SYNTHESIS AND CHARACTERIZATION OF STRONTIUM OXIDE NANOPARTICLES USING HENNA LEAVES EXTRACT AND ITS APPLICATION TOWARDS MCR REACTIONS

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Science Degree in Chemistry

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **J. Jenifer Shameni (39030009) and S. Rubiny (39030027)** who carried out the project entitled "**SYNTHESIS AND CHARACTERIZATION OF STRONTIUM OXIDE NANOPARTICLES USING HENNA LEAVES EXTRACT AND ITS APPLICATION TOWARDS MCR REACTIONS"** under my supervision from December 2021 to April 2022.

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Submitted for Viva voce Examination held on____________________

INTERNAL EXAMINER **EXTERNAL EXAMINER**

DECLARATION

We **J. JENIFER SHAMENI and S. RUBINY**, hereby declare that the Project Report entitled **"SYNTHESIS AND CHARACTERIZATION OF STRONTIUM OXIDE NANOPARTICLES USING HENNA LEAVES EXTRACT AND ITS APPLICATION TOWARDS MCR REACTIONS"** done by us under the guidance of **Dr. T. Krithiga MSc., M. Phil., Ph.D.,** Associate Professor Department of Chemistry, Sathyabama Institute of Science and Technology is submitted in partial fulfilment of the requirements for the award of Bachelor of Science degree in Chemistry.

DATE:

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J. JENIFER SHAMENI

S. RUBINY

ABSTRACT

Strontium oxide nanoparticles were green synthesized by using strontium nitrate and sodium hydroxide at room temperature in presence of henna leaves extract which is very simple and cost effective method. The characterization of synthesized strontium oxide nanoparticles was done using X-ray diffraction, scanning electron microscopy (SEM) and Fourier transform infra-red(FTIR) spectroscopy.

X-ray diffraction pattern indicates that the nanoparticles are crystalline in nature. The crystalline size of strontium oxide nanoparticle was calculated by Debye-Scherer formula and the crystallite size was found to be 40 nm.The surface morphology of nanoparticles was observed and investigated using SEM. The material at room temperature, shows crystallite of pseudo spherical shape with strong agglomeration of particles. The presence of Strontium – Oxygen bond (Sr – O) bond in the synthesized sample was confirmed by the peak at 821.23cm^{-1} in the FTIR spectrum of strontium oxide nanoparticles.

The catalytic activity of the synthesized Strontium oxide nanoparticles was tested by the one-pot synthesis of ethyl-6-amino-5-cyano-4-(4-hyroxy-3methoxyphenyl) -2-methyl-4H-pyran-3-carboxylateThe product formation was confirmed by the FTIR and Mass Spectrometry.

TABLE OF CONTENT

LIST OF ABBREVIATION

FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction
MCR	Multi Components Reaction
SrO	Strontium oxide
Nm	Nanometre

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1METAL OXIDE CATALYST

The metal oxide is found to be effectively catalysed in the midst of the 195s, especially when the reaction of oxidation and acid is effectively catalysed. They are involved in many petrochemicals, intermediate products, thin and pharmaceutical chemicals and biomass conversion reactions. They are also the basis of metal (monoorplurimetallic) catalysts for hydrogenation catalysts for metal oxide catalysts. The main catalytic domain covers oxidation (selective or general,), acid and basic catalysts, photo catalysts, dematic and biomass therapy. Dedicated to gas heterogeneous catalysts, this article believes that the interface between two environments, that is, a solid catalyst and reagent, and a reaction of the product medium

Metal oxides may simply be used as carriers for the active phase. B. Silica, alumina, silica-alumina, mesoporous oxides, MOF, etc. These can affect catalytic properties due to the synergistic, electron-conducting, and / and thermal-conductivity effects ensuring from the interactions of metal-oxide-transit

Among the various fields of heterogeneous catalysis, metal oxide catalysis is industrially used, such as silica, alumina, clay, zeolites, TiO2, Zno, ZrO_2, porous and mesoporous metal oxides. It is one of the most important because it covers most of the processes and catalyst families. ,Keggin or Dawson type polyoxometallate (POM), phosphate family (eg VPO, FePO4, silica-phosphate (SPA)), multi-component mixed oxide (molybdate) Salts, antimonates, tungstate, MoVTe). (Sb) NbO etc.), perovskite, hex aluminate, etc.

The metal oxide is found to be effectively catalysed in the midst of the 195s, especially when the reaction of oxidation and acid is effectively catalysed. They are involved in many petrochemicals, intermediate products, thin and pharmaceutical chemicals and biomass conversion reactions. They are also the basis of metal (monoorplurimetallic) catalysts for hydrogenation catalysts for metal oxide catalysts. The main catalytic domain covers oxidation (selective or general,), acid and basic catalysts, photo catalysts, dematic and biomass therapy. Dedicated to gas heterogeneous catalysts, this article believes that the

interface between two environments, that is, a solid catalyst and reagent, and a reaction of the product medium.

Metal oxide is a kind of inorganic material with special and diverse properties and applications like sensors, catalysts, fuel cells, etc. The oxide surfaces are terminated with O_2- oxide anions, since their size is much larger than that of the Mn cations. As a result, the symmetry and coordination of the Mn cations is lost surface. Furthermore, the surface of an oxide can contain different types of defects and environments (fold, ladder, ladder), plays a decisive role in the phenomenon of catalysis. This area unsaturation is usually compensated by reaction with water vapour, leading to the formation of the hydroxyls follow: O_2- $H_2O \rightarrow 2OH^-$. The OH group is the conjugate acid of the oxygen ions of the O2− crystal lattice, is the strong base and the conjugate base of the water molecule.[Jacques C.Védrine et al,2019]

1.2 METAL OXIDE NANOPARTICLES

In recent years, nanoparticles have been developed for agricultural use as Nano pesticide and Nano fertilizer (including the use of nanoparticles as Nano carriers for pesticides, fertilizers. In recent years, nanoparticles have been developed for agricultural use as Nano pesticide and Nano fertilizer consisting of nanoparticles as a Nano carrier for pesticides and fertilizers. Including nanoparticles, such as Nano zeolite (the building block of silicates [SiO_4] - and aluminate tetrahedral [AlO_4] -) as well as hydrogels (consisting of various polymers such as chitosan and alginate), which contributes to improved soil quality and Nano sensors (to monitor the health of plants and soil

Over the past decades, nanoparticles have been used in various household and industrial products. Due to the increasing use of nanoparticles in commercial products, various industries are developing new nanoparticles to improve their services and products.

Nanoparticles are classified as matter with at least one dimension <1 nm in diameter. Nanoparticles are not new to the environment and exist naturally as minerals, clays and bacterial products. It has been used since ancient times as a colorant for metals, but the systematic design and fabrication of nanoparticles for various uses has only begun in recent decades. Engineered nanoparticles are designed to have properties not found in large samples of the same material. The engineered nanoparticles are composed of a wide variety of materials and vary in size and shape along with a set of synthetic surface molecules, which distinguish them from natural materials. Metal and metal oxide nanoparticles exhibit different physicochemical properties and differ from their native bulk compounds in several respects, including surface, optical, thermal, and electrical properties of them.

Nanoparticles of metals and metal oxides of titanium dioxide(TiO_2), silver, zinc oxide, cerium dioxide, copper, copper oxide, aluminium, nickel and iron are most commonly used in industries and because which are mainly studied for their effects on plants. Several non-metallic nanoparticles, such as single-walled carbon nanotubes and fullerenes, have been thoroughly investigated to reveal their mechanism of nontoxicity (Joner et al., 28). On the other hand, the ability to grow as well as increase seed germination for different organs of maize, tomato, rice and soybean was observed when exposed to single-walled carbon Nano horns. [Roy.A, Bulut.O et al, 2019].

1.3 CHEMICAL SYNTHESIS OF METAL OXIDE NANOPARTICLES

Chemical Synthesis of Metal oxide nanoparticles has attracted great attention as a reliable, sustainable and environmentally friendly protocol for the synthesis of a wide variety of Nano materials/materials, including metal oxide Nano composites. As such, green synthesis is considered an important tool for reducing the destructive effects associated with traditional Nano particle synthesis methods commonly used in the laboratory and in industry. The new era of "green synthesis" methods / approaches is attracting a lot of attention in the research and development of materials science and technology today. Basically, the green synthesis of Nano materials/materials, produced through a process of adjustment, control, cleaning and treatment, will directly contribute to the improvement of the environmental friendliness of the plant. They. Therefore, some of the basic principles of "green synthesis" can be explained by several components such as waste prevention/reduction, reduction of derivatives/pollution and use of solvents/additives. Safer (or non-toxic) as well as renewable raw materials.

The chemical industry has been using natural gas and crude oil as its primary raw material. However, today there is a trend towards switching from fossils to renewable raw materials such as carbohydrates and triglycerides obtained from biomass [18]. A partial switch to renewable energy sources is desirable for reasons such as biocompatibility, biodegradability and reduced toxicity. Products based on renewable raw materials are obtained through photosynthesis from carbon dioxide and water and, after use, are returned to the biosphere through biodegradation in the form of CO2 and H2O. They are becoming more stable and cheaper compared to the rapidly rising gas and oil prices. The development of “green” products that can replace oily products and the introduction of “green synthesis” methods for producing chemicals from biomass are key to the transition to renewable feed stocks. For example, chemical reaction catalysts for chemical reactions can be obtained from biomass.

In this study, we focus on biosynthetic procedures for MONP synthesis, including comparison between green synthesis and classical chemical methods, and several new directions for green synthesis of Nano particles from various plant parts, especially plant leaf extracts. Will be Reducing compound plants are the preferred choice for precious metal synthesis. Metal oxidevions can be reduced to the corresponding metal without other chemicals under microwave irradiation conditions using the mild solvent water.[Lalitha Gnanasekaran, R.,et al,2017]

1.4 GREEN SYNTHESIS OF METALOXIDE NANOPARTICLES

A variety of microorganisms and plant extracts have been used to efficiently synthesize metal nanoparticles for green synthesis. Therefore, the synthesis of nanoparticles by green synthesis method is the most convenient, easiest and most environmentally friendly way and minimizes the side effects of chemical and physical methods by preventing prevent the use of harmful chemicals and the formation of toxic/hazardous by-products. Nanoparticles are widely used because of their superior properties and have been intensively studied in recent years. The physical and clinical effects of antibacterial, antioxidant and non-toxic nanoparticles obtained from green synthesis are becoming increasingly important. Future studies will likely focus on obtaining nanoparticles with maximum antibacterial effect and minimal toxicity. For this reason, the synthesis of metal nanoparticles, in particular by non-toxic green synthesis, is used in many application areas such as cancer treatment, drug delivery, and sensor fabrication. Biology is of great importance today.

Green synthesis is necessary to avoid the generation of unwanted or harmful by

products through the establishment of reliable, sustainable and environmentally friendly synthesis processes. The use of ideal solvent systems and natural resources is essential to achieving this goal. Green synthesis of metal nanoparticles has been applied to accommodate various biomaterials. Among the green synthesis methods available for metal/metal oxide nanoparticles, the use of plant extracts is a fairly simple and easy process to produce conventional nanoparticles. Large tissue compared with bacterial and fungal mediated synthesis. These products are collectively referred to as biological nanoparticles. [Aravind Kumar.J et al,2021][Banerjee s. et al, 2012]

1.5 CATALYST INTRODUCTION

Strontium oxide is an alkaline earth flux that melts at 446°F (243°C), but the substance initiates melting activity above 1994°F (19°C). The properties of strontium oxides are difficult to describe, so they are often compared with other alkaline earth oxides such as calcium and barium oxide. Strontium oxide has strong melting and coloring properties like barium oxide, but not as strong. It adds strength and durability to a glaze like calcium oxide and it melts very slowly, increasing the melting range of a glaze**.**

Strontium has moderate viscosity and surface tension and medium to high expansion and contraction rates similar to calcium oxide. It is non-volatile at ceramic temperatures, sparingly soluble, and has no known toxicity. The less soluble source of strontium oxide is strontium carbonate, which usually contains CaO.

1.6 STRONTIUM OXIDE STRUCTURE

Strontium oxide, SrO_3, is a metal oxide commonly produced by the reaction of strontium with oxygen or by the decomposition of strontium carbonate. It is commonly used in the glass, ceramics and electronics industries and has other specific applications such as fuel cells and sputtering target materials

Strontium hydroxide, $Sr(OH)_2$, is a strong base, although it is not as strong as barium hydroxide or the alkali metals.

Strontium oxide is an alkaline earth flux that melts at 446°F (243°C), but the material starts fluxing above 194°F (19°C). The properties of strontium oxide are difficult to explain and are often compared to other alkaline earth oxides such as calcium and barium oxides. Both are similar. Although strontium oxide is not strong, it has fluxing properties and a strong color response similar to that of barium oxide. It gives strength and durability to glazes such as calcium oxide and extends the melting interval of the glaze because it melts very slowly.

SrO is additionally utilized in drugs, pyrotechnics, pigments, greases, soaps, and as a chemical intermediate. It's conjointly made as high-purity metallic element compound mobile sputtering targets with the very best attainable density and smallest attainable average grain sizes to be used in semiconductor, electrical phenomenon, and coating applications by chemical vapor deposition and physical vapor deposition and optical applications. One of the explanations why rare metals area unit restrained from wide use in production is their high price.[Greenwood .N.N et al,1997]

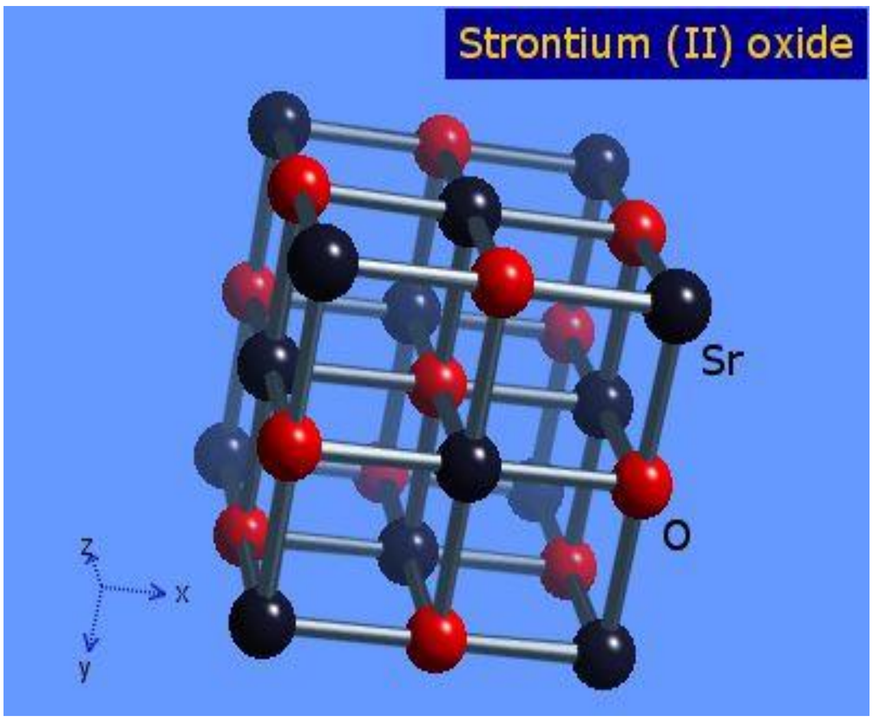


Fig: 1.1 Strontium oxide structure (SrO)

Source:https://www.webelements.com/compounds/strontium/strontium_oxide.html

1.7HENNA LEAVES

Henna leaves by the common names, henna, Inai, hina, reseda, Egyptian clover, used as a dye for skin, hair and nails, as well as for fabrics - silk, wool and leather [36]. Biologically, it is used in the treatment of hair and scalp problems as well as eye syndromes and hair loss. Henna leaves are rich in the compound naphthoquinone, a colouring molecule, lawsone, in addition to gallic acid, mucilage, and traces of alkaloids. Leaves soaked in ether or alcohol produce henna tannic acid and the green resin of olive oil.

Lawsonia inert Linn. (Lythraceae) is a very useful medicinal plant in all parts of the world. Powdered henna leaves are used to color hair, nails and beards (Chengaiah et al., 21). Inert Lawsonia leaves are used to treat polio and measles by the Yoruba tribe of south-western Nigeria. Henna seeds are said to have a deodorizing effect and are used in most cases of gynaecological disorders such as menorrhagia.

Henna leavesis a well-known ethno botanical species that has been used in cosmetics and medicine for over 9, years. Its use in traditional Indian folk medicines is well documented. [Singh.D.K et al,2015]

Fig 1.1 Henna leaves

Source:[https://www.google.com/search?q=lawsonia+inermis+plant&rlz=1C1VDKB_enIN981IN981&sxsrf=ALiCzsbarzj6D-TMfW30xY5-nUbB9qJYIA:1651819491514&source=lnms&tbm=isch&sa=X&ved=2ahUKEwjeh5mQo8r3AhW6L6YKHcakDOoQ_AUoAXoECAMQAw&biw=1366&bih=657&dpr=1#imgrc=dqofhXpJuG-qCM]

1.7.1 BIOLOGICAL DESCRIPTION

Henna leavesis a tall, branching, bare shrub or small tree up to 26 m in height that can be thorny. The bark is greyish-brown, glossy when young, and older plants have twigs with thorns at the tips. Young twigs are tetrahedral and green, but turn red with age.

Leaves opposite, whole, sessile, elliptical to broad lanceolate, 1.55 x .52 cm, smooth, hairy; the superior vein is concave.

Flowers are small, white, many; at the tip of a large, fragrant pyramid, 1 cm in diameter, at the bud with 4 wrinkled petals. The calyx has 2 mm tubules and 3 mm spreading lobes; petals are large ovoid, white or red; stamens 8, arranged in pairs at the edge of the calyx; The ovary has 4 segments, the type is up to 5 mm long, erect. Small spherical brown spherical fruit 48 mm in diameter, many seeds, irregularly opened, 4 segment split, tough form. Seeds 3 mm diameter, angular, thick seed coat[Beg, A.Z. et al 2002,].

1.7.2 PHYSIOCHEMICAL CHARACTERISTICS

Physiochemical disquisition of splint showed that the total ash was (14.60), acid undoable ash (4.50), water answerable ash (three. Zero), loss on drying (four.5), alcohol answerable extractive value (3.8 w/ w) and waterless extractive fee (5.0 w/ w) (22)[Ahmad, I. et al,2002].

1.7.3 CHEMICAL CONSTITUENTS

The phytochemical analysis of the aqueous extract of Henna leaves reveals the presence of carbohydrates, phenolic compounds, flavonoids, saponins, proteins, alkaloids, terpenoids, quinones, coumarins, xanthones, 6ts, 2-three% resin and seven-eight% tannins. Henna leaveshas 2-hydroxy-1, 4-naphthoquinone. HPLC evaluation showed that the extracts of Henna leaves [Jaber.K.K et al 2008]

1.7.4 ANTIMICROBIAL EFFECTS

Henna leavesleaves (ethanol, ethyl acetate and n-hexane) was found for their antibacterial (1000 μg/ ml) against Gram negative and Gram-positive bacterial strains (Proteus mirabilis, Pseudomonas aeruginosa, Staphylococcus epidermidis and Enterococcus faecalis) using slice prolixity assay system. All excerpts held antibacterial exertion against all the tested bacteria. Ethanol excerpt showed the loftiest antibacterial goods followed by ethyl acetate and n-hexane excerpts (55).

Antibacterial exertion of Henna leaves was determined against six bacterial strains (Escherichia coli (MTCCNo. 40), Staphylococcus aureus, Bacillus subtilis (MTCCNo. 10619), Salmonella typhi (MTCCNo. 3231), and Klebsiella and Pseudomonas aeruginosa (MTCCNo. 424)) by slice prolixity system. Crude ethanolic, hexane, ethyl acetate and waterless methanol bit held antibacterial exertion against all the tested bacterial strains especially when used as 20 mg/ fragment (52). The ethanol excerpt of Henna leavesleaves wielded antibacterial effect against Bacillus subtilis, Salmonella typhi, Sal. paratyphi, Pseudomonas aeruginosa, and Staphylococcus aureus, the MIC values of the ethanol excerpt were 800, 1200, 1600, 4000, and 1200 µg/ ml, independently (56). Antibacterial exertion of Henna leaves excerpts was studied. [Rubiay.A.I et al,2008]

1.8 MULTI COMPONENTS REACTION

Multicomponent Reaction (MCR) is a synthetic process that produces a single product from three or more reactants in a single reactor through a series of elemental reactions. Possible reagent combinations. The first recorded example of this reaction, Strecker's synthesis of amino nitrile from aldehydes, appeared in early 185, and shortly thereafter industrialized, particularly for the production of methionine, a common amino acid used as a raw material for drug synthesis.

Multi-component response (MCRS) contributes to the newly educated products that are all or most atoms in the convergence response that responds to the formation of the product with three or more source materials. MCR products are assembled according to cascades of basic chemical reactions. Therefore, everything finally moves to the steps that everything cannot be turned off and you have a counter-balanced equilibrium network to get the product. This task is to perform MCR in a way that the network of the primary mode of the main product is not to provide side products. The result depends on the reaction conditions of the type of solvent, temperature, catalyst, concentration, source material and functional group. These considerations are particularly important with respect to the development and discovery of novel MCR.

Multicomponent reaction (MCR) is a synthetic methodology in which three or more reactants come together in a single reaction vessel to form a new product. The characteristic aspect of MCR is that the final products contain almost all portions of substrates, generating almost no by-products. That makes MCRs an extremely ideal and eco-friendly reaction system. Target compounds can be obtained in one pot with much fewer steps. Therefore, MCRs have been paid much attention in various research fields, such as discovery of lead compounds in medicinal chemistry, or combinatorial chemistry[Brauch .S et al ,2013]

CHAPTER 2
LITERATURE SURVEY

Athar, Taimur published Synthesis and Characterization of Strontium Oxide Nanoparticles via Wet Process. They demonstrated the synthesis of Strontium Oxide (SrO) Nano powder how it was synthesized by hydrolysis of single source molecular precursor $SrCl_2 \cdot 6H_2O$ with potassium hydroxide. The structure, morphology and properties of the particle were characterized by XRD, TEM, Raman and UV-vis-spectroscopy. The mean particle size (28.6 nm) was calculated by using X-ray diffraction pattern. A crystalline nature and with controlled particle distribution and phase purity were established with XRD and FT-IR

Periasamyanbu, subash C. B. Gopinath, midhatnabilsalimi, iswaryletchumanan, sreeramanansubramaniam. Green synthesized strontium oxide nanoparticles by elodea canadensis extract and their antibacterial activity. Journal of nanostructure in chemistry 2021, 2 https://doi.Org/10.1007/s40097-021-00420-x

Anbu.PGopinath.M.NSalimi,IswaryLetchumnanS.subramaniam,green synthesis strontium oxide nanoparticles from an aquatic plant extract described FTIR results confirmed functional group reserved as caping agents during Strontium oxide Nanoparticles.

Ajay. K.D et al used Sulfated Titania (TiO_2-SO_4^{2-}) as a heterogeneous solid acid catalyst in multicomponent reactions in water to produce benzylamino coumarin derivatives by microwave irradiation (100°C) [Ajay. K.D et al, 1998]. Elemental analysis, Melting points, IR, 1H, and 2D NMR and 13C NMR, spectral data were used to validate the structure of those benzylamino coumarin derivatives.

Banerjee s. Green synthesis and characterization of metal Nanoparticles and its antimicrobial properties. Ph.D. Thesis. Jadavpur: Jadavpur University; 2012. The synthesis of highly crystalline metal oxide nanoparticles

Singhal G, bhavesh R, kasariya K, sharma AR, singh RP. Biosynthesis Of strontium nanoparticles using octimum sanctum (tulsi) leaf extract and Screening its antimicrobial activity. Chracterization of strontium nanoparticles was done using X-ray diffraction, SEM, FTIR.

Karpagavinayagam. P, vedhi. C Green synthesis of iron oxide nanoparticles using *Avicennia marina* flower extract Nanoscience and technology play an motion towards the eco-friendly, simple, sustainable and cost-effective green chemical methods for synthesizing materials rather than toxic chemical methodology. V.O. Chidambaram College Version of Record 28 November 2018. https://doi.org/10.1016/j.vacuum.2018.11.043

CHAPTER-3

AIM AND SCOPE

3.1 AIM

The main aim of the project is to synthesize strontium oxide nanoparticles by green synthesis method using henna leaves extract. The green synthesis of Strontium oxide nanoparticles was carried out using strontium nitrate and sodium hydroxide at room temperature in presence of henna leaves extract as reducing agent. This method is cost effective and simple and environmentally safe. SrO Nanoparticles were characterized with the help of FTIR, SEM, XRD spectroscopy techniques. The synthesized SrO nanoparticles were tested for its catalytic activity by the one pot synthesis ofethyl-6-amino-5-cyano-4-(4-hyroxy-3methoxyphenyl) -2-methyl-4H-pyran-3-carboxylate and the product was confirmed by FTIR and Mass spectroscopy.

3.2 SCOPE

- Synthesizing strontium oxide nanoparticles using henna leaf extract.
- Characterization of strontium oxide nanoparticles by FTIR Spectroscopy,XRD, SEM-EDX.
- To test its catalytic application of Strontium oxide by one pot Synthesis of substituted 4-H Pyrans using synthesized strontium oxide nanoparticle.
- Product confirmation byFTIR and Mass spectrometry.

CHAPTER-4

MATERIAL AND METHODOLOGY

4.1 MATERIALS

Materials used in this reaction are strontium nitrite, sodium hydroxide, vanillin, malononitrile, ethyl acetoacetate and ethanol. Which are bought from Merck chemicals and is used it is. Henna leaves are collected and dried in sunlight.

4.2 CHARACTERIZATION TECHNIQUES:

4.2.1 FTIR (Fourier Transform Infrared Spectroscopy)

FTIR analysis is used to identify organic, inorganic, and polymeric materials using infrared light to scan samples. Changes in the pattern of the suction bands clearly indicate a change in the composition of the material. FTIR is useful for identifying and identifying unknown objects, detecting contaminants, detecting additives, and detecting decay and oxidation.

The modern FTIR spectrometer includes a power supply, mobile sampler, detector, amplifier, A/V converter and computer. The radiation from the source passes through the interferometer and then reaches the detector. The signal is amplified by an analog-to-digital converter and amplifier, converted to a digital sign, and then the signal is sent to a computer to perform a Fourier transform.

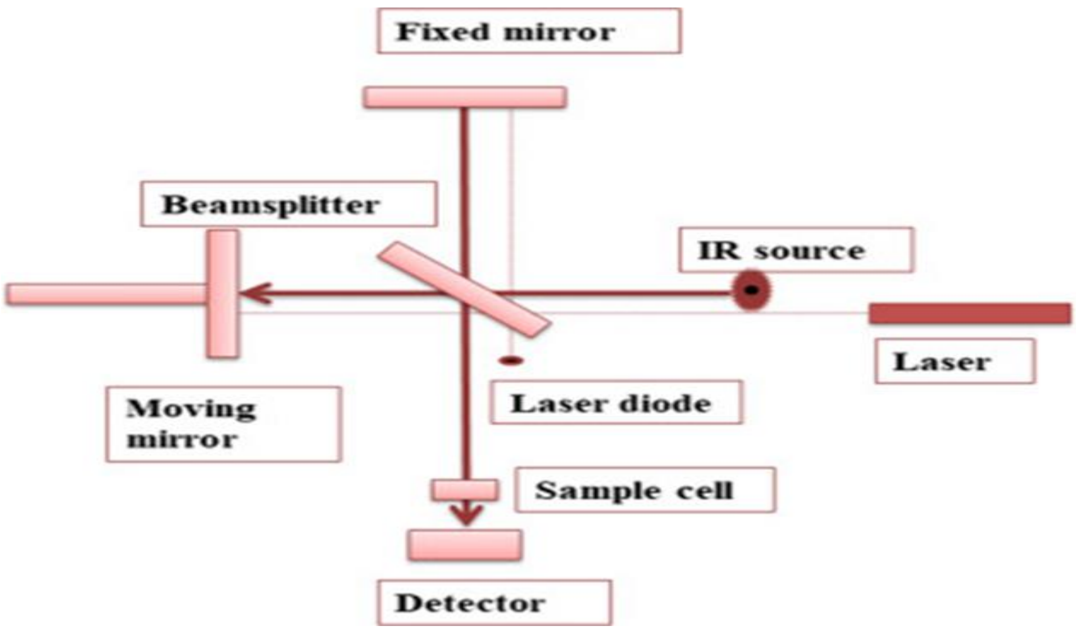


Fig:4.1 FTIR Schematic diagram

Source: https://www.sciencedirect.com/topics/engineering/fourier-transform-infrared-spectroscopy

4.2.2 SEM (Scanning Electron Microscopy)

Scanning electron microscope (SEM) provides high resolution, high magnification images of samples a material that emits a finely focused electron beam onto a sample. This beam interacts with the molecule Sample composition. These interactions produce a series of measurable electronic energies to be analyzed. Create a 3D image through a scanning electron microscope. The electron beam emitted to the sample also produces X-rays. Energy dispersive X-ray (EDX) The device collects x-rays and converts them into useful information. Each element has a set of properties X-ray line. Use energy dispersive X-ray technology to identify elements and measure composition of the sample material. The output of EDX analysis is a spectrum. The EDX spectrum is X-ray images are often taken at each energy level. The EDX spectrum usually shows the corresponding peak Energy level. These tips generally specific to the item. The higher peaks in the spectrum the concentration of this element is high. Overlapping peaks from the mixture Deployed on a dedicated computer software. Energy dispersive X-ray Often the system is an attachment

Watch with an electron microscope instrument. Usually scan the electron microscope Visual analysis and energy distribution X-rays provide elemental analysis. Scanning electron microscope Energy dispersive X-rays are a powerful tool for classifying and distinguishing materials because they can be done simultaneously. Study the morphology and elemental composition of objects. Some of the typical uses of SEM / EDX Identification and classification of various material structures, investigation of surface morphology, particles Contamination detection, structural analysis, forensic investigation, corrosion and oxidation detection Problems, products and processes error.

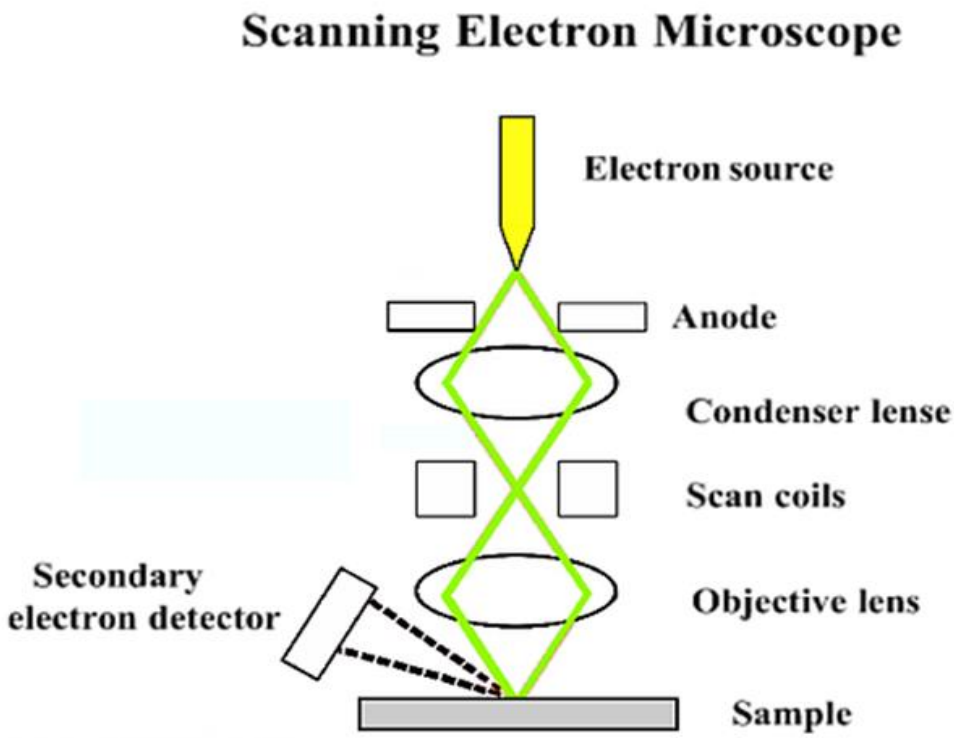


Fig: 4.2 SEM Schematic diagram

Source: https://www.thermofisher.com/blog/microscopy/what-is-sem-scanning-electron-microscopy-explained/

4.2.3 XRD

Powder X-ray diffraction (XRD) is a common characterization method for Nano scale materials. Analysis of Samples with powder XRD provide important information these are phase identification, sample purity, Crystal size and possibly morphology. As mass Technology that makes it possible to correlate the information it provides Microscopic data to test whether microscopic observations are small the number of particles is representative of most of sample. Despite its importance and ubiquity, the information is The XRD data of the Nano scale material contained in the powder is it is always fully utilized and in some cases misunderstood. This editorial is with Nano science Nanotechnology community with short tutorials on some of them often an important aspect of powder XRD data Occurs when analyzing samples of Nano scale materials focusing on inorganic nanoparticles of various sizes, Shape and dimensions. In this way, researchers collide with each other Powder XRD

X-ray diffraction (XRD) is one of the most widely used methods to differentiate NPs. Typically, XRD provides information about crystalline structure, phase environment, lattice boundaries and crystalline character sizes. The latest parameter is measured using a Scherer scale using the XRD maximum resolution of a particular sample. The advantage of XRD techniques, which are usually made in powder form samples, is usually after setting up their corresponding colloidal solutions, that it results in statistical representation, medium volume values. Particle formation can be determined by comparing the location and strength of the peaks with the reference patterns found in the International Center for Diffraction Data (ICDD, formerly known as the Joint Committee on Powder Diffraction Standards, JCPDS) database. However, it is not suitable for amorphous substances and XRD peaks are very broad in particles with a size less than 3.

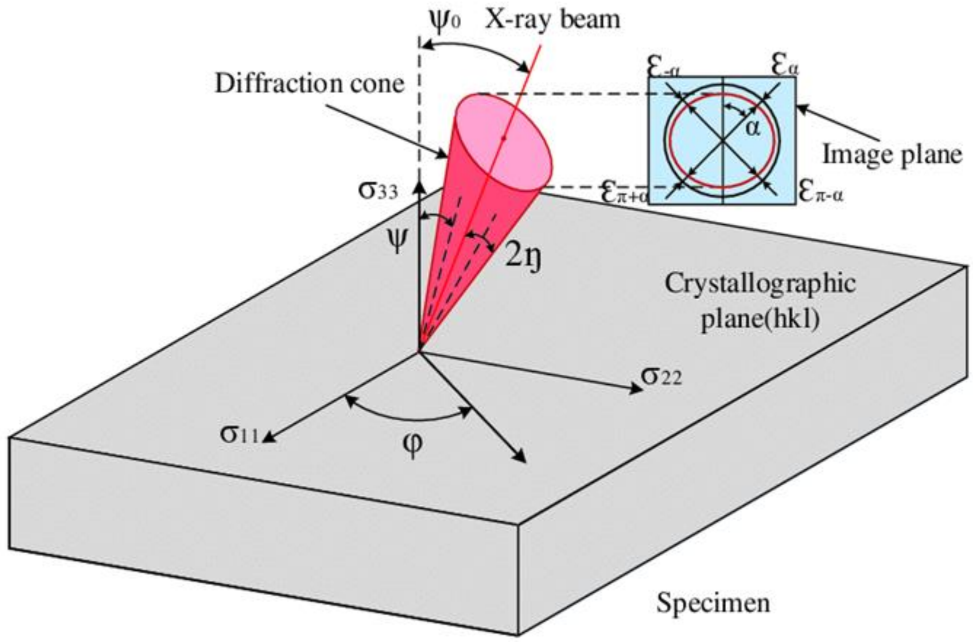


Fig:4.3 XRD Schematic diagram

Source: https://www.researchgate.net/figure/X-ray-diffraction-XRD-schematic-diagram-X-ray-diffraction-XRD-schematic-diagram_fig1_325774512

4.2.4 NMR (Nuclear magnetic resonance spectroscopy)

Nuclear magnetic resonance spectroscopy most commonly known as NMR spectroscopy or magnetic resonance spectroscopy (MRS), is a spectroscopic technique for observing local magnetic fields around a nucleus. This is a spectroscopy based on the absorption of electromagnetic radiation in the high frequency range of 4 to 9 MHz by the atomic nucleus.NMR has become an excellent technique for determining the structure of organic compounds of all spectroscopy's that is usually the only expectation of a complete analysis and interpretation of the entire spectrum.

NMR differs in that the discrete energy levels at which transitions occur are artificially created by placing the nuclei in a magnetic field.

The signal in the NMR spectrum is characterized by four attributes: resonant frequency, multiplicity, line width, and relative intensity. The analytical usefulness of NMR techniques lies in the fact that nuclei of the same type have different resonance frequencies when placed in different molecular environments.

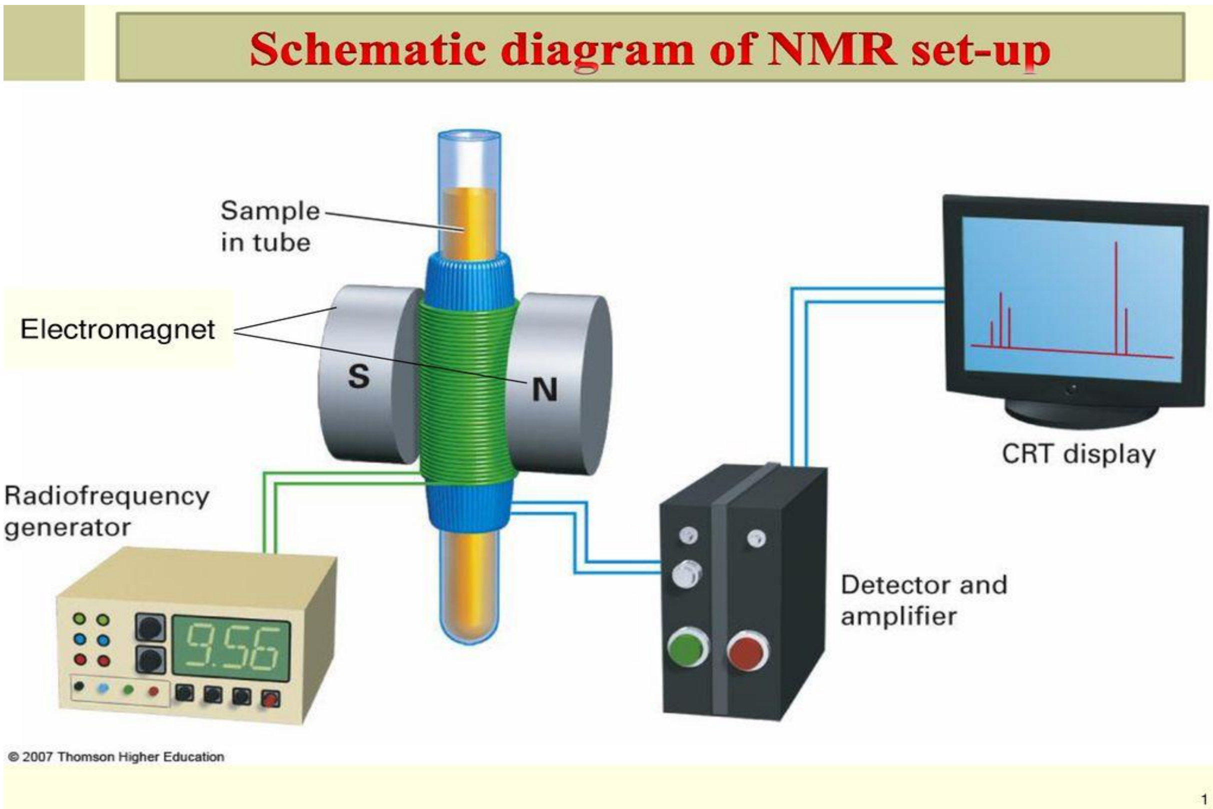


Fig: 4.4 NMR Schematic diagram
Source:https://slideplayer.com/slide/16246079/

4.3 METHODOLOGY

4.3.1 SYNTHESIS OF HENNA LEAF EXTRACT

In this preparation, fresh leaves of Henna leaves are washed, rinsed in double distilled water, and dried in the sun for 4-5 days. 5 g of dried leaves are heated to 80° C in 100 ml of doubly distilled water and stirred with a magnetic stirrer. After cooling the boiled solution, centrifugation is performed to obtain a leaf extract.

4.3.2 SYNTHESIS OF STRONTIUM OXIDE

To 100ml of Strontium Nitrate solution 30 ml of leaf extract is mixed and heated at 80℃ for 30 minutes. The pH of the solution is adjusted to 11 using 2M NaOH and it is added while stirring and heated at 70℃ for about 30 minutes. A Reddish Brown precipitate of Strontium Nitrate is obtained which is washed using water, filtered and kept to dry in hot air oven at 70℃ and the SrO Nano particles are obtained, which is used as a green catalyst.

4.3.3 ONE POT SYNTHESIS:

Synthesis of **ethyl-6-amino-5-cyano-4-(4-hyroxy-3methoxyphenyl) -2-methyl-4H-pyran-3-carboxylate (2):**

A mixture of vanillin (1.0 mmol, 0.106g), malononitrile (1.0 mmol, 0.066g), Ethyl acetoacetate (1.0 mmol, 0.13g) was added and refluxed at 80^{o}C for 2hr using Ethanol (10ml) as a solvent. After completion of the reaction, as indicated by TLC, the solvent was evaporated and the crude product was extracted from ethyl acetate and water.Yield 70%

CHAPTER-5
RESULTS AND DISCUSSION

5.1 CHARACTERIZATION OF STRONTIUM OXIDE NANOPARTICLE

5.1.1 FTIR Spectrum Strontium Oxide nanoparticles

FTIR spectrum of strontium oxide nanoparticle in figure 5.1 shows the peaks at 821.23 cm^{-1} which is due to Sr – O bond. A sharp peak at 1341.87cm-1, 1638.44cm-1, and 2361.50 cm-1 was because of H-O-H bending. The peak at 3000-3500 cm-1 region represents the –OH group and interstitial water molecule in the material.

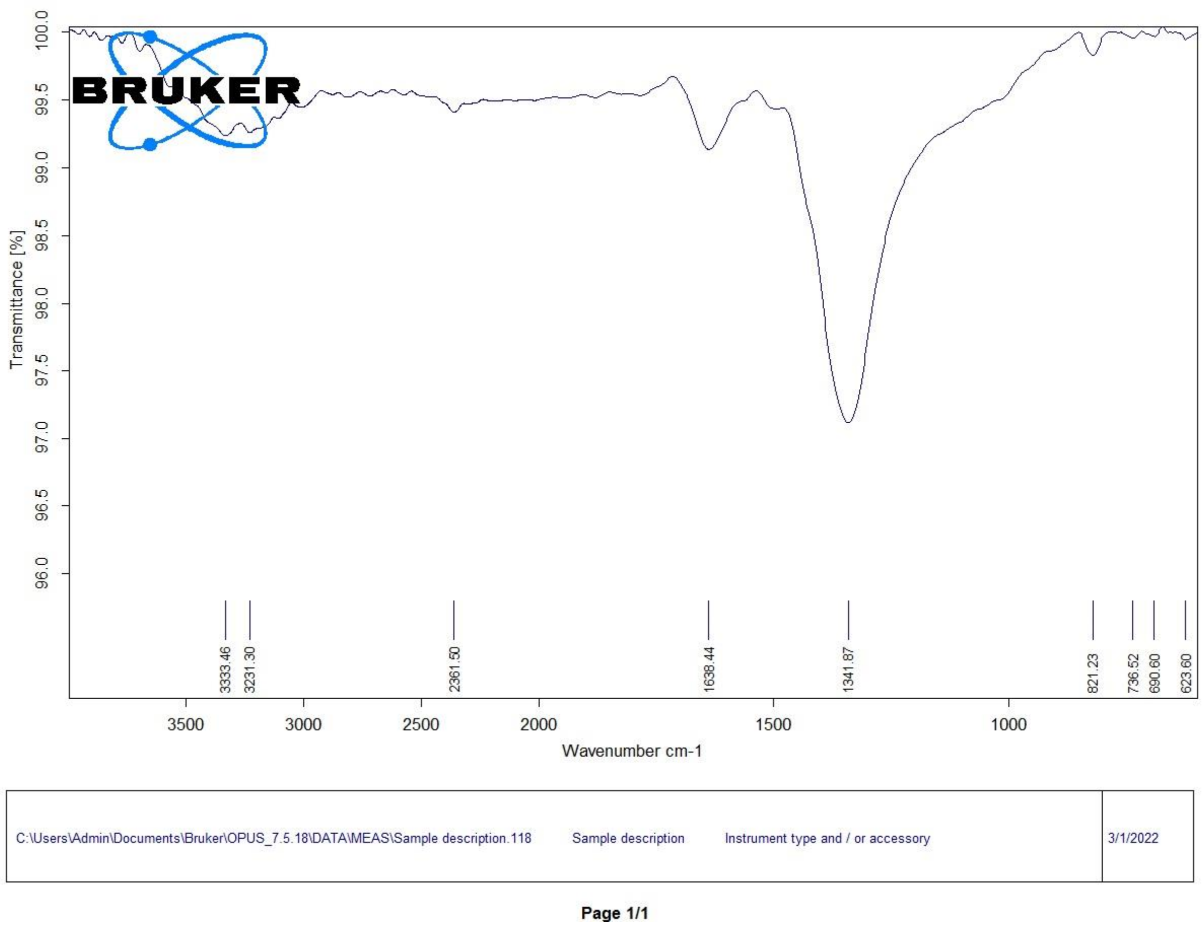


Fig 5.1 FTIR spectrum of Strontium oxide nanoparticle

Vibrational frequency	Functional group identification
3333.46 cm^{-1}	O-H Stretching of absorbable water Molecule on SrO.
2361.50 cm^{-1} 1638.44 cm^{-1} 1341.87 cm-1	O-H-O Stretching
821.23cm-1	Sr – OStretching

Table:5.1 FTIR Spectra of SrO Nanoparticles

5.1.2 SEM and EDAX image of Strontium oxide nanoparticle

The SEM image of strontium oxide nanoparticle is shown in the Fig. 5.2. The image explains that strontium oxide nanoparticle obtained have pseudo spherical shape. The SEM image also shows the presence of agglomeration of SrO nanoparticles. The EDAX spectra shows the presence of Sr (30.92 wt%) and O (49.49%) in the SrOnanoparticle catalyst**.**

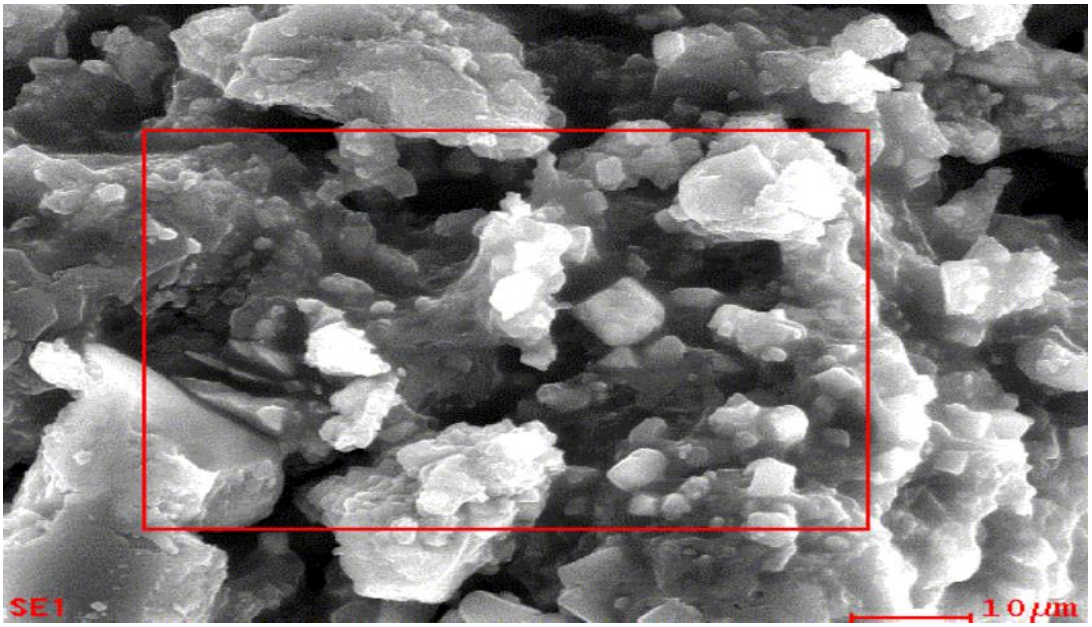
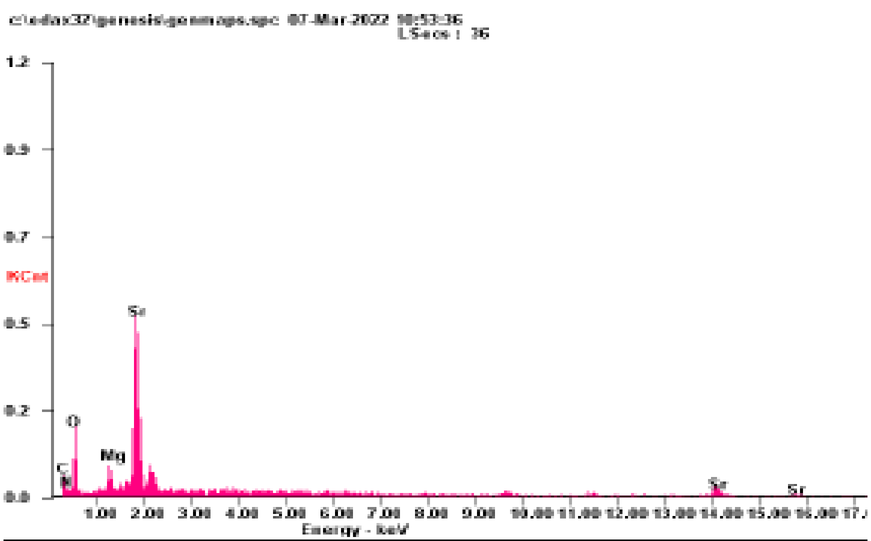


Fig: 5.2 SEM Image of strontium oxide nanoparticles



Element	Wt%	At%
NK	14.00	21.38
OK	49.49	66.16
MgK	05.59	04.92
SrK	30.92	07.55
Matrix	Correction	ZAF

Table 5.2 Elementals analysis of SrO

Fig: 5.3 EDAX Spectrum of SrO_2

5.1.3 XRD IMAGE OF STRONTIUM OXIDE NANOPARTICLES

X- ray diffraction pattern shows crystalline nature of nanoparticle. Diffraction peaks matches with the database (JCPDS file #6-520) which expressed the cubic structure. The Debye-Scherer equation $D = 0.94\lambda / \beta \cos\theta$ was used to calculate the crystalline size, where β is the full width at half maximum of peak. λ represent the X-ray wavelength and q referred as Bragg diffraction angle. The crystalline size is about 80nm. The x ray diffraction pattern of strontium oxide nanoparticles is shown in Figure. 5.3

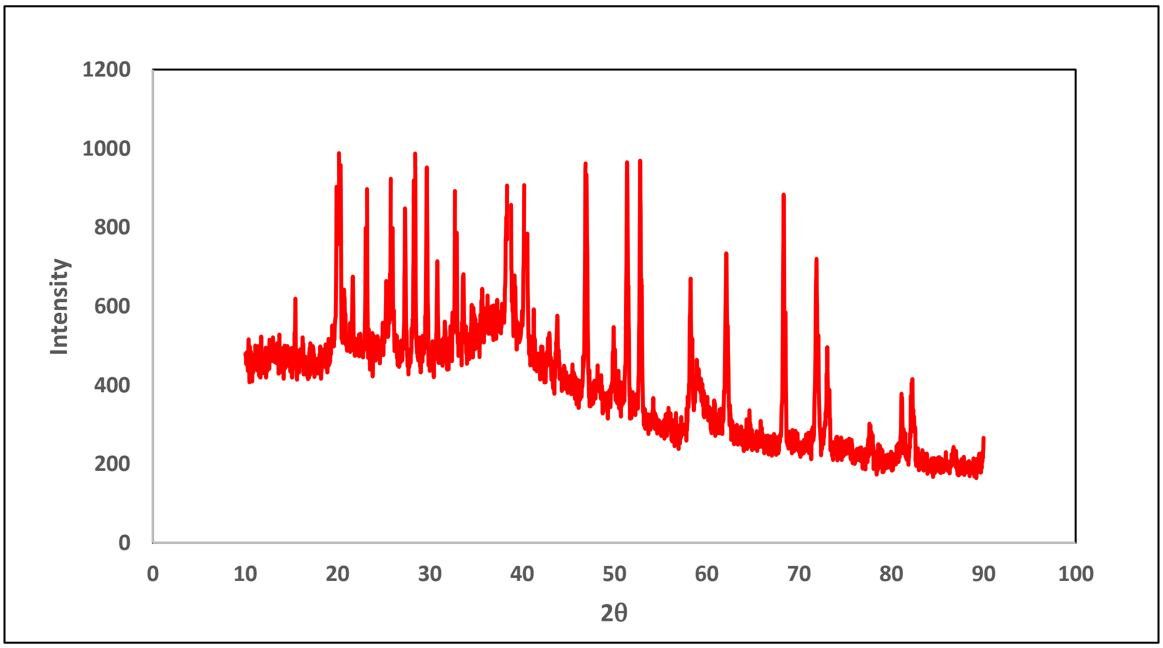
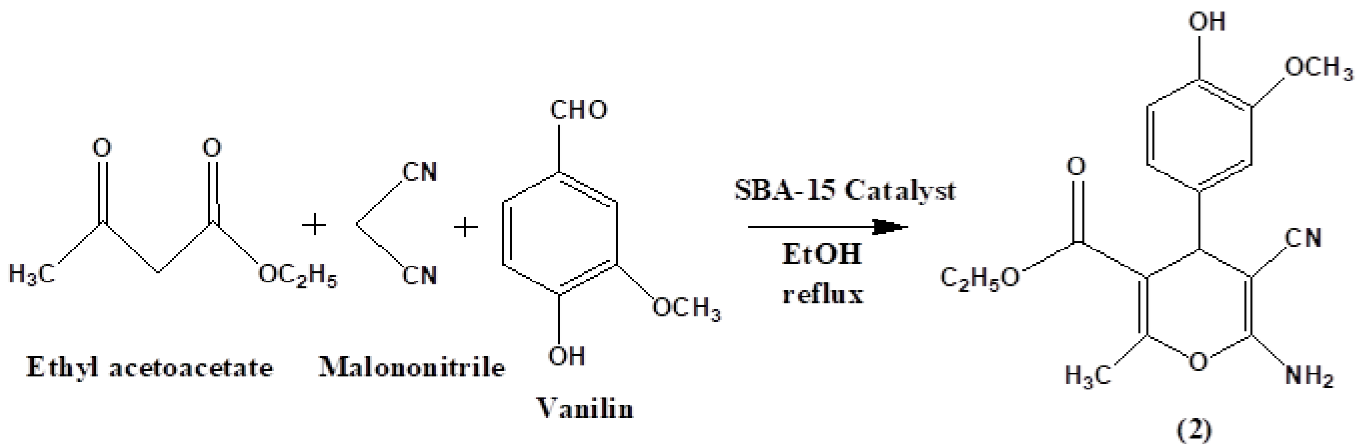


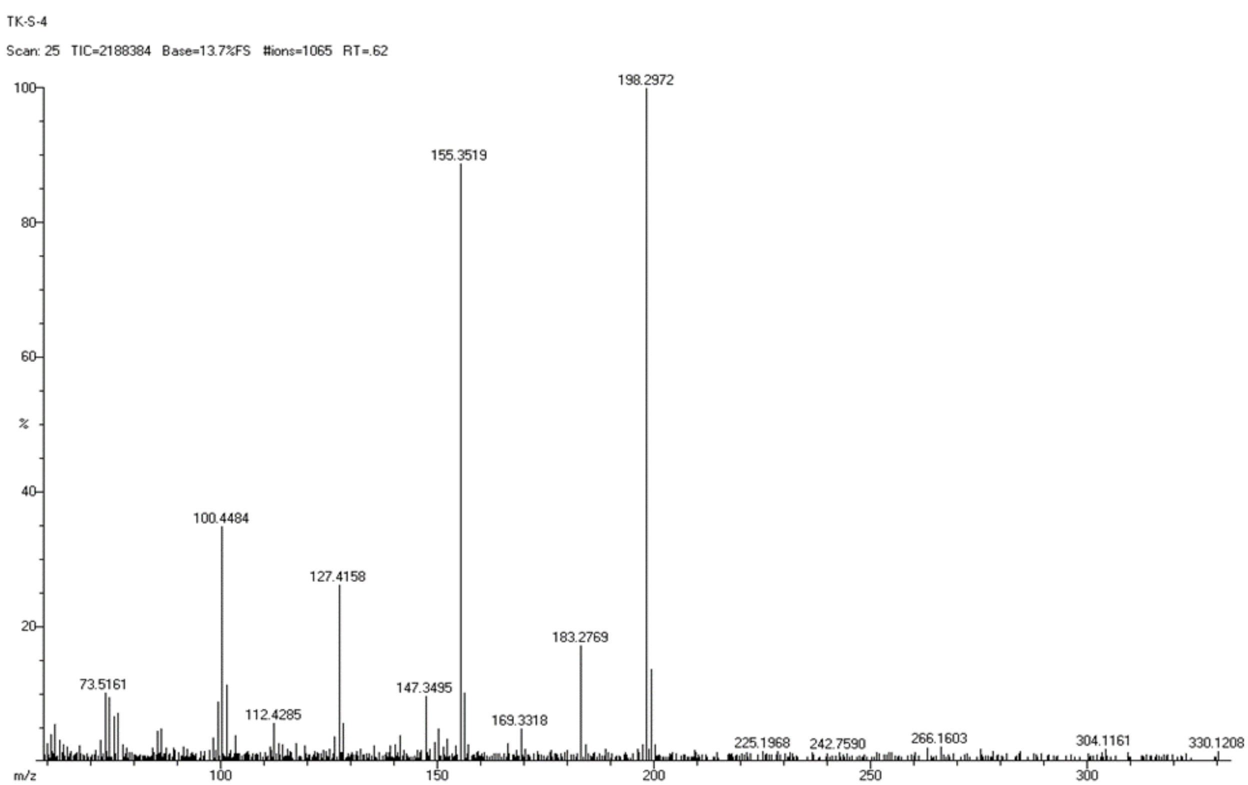
Fig: 5.4 XRD pattern of strontium oxide nanoparticles

5.1.4: Spectral Data of ethyl-6-amino-5-cyano-4-(4-hyroxy-3methoxyphenyl) -2-methyl-4H-pyran-3-carboxylate (2):



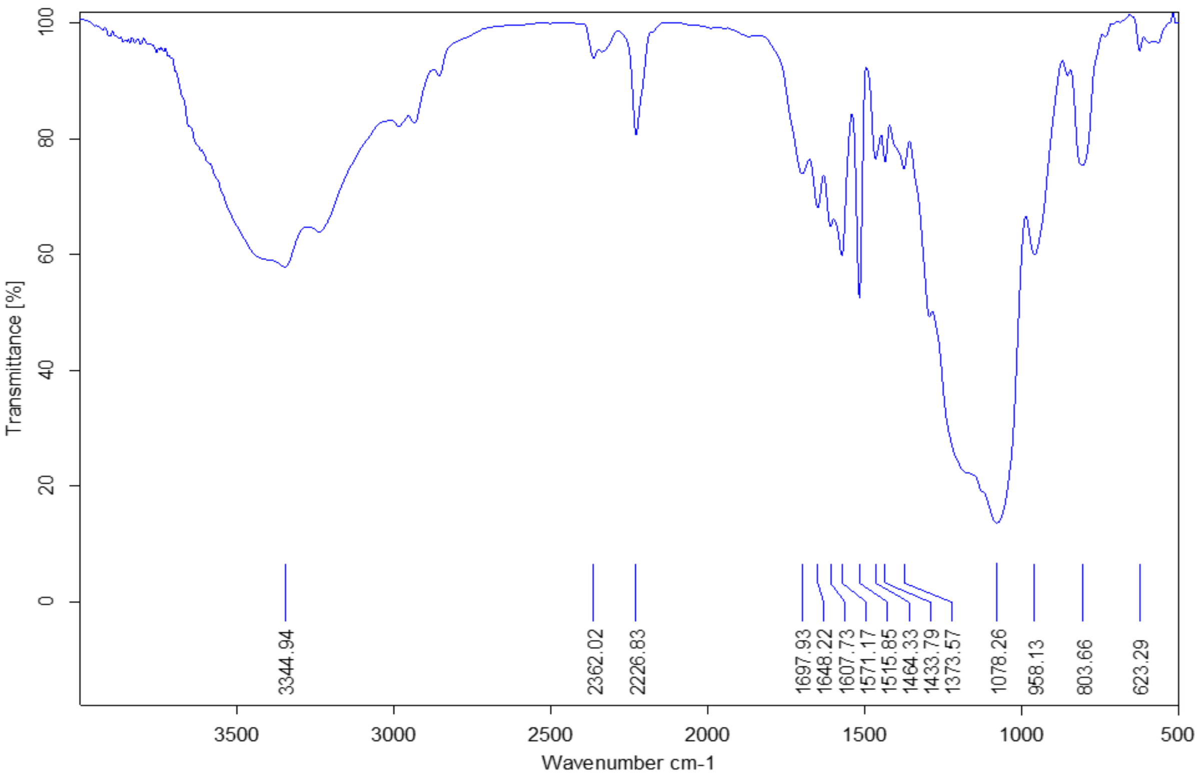
MASS DATA:

GCMS Data Exact mass 330.12 Observed mass 330.12 (M+), 330.34 (M+ H+)



FTIR DATA:

(KBr / cm-1): 1607 (NH), 2226 (CN), 1697 (C=O), 1078 (C-O), 3344(OH)

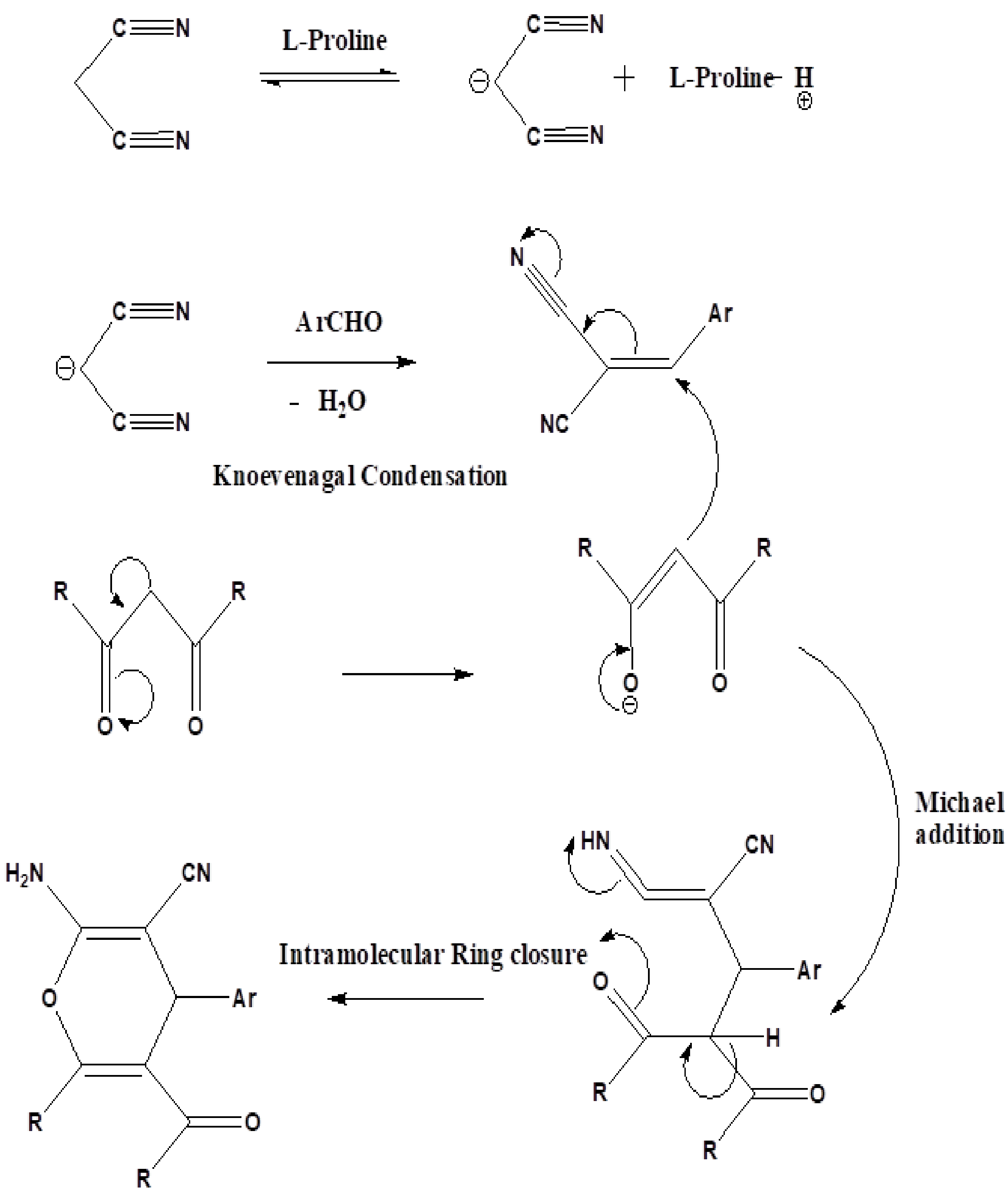


5.1.5

Page 1 of 1

MECHANISM OF THREE COMPONENTS REACTION OF 4H PYRANS

The mechanism of formation of 4Hpyran by the three-component coupling strategy generally begins with the Knoevenagel condensation between the active methylene compound and the aldehyde, followed by the Michael addition by the less reactive active methylene compound, and finally the intermolecular ring closure. This mechanism is very possible in the case of traditional approaches to the synthesis of these compounds. The first condensation of aromatic aldehydes and malononitrile in the presence of proline results in the formation of allylidene malononitrile with the loss of one water molecule. Nucleophile addition of enolizable ethyl acetoacetate to allylidene malononitrile followed by intramolecular cyclization of the resulting species yields 2amino4Hpyrans. In addition, in this study, a reduction in yield was observed when the three components were combined simultaneously compared to the combination of malononyltyl and aldehyde and the addition of the nucleophile 5 minutes later. When two active methylene compounds are present with an aldehyde, the first condensation reaction can occur first with malononitrile or dime done, each producing a different intermediate. Since malononitrile is a more reactive active methylene compound, the arylidene malononitrile intermediate should predominate. Using these formed intermediates, condensation can produce at least four products.



L-Proline
L-Proline- H
ArCHO
- H2O
Knoevenagal Condensation
Michael addition
Intramolecular Ring closure

CHAPTER 6

CONCLUSION AND REFERENCES

6.1 CONCLUSION

Due to the rich biodiversity of plants, the green world has potential for the synthesis of noble metal nanoparticles Green synthesis technology is a clean, non-toxic and environmentally friendly technology for synthesizing metal nanoparticles and is of great interest due to its commercial perspective and feasibility. However, to make these methods cost-effective and comparable to traditional methods for large-scale production of nanoparticles, improving reliable and environmentally friendly methods for synthesizing metal nanoparticles. In addition, most of these strategies are still in development and there are challenges to overcome. Metal nanoparticles produced by plants and / or plant extracts are more stable than those produced by other organisms and are required for the biosynthesis and stabilization of nanoparticles in large quantities of proteins, enzymes, and biomolecules. It has an amazing ability to be optimized for the formation of molecules. Production of metal nanoparticles by applying "green synthesis". Strontium oxide nanoparticle Synthesized using the sol-gel method. Characterization Analysis of SrO nanoparticles was performed by X-ray Diffraction, SEM and Fourier transform infrared (FTIR). This shows the X-ray diffraction pattern Nanoparticles are crystalline in nature. The crystalline size of strontium oxide nanoparticle was calculated by Debye-Scherer formula and the crystallite size was found to be 40 nm.The surface morphology of nanoparticles was observed and investigated using SEM. The material at room temperature, shows crystallite of cubical shape with strong agglomeration of particles. The presence of Strontium – Oxygen bond (Sr – O) bond in the synthesized sample was confirmed by the peak at 821.23cm^{-1} in the FTIR spectrum of strontium oxide nanoparticles.

The catalytic activity of the synthesized Strontium oxide nanoparticles was tested by the one-pot synthesis ofethyl-6-amino-5-cyano-4-(4-hyroxy-3methoxyphenyl) -2-methyl-4H-pyran-3-carboxylateThe product formation was confirmed by the FTIR and Mass Spectrometry.

6.2 REFRENCES

1. Jacques C. Védrine / Importance, features and uses of metal oxide catalysts in heterogeneous catalysis/Chinese Journal of Catalysis 40 (2019) 1627–1636

2. Roy.A, Bulut.O, Some.S, . Mandal.A.K, Yilmaz.M .K Green synthesis of silver nanoparticles: biomolecule-nanoparticle organizations targeting antimicrobial activity,RSC Adv, 9 (2019), p. 2673

3.Lalitha Gnanasekaran, R. Hemamalini, R. Saravanan, K. Ravichandran, F., Synthesis and characterization of metal oxides (CeO_2, CuO, NiO, Mn_3O_4, SnO_2 and ZnO) nanoparticles as photo catalysts (2017), doi: 10.1016/j.jphotobiol.2017.05.02

4. Aravind Kumar.J, Krithiga.T, Manigandan.S, Sathish.S,Corrigendum to “A focus to green synthesis of metal/metal based oxide nanoparticles: Various mechanisms and applications towards ecological approach” [J. Clean. Prod. 324 (2021) 129198]

5. Ajay K.D, Sethuraman. R, Katikaneni. S.P.R , and Idem. R.O, Synthesis and Characterization of Sulfated Titania Solid Acid Catalysts, Industrial & Engineering Chemistry Research, 1998, Vol 37, pp. 3869-3878.

6. Brauch. S, Sander S. V. B, Bernhard. W, Higher-Order Multicomponent Reactions: Beyond Four Reactants, Chemical Society reviews,Vol-42, 2013/02/21, Doi-10.1039/c3cs355055

7. Greenwood .N.N,Earnshaw.A in *Chemistry of the Elements*, 2nd edition, Butterworth, UK, 1997

8. Singh.D.K et al. / cultivated crops of Industrial Crops and Products 65 (2015) 269–286

9. Ahmad, I., Beg, A.Z., . Antimicrobial and phytochemical studies on 45 Indian medicinal plants against multi-drug resistant human pathogens. - 2002 J. Ethnopharmacol. 74, 113–123

10. Rubiay.A.I, Jaber.K.K , Al-Mhaawe, B.H., Alrubaiy, L.K.,. Antimicrobial Efficacy of Henna Extracts, Oman Med. J. .2008 23, 253–256.

11.Prathna TC, ashok MC, mukherjee RA. Biomimetic synthesis of Nanoparticles. Sci technolappl 2012; 20:76-80.

12. Murugesan s, bhuvaneswari s, sivamurugan v. Green synthesis, Characterization of silver nanoparticles of a marine red alga Spyridiafusiforms and their antibacterial activity. Int J pharm pharm Sci 2017; 9:192-7.

13. Willard ma, kuriharalk, carpenter ee, calvin s, harrisvg.Chemically prepared magnetic nanoparticles. Int mater rev 2014; 49: 125-170

14. Banerjee s. Green synthesis and characterization of metal Nanoparticles and its antimicrobial properties. Ph.D. Thesis. Jadavpur: Jadavpur University; 2012. P. 9-1

15. Sharma NC, sahi SV, nath S, parsons JG, gardea-torresdey JL, pal T. Synthesis of plant-mediated gold nanoparticles and catalytic role of biomatrix-embedded nanomaterials. Environ sci technol 2007; 41: 5137-42.

16. Bar H, bhui DK, sahoo GP, sarkar P, de SP, mirsa A. Green synthesis of silver nanoparticles using latex of jatropha curcas. Colloids surf A: physicochemeng asp 2009a; 339:134-9.

17. Bar H, bhui DK, sahoo GP, sarkar P, pyne S, mirsa A. Green synthesis of silver nanoparticles using seed extract of jatropha curcas. Colloids surf A: physicochemeng asp 2009b; 348:212-6

18. Singhal G, bhavesh R, kasariya K, sharma AR, singh RP. Biosynthesis Of silver nanoparticles using octimum sanctum (tulsi) leaf extract and Screening its antimicrobial activity. J nanopart res 2011; 13:2981-81

19. Areejshahbaz, nazim hussain, muhammadzafarsaleem, muhammadusamasaeed, muhammadbilal, hafiz M.N. Iqbal. Nanoparticles as stimulants for efficient generation of biofuels and renewables. Fuel 2022, 319, 123724. https://doi.Org/10.1016/j.Fuel.2022.123724

20. Iltaf khan, mingshengluo, sohail khan, humairaasghar, muhammadsaeed, shoaib khan, aftab khan, muhammadhumayun, linguo, buchangshi. Green synthesis of sro bridged lafeo3/g-c3n4 nanocomposites for CO2 conversion and bisphenol A degradation with new insights into mechanism. Environmental research 2022, 207, 112650. Https://doi.Org/10.1016/j.Envres.2021.112650