures transitions from the ground state to the excited state.

Molecules containing bonding and non-bonding electrons (n-electrons) can absorb energy in the form of ultraviolet or visible light to excite these electrons to higher anti-bonding molecular orbitals.

The more easily excited the electrons (i.e. lower energy gap between the HOMO and the LUMO), the longer the wavelength of light it can absorb. There are four possible types of transitions (π–π*, n–π*, σ–σ*, and n–σ*), and they can be ordered as follows :σ–σ* > n–σ* > π–π* > n–π*.