OPTIMISATION OF PROCESS PARAMETERS USING THE TAGUCHI METHOD FOR BIO-SYNTHESIS OF ZINC OXIDE NANOPARTICLES

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CANDIDATE'S DECLARATION

We, Dakshina Singh (2K22/MSCCHE/59) and Riya Yadav(2K22/MSCCHE/33) hereby certify that the work which is being presented in the thesis entitled **"Optimization of Process parameters using the Taguchi Method for Biosynthesis of Zinc Oxide Nanoparticles"** in partial fulfillment of the requirements for the award of the Degree of Masters in Chemistry, submitted in the Department of Applied Chemistry, Delhi Technological University is an authentic record of our own work carried out during the period from September 2023 to May 2024 under the supervision of Dr. Poonam Singh .

The matter presented in the thesis has not been submitted by me for the award of any other degree of this or any other Institute.

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ABSTRACT

Nanotechnology has ushered a new age in research due to its extensive role in various applications. Metal oxide nanoparticles (MONPs) have garnered attention in academic and industrial circles due to their high surface area-to-volume ratio and unique physico-chemical properties, resulting in their potential applications in fields of bio-medics, sensors, catalysis, wastewater remediation, etc. Top-down and the bottom-up approaches are the principal methods used to fabricate nanoparticles, which generally include physical, chemical and biological methodologies. However, certain disadvantages are associated with these conventional methods, including high energy consumption, waste production, low purity, and use of hazardous precursors and organic solvents. Hence, there is an increasing demand for environmentally friendly methods for MONPs fabrication. Plant-based resources are commonly utilized for environmentally friendly and cost-effective green synthesis of nanoparticles. Their non-toxic nature and economic viability make them attractive options. In this study, a straightforward and eco-friendly approach was employed to fabricate metal oxide nanoparticles (MONPs). The extract of *Coriandrum sativum* seeds has been utilized for the green synthesis of zinc oxide nanoparticles. Several runs were designed using the Taguchi method to study the effect of each parameter i.e, time, concentration of zinc acetate, volume of plant extract and calcination temperature on synthesis of ZnO nanoparticles across the different conditions. The percentage contribution of the process parameters was studied in response to wavelength and yield. The prepared material was characterized by using UV-Vis spectroscopy, powder X-ray diffraction technique and FT-IR analysis.

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LIST OF ABBREVIATIONS

w.r.t	with respect to
ZnO	Zinc oxide
ZnAc	Zinc Acetate
temp.	Temperature
Conc.	Concentration
DTU	Delhi Technology University
eV	Electron volt
FTIR	Fourier Transform Infrared Spectroscopy
g	Grams
hrs	Hours
IR	Infrared
eV	Electron volt
M	Molar
XRD	X-Ray Diffraction
pH	Potential of Hydrogen
NPs	Nanoparticles
nm	Nanometer
MONPs	Metal oxide nanoparticle

CHAPTER 1

INTRODUCTION AND LITERATURE REVIEW

1. Introduction

Metal oxides (MOs) have gained attention in academic and industrial circles due to their enhanced reactivity, surface area and versatile applications ranging from catalysis to sensing for biomedical uses, making them a valuable material in research fields as well. The inclination of researchers towards the metal oxide-based nanoparticles is also due to the larger surface-to-volume ratio along with small size, which tends to differ in the catalytic, biocompatibility along with a change in physicochemical properties [1].

Nanotechnology is the fastest-growing field in applied sciences today, having great potential to bring a new revolution. It mainly helps deal with and develops new nanomaterials while studying their various day-to-day applications. It not only helps in solving environmental issues but also considerably detects technological issues in the field of catalysis, solar energy conservation, etc. Nanotechnology enables the enhancement of surface-to-volume ratio along with alteration in catalytic, magnetic, and optical activities of MOs. The metal oxide nanoparticles (MONPs) exhibit unique properties emerging from their bulk counterparts, representing the building blocks of reformative potential across multiple scientific disciplines. They surpass the limitations of conventional MOs, serving as a playground for research and development. These MONPs exhibit various applications, from drug delivery to power science, the space industry, and biomedical science. Now, for the preparation of MONPs, there are conventional ways, which include top-down and bottom-up approaches. Each of these approaches has a different strategy for creating nanoparticles, and they all have their own pros and disadvantages. Implying various chemical and physical techniques such as lithography, milling, and laser ablation, bigger or bulk materials are converted into tiny pieces until the NP size is achieved. In contrast to the top-down method, the bottom-up method entails assembling smaller nanoparticles, molecules, or atoms to generate bigger ones. This technique creates nanoparticles from molecular or atomic precursors via chemical synthesis or self-assembly mechanisms [2]. Several areas of physics, biotechnology, information technology, chemistry, material science, and environmental technology have shown considerable interest in nano-structured transition metal oxides and nano-scale semiconductors as future technologies.

1.1 Disadvantages of Traditional Methods

There are various methods for the preparation of MONPs using conventional methods such as vapour condensation, hydrolysis, sol-gel method, etc. [4] having certain drawbacks associated with them such as use of toxic chemicals, energy intensive which contributes to higher production costs, low output and environmental safety concerns [3]. Also, due to the impurities generated from the by-products affects the purity of the nanoparticles making them unsafe for further use in medical field.

In traditional chemical synthesis, the Transition metal oxides are usually prepared by using toxic reducing agents (such as sodium borohydride, hydrazine, etc.) which further results in poor size control of the NPs being formed. To avoid the accumulation of MONPs, capping agents are added, such as ligands, polymers, etc., to maintain steric stabilization. High operational cost along with toxic precursors resulting in low yield of the desired product not only takes longer reaction time but also limits the large-scale applications [1]. Nevertheless, MONPs have been synthesized using plant-mediated methods, which not only have significant stability, greater reducing capacity but also aim at following green chemistry principles. Moreover, the MONPs prepared using the green synthesis also show great properties by achieving variable sizes and shapes [4].

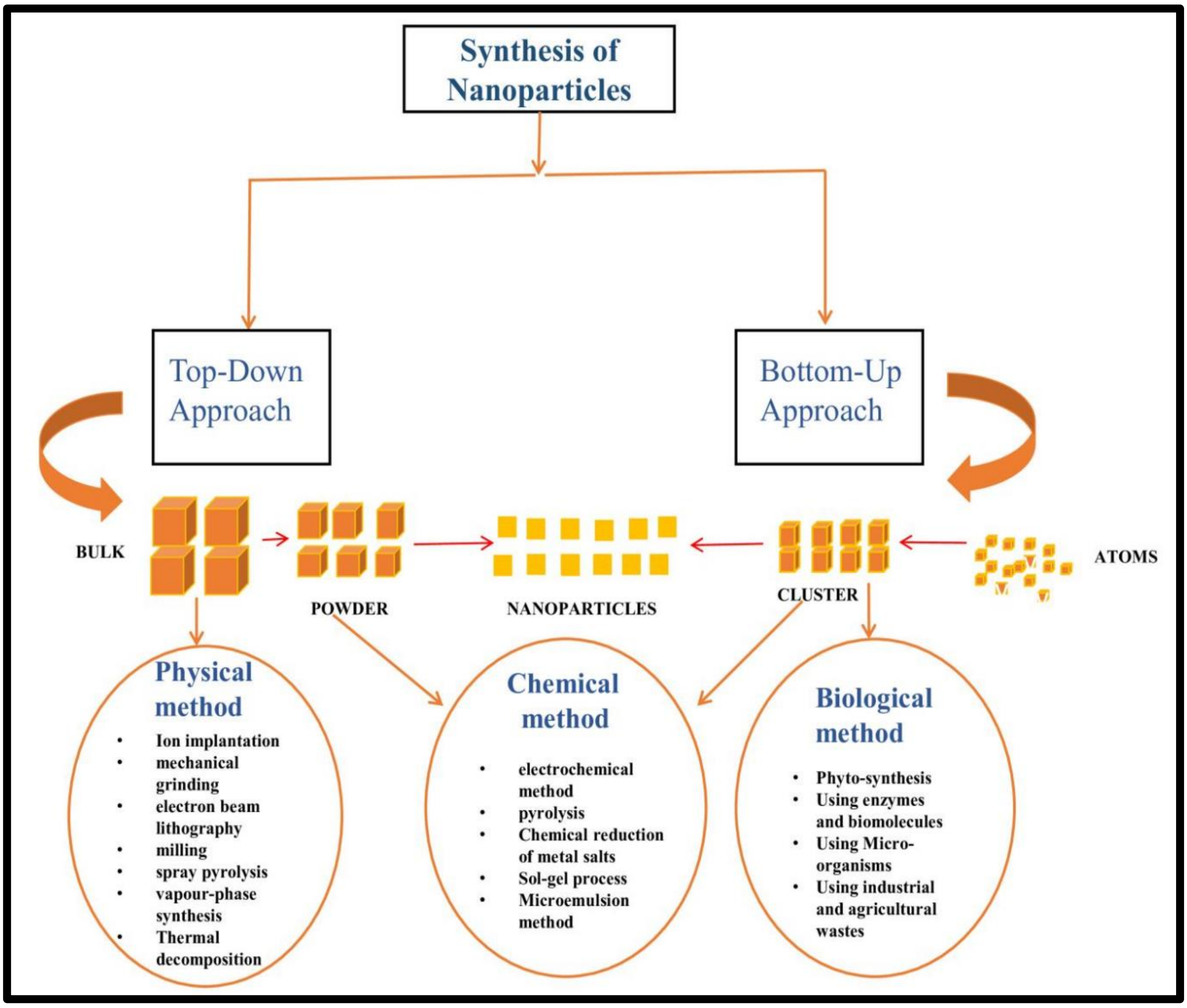

Fig.1.1 Different approaches to synthesis of Metal oxide nanoparticles (MONPs).

1.2 Green synthesis of nanoparticles

Embracing a greener approach for the preparation of nanoparticles is the need of the hour due to the adverse effects of traditional methods. A more sustainable alternative green synthesis of NPs or MONPs offers more energy efficient methods and improved bio-compatibility of NPs, reducing toxicity and making them economically viable [5]. Greener approach minimizes toxic chemicals and reduces energy consumption and waste generation, aligning itself with the twelve principles of green chemistry and circular economy by utilizing the natural resources. Opting for green synthesis employs plant as well as microorganisms extract mediated synthesis. Biosynthesis of nanoparticles is carried out using plant extracts, enzymes, bacteria, and fungi without producing harmful by-products. Plant extracts are easily accessible, safer to use and functions as a reducing and capping agent to decrease metal-based precursors [6]. These extracts are made from many plant components, including seeds, leaves, and flowers, and their capacity to create nanoparticles (NPs) is being studied. Flavonoids, saponins, terpenoids, sugars, alkaloids, and proteins are among the phytochemicals that are thought to be responsible for the plant extract's capacity to synthesize MONPs [5]. Green synthesis of MONPs tailors the physical, chemical, biological properties but also the functionalities by which they hold several applications in the field of biomedical as well as in sectors of energy storage, sensing, packaging materials, etc., contributing towards sustainable designing and development. Numerous applications in the field of biotechnology, catalysis, sensors, coatings, drug delivery, optical devices, and agriculture arises from the use of plant extract from bark, leaves, or roots [7].

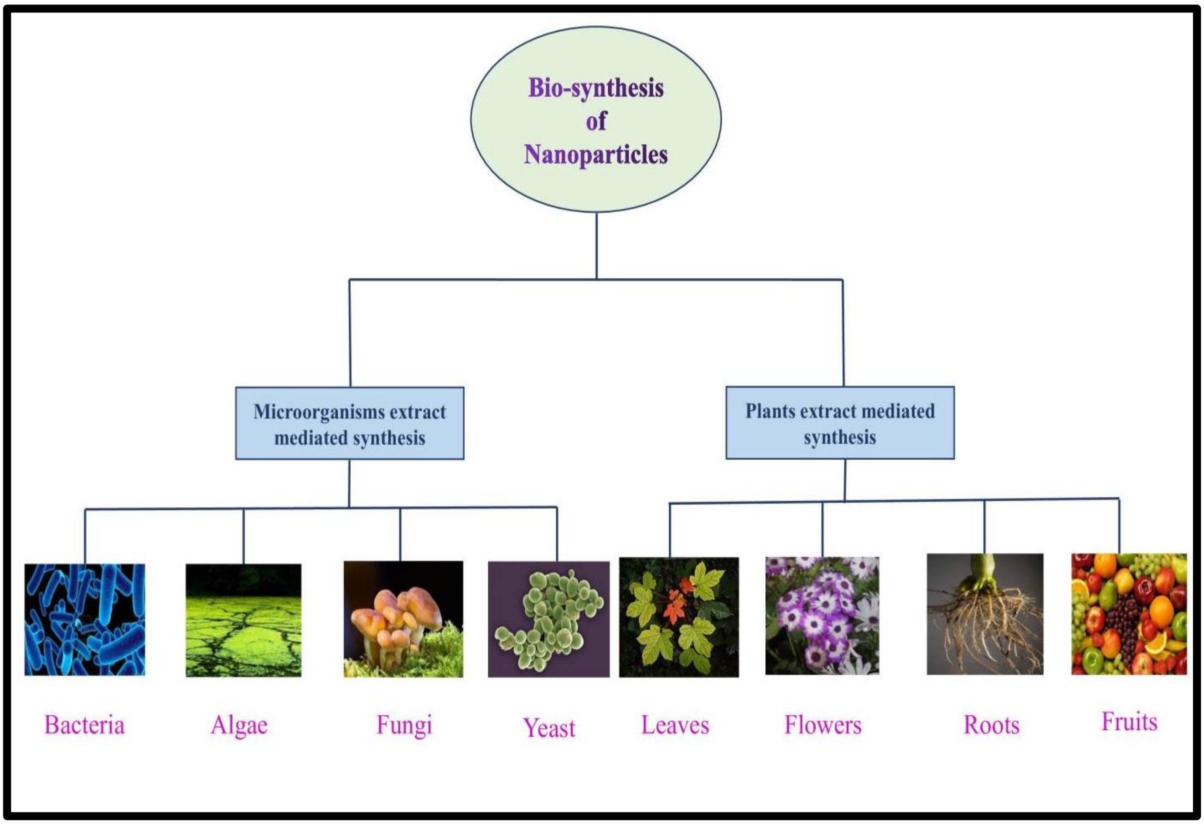

Fig.1.2 Various precursors used for the green synthesis of MONPs

Coriander *(Coriandrum sativum L.)* is a plant of the *Umbelliferae* family that is a well-regarded ayurvedic medicinal tree identified as ‘dhania’ [8]. Coriander has renowned properties in traditional medicines; it was used as a digestive aid, anti-oxidant, and anti-inflammatory, majorly in the kitchen as a spice in various cuisines, seasoning and garnishes [9]. It is also used for aromatherapy possessing relaxing and calming properties, natural insect repellent, and in skincare products possessing skin benefits. Some of the common applications includes- as a diuretic, an antidepressant and a memory enhancing medicine [10]. Also, due to its polyphenolic compounds and further anti-diabetic and anti-mutagenic effects it has been shown to exhibit great anti-oxidative capacity [9]. Since ancient times, coriander leaves and seeds have been valued much for their pleasant tastes as well as for their ability to limit food deterioration and have positive health effects, which are both attributed to their potent antioxidant qualities. Coriander's active bio compounds function as both stabilizing and reducing agents at the same time [6].

Numerous components are procured from varied parts of the Coriandrum sativum plant such as – phytosterols which are derived from the stem of the coriander plant, flavonoids which showcase potential health benefits with bio-active compounds obtained from coriander leaves along with linalool. Essential oils contain terpenes and other aromatic compounds which are gained from coriander seeds along with coriandrin exhibiting antioxidant property [11]. Naturally occurring nitrogenous component – alkaloids as well as polyphenols are secured from the whole plant extract. In the present work an attempt has been made to utilize coriander seeds for the preparation of MONPs. It is an idealized plant for the biosynthesis of nanoparticles as it is cheap and easily available in the market so can be easily acquired.

Coriander seeds extract have various applications as a novel reducing agent which has gained focus not only in pharmaceutical aspect but also has provided with environment friendly solutions by acting as an antioxidant and antimicrobial agent which is incorporated in food packaging materials to prolong their shelf value by preventing oxidation as well as microbial growth[6]. It is also used as a reducing agent in dyeing processes for textile industry. It also reduces metal ions concentration to less toxic form which then contributes to remediation of contaminated water i.e. for the treatment of wastewater. Depending upon the metal there are different methods for the preparation of metal oxide nanoparticles. Each metal oxide requires a specific property to work with such as reagents (zinc sulphate heptahydrate, zinc nitrate hexahydrate, zinc acetate dehydrate, etc.), precipitating agents (NaOH, NH_4OH, etc.) being used, reaction conditions under which they are being processed as well as the stabilizing agents. As recorded by the previous studies it has been observed already a huge amount of work has been done using metals like titanium (TiO_2), calcium (CaO), Iron (Fe_2O_3), Mn (MnO_2, MnO), Mg (MgO), K (K_2O) , Na (Na_2O_2), etc. including various multifunctional metal oxides were prepared such as $ZnCr_2O_4$, $ZnCrO_4$, etc.

In current work, Zn was used as a metal for the preparation of MO NPs which possess different properties such as magnetic properties depending upon the method of preparation as well as doping which is used in magnetic sensors, electrical conductivity as they usually exhibit semiconductor properties, antimicrobial and anti-fungal property, ZnO NPs also helps in degradation of organic pollutants when exposed to ultraviolet light thus exhibiting photolytic activity. Also depending upon the shape, size, and morphology ZnO NPs display distinct characteristics such as optical as well as electrical properties which helps in further application in the industries. Due to their great versatility they offer distinct applications – such as in cosmetic industries for skincare products like sunscreen exhibiting UV- blocking properties [12] similarly in paints and coatings for its anti-corrosive nature and antimicrobial properties. As ZnO NPs demonstrate photocatalytic activity it is also used for water purification. Featuring several advantages, their toxicity and environmental impact are the areas of on-going research especially in the bio-medical sector. Zinc oxide nanoparticles have several applications in the treatment, targeting and diagnosis of lung, breast, skin and many other cancers or tumors.

Numerous plant-mediated methods exist for synthesizing nanoparticles, and the literature includes reports on using plant extracts—such as aloe vera—for green synthesis of ZnO nanoparticles, leveraging their bioactive components, *Azadirachta indica* commonly named as neem exhibiting anti-microbial properties[13], *Moringa oleifera*[14], turmeric botanical name *Curcuma longa*[15], banana (*Musa spp.*)[16], green tea *(Camellia sinensis)* [17] which is rich in polyphenols – unveiling reducing and capping abilities, etc. However in this current study we have utilized coriander for the preparation of zinc oxide nanoparticles.

1.3 Influence of Various Parameters on Synthesis of NPs:

It is found that Zinc oxide NPs have great dependency on these variables - temperature, pH, conc. of plant extract, conc. of Zinc acetate solution and also on calcination temperature; influencing the synthesis of NPs [7].

- **Effect of pH:** Positively and negatively charged ions present in the reaction medium affects the morphology of the nanoparticles. In presence of an acidic medium when pH<7 the concentration of hydroxyl ions is low which results in alteration and hindrance in hydrolysis and condensation processes resulting in smaller aggregates formation due to corrosion of ZnO crystals whereas at pH=7 which is neutral the hydroxyl ion conc. as well as hydrogen ion conc. is equal due to which there is no interaction at the interface of crystals. When the reaction mixture is basic in nature i.e. having pH>7, the concentration of hydroxyl ions is greater resulting in strong attraction between the positively charged as well as negatively charged ions of the crystals ultimately resulting in formation of smaller ZnO NPs [18]. As the size reduces which the increase in pH it also results in the increase of band gap value [19]. Synthesis of ZnO NPs with controlled pH values has vast applications in optoelectronic devices along with improved properties. It is also known that with increase in pH level of the medium there is increase in metal-oxygen binding capacity which results in stabilizing the nanoparticles due to lesser tendency of agglomeration [1].

- **Effect of Temperature:** Temperature not only determines the crystallinity of the NPs but also determines the yield obtained. Chemicals processes for the synthesis of nanoparticles can be carried out at room temperature opposite to that, the physical method employees the use of high temperature conditions [18]. It is generally observed that with increase in temperature the size of the nanoparticles decreases and is comparatively smaller in case of green synthesis as compared to those produced by chemical method [20]. This inference is drawn from the G. Indramahalakshmi who employed green synthesis of ZnO NPs and regardless of temperature conditions; smaller nanoparticles develop even when conc. of precursor is low. Also with increase in temperature the total reaction rate increases i.e. high temperature is conductive to nucleation resulting in increase in rate of formation of NPs [21].

- **Concentration of Precursor:** Zinc acetate (Zn $(CH_3COO)_2$, Zinc nitrate(Zn$(NO_3)_2$, Zinc sulphate ($ZnSO_4$) and Zinc chloride ($ZnCl_2$) are different zinc salt precursors used for the synthesis of ZnO NPs. They have been widely investigated which implies that various shapes of ZnO nanoparticles can be produced with little to no effect on crystal size but in turn influences the morphology. Also majority of the size of ZnO NPs depends on the process being implied for the production [18].

- **Calcination Temperature:** Calcination is the process of heating a solid chemical compounds in the presence of fraction of air by raising it to high temperatures to cause thermal decomposition, removal of volatile fraction as well as phase transition. This temperature is crucial as it affects the compound's physical and chemical properties. According to the studies, ZnO nanoparticles synthesized via co-precipitation method using zinc acetate dihydrate and sodium hydroxide (NaOH) as precursor material maintaining a specific pH by further increasing the temperature. The impure ZnO NPs further formed were calcinated at different temperatures ranging from 200 °C, 400 °C to 500 °C, ultimately observing an increase particle size from 30, 41, and 44 nm, respectively [22] which tends to affect their purity as well as morphology.

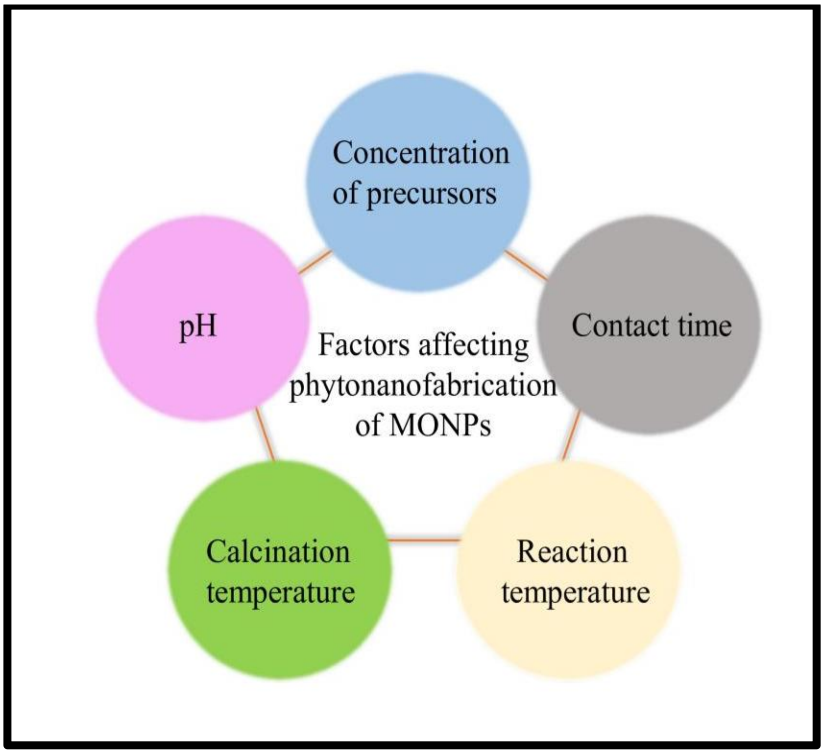

Fig 1.3 Factors affecting synthesis of ZnO NPs

1.4 Optimization Process:

It is necessary to design experiments that are efficient. An experiment can be highly influenced simply by the quantitative as well as qualitative factors, so it is necessary to obtain the maximum information from the smallest possible number of trials [23]. The aim is to identify those factors which are majorly impacting the experimental designs. The process of optimizing an experiment is much more material efficient as well as time efficient. It focuses on determining optimum conditions or factors such as temperature, pH, etc. to achieve the best output possible for the given chemical process. One of the most widely used optimization technique is DOE i.e. design of experiment.

Design of Experiment is a statistical method whose basic aim is to develop a model that mathematically derives an output based on the experimental inputs based on the reaction factors such as temperature, reaction time, etc. [24]. In order to optimize an experiment it is necessary to seek maximum response by identifying the factor on which basis the experiment is to be optimized such as reaction rate, percentage yield, absorption, size, etc. One factor or single factor optimization does not need an advance methods it can simply be done using factors such as spectral bandwidth and wavelength whereas optimization done keeping two or more factors into consideration is much more complex [23].

1.5 Taguchi Method

It is again a type of statistical method which is generally referred to as a robust method designed and developed by Dr. Genichi Taguchi, Japanese engineer as well as a statistician [23]. It is a basically used to process and investigate multiple factors which directly affects the mean and variance of a process. Taguchi involves designing an experiment which includes orthogonal arrays to organize factors which directly affects the process. Experimental runs are determined using the formula:

$$N = (L - 1)P + 1. \quad [1]$$

Where,N=smallest probable number of experimental runs

P= number of process parameters

L= no.of levels

This method tends to test combination of pairs rather than testing all the possibilities which directly results in decreasing the number of experimental runs, ensuring use of less resources as well as reduced time. Comparing it with traditional DoE, Taguchi method treats noise as a major focus for analysis whereas for the former it is treated as nuisance variable. The data obtained from these arrays are plotted and further analyzed by performing ANOVA, visual analysis, or by calculating the S/N ratio, etc. Quality of the product is increased using taguchi method of analysis. It not only operates optimally over a variety of factors or conditions but also determines the best design by strategically designing the experiments [25].

ANOVA Method: Also called Analysis of Variance which is used to calculate differences between two or more means and deduce the impact of percentage contribution of each parameter being analyzed [26].Understanding the distinction between categorical independent variables and quantitative dependent variables is crucial. The alternative hypothesis asserts that at least one group significantly deviates from the overall mean of the dependent variable, while the null hypothesis posits no difference between group means . One-way ANOVA deals with a single independent variable and multiple groups, whereas two-way ANOVA involves two independent variables, particularly useful for studying interactions between factors. It employees the use of F-test which calculates and compares variance within groups to variance between the groups [28]. If the variance within the groups is smaller than the variance between the groups there is significant detection of difference which results in rejection of the null hypothesis.

Signal to Noise Ratio (S/N Ratio): For the measurement of variance from design experiment Taguchi method utilizes Signal to Noise ratio. It is the ratio of mean (signal) to the standard deviation (noise). In Taguchi model there are three characteristics values for conversion in Signal to Noise ratio (S/N Ratio) which are - “Nominal is the best”, “Smaller is better”, “Larger is better”. These three values describe the quality of the desired product [25].

For “Smaller is Better” the size of the nanoparticles have to be minimized which in results offer larger surface-to-volume ratio, enhancing reactivity which helps to optimize properties of the nanoparticles.

“Nominal is Better” S/N Ratio is specifically used to achieve targeted values such as shape, size, surface charge, concentration, etc. So the overall significance of S/N Ratio is ensuring successful performance of the nanoparticles. It is also employed in imaging and has a great impact on drug delivery as well.

"Larger is Better" S/N Ratio is used to maximize specific properties (e.g., conductivity, catalytic activity) or response. Larger nanoparticles may be desirable for efficient catalysis or enhanced electrical conductivity. The "Larger is Better" S/N ratio aligns with this goal. It comes into play when we seek to enhance a particular property of nanoparticles. This empowers us to fine-tune nanoparticle behaviour, ultimately influencing their functionality.

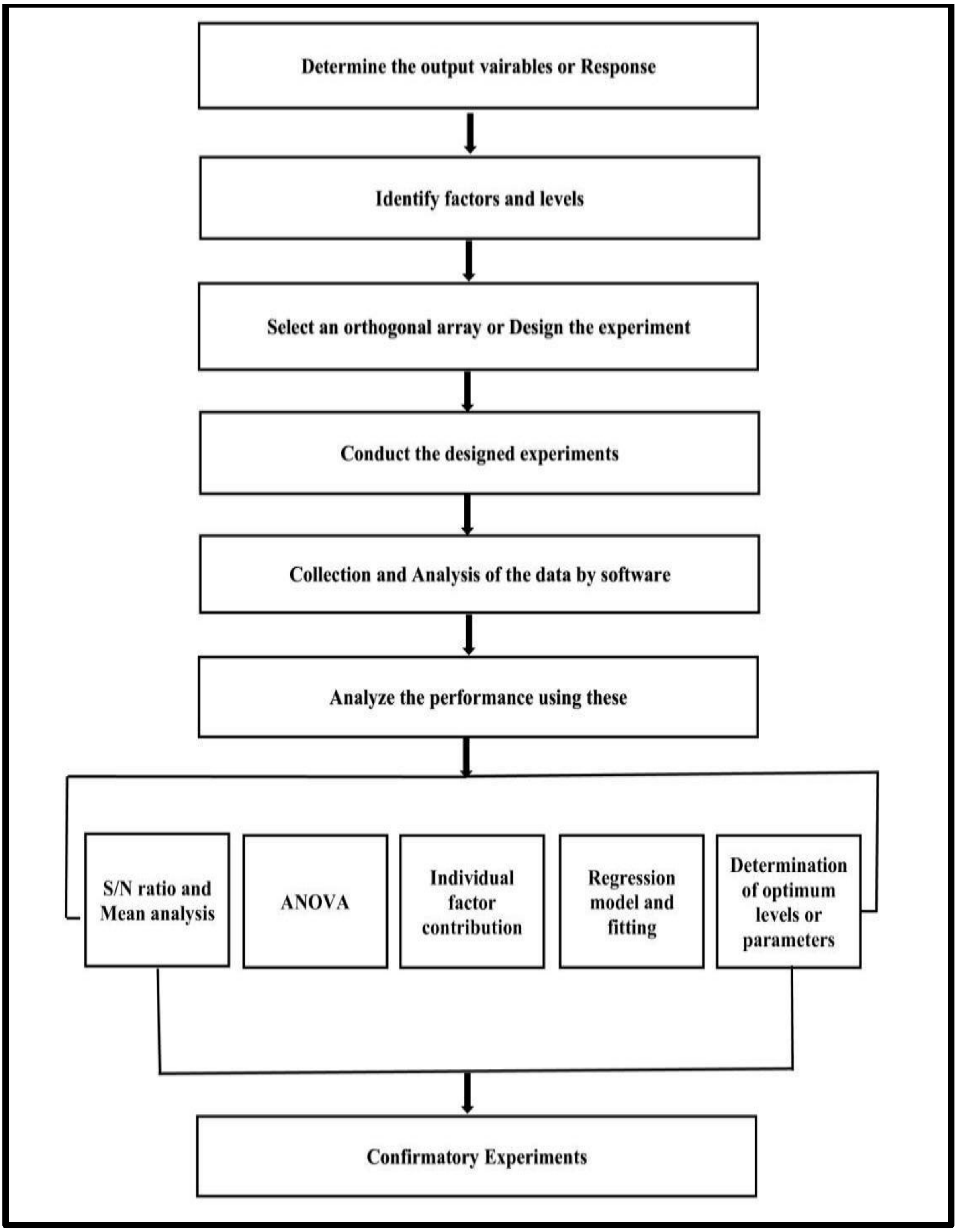

Fig.1.4 Steps involved in determining Taguchi model

CHAPTER- 2

EXPERIMENTAL SECTION

2.1 CHEMICALS AND REAGENTS

Zinc acetate dihydrate, sodium hydroxide, distilled water (DW) were used to prepare all the solutions, ethanol (CH_3CH_2OH).

2.2 *Coriandrum sativum l.* SEED EXTRACT PREPARATION

Coriander seeds were washed with DW to remove dust particles and impurities and were left to dry at 40℃. After drying the seeds were crushed using a mortar and pestle. The 20g of seeds were mixed with 100 ml of DW with constant stirring and heating at 60℃ for 30 minutes. Later was kept to cool down at room temperature and was left overnight. The extract was filtered and collected and later stored in the refrigerator at 4℃ for further use.

2.3 BIOSYNTHESIS OF ZnO NPs

Green synthesis of ZnO NPs was done by taking 50ml, 0.5M zinc acetate dihydrate solution, to which 50 ml of seed extract was added. Further the process was carried out in the presence of sodium hydroxide maintaining a pH of 8 to 11. The mixtures were stirred for 2 hours at room temperature. The obtained product was filtered and washed in minimum amount of ethanol and distilled water. The sample obtained was calcinated at different temperature. The process of preparation of ZnO NPs was followed by modifications in the literature as mentioned in the table below by altering the time, concentration of zinc acetate, volume of extract (mL), as well as the calcination temperature.

2.4 STATISTICAL ANALYSIS

The synthesis of nanoparticles was methodically investigated using the Taguchi model to discern the influence of various parameters and to ascertain the optimal conditions for synthesis. The study meticulously examined four independent variables: the stirring duration (time), the concentration of zinc acetate, the calcination temperature, and the volume of the extract, as documented in Table 2.1. These factors were analyzed to determine their impact on nanoparticles production. The statistical significance of the influence exerted by each variable was quantified through Analysis of Variance (ANOVA), facilitated by the Minitab 17 software.

Table2.1 Taguchi L9 Orthogonal array (OA) design

Sample	Time	Concentration of ZA	Volume of PE	Calcination temperature
R1	1.0	0.5	10	300
R2	1.0	1.0	20	400
R3	1.0	1.5	30	500
R4	1.5	0.5	20	500
R5	1.5	1.0	30	300
R6	1.5	1.5	10	400
R7	2.0	0.5	30	400
R8	2.0	1.0	10	500
R9	2.0	1.5	20	300

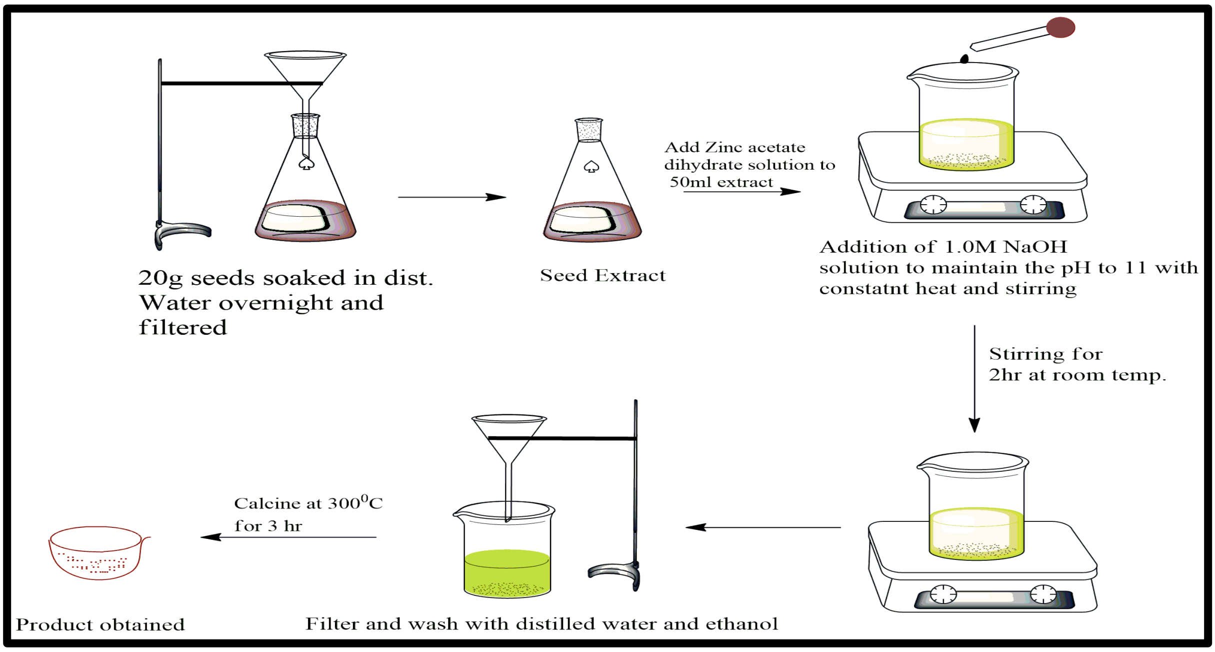

Fig. 2.1 Schematic representation of Synthesis of ZnO NPs using seed extract of *Coriandrum sativum*

2.5 CHARACTERIZATION TECHNIQUES

2.5.1 POWDER X-ray DIFFRACTION ANALYSIS [PXRD]

PXRD is a analytical technique used to determine the crystal structure of powdered materials. Figure 3. shows a typical X-ray diffractometer. Sealed tubes, synchrotron radiation sources, and rotating anodes are usually used to generate X-rays. PXRD has several advantages such as has fast operational procedure which is convenient to use along with it being low cost maintenance, effective resolution, etc. Samples employed for analysis could be inorganic complexes, fiber, pharmaceutical samples, metals, polymers, semiconductors. This technique is used to identify and monitor the quality of the material which is being manufactured [29]. The peak position analyses the chemical composition, phase structure and other parameters whereas the peak shape contributes crystallite size.

$$D = 0.94\ \lambda / \beta \cos \theta \qquad [2]$$

Here, D represents the average crystallinity, λ is the wavelength (Å), β is FWHM (in radian) whereas degree of diffraction is indicated by θ.

Fig. 2.2 Bruker D8 Advanced X-Ray Diffractometer.

2.5.2 FOURIER TRANSFORM INFRARED SPECTROSCOPY [FTIR]

The FTIR analysis is a type of analytical technique that uses infrared radiation to identify and quantify a sample's chemical composition. Figure 4. shows a typical FTIR Spectrophotometer**.** FTIR analysis measures the interaction between a beam of infrared light and a sample. In addition to providing information about the samples' chemical composition, the spectrum also identifies the functional groups present. As a result, no two compounds produce similar IR spectra. In this way, an IR spectrum can be used as an identification fingerprint. Different kinds of materials can be qualitatively analyzed using IR spectroscopy. IR region is usually categorized into three regions i.e., far - IR (14000-4000 cm^{-1}), mid - IR (4000 - 400 cm^{-1}) & near - IR (400 - 10 cm^{-1}).

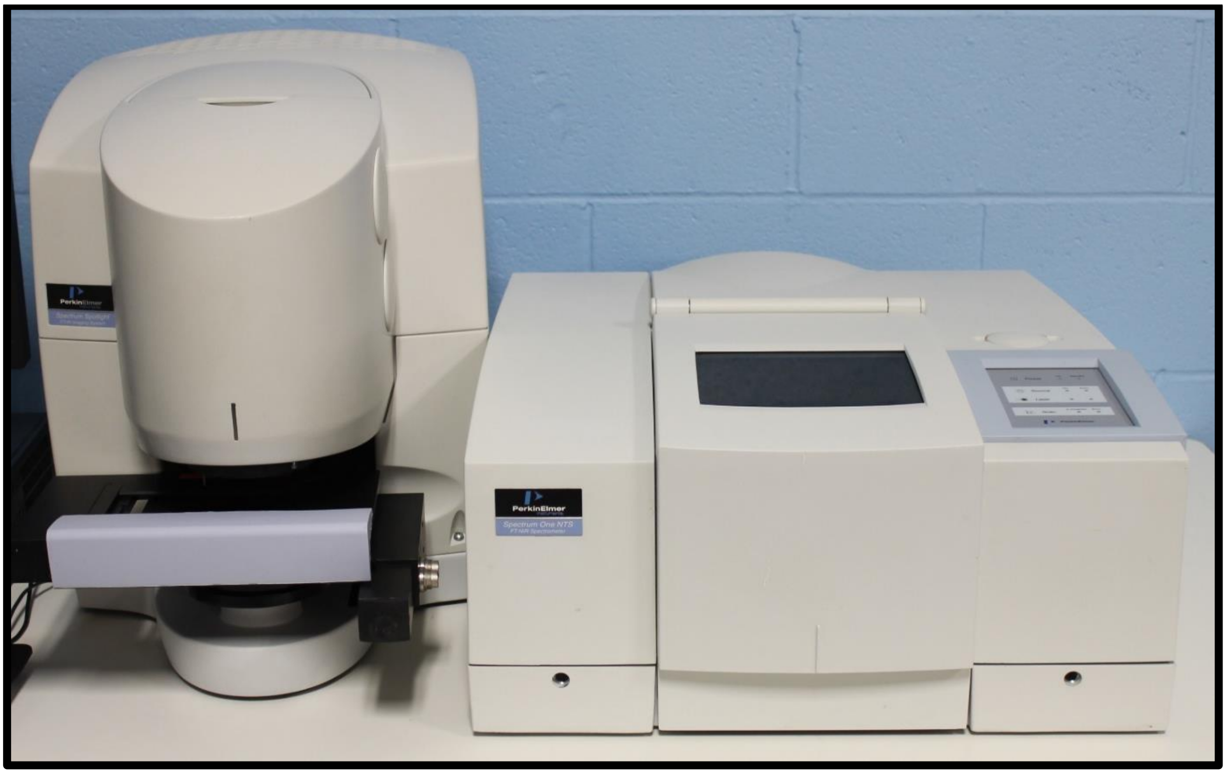

Figure 2.3 Perkin-Elmer-200i FTIR Spectrophotometer

2.5.3 UV-Visible SPECTROPHOTOMETER:

The UV-visible spectroscopy is frequently employed for characterizing various metal nanoparticles within the size range of 2–100 nm[1].The formation of nanoparticles was substantiated through the analysis of an aqueous solution using a UV-Visible spectrophotometer, which scanned the wavelength ranges from 200 to 800 nm. It is also applied to characterize the rate of chemical reaction. Several organic samples can be studied using UV spectroscopy which has high degree of conjugation. A UV-Vis spectrophotometer can also be used as a detector in HPLC. Beer- Lambert's law is used for quantitative analysis for determination of concentration of the absorbing species.

Beer-Lambert law: A=εCL, where "A" is the absorbance, "C" is the concentration of the absorbing species, "ε" is the molar absorptivity or extinction coefficient which is constant for a particular wavelength and species and "L" is the path length.

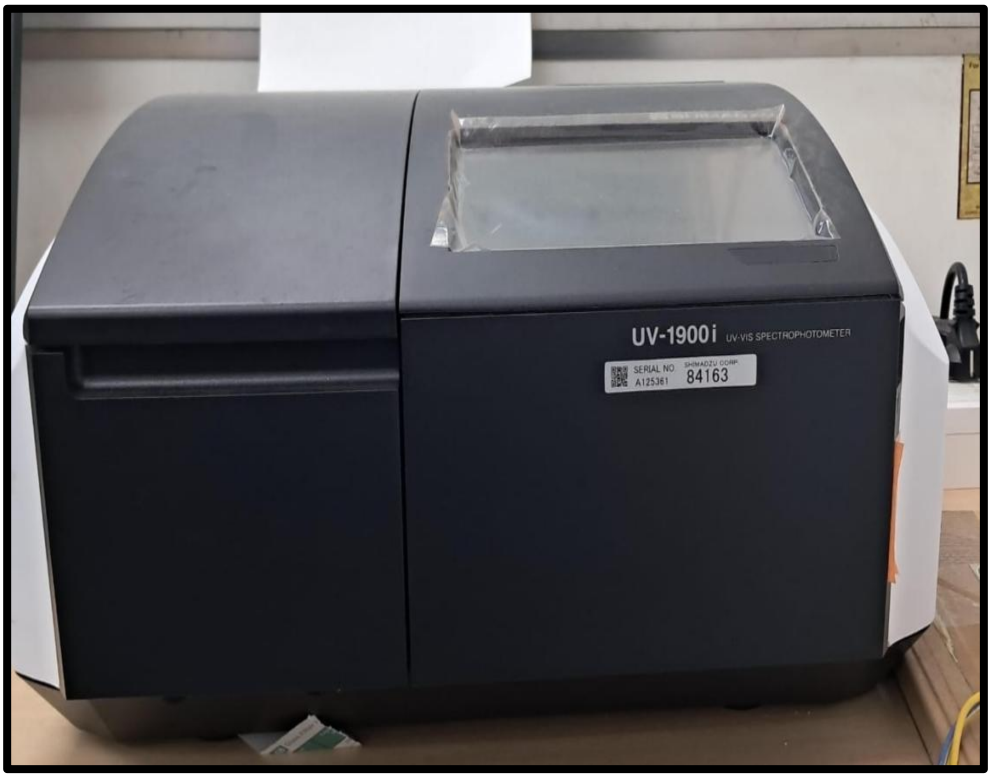

Fig.2.4: Shimazdu 1900 UV Spectrophotometer

CHAPTER-3

RESULTS AND DISCUSSION

3.1 EXPERIMENTAL DESIGN AND OPTIMIZATION

3.1.1 Modelling:

The optimization of the green synthesis of ZnO NPs using *Coriandrum sativum* extract was achieved by the Taguchi technique. This technique is used to study the independent influence of volume of extract, concentration of Zinc acetate, stirring time and calcination temperature on the preparation of ZnO NPs. In this method, the better model was preferred which is used to maximize the specific property which in case here is yield and absorbance. The optimum condition can be determined by Signal to noise ratio. S/N ratio of all the factors taken in consideration is shown in **Table 3.1** with respect to the wavelength and **Table 3.4** with respect to the yield. The S/N ratio selected for study was larger, since the aim of the study is to maximize the response of the NPs properties. Therefore, the larger the value of S/N ratio of factor, the better the effect is on the selected parameter. The results marks that the most influential parameter for ZnO NPs synthesis was calcination temperature followed by the concentration of Zinc acetate solution in response with wavelength whereas concentration of Zinc acetate dominated followed by calcination temperature as most influential parameter in response with the yield of ZnO NPs. The results of S/N ratio from **Table 3.1** and **Table 3.4** shows that the sample R3 is the best one out of the 9 sample runs w.r.t both parameters wavelength and yield. To study the percent contribution of the synthesis parameters ANOVA analysis was done. **Table 3.3** shows the ANOVA study in response with wavelength. From ANOVA study, it was confirmed that the calcination temperature has significant influence on the wavelength of the ZnO nanoparticles. ANOVA study in response with the yield is shown in **Table 3.6**, wherein the highest contribution factor is concentration of Zinc acetate. The highest contribution value of Zinc acetate reveals that it has most positive impact on the yield of

ZnO NPs; however, both the volume of extract and stirring time has the least impact on the ZnO nanoparticles synthesis.

Table 3.1: Four factor-Three layered (L9) Taguchi design for larger is better Signal to noise (S/N) ratio w.r.t Wavelength:

Sample	Time	Concentration of ZA	Volume of extract	Calcination temperature	Wavelength (nm)	S/N ratio
R1	1.0	0.5	10	300	363.0	51.1981
R2	1.0	1.0	20	400	372.0	51.4109
R3	1.0	1.5	30	500	374.5	51.4690
R4	1.5	0.5	20	500	371.0	51.3875
R5	1.5	1.0	30	300	364.5	51.2340
R6	1.5	1.5	10	400	372.0	51.4109
R7	2.0	0.5	30	400	368.0	51.3170
R8	2.0	1.0	10	500	373.0	51.4342
R9	2.0	1.5	20	300	365.0	51.2459

Table 3.2: Response Table for Signal to Noise Ratios larger is better w.r.t wavelength:

Level	Time	Zinc acetate	Volume of extract	Calcination temperature
1	51.36	51.30	51.35	51.23
2	51.34	51.36	51.35	51.38
3	51.33	51.38	51.34	51.43
Delta	0.03	0.07	0.01	0.20
Rank	3	2	4	1

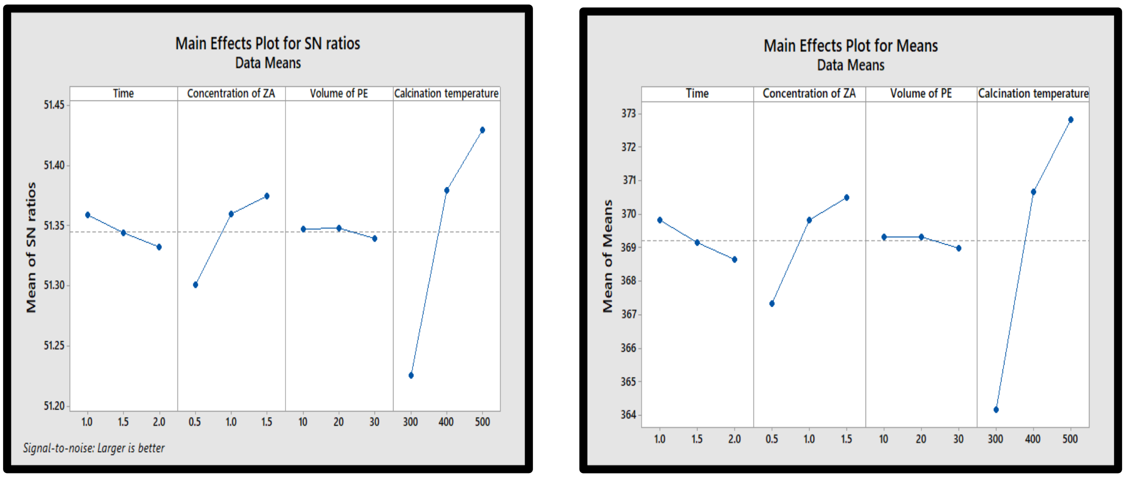

Fig 3.1: Plot for S/N ratio and Mean for ZnO w.r.t wavelength

Table 3.3: ANOVA analysis for Wavelength

Source	Degree of Freedom	Sum of Squares	Mean Squares	% contribution
Time	2	2.056	1.457	1.027
Conc. of ZnAc	2	16.722	8.3611	11.854
Vol. of extract	2	0.222	0.1111	0.157
Calcination temperature	2	122.056	61.0278	86.530
Total	8	141.056		100

Table 3.4: Four factor-Three layered (L9) Taguchi design for larger is better Signal to noise (S/N) ratio w.r.t yield:

Sample	Time	Concentration of ZA	Volume of extract	Calcination temperature	Yield(g)	S/N ratio
R1	1.0	0.5	10	300	0.2889	-10.7850
R2	1.0	1.0	20	400	1.0242	0.2077
R3	1.0	1.5	30	500	1.0631	0.5315
R4	1.5	0.5	20	500	0.9250	-0.6772
R5	1.5	1.0	30	300	0.5841	-4.6703
R6	1.5	1.5	10	400	0.7733	-2.2330
R7	2.0	0.5	30	400	0.3144	-10.0503
R8	2.0	1.0	10	500	0.9427	-0.5125
R9	2.0	1.5	20	300	0.9326	-0.6061

Table 3.5: Response Table for Signal to Noise Ratios larger is better w.r.t yield:

Level	Time	Zinc Acetate	Volume of extract	Calcination temperature
1	-3.3486	-7.1709	-4.5102	-5.3538
2	-2.5268	-1.6584	-0.3585	-4.0252
3	-3.7230	-0.7692	-4.7297	-0.2194

Delta	1.1962	6.4016	4.3712	5.1344
Rank	4	1	3	2

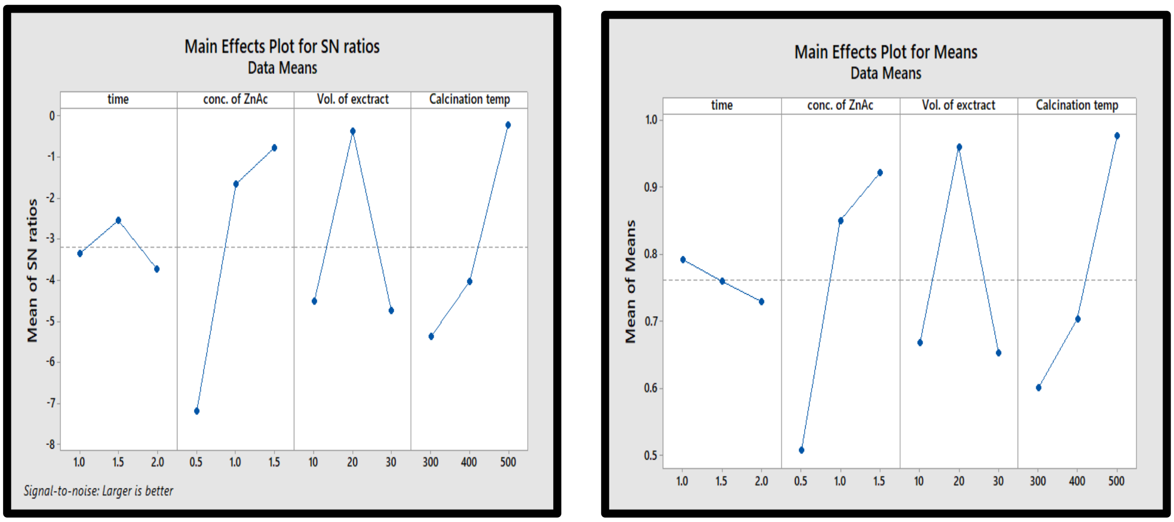

Fig 3.2: Plot for S/N ratio and Mean for ZnO w.r.t yield

Table 3.6: ANOVA analysis for Yield

Source	Degree of Freedom	Sum of Squares	Mean Squares	% contribution
Time	2	2.246	1.1232	1.46
Conc. of ZnAc	2	72.159	47.03	47.03
Vol. of extract	2	36.392	23.70	23.722
Calcination temperature	2	42.611	21.3057	27.77
Total	8	153.409		100

3.1.2 Optimization of synthesis of ZnO NPs:

The response graphs corresponding to the mentioned factors for parameters wavelength and yield are presented in Fig.3.1 and Fig.3.2 respectively. From Fig.3.1, the optimal conditions for the synthesis of Zinc oxide nanoparticles are the calcination temperature at level 3, conc. of Zinc acetate at the level 3, the volume of extract at level 1, and time at level 1.Whereas, for Fig.3.2, the optimal conditions for synthesis of ZnO nanoparticles are calcination temperature at level 3, conc. of Zinc acetate at the level 3, the volume of extract at level 2, and time at level 2.

The optimum condition for the synthesis of ZnO NP was found to be as follows: stirring time = 1hr, conc. of zinc acetate = 1.5M, volume of extract=10ml and Calcination temp. = 500℃ in order to obtain maximal wavelength. The best parameters for the production of nanoparticles of ZnO were obtained at; Calcination temperature=500℃; stirring time=1.5hr; conc. of zinc acetate=1.5M and volume of extract=20 ml as mentioned in table 3.7 in order to obtain maximum yield.

Table 3.7: Factor levels for predictions

Parameter	Time	Concentration of ZnAc	Volume of extract	Calcination temperature	S/N ratio
Wavelength	1	1.5	10	500	51.4768
Yield	1.5	1.5	20	500	5.72447

3.2 CHARACTERIZATION

3.2.1 UV-Vis spectroscopy:

The UV-Vis absorption intensity exhibits strong dependence on the concentration of ZnO nanoparticles (NPs). According to an analysis of Figure 3.3, ZnO NPs' increasing intensity with each experimental run indicates that their solubility and dispersion in the solutions have improved. Variations in wavelength positions further indicate differences in morphology and size of the prepared ZnO NPs. Notably, the maximum absorption spectra i.e. 374nm and 373 nm, correspond to characteristic electron transitions from n → π* and π → π*. Table 3.1 presents the actual maximal wavelength values from nine experimental runs. The maximal wavelength (374.5nm and 373nm) indicates the formation of ZnO nanoparticles. Fig.3.4 indicates the UV−visible absorption spectra of sample R3 with calcination temperature of 500°C. Its maximal wavelength is at 374 nm with band gap value of ~3.31 eV.

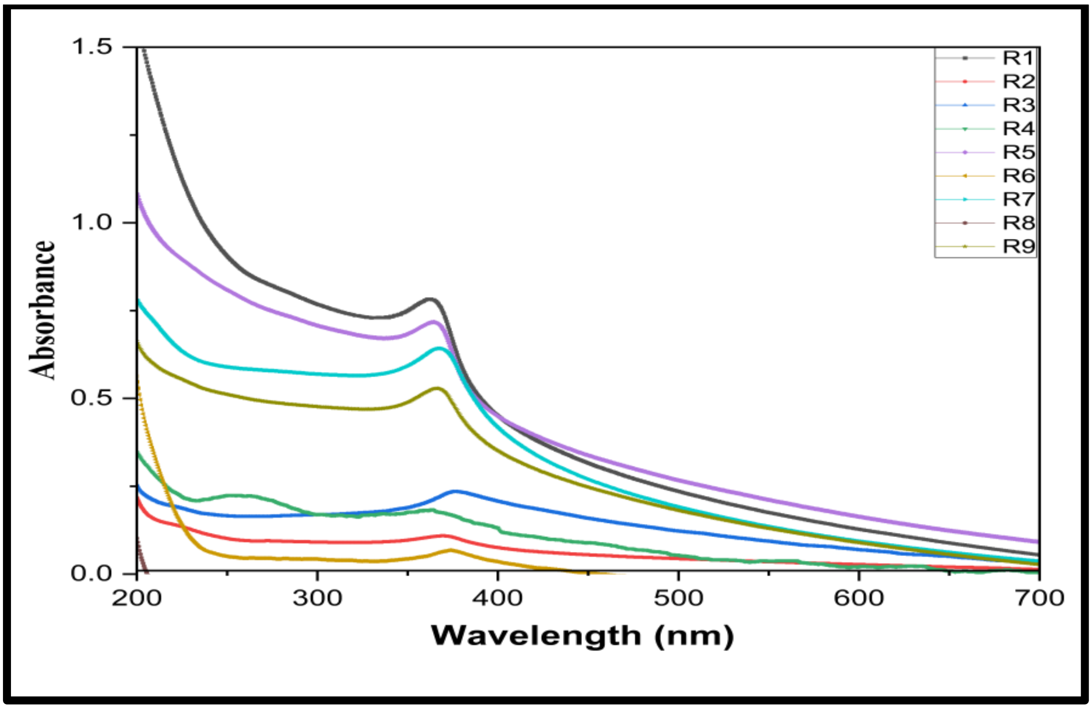

Fig 3.3: UV-Vis spectra for synthesized ZnO NPs

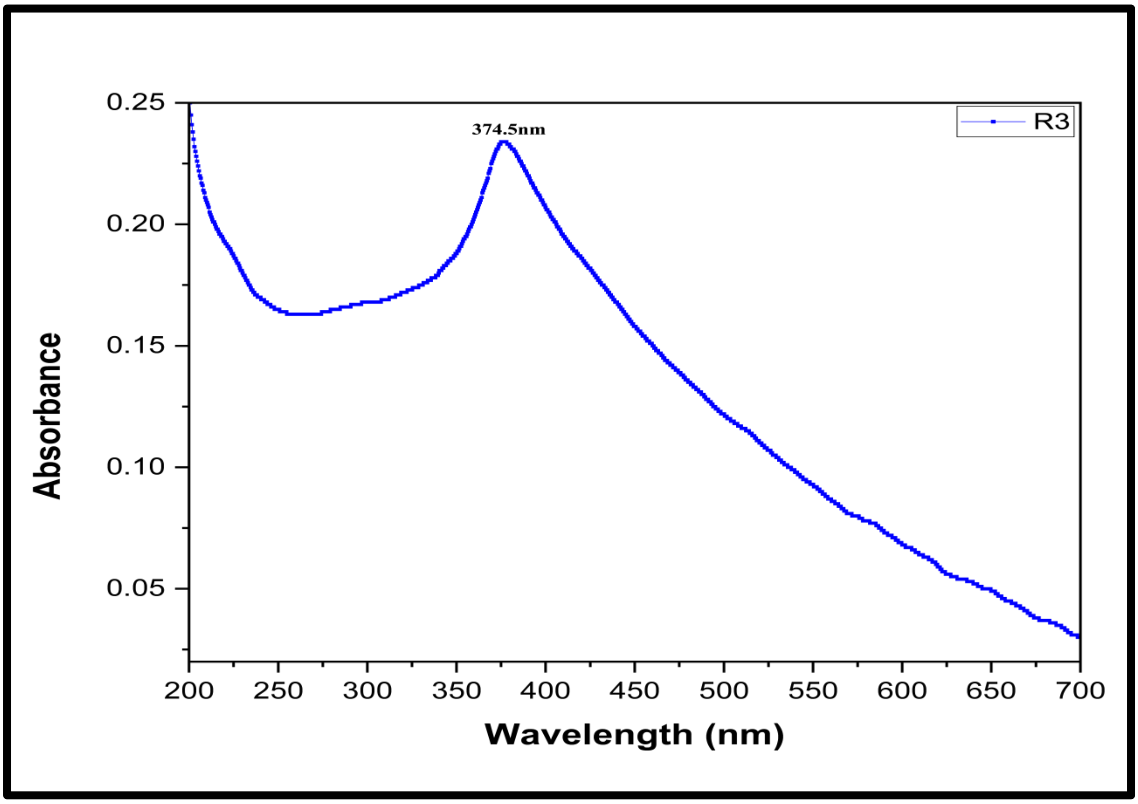

Fig 3.4: UV-Vis spectra of sample R3

3.2.2 X-Ray Diffraction:

The bio-synthesized ZnO NPs were evaluated for their crystalline structure and phase purity using X- ray diffraction (XRD) analysis as seen in Fig. 3.5. The XRD pattern showed a significant peak corresponding to its hkl plane at 32.14° (100), 34.82° (002), 36.64° (101), 47.94° (102), 57.0° (110), 63.02° (103), 66.74° (200), 68.3° (112), and 69.38° (201). The average crystallite size of the ZnO NPs was determined using Scherer's formula and was found to be 19.87nm.

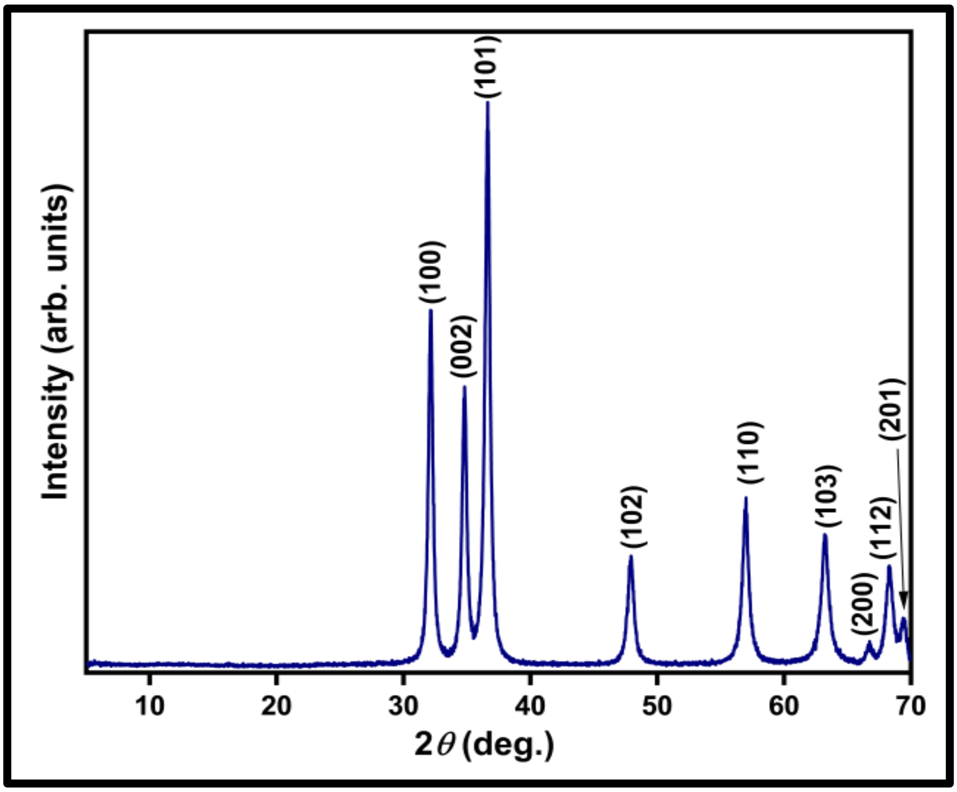

Fig 3.5: XRD pattern for ZnO NPs of sample R3

3.2.3 Fourier Transform Infrared (FTIR):

The functional group present in the ZnO NPs sample were identified using FTIR technique and the results are shown in fig. 3.6. The IR spectrum was recorded around the range of 500-4000cm^{-1}. The peak at 552cm^{-1} signifies the presence of metal-oxide bond i.e. ZnO bond. The broad peak at 3364cm^{-1} corresponds to the O-H band stretching vibrations present in the sample system of absorbed water molecule. The peak around 1413cm^{-1} is due to the presence of C-H bending vibrations of aromatic rings and the peak around 1564cm^{-1} is for C=C stretching. The absorption peak at 2992cm^{-1} indicates the C-H stretching frequency whereas the peaks 652cm^{-1} and 779cm^{-1} the value corresponds to the out-of-plane bending vibrations of aromatic compounds, specifically involving the bending motion of hydrogen atoms attached to carbon atoms within an aromatic ring.

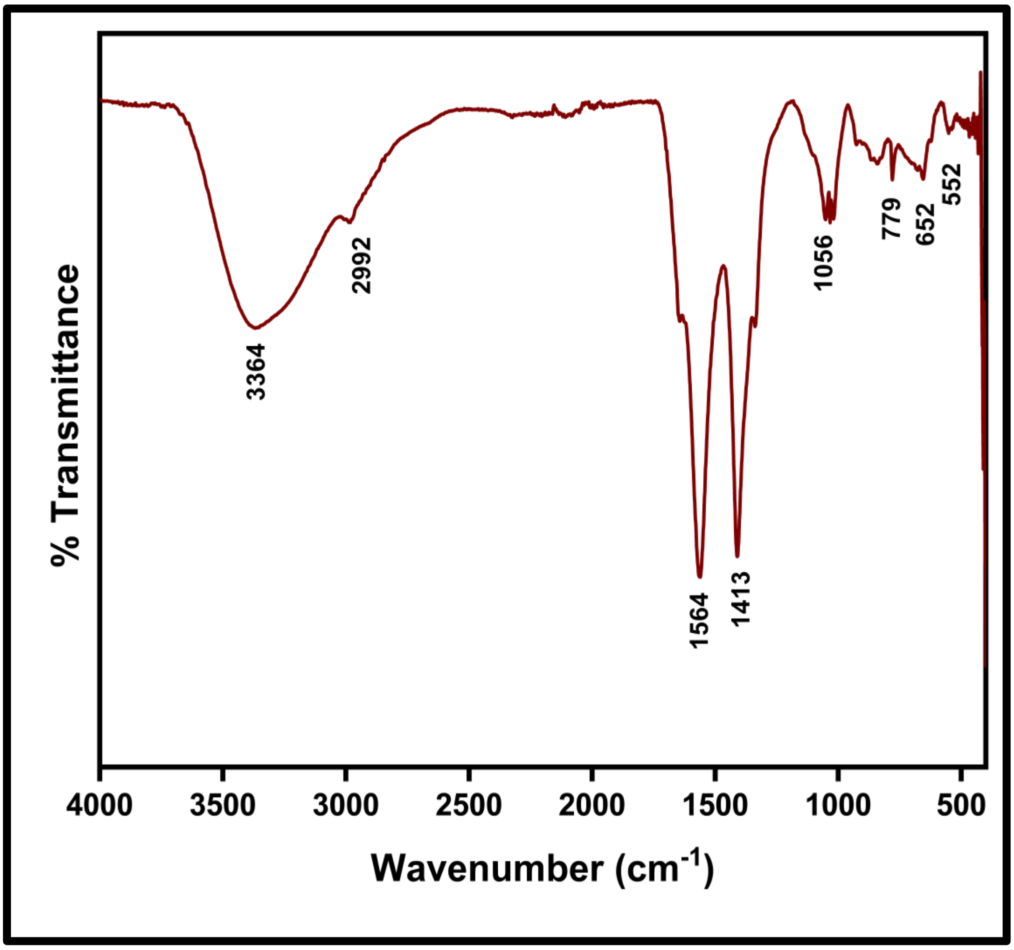

Fig 3.6: FT-IR spectra for ZnO NPs of sample R3

CHAPTER 4

CONCLUSIONS AND FUTURE PROSPECTS

4.1 CONCLUSION

In this study, the nanoparticles of ZnO were synthesized using *Coriandrum Sativum L*. seed extract using green synthesis which is green, cost-effective and simple. Preparation of ZnO nanoparticles were confirmed using the characterization techniques: FTIR, XRD, UV-Visible. UV-Vis study was conducted to find the highest wavelength value. The FTIR spectra demonstrate the impact of plant extracts on NP extraction and confirm the presence of ZnO bonds. Additionally, XRD results indicate that the particle size of the synthesized NPs falls within the range of 19.87 nm. The synthesis parameters of production of ZnO nanoparticles were optimized using Taguchi method. The parameters time (hr), conc. of ZnAc, volume of plant extract and calcination temperature was observed during the experiment. The result shows the positive influence of calcination temperature in synthesis of ZnO NPs in response with wavelength which indicates with the increase of calcination temperature increases the formation of ZnO NPs.Whereas, in response with yield concentration of zinc acetate influences the production of ZnO NPs the most. The percentage contribution of these parameters for the synthesis of ZnO NPs in response with wavelength and yield were studied.

4.2 FUTURE PROSPECTS

Zinc oxide nanoparticles (ZnO NPs) have attracted significant amount of attention due to their wide range of applications and unique properties. They have promising future prospects particularly in antimicrobial applications, dye degradation and many more.

- The synthesis of ZnO NPs may exhibit strong anti-microbial properties making them effective against a broad spectrum of microorganisms. Anti-microbial activity of ZnO NPs attributes to various mechanisms such as reactive oxygen generation species, interference with DNA along with protein synthesis making it an excellent candidate for its usage in food packaging, wound dressing, medical devices and what not.
- These nanoparticles are known for their great absorbance capacity especially in ultraviolet region. This property of ZnO NPs makes it a superior protector against harmful UV rays. It has great applications as a sunscreen and protective coatings. In future, it can be used in textiles, packaging and building materials to offer enhanced UV shielding.
- Organic dyes are common pollutants in wastewater released form textile and dyeing industries. ZnO NPs have shown significant potential in degradation of dyes. In future they can be used in wastewater treatment technologies.
- ZnO NPs may also be studied for use as supercapacitors because of their excellent electrochemical properties and large surface area.

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