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Microwave Combustion Synthesis and Characterization Studies of Zinc Oxide Nanoparticles using *Sesamum indicum* Plant Extract as Fuel

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ABSTRACT

In the prevailing study, ZnO nanoparticles had been organized via way of microwave combustion technique, using the *sesamum indicum* plant extract as fuel and reducing agent. The structural section and morphology had been investigated with the use of XRD, HR-SEM and HR-TEM. The crystallite length changed into calculated the use of Scherrer formula, and it lies with inside the variety of 20-22 nm. The elemental evaluation changed into investigated via way of means of EDX analysis. Also, absorbance and emission spectra had been measured via way of means of the use of diffuse reflectance (DRS) and photoluminescence (PL) studies. The band hole changed into measured the use of Kubelka-Munk version and it indicates 3.21 eV for ZnO nanoparticles. The photocatalytic degradation of methylene blue dye (MB) was carried out and obtained greater degradation efficiency.

Key words: *Sesamum indicum*, Plant extract, Photocatalysis, ZnO nanoparticles, Microwave combustion

Mesoporous materials are important in both macroscopic and nano applications. Nanoporous materials have been used in many areas such as electronical and biotechnological applications, etc. In particular, mesoporous titania with controlled pore structures is predictable to be useful in photocatalysis and solar cells [1-3]. The size and stability of nanoporous materials encapsulated in nanospaces are important key factors for the catalytic activity in many areas. Individual nanosheets or nanoflakes have sub-nanometric thicknesses and possess different properties depending on their elemental compositions [3-10]. ZnO have been synthesized with different morphologies, such as nanoflakes, nanowires, nanoflowers, and nanorods. Among various nanostructures, ZnO nanoflakes have been widely considered, because of their flexibility for the electron transfer and increase in the electron diffusion length [11-13]. ZnO nanostructures have been prepared by different methods, including, chemical vapor deposition, pulse laser deposition, sputtering, electro deposition, template based methods, microwave heating and thermal evaporation [12-18] etc. Fast development in nanotechnology is predictable to inspire the improvement of progressive substances with state-of-the-art functions. Nanotechnology has some of analytical elements which might be precious to the nanoscopic sciences on the atomic or molecular levels [19-23]. Generally, nanoporous substances have well-described pore systems, which may be fashioned through using a fixed of traditional and easy procedures. Mesoporous substances are vital in each macroscopic and nano applications. Nanoporous substances were used in lots of regions which include electronical and biotechnological applications, etc.

ZnO is a wide band gap semiconductor (3.37 eV) [24] with large exciton binding energy (60 meV) has a variety of interesting physical and chemical properties, thus making it suitable for various industrial applications, such as solar cells, gas sensors and laser diodes [38]. Among the various applications of ZnO nanostructures, photocatalytic activity is the most important application for the environmental protection. Furthermore, ZnO is a suitable alternative to TiO_2 which is universally recognized as the most photo-active catalyst with band gap energy of 3.2 eV and it can able to absorb solar spectrum over a larger fraction than TiO_2 [39, 40]. ZnO nanostructures have been prepared by different methods, including, chemical vapor deposition, pulse laser deposition, sputtering, electro deposition, template-based methods, microwave heating and thermal evaporation [25] etc., with different morphologies, such as nanoflakes, nanowires, and nanorods [26]. However, these methods suffered from the following drawbacks, such as high temperature, sharp thermal gradient, long reaction time, and hard-control process. Furthermore, ZnO has emerged as an attractive candidate in catalysis, due to its enhanced catalytic behaviour. In addition, ZnO is act as photocatalyst

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for the photodegradation of ortho, meta and paracresol (o, m, p-cresol) under visible light irradiation.

Zinc oxide is a wurtzite, hexagonal II-VI semiconductor with direct band gap of 3.3 eV and high exciton binding energy (60 MeV) which leads to higher UV emitting efficiency at room temperature [25]. It has been widely used in the optoelectronic applications, such as functional devices, thermo electronic materials, UV protection, photo catalyst, solar cells, blue light emitting devices, lasers, field emission displays, super hydrophobic coatings, etc., due to its excellent physical and chemical properties [25]. The shape and size of ZnO govern its electrical and optical properties and various morphologies, of ZnO NPs like springs, tower, flower, belt, ring structures were reported based on the different reaction conditions [7-11]. The synthesis of ZnO NPs by various techniques such as hydrothermal, microwave assisted methods, electrochemical, sonochemical, chemical co-precipitation, sol-gel methods were reported [25]. Thermal decomposition of various precursors like zinc oxalate, zinc nitrate, zinc carbonate and zinc acetate, were used to synthesis zinc oxide NPs. The nanosized ZnO was synthesized from colloidal Zn $(OH)_2$ by heating at various temperatures using rotary evaporators. Chemical precipitation, an inexpensive method, is becoming interesting and attractive for producing small grain size, crystalline and homogeneous ceramics at relatively low temperature and reduced processing time [16]. The synthesis of NPs by precipitation method easily forms agglomerated particles which can be controlled by using the surfactant.

In the prevailing work, ZnO nanosparticles had been synthesized the use of one-of-a-kind techniques which include microwave combustion technique using *sesamum indicum* plant extract as a fuel and reducing agent. The received samples had been characterized through XRD, HR-SEM, HR-TEM, EDX, DRS and PL spectroscopy. The photocatalytic performances of the products had been investigated.

MATERIALS AND METHODS

Preparation of ZnO nanoparticles

ZnO nanoparticles had been synthesized the usage of the precursors, zinc nitrate and *sesamum indicum* plant extract as a fuel. In stoichiometric amounts, they had been dissolved one at a time in 10 ml of deionized water and stirred for 15 minutes. In microwave combustion method, the answer turned into located in a microwave-oven (2.45 GHz, 750 W) for 10 minutes. Initially, the answer boiled and underwent dehydration observed with the aid of using decomposition with the evolution of gases. When the answer reached the factor of spontaneous combustion, it turned into vaporized and right away has become a strong. This turned into categorized as ZnO nanoparticles.

Characterization techniques

The structural and phase characterization was achieved using a Philips X'pert X-ray diffractometer with CuKα radiation at λ=1.540Å. Surface morphological studies and EDX of ZnO have been performed with a Jeol JSM6360 HR-SEM technique. The TEM images were carried out by Philips -TEM (CM20). The diffuse reflectance UV-vis. spectrum was recorded using Cary100 UV-vis. spectra to estimate their optical band gap energy. The PL properties were verified using Varian Cary Eclipse Fluorescence Spectra.

RESULTS AND DISCUSSION

X-ray diffraction analysis

The structural stages of ZnO nanoparticles have been decided through X-ray diffraction sample. The XRD styles have been recorded two times on batches of samples for reproducible outcomes. Fig. 1 suggests the XRD sample of as organized ZnO nanoparticles. The diffraction peaks of ZnO nanoparticles divulge that it has hexagonal wurtzite structure (JCPDS No. 80-0074), and no different impurity peaks are found. The sample suggests very sharp peaks, indicating that the ZnO nanoparticles have exact crystallization. As proven in Fig.6, the found diffraction peaks at 2θ = 31.67, 34.32, 36.15, 46.44, 56.48, 62.75, 67.85 and 68.95° are related to [100], [002], [101], [102], [110], [103], [112] and [201] planes, respectively. The lattice parameters have been calculated [9] the use of the system. The lattice parameters of ZnO nanoparticles are a = 3.253 Å and c = 5.215 Å. Therefore, it's far clean that the training approach has no or negligible impact at the lattice parameters of ZnO nanoparticles. The common crystallite length changed into calculated [18] the use of Scherrer system.

The common crystallite length 'L' calculated from the diffraction peaks changed into determined to be round 20-22 nm for ZnO nanoparticles. A growth within side the crystallite length of ZnO nanoparticles is feasible in the course of calcinations, due to the fact the traditional combustion approach is excessive-electricity ingesting and calls for instead lengthy response times. Hence, on publicity of the crystallites to excessive temperature for a protracted time, a growth within side the length of the crystallites takes area thanks to solid-nation diffusion.

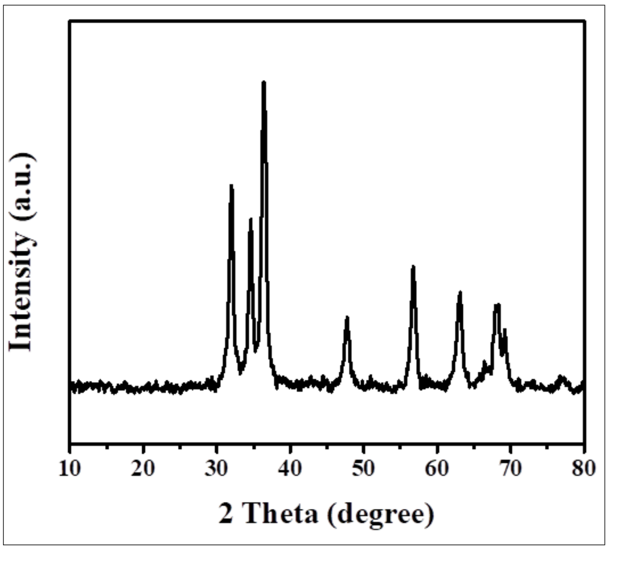

Fig 1 XRD pattern of zinc oxide nanoparticles

Table 1 Average crystallite size and lattice constant of Zinc oxide nanoparticles

Samples	Crystallite size (nm)	Band gap (eV)	a (Å)	c (Å)	u=c/a
ZnO	21	3.26	4.333	3.324	0.569

SEM analysis

The HR-SEM photographs of ZnO nanoparticles are proven in (Fig 2) and suggests the presence of ZnO nanoparticles with exact separation, various in diameter among 20-22 nm, which encompass a massive quantity of homogeneous nanoparticles and agglomerated to shape round like debris. Therefore, we are able to infer that ZnO nanoparticles were shaped with exact separation in the course of the unexpected combustion method within side the microwave combustion approach. Furthermore, ZnO round like nanoparticles can be shaped in thermal decomposition method and converted to homogeneous and agglomerated round debris while calcinizing at combustion approach. The ZnO nanoparticles have been now no longer tightly organized and there has been a way among the ZnO nanoparticles, which can growth the green reactive location and enhance its characteristics. Hence, we recommend the feasible mechanism as follows; The ZnO nanoparticles have been organized the use of a microwave oven operated at a strength of 750W withinside the gift observe, could have produced extensive quantity of warmth in the course of the combustion method. This could have resulted withinside the formation of ZnO nanoparticles in line with the subsequent whole combustion the use of sesamum indicum as a fuel. During combustion, the gaseous merchandise launched has been N_2, CO_2 and H_2O as water vapours. We anticipate that the white fumes found in the course of combustion have been launched withinside the shape of the above gaseous merchandise.

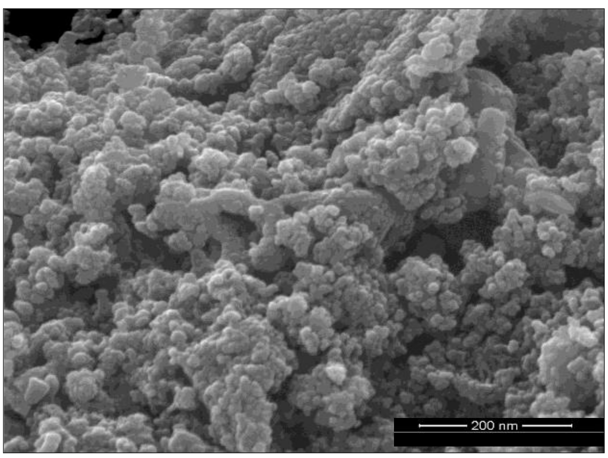

Fig 2 SEM images of zinc oxide nanoparticles

TEM analysis

In order to look at the feasible mechanism for the formation of ZnO nanoparticles to decide whether or not any secondary section exist withinside the samples, a meticulous structural characterization changed into finished through excessive decision TEM. The HR-TEM characterization of ZnO nanoparticles screen that they're unmarried crystalline nature (Fig 3a). The magnified picture of HR-TEM on ZnO nanoparticles quit suggests that it's far related to defects and crystal dislocations. The stress prompted in ZnO as defects and dislocations can behave as an excessive electricity pouch facilities and is a fantastic potential of this cloth for use withinside the regions of catalysis to be hired as adsorbents or oxidation catalyst for the elimination of very exceptional poisonous compounds and additionally for exact photovoltaic overall performance (performance), respectively. The measurement of the ZnO nanoparticles is in supplement with the SEM outcomes. The inset (Fig 3b) is the chosen location electron diffraction (SAED) sample of the ZnO nanoparticles, which suggests that they're properly crystalline in nature.

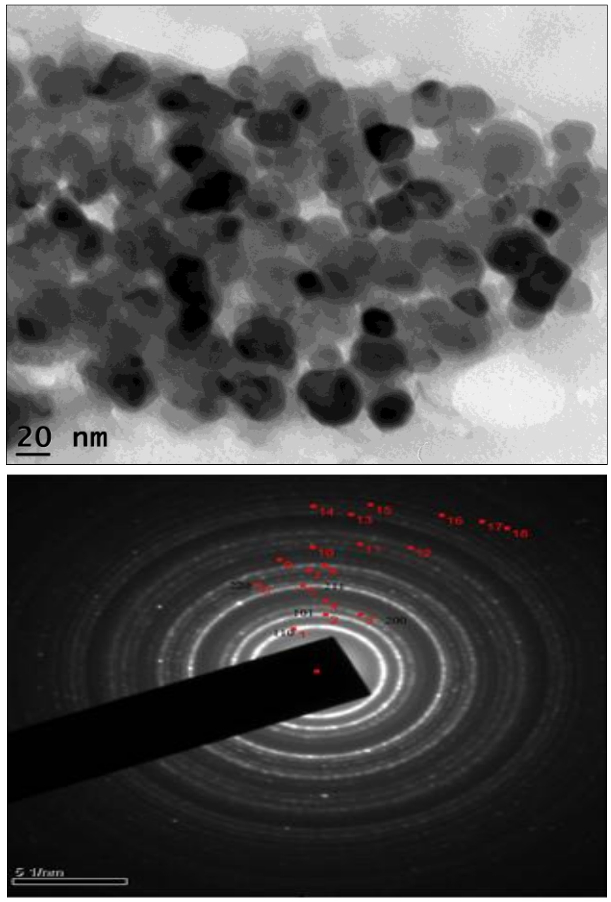

Fig 3 TEM images of zinc oxide nanoparticles

EDX analysis

Th (Fig 4) suggests the chemical composition of the ZnO nanoparticles that have been decided through EDX. It well-known shows the presence of O and Zn factors and no indicators of different factors gift suggest that the as-synthesized merchandise is natural. The found percent of Zn and O suits properly with the quantity of Zn and O used withinside the respective precursors (Fig. 4), which shows that no lack of factors befell in the course of the synthesis.

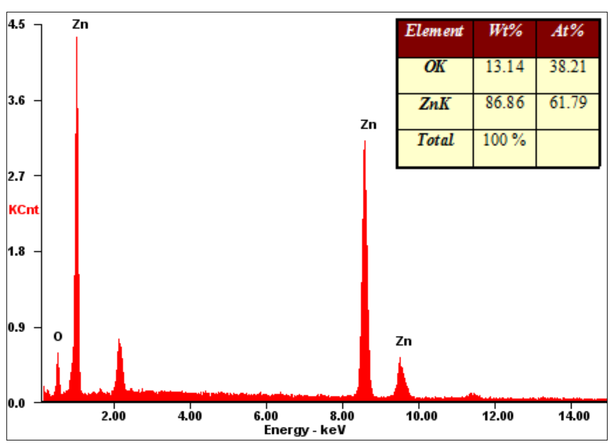

Fig 4 EDX spectra of zinc oxide nanoparticles

DRS spectral analysis

The electricity band hole (Eg) of ZnO changed into received from the optical diffuse reflectance spectral analysis (DRS) recorded at room temperature and is proven in (Fig 5). The band hole of the samples may be evaluated from the Eg measurements the use of Kubelka-Munk model [19]. A graph is plotted among $[F(R)h\upsilon]_2$ and hυ, and the intercept fee is the band hole electricity (Fig 5). The expected band hole of ZnO nanoparticles is 3.22 eV. Compared with the majority band-hole fee of ZnO (3.37 eV), the absorption edges of the ZnO nanoparticles have blue shifted, and ZnO nanoparticles have crimson shifted, which is probably attributed to the electricity stages which can be prompted through the floor and interface defects in those nanostructures.

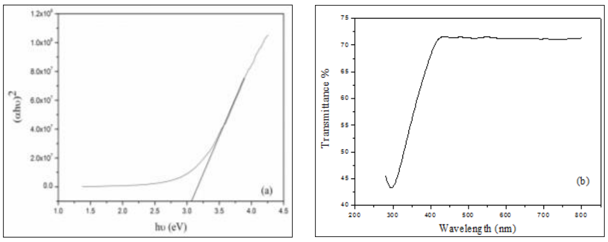

Fig 5 Optical transmittance spectra and band gap values of ZnO nanoparticles

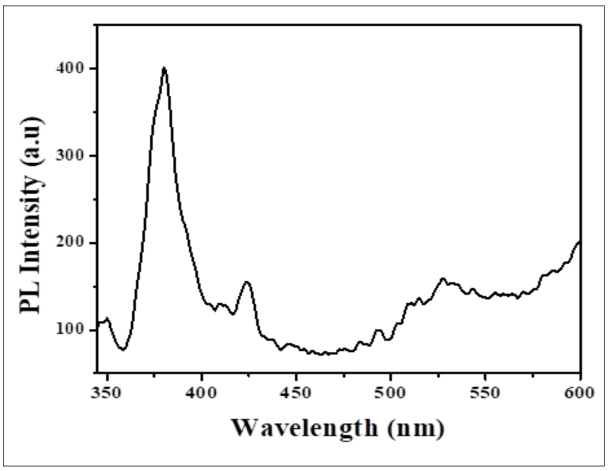

Fig 6 PL spectra of ZnO nanoparticles

PL spectral research

The PL spectra of ZnO nanoparticles are proven in (Fig 6) that changed into recorded with a view to observe the defects and different impurity states. For ZnO nanoparticles, the PL spectrum suggests an emission band withinside the UV area, focused at round 380 nm, that's attributed to the close to band-aspect emission of ZnO, and it's far because of the recombination of loose excitons thru an exciton–exciton method. However, ZnO nanoparticles exhibited a top close to 381 nm similar to UV-close to band-aspect emission indicating that the quantum confinement may be very weak [11, 13, 20]. For the samples, ZnO nanoparticles, luminescence peaks have been found withinside the seen area among 424 and 508 nm this is because of the presence of defects and oxygen vacancies. The top at 424 nm corresponds to the violet emission that can be because of radiative defects associated with the interface traps current on the grain boundaries. The top at 493 and 527 nm can be ascribed to oxygen vacancies, which provide upward push to inexperienced emissions.

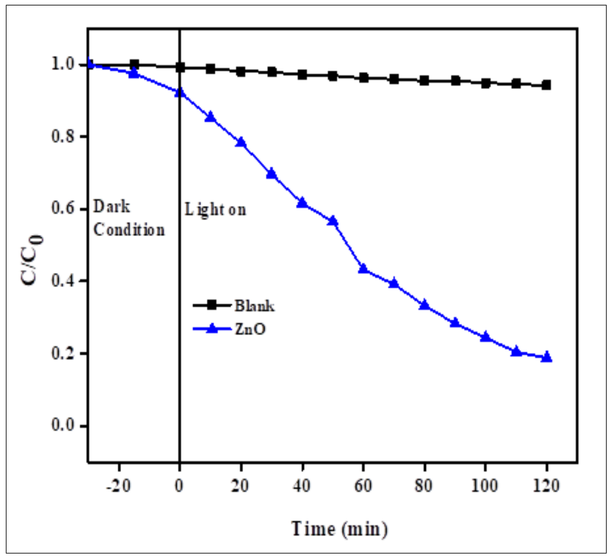

Fig 7 PCD performances of ZnO nanoparticles

Photo-catalytic degradation studies

The ZnO nanoparticles gadgets are properly-dispersed and feature excessive crystallinity, thereby main to excessive PCD performance towards the degradation of methylene blue dye [13], [22] showed in the (Fig 7). This can be because of the -dimensional period of ZnO nanoparticles, which growth the electron dispersion period, while as in comparison to ZnO nanoparticles [22-23]. However, the ZnO nanoparticles gadgets have a decrease mobileular overall performance [24] because of greater aggregation that could cause the manipulate of photocurrent and distort the plasmonic impact through broadening their spectral enrichment. The aggregated ZnO nanoparticles have efficiently reduced the floor location, and with direct touch to the dye molecules, it led to a increase quantity of adhered dye molecules to transform daylight thereby main to a increase PCD [25-26]. The electrons generated through the photo-absorption thru aggregated ZnO nanoparticles layer might also additionally have a decrease injection charge to ZnO nanoparticles in comparison to the ones absorbed through the dye, accordingly main to a lower in PCD. Finally, the formation of ZnO nanoparticles might also additionally introduce greater interface defects to the complete nanostructure medium, ensuing in an increasing of the PCD.

CONCLUSION

PCD primarily based totally on ZnO nanoparticles have been synthesized through microwave combustion using *sesamum indicum* plant extract as a fuel. A tool primarily based totally on ZnO nanoparticles confirmed a better performance while as in comparison to that of ZnO nanoparticles primarily based totally tool. The natural ZnO nanoparticles gadgets are properly-dispersed with excessive crystallinity, thereby main to excessive mobileular performance. ZnO nanoparticles withinside the shape of aggregates, could have brought about the deformation of plasmonic impact and thereby it suggests decrease performance.

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