



# Intranasal curcumin regulates chronic asthma in mice by modulating NF- $\kappa$ B activation and MAPK signaling

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## ARTICLE INFO

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## ABSTRACT

**Background:** Curcumin, a natural product found in the plant *Curcuma longa*, has been reported to have diverse range of molecular targets that influence numerous biochemical and molecular cascades including anti-inflammatory and antioxidant properties.

**Purpose:** The aim of the study was to investigate the therapeutic potential of intranasal curcumin on ovalbumin (OVA)-induced chronic asthma and to elucidate underlying molecular mechanisms.

**Study design/method:** Mice were sensitized and exposed to 2% OVA aerosol for 2 times in a week for five consecutive weeks to study effect of intranasal curcumin on various MAPK pathway enzymes involved in chronic asthma and its effect on the activation of nuclear factor  $\kappa$ B (NF- $\kappa$ B).

**Results:** Curcumin treatment decreased the ROS level in BALF and nitrite level in blood serum of chronic asthmatic mice. Curcumin treatment had significantly decreased the phosphorylation of JNK, ERK1/2, and p38 and COX-2 expression thereby nuclear factor  $\kappa$ B (NF- $\kappa$ B) activation and expression in lung tissues.

**Conclusion:** These results suggest that intranasal curcumin protects against asthma via action on mitogen-activated protein kinase (MAPK)/NF- $\kappa$ B signaling pathways.

## Introduction

Chronic asthma has been considered as a disease characterized with many airway abnormalities including airway inflammation and remodeling. Airway abnormalities particularly the increased production of various cytokines, chemokines and growth factors, leads to the activation of intracellular signaling pathways. There are many pathways which are linked to signaling molecules and their crosstalk lead to the development of asthmatic disease. Studies suggest that there is an increased expression of multiple proteins in the respiratory tract, including pro-inflammatory proteins, cytokines, chemokines, and adhesion molecules. The time course and intensity of asthmatic responses by resident and circulating cells may be regulated by various inflammatory signaling, including Src family kinases (SFKs), protein kinase C (PKC), growth factor tyrosine kinase receptors, nicotinamide adenine

dinucleotide phosphate (NADPH)/reactive oxygen species (ROS), PI3K/Akt, MAPKs, nuclear factor-kappa B (NF- $\kappa$ B), activator protein-1 (AP-1), and other signaling molecules. These signaling molecules target proteins involved in airway inflammation as well as remodeling process (Lee and Yang, 2013).

The mitogen-activated protein kinase (MAPK) pathway is one of the major signal transduction pathways, which represent a converging point for many other signaling pathways, including tyrosine and serine/threonine kinases, G proteins, and calcium signaling. MAPKs regulate a variety of biological processes like cell growth and proliferation, chemotaxis, degranulation, and other secretory processes. Studies have implicated that the MAPK family of phosphoproteins are critical signaling molecules that leading to the generation of pro-inflammatory mediators. Airway remodeling is thought to be a consequence of long-term inflammation and MAPKs are key signaling molecules that drive pro-

**Abbreviations:** AP-1, Activator protein; BALF, Bronchoalveolar lavage fluid; Cox-2, Cyclooxygenase-2; cPLA2, Cytosolic phospholipase A2; DCFH-DA, Dichlorofluorescein diacetate; ERK 1/2, Extracellular signal-regulated kinase; GFTKR, Growth factor tyrosine kinase receptors; ICAM-1, Intercellular adhesion molecule-1; IL-1, Interleukin-1; JNK, Jun-N-terminal kinase 11; Lox-5, Lipoxygenase-5; MMP-9, Matrix metalloproteinase-9; NADPH, Nicotinamide adenine dinucleotide phosphate; NO, Nitric oxide; NOS, Nitric oxide synthase; NF- $\kappa$ B, Nuclear factor-kappa B; OVA, Ovalbumin; p 38K, p38 kinases; MAPKs, Mitogen activated protein kinase; PKC, Protein kinase C; ROS, Reactive oxygen species; SFKs, Src family kinases; TNF- $\alpha$ , Tumor necrosis factor- $\alpha$ ; VCAM-1, Vascular cell adhesion molecule-1

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# Curcumin Modulates Paraquat-Induced Epithelial to Mesenchymal Transition by Regulating Transforming Growth Factor- $\beta$ (TGF- $\beta$ ) in A549 Cells

Namitosh Tyagi,<sup>1</sup> **D. K. Singh,**<sup>2</sup> D. Dash,<sup>3</sup> and Rashmi Singh<sup>1,4</sup>

**Abstract**— Paraquat (PQ), a widely used potent herbicide, generates superoxide anions and other free radicals, leading to severe toxicity and acute lung injury. PQ induces pulmonary fibrosis through epithelial to mesenchymal transition (EMT) characterized by increased number of myofibroblasts. Time-dependent PQ-induced EMT has been evaluated in present investigation where intracellular ROS levels were significantly enhanced after 24 h of PQ intoxication. Anti-inflammatory effects of curcumin have been studied where alveolar epithelial cells (A549 cells) were incubated with curcumin (30  $\mu$ M) for 1 and 3 h before PQ intoxication (700  $\mu$ M). Western blot and immunocytochemistry studies revealed that pretreatment of A549 cells with curcumin for 3 h before PQ exposure has maintained E-cadherin expression and inhibited PQ induced  $\alpha$ -smooth-muscle actin ( $\alpha$ -SMA) expression. Transforming growth factor- $\beta$  (TGF- $\beta$ ) that seems to be involved in PQ-induced EMT was enhanced after PQ intoxication, but curcumin pretreatment has effectively inhibited its expression. Immunostaining studies have shown that curcumin pretreatment has significantly reduced matrix metalloproteinase-9 (MMP-9) expressions, which were elevated after PQ intoxication. These results demonstrate that curcumin can regulate PQ-induced EMT by regulating the expression of TGF- $\beta$ .

**KEY WORDS:** Paraquat; TGF- $\beta$ ; Matrixmetalloproteinases-9; Fibroblasts and fibrosis.

## INTRODUCTION

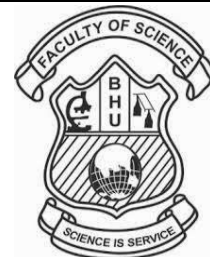
Paraquat (PQ) is widely used as highly toxic weed killer (herbicide), which can cause multiple organ failure in human and animals [1, 2]. Paraquat poisoning is a major medical problem due to lack of effective treatment. The lung is the principle target organ, and respiratory failure (irreversible lung injury) is the most common cause of death from PQ poisoning because its amount concentrate in lung tissue than any other organ [3–5]. PQ accumulation in lung occurs through type II pneumocytes *via* a polyamine uptake system. The respiratory epithelium is the

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# Effects of Pesticides on Haematological Parameters of Fish: Recent Updates

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**Abstract:** Since the blood takes part directly or indirectly in various physiological processes of the body, haematological parameters are considered important biomarker of alterations in metabolism or physiology. Indiscriminate use of pesticides has polluted different water bodies with adverse effects on the health of aquatic biota including fishes. Toxicological effects of agrochemicals including pesticides can be observed by monitoring haematological parameters. Present review deals with study of effects of pesticides on the important blood parameters such as erythrocyte count, haemoglobin content, packed cell volume, erythrocyte sedimentation rate, absolute values, leucocyte count, coagulation time and thrombocyte count of freshwater fish species. The review also aims to explain possible mechanism of pesticide induced alterations in the parameters and deleterious consequences on fish health. This could help to explore the future prospects of research in the concerned field. Haematological parameters of fish could thus serve as sensitive index to examine health status and to ascertain the toxic effects on ecosystem under pesticide exposure in the era of increased pesticide utilisation.

**Index Terms:** Fish, Pollution, Pesticides, Haematological parameters, Toxic effects

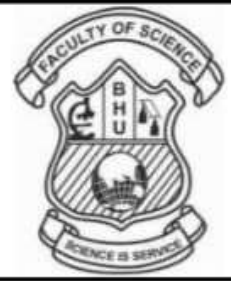
## I. INTRODUCTION

Now-a-days, detrimental ecological consequences posed by indiscriminate use of pesticides in agriculture are of great concern in general. Moreover, the increasing trend of Indian population requires self-sufficiency in food production by improved tools and techniques and effective chemicals. Pesticides are one of those agrochemicals that are widely used in agriculture to control different types of pests e.g. insects, unwanted weeds, parasitic nematodes and fungus (Tudi et al., 2021). They are being extensively used not only in the developed countries but also in developing countries to increase food production and its quality. Thus the pesticides have

occupied significant and inseparable association with the advance agricultural achievements in our country. In pesticide production and consumption, India is the biggest country in Asia and twelfth in the world while at fourth position for export (Devi et al., 2017).

In past few decades, a considerable number of researches have been carried out in relation to the adverse effects of toxic pesticides on environment and biodiversity. These chemicals are particularly the persistent ones and bring about disturbances in ecological balance in nature and various kinds of health hazards by leaving residues. A very small portion of total pesticides is in fact effective in killing or controlling target pest, while remaining large amount is released in the environment including aquatic ecosystem to have negative impact on non-target species (Tudi et al., 2021; Özkara et al., 2016). There are overwhelming evidences depicting the adverse effects of pesticides on aquatic ecosystem (Barlas 1999; Aktar et al., 2009). They pollute water by discharging surplus formulation after spraying operations into rivers, ponds and lakes, pouring the washing water of spraying equipments into water bodies; extending spread crops to the water's edge, accidental spillage of agrochemical formulations, run-off and erosion from treated areas, fallout from air pollution by agrochemical industrial effluents etc.

Water pollution is the cause of death of several interdependent aquatic forms of life and also a source of bio-magnification of persistent pesticides. This can result in local effect on environment and mortality of fish. Fishes are particularly sensitive to any change in physicochemical as well as biological characteristics of aquatic bodies. The toxic chemicals in aquatic environment are proved to be dangerous for the survival of fish (Caldas et al. 1999; Lamai et al. 1999; Sayeed et al. 2000; Isenring, 2010; Zacharia, 2011). Therefore, fish are regarded as very sensitive biological indicators of any adverse change in the



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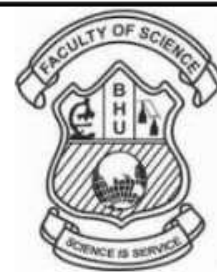
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# Plants as a Source of Potential Antioxidants and Their Effective Nanoformulations

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**Abstract:** Antioxidants act as weapons to neutralize the damaging effects of free radicals that are produced in the living systems during cellular activities and are responsible for a number of diseases. Plants are the major source of antioxidants in the form of enzymatic as well as non-enzymatic which is proved by several pieces of research on various natural products that have the potent capability to mimic the deleterious effects of free radicals. Among several antioxidants, the non-enzymatic antioxidants such as polyphenols, vitamins, carotenoids, glutathione, etc. have been isolated and screened for anti-oxidant properties from several food sources. Unfortunately, the effects of these natural antioxidants are limited due to their reduced absorption, hindrance in crossing the cell membranes and its degradation in the pathway during delivery, etc. To overcome these troubles and for effective, targeted delivery of antioxidants, its gradual and sustained release, nanoformulations of natural antioxidants are the key players in the enhancement of effectiveness and their release at the specific targeted site in the treatment of several diseases. This review focuses on the current advancements in the field of natural antioxidants and their nanoformulations with potent and enhanced action in minimizing the damaging effects of free radicals.

**Index Terms:** Antioxidant, free radicals, reactive oxygen species, nanoformulation, nanoparticles.

## I. INTRODUCTION

Free radicals are molecular species containing unpaired an electron capable of independent existence (Lobo et al., 2010). The presence of the unpaired electron in the atomic orbital of free radicals makes them highly unstable and reactive, which facilitates them to donate or accept electrons and act as either oxidant or reductant (Young et al., 2001). The presence of the unpaired electron in the atomic orbital of free radicals makes them highly unstable and reactive, which facilitates them to

donate or accept electrons and act as either oxidant or reductant (Young et al., 2001). The major free radicals associated with health disorders are reactive oxygen species (ROS), reactive nitrogen species (RNS) and reactive sulphur species (RSS) e.g. oxygen singlets, superoxides, hypochlorite, hydrogen peroxide, hydroxyl radical, nitric oxide, and peroxy radicals (Lobo et al., 2010). Free radicals play an essential role in various biological activities like a non-specific host defence mediators, cell signalling, killing of tumour cells. However, their excess damages biologically relevant molecules such as carbohydrates, proteins, lipids and nucleic acids (Young et al., 2001; Pham-Huy et al., 2008) resulting in cellular damage and homeostatic disruption. Free radicals are generated during the normal essential metabolic processes in the human system as a by-product of both enzymatic and non-enzymatic reactions, however, exposure to physical agents such as X-rays, ozone, nicotine, air pollutants and certain industrial chemicals (Bagchi and Puri, 1998; Devasagayam et al., 2004). The endogenous production of free radicals takes place through enzymatic and non-enzymatic reactions of respiratory chain, cytochrome P-450 system, prostaglandin synthesis, mitochondria, peroxisomes, xanthine oxidase, arachidonate pathway etc. (Lobo et al., 2010; Phaniendra et al., 2015; Uttara et al., 2009). The free radicals produced generally leads to damage of cellular membranes, subcellular organelles, and biomolecules leading to impairment of normal cellular functioning (Phaniendra et al., 2015). At the systematic level free radicals induced oxidative stress results in the hastening and development of several health disorders including Alzheimer, Parkinson, acute renal failure, cancer, radiation injury, diabetes, cardiovascular diseases and even ageing (Koltover, 2010; Gupta et al., 2015).

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# Omega 3 PUFA

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## Introduction

Fat, or more appropriately lipid, is an essential macronutrient which not only provides nutrition but also is essential for the delivery of energy and cell growth. Lipids are mainly comprised of carbon and hydrogen atoms which puts them in the hydrophobic category. Chemically, lipids are the esters of fatty acids with an alcohol. Alcoholic moiety in lipids may be glycerol as in triglycerides and phospholipids, sphingosine as in many glycolipids, or triacontanol as in waxes. Fatty acids are long-chain carboxylic acids containing more than four carbon atoms in their long hydrocarbon tail. Structurally, fatty acids are categorized into two classes—saturated and unsaturated fatty acids. Saturated fatty acids do not contain a double bond in the chain, e.g., myristic acid (C14:0), palmitic acid (C16:0), stearic acid (C18:0), etc. Lipids or triglycerides containing only saturated fatty acids are solid at room temperature, and are known as fats with high melting points. Unsaturated fatty acids contain one or more double bonds in their long hydrocarbon chain. Lipids or triglycerides having abundant unsaturated fatty acids are called oils, and remain in a liquid state at room temperature, i.e., they have a low melting point. Further, unsaturated fatty acids are of two types—monounsaturated fatty acids (MUFAs) with only one double bond and polyunsaturated fatty acids (PUFAs) having two or more double bonds. MUFAs include palmitoleic acid (C16:1, *cis*- $\Delta^9$ ) and oleic acid (C18:1, *cis*- $\Delta^9$ ). There is a long list of PUFAs, which have more than one double bond.

On the basis of nutritional requirements, fatty acids are classified as nonessential and essential fatty acids. Most organisms are capable of

# Effect and Importance of Compatible Solutes in Plant Growth Promotion Under Different Stress Conditions



Ashutosh Paliwal, Abhishek Verma, Harshita Tiwari, Manoj Kumar Singh, Jalaj Kumar Gour, Ashwini Kumar Nigam, Rohit Kumar, and Vimlendu Bhushan Sinha

**Abstract** A plant requires favorable conditions for their growth. When minerals, temperature, light, pH, and water are present in their optimum concentration, plant grows well, but any change in their level affects the growth of the plant. A situation that does not promote plant growth is called stress. For optimum growth of plants in stress conditions, the plant accumulates compatible solutes, an organic compound that is nontoxic and nonreactive and manages osmotic pressure in plants. Plants synthesize different types of compatible solutes in different kinds of biotic as well as abiotic stress. Compatible solutes also scavenge reactive oxygen species (ROS), and in this way, it could protect plants from oxidative stress. In this chapter, we review different types of compatible solutes and their impact on stress conditions. We also summarize the role of compatible solutes in plant growth promotion.

**Keywords** Stress · Glycine betaine · Plant growth · Compatible solutes

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# Chapter 11

## Compatible Solute Engineering: An Approach for Plant Growth Under Climate Change



Ashutosh Paliwal, Harshita Tiwari, Manoj Kumar Singh,  
Ashwini Kumar Nigam, Jalaj Kumar Gour, Rohit Kumar,  
and Deepak Kumar

**Abstract** Agriculture is not only a livelihood source but also fulfils the nutritional requirement of humans. To feed the world population increasing at an alarming rate, crop production must be increased. However, crop production is adversely affected by various abiotic factors or changes in climatic conditions worldwide. Climate exhibits a direct impact on the yield of the crop. In stress condition, the physiological state of plants gets altered that ultimately affects the overall growth of plants. Plants exhibit different mechanisms to survive these stress conditions. Plants synthesize and accumulated non-toxic, non-reactive organic compounds to counter the unfavourable conditions, and these compounds are known as compatible solutes. Compatible solutes exhibit beneficial characteristics by which plants could survive in hostile environments. Hereby, we discussed the role of few important compatible compounds and their role in plant growth as well as in different stress conditions. We have also summarized the effect of inducible changes in the compatible solutes on plant growth under different climatic conditions.

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# Evaluation of Antioxidant Potential of *Hedychium spicatum* Rhizome Extracts from Bhowali Region, Uttarakhand, India

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## ABSTRACT

**Background:** It is well-known that different extraction methods, including the technique, solvents, time and temperature extensively influence the antioxidant activity of plant secondary metabolites. In our study, *Hedychium spicatum* was used as a plant sample, collected from Bhowali region, Uttarakhand, India, using two extracting solvents (acetone and chloroform) to explore the antioxidant activity, total phenolic and flavonoid contents (TPC and TFC).

**Methods:** Initial phytochemical analysis was performed by evaluating the TPC and TFC content by Folin-Ciocalteu and AlCl<sub>3</sub> colorimetric assay. For the estimation of antioxidant activity of test samples, 2,2-diphenyl-1-picrylhydrazyl (DPPH) and ferric reducing antioxidant power (FRAP) methods were used for the determination of free radical scavenging activity, respectively.

**Results:** The FRAP results of acetone and chloroform samples was 335.782 and 254.116  $\mu\text{M FeSO}_4 \cdot 7\text{H}_2\text{O/g}$  of dry extract, respectively. IC<sub>50</sub> values for acetone and chloroform extract were calculated and used to interpret DPPH radical scavenging activity. Both tested extracts exhibited potent DPPH radical scavenging activity having IC<sub>50</sub>, 113.11 and 294.23  $\mu\text{g/ml}$  for acetone and chloroform extract, respectively. The result of TPC (12.82 mg equivalent to gallic acid) and TFC (13.998 mg equivalent to quercetin/gram) of dry extract respectively.

**Conclusions:** The overall results exhibit the high antioxidant potentiality of acetone extract as compared to chloroform extract, which could be due to its high phenolic and flavonoid content presence.

**Key-words:** Antioxidant potential, free radical scavenging activity, *Hedychium spicatum*, Medicinal plants, Plant extract, IC50

## INTRODUCTION

In modern time, attention has increased to find natural antioxidants as an alternative option for medicines, cosmetic and foods items, as a substitute for synthetic antioxidant compounds to minimize the possibility of toxicity [1]. Medicinal plants have been investigated as potential sources of natural anti-oxidants secondary metabolites and other activities like anti-cancer, antibacterial, anti-mutagenic [2-4].

Phytochemicals containing phenols are present in plants which help in protecting them from ultra-violet rays, grass-eating animals and other different forms of biotic/abiotic factors [5]. The production of secondary metabolites by the plants are usually affected by different factors such as the strength of sunlight, altitude, elevated temperature, seasonal variation, different stress conditions including biotic and abiotic factors, rainfall, maturity at harvest [6].

*H. spicatum* is well-known as 'spiked ginger' Lilly or perfume ginger. It is associated with the family Zingiberaceae. *H. spicatum* is mainly found in Himalayan regions of India, and it's also natives to China, Myanmar, Thailand, and Ethiopia. It's a rhizomatous aromatic green herb with a hearty stew. The rhizome of *H. spicatum* is utilized in the prevention of several diseases in the

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ISCAS 2017

## Synthesis and morphological study of Mn doped ZnS films

Gautam Sheel Thool<sup>a</sup>, Mitisha Baid<sup>a</sup>, Ajaya Kumar Singh<sup>a\*</sup>, Narendra Pratap Singh<sup>b</sup>

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### Abstract

Low temperature chemical bath deposition (CBD) technique has been employed to synthesize Manganese (Mn) doped ZnS films. The synthesized material was systematically characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDAX), Fourier transform infrared spectroscopy (FTIR), and photoluminescence (PL) techniques. XRD spectra revealed the existence of cubic zinc blende structure and nanocrystalline nature of synthesized material. PL spectra shows an intense band at 486 nm (2.55 eV), while the intensity of band obtained at lower energy region 375, 416 nm were decreased in the synthesized film, which indicate that incorporation of Mn decreased the surface defect in ZnS films. Surface structure of Mn doped ZnS nanoparticle was confirmed by FTIR. SEM images depicted the spherical shaped particles evenly distributed throughout the substrate. It was noticed that presence of Mn enhances the agglomeration process, resulting the larger sized particles.

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*Keywords:* Chemical bath deposition; Thin film, Manganese; ZnS; Photoluminescence,

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## 1. Introduction

Semiconductor nanoparticles have attracted a great deal of attention during the last decade due to their unique properties and potential applications.<sup>1-3</sup> New semiconductor nanomaterial possess novel optical and electronic properties, which can be technologically useful, compared to the corresponding bulk materials. Generally, II-VI semiconductor compound with a wide direct band gap nature were widely utilized in opto-electronics, spintronics devices.<sup>4</sup> In this connection, zinc sulfide (ZnS) is an important semiconductor material of II–VI group with a wide band gap energy of 3.67 eV at room temperature having the potential applications in various optical devices, i.e. fluorescence, infrared transparent, blue light photoluminescence and electroluminescence devices.<sup>5,6</sup> It is used as a reflector in optics because of its high refractive index, and due to high transmittance in the visible range, used as a dielectric filter.<sup>7</sup> It is also consider as a better alternative of CdS buffer in thin film hetero-junction solar cell.<sup>8,9</sup> In comparison with toxic CdS buffer, it is relatively cheaper, non-toxic, and contains abundant elemental constituents, and its wide band gap energy reduces window absorption loses, enhances short circuit current density of the cell. Recently, around 17.4% conversion efficiency has been achieved by ZnS buffer used CIGS based hetero-junction thin film solar cell.<sup>10</sup>

Doping is the most powerful technique to tailor the optical and electrical properties of semiconductors which allow us to tune desired properties for a particular application. Being a semiconductor with large band gap and high exciton binding energy (40 meV), ZnS particularly suitable for luminescent host material for transition metals ions (TM), acts as luminescent center<sup>11</sup> and commercially applied as a phosphor and a thin film electroluminescent material. It has a good luminescence property, shows blue and green emission, originates from self-activated and defect center. The metal doped ZnS film has great advantage in opto-electronic devices and the presence of metal dopant can modify the emission color of ZnS. Metal doped ZnS film shows band gap energy near visible region due to radiative recombination center, traps and surface defect.<sup>12-13</sup> Hence, several researches are made to study the effect of metal such as Mn, Ni, Co, Cu etc. on ZnS films.<sup>14-17</sup>

Among all TM, Mn is being the best choice of dopant due to the fact that properties of material are directly affected by the concentration of Mn ions because they can alter the energy band and form luminescence centers of different energy levels. Hence, Mn doped ZnS nanoparticles are found to have great potential applications in displays, sensors, lasers, and biomedical labeling.<sup>18,19</sup> For about 30 years Mn:ZnS has been used as a emission layer in thin film electroluminescence devices.<sup>20</sup> ZnS doped with magnetic Mn<sup>2+</sup> ion form a diluted magnetic semiconductor (DMS) which have interesting magnetic and magneto-electronic properties such as ferromagnetism, spin glass behavior, paramagnetism.<sup>21-23</sup> The magnetic spin and electron charge recombination of DMS will provide an opportunity to utilized in spintronic device such as magneto-optic devices, electro-optic modulators, photoconductors, field effect transistor etc..<sup>24-27</sup> A.N. Karov et al. showed that the Ag co-doping enhances the luminance of Mn doped ZnS thin film electroluminescent device.<sup>28</sup> Experimental study showed that shortening decay time of Mn<sup>2+</sup> doped ZnS nanocrystal is due to the purple/blue emission of ZnS nanocrystal not due to the presence of doped Mn<sup>2+</sup> centre. Hence, study of high photoluminescence efficiency of Mn<sup>2+</sup> doped ZnS nanocrystal is of great interest.<sup>29,30</sup>

A variety of techniques have been employed to synthesize Mn doped ZnS nanostructure films such as chemical vapor deposition,<sup>31</sup> CO<sub>2</sub>-laser assisted evaporation,<sup>32</sup> rf-magnetron sputtering technique,<sup>33</sup> vapor phase transport method,<sup>34</sup> successive ionic layer adsorption and reaction technique (SILAR),<sup>35</sup> chemical bath deposition (CBD)<sup>36</sup>. CBD is aqueous solution growth method which has attracted special interest for researchers, due to its simplicity, inexpensive equipment, low deposition temperature and can be employed for large area batch processing or continuous deposition. CBD works at relatively low temperature (below 100 °C), hence allow us to use organic capping agents to control the size and shape of nanostructured material, and also the film defects caused by high temperature can be avoided. Moreover, this method allows controlling the growth factors simply by varying the solution pH, temperature and bath concentration with ease.

In this paper, we synthesized Mn doped ZnS nanostructure film and systematic studies were carried out which included structural, morphological, and photoluminescence studies.

## 2 Experimental Details

### 2.1 Materials

Zinc acetate dihydrate [ $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ , Merck India Ltd], manganese(II) sulfate monohydrate ( $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ , Molychem, India), hexamethylenetetramine (HMT) ( $\text{C}_6\text{H}_{12}\text{N}_4$ , Alfa Acer, India) and thioacetamide ( $\text{CH}_3\text{CSNH}_2$ , Finar Chemicals, India) were used as precursors. All the chemicals used were of analytical reagent grade and used without further purification. All the solutions were freshly prepared with Milli-Q water (18 M $\Omega$  Millipore purified water system). The commercial microscopic glass slides with size  $1.45 \times 75 \times 25 \text{ mm}^3$  were used as a substrate for the deposition of Mn doped ZnS nanostructure films. Before deposition, the substrates were boiled for 2 hours in chromic acid, cleaned with single distilled water (SD), double distilled water (DD), degreased with acetone, ultrasonically cleaned by DD and finally dried in air.

### 2.2 Preparation and optimization of chemical bath

Aqueous solutions of 0.5 M [ $\text{Zn}^{2+}$ ], 0.1 M [ $\text{Mn}^{2+}$ ] as dopant, 0.5 M [HMT] and 1 M of thioacetamide as sulfur source, were used to prepare Mn doped ZnS nanostructure film. First 5 ml of [ $\text{Zn}^{2+}$ ] solution was placed in a beaker and 5 ml of [HMT] was added with continuously stirring for 15 minutes to get a clear and homogenous solution. Then, 5 ml of thioacetamide was added in above solution and make the volume 50 ml with Milli-Q water. The pH of the bath was maintained at 5-6 and the substrate was then placed tilted around  $60^\circ\text{C}$  inside the beaker and heated upto  $80^\circ\text{C}$  for about 15 hours to complete a single deposition run. For second deposition runs, the film obtained from the first run was submerged into freshly prepared bath for 15 more hours. The same bath was repeated up to four times. At the end of the deposition run, the layer was rinsed in Milli-Q water. For the preparation of Mn doped ZnS nanostructure film, [ $\text{Mn}^{2+}$ ] solution was added in [ $\text{Zn}^{2+}$ ]: [ $\text{Mn}^{2+}$ ] = 100 : 1 molar ratio. After deposition, the substrate was removed, washed with DD, ultrasonically cleaned for 2 min to remove loosely attached particles and then finally dried at room temperature.

### 2.3 Characterization techniques

Mn doped ZnS nanostructure films were subjected to different characterization techniques. The phase purity and microstructure of films were studied by Bruker D-8 Advance X-ray diffractometer with  $\text{CuK}\alpha$  X-ray radiation ( $\lambda=0.15496 \text{ nm}$ ). Surface morphology and elemental analysis of the films was examined by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDAX) by Hitachi S3000N SEM and LINK ISIS-300 Oxford energy dispersive X-ray spectroscopy fitted to SEM Hitachi S-520. Photoluminescence (PL) measurement was performed by Cary eclipse fluorescence spectrophotometer at room temperature. FTIR spectra were obtained using Thermo Nicolet Nexus 670 spectrometer with  $4 \text{ cm}^{-1}$  resolution.

## 3 Results and Discussion

### 3.1 Structural analysis

The X-ray diffraction patterns of Mn doped ZnS nanostructure films have been recorded in the  $2\theta$  range of  $20\text{--}80^\circ$  (Figure 1). All the diffraction peaks i.e. (111), (220) and (311), were in good agreement with the standard data for ZnS (JCPDS card no. 05-0566) indicated the formation of a cubic phase. Only diffraction peaks of ZnS present in the XRD patterns due to the small doping amount of Mn ions, hence the formation of Mn based precipitate on ZnS nanoparticles can be excluded within the limitation of XRD measurement. It was observed that presence of Mn did not alter the crystallization of ZnS nanoparticles i.e. its cubic structure remains unchanged and from the graph it was observed that no secondary phases are present in the synthesized film.<sup>37</sup> The broadening of the XRD lines is attributed to the nanocrystalline nature of the synthesized materials.

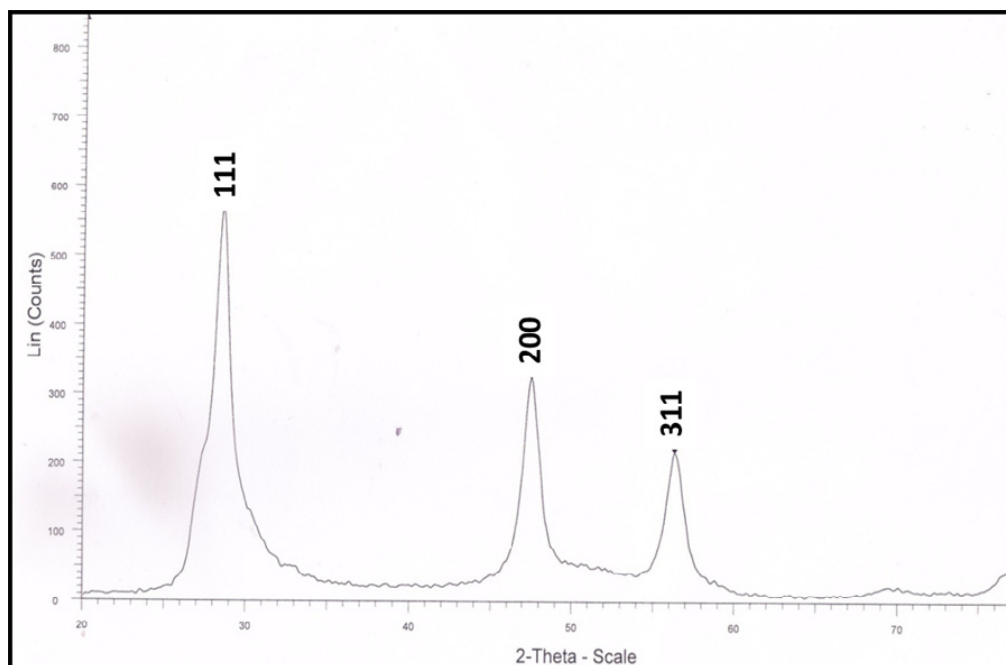


Figure 1: XRD spectra of Mn doped ZnS film.

### 3.2 Morphological analysis

Scanning electron micrographs of Mn doped ZnS films deposited on glass surface are shown in Figure 2(a) and (b). It was clearly observed that Mn doped ZnS film composed of spherical shaped particles and has a compact layer throughout the surface. Incorporation of Mn into ZnS lattice, does not change the shape of particles but size of the particles have increased and the average diameter of the film is in the range of 108-141 nm. It can be concluded that presence of Mn enhance the agglomeration process, resulting the larger sized particles. The chemical composition of the synthesized materials was measured by EDAX analysis. A typical EDAX spectrum of Mn doped ZnS film is shown in (Figure 3) which confirmed the presence of Mn in ZnS nanoparticles. It also revealed that only Zn, S and Mn are the constitute element of nanoparticles. The weight percentage and atomic percentage of the elements in the sample has shown in inset of Figure 3 and atomic percentage of Mn was found to be 0.06% with respect to  $[Zn^{2+}]$ .

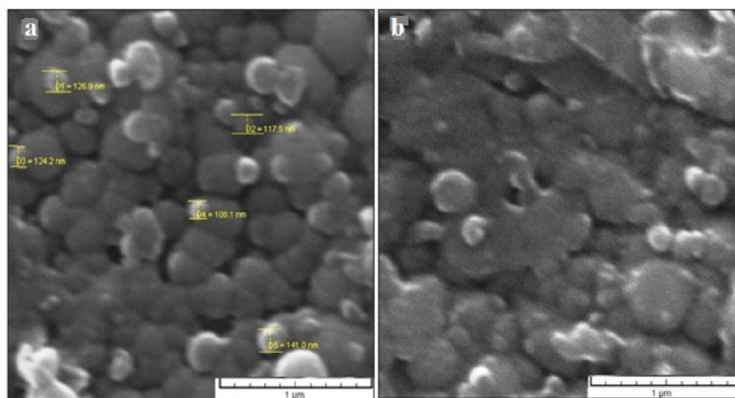
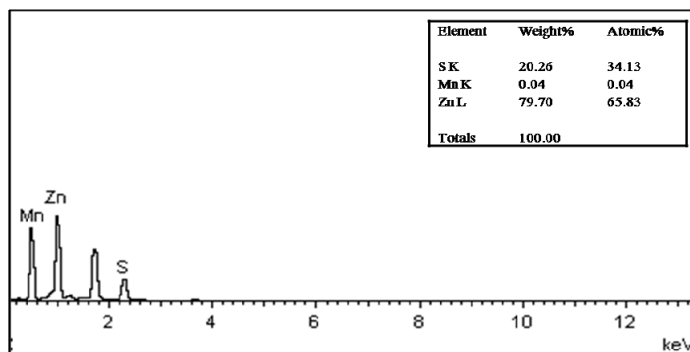


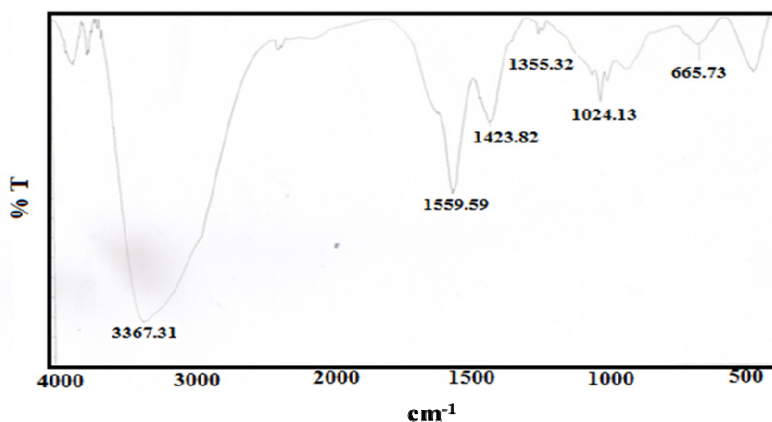
Figure 2: (a) and (b) SEM images of Mn doped ZnS at different part of the film.



**Figure 3:** Representative EDAX spectrum of Mn doped ZnS film. Inset showing weight and atomic percentage of element.

### 3.3 FTIR Spectra

FTIR spectroscopy is an efficient tool to detect the different functional groups participating in the formation nanostructure materials. To derive the surface structure of Mn doped ZnS nanoparticles, we carried out FTIR measurement in the wave number range of 400 to 4000  $\text{cm}^{-1}$  using the KBr method at room temperature shown in Figure 4. The broad peak in the range of 3000-3500  $\text{cm}^{-1}$ , centered at 3367.31  $\text{cm}^{-1}$  correspond to -OH group revealed the existence of absorbed water on the surface of nanoparticles. The band at 1559.59  $\text{cm}^{-1}$  is due to the C=O stretching vibrational mode appeared from the absorption of atmospheric  $\text{CO}_2$  on the surface of the nanoparticles.<sup>38</sup> Other peaks observed at 1423.82, 1355.32 and 1024.13  $\text{cm}^{-1}$  are due to C-H bonding which indicated presence of HMT residue on the surface of the nanoparticles. The peak at 665.73  $\text{cm}^{-1}$  is assigned to the Zn-S bond, which corresponds to sulfides.<sup>39</sup>



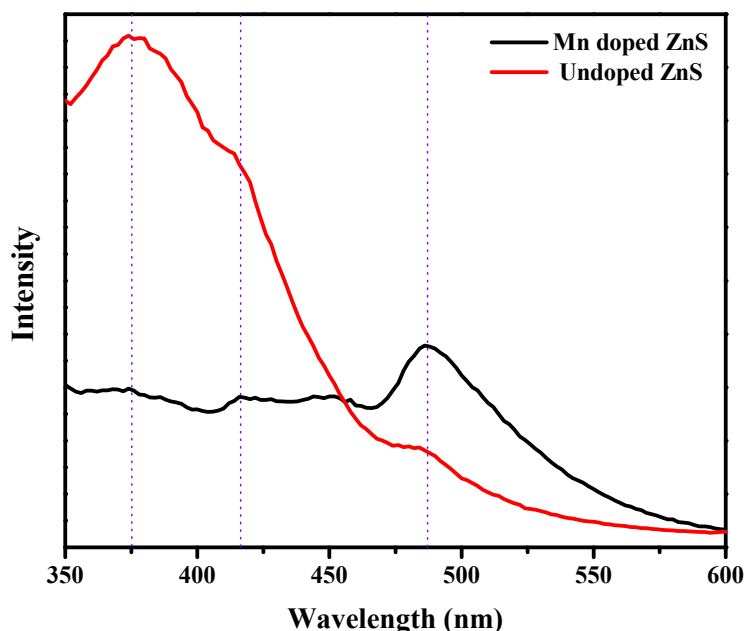
**Figure 4:** Representative FTIR spectrum of Mn doped ZnS film.

### 3.4 Photoluminescence spectra

In order to study the doping induced defects on the photoluminescence behavior of ZnS nanoparticles, emission spectra were recorded in the wavelength range 350–600 nm with the excitation wavelength of 320 nm at the room temperature as shown in Figure 5. The PL spectra of both undoped and Mn doped ZnS films consist of three bands located at 375 nm (3.31 eV), 416 nm (2.98 eV) and 486 nm (2.55 eV). The peaks are obtained at lower energy to that of energy band gap of ZnS film.<sup>40</sup> The intensity of the band located at 486 nm was found to be increased with Mn doping. While the band arises at 375 nm was completely quenched for Mn doped ZnS film. The quenching of PL intensity in Mn:ZnS film is due to the presence of Mn, which introduce a non-radiative density defect in ZnS film.<sup>41</sup> The band position was unchanged i.e. no shifting of peaks was observed but drastic change in intensity was seen. This change in intensity indicate a lower defect concentration in Mn doped ZnS film and a higher level of point defect in undoped ZnS film, this is because the self activated center associated to the lattice defect of ZnS decreases in Mn doped ZnS film.<sup>42</sup> The sharp intensity peak near UV-Vis range around 375 nm and 416 nm for



undoped ZnS is associated with the radiative recombination between the valence band and conduction band. The blue emission peak around 416 nm is mainly due to the sulfur vacancy defect and is associated to the radiative recombination of the  $e^-$  - hole pair between the defect states and the valence band.<sup>43,44</sup> Direct band to band emission and donor like sulfur vacancy might be cause of emission at 3.31 and 2.98 eV. The decreased PL intensity of doped ZnS film indicate decrease in surface defect and suggest the metal incorporation into the ZnS host lattice.



**Figure 5:** Room temperature photoluminescence spectra of undoped and Mn doped ZnS films.

#### 4. Conclusions

Mn doped ZnS films have been successfully synthesized via low temperature chemical bath deposition method. XRD study showed cubic structure and nanocrystalline nature of the Mn doped ZnS films. It was observed that incorporation of Mn into the ZnS lattice does not alter the crystallization process of ZnS nanoparticles. The presence of Mn in ZnS nanoparticles was confirmed by EDAX analysis. SEM analysis revealed that the presence of Mn in ZnS lattice enhance the agglomeration process, hence led to formation of larger crystals. FTIR analysis showed the presence of HMT residue on Mn doped ZnS nanoparticles. PL analysis shows the decrease in radiative defect concentration, surface defect i.e. sulfur vacancy in Mn doped ZnS film.

#### Acknowledgements

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ICMAM-2018

## Synthesis and characterization of PEDOT:PSS/ZnO nanowires heterojunction on ITO coated plastic substrate for light-emitting diodes

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### Abstract

In this paper we report on heterojunction between the hole transporting polymer poly(3, 4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT: PSS) and zinc oxide (ZnO) nanowires grown on an indium tin oxide (ITO) coated polyethylene terephthalate (PET) plastic substrate. For the fabrication of heterojunction simple and low cost solution methods are used. The deposited films and heterojunction are characterized by scanning electron microscope (SEM), X-ray diffraction (XRD), photoluminescence (PL) and electroluminescence (EL) measurements. Electroluminescent and photoluminescent spectra of the hybrid heterojunction show one ultraviolet (UV) near-band-edge emission peak. The current-voltage characteristic confirms the junction formation between the polymer and ZnO nanowires and shows good rectifying p-n junction diode type behaviour of the fabricated structure. The charge transfer process in heterojunction is explained by band energy diagram.

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**Keywords:** ZnO nanowires, PEDOT: PSS, p-n- heterojunction, flexible electroluminescent diode.

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# Nanocomposites for dye remediation from aqueous solutions

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## 11.1 Introduction

On the earth, for the survival of life, water is essential. Clean water is only less than 1% and also contaminated with different types of pollutants such as municipal wastes, industrial wastes, and agricultural wastes. Amongst different pollutants, dyes coming out from

industries are of major concern. Dyes are used in industries such as paper, textile, tanning, rubber, printing, and plastics. High risk to the environment occurs due to large amounts of dyes coming to water bodies. Dyes have adverse effects to humans and aquatic organisms. Toxic effects of dyes occur due to the presence of heavy metal ions in them as well as they reduce transmission of sunlight. Water-containing dyes are responsible for carcinogenicity, mutagenicity, and dysfunction of human's liver, kidney, reproductive system, brain, and central nervous system. In order to decontaminate dyes from water, different methods have been discussed (Srivastava et al., 2020). Methods with their demerits are given in Fig. 11–1. Conventional methods like distillation and reverse osmosis are costly. Precipitation and biological methods produce a huge amount of sludge, which creates a lot of disposal problems. Flocculation, coagulation, and chlorination processes require additional chemicals, making the process costly and sometimes introducing toxic chemicals in the water bodies. Membrane and adsorption technologies have problems about regeneration and fouling. However, the best method for water purification is the combination of different technologies depending on the requirement. Normally, dyes are quite stable and do not easily degrade because of their complex structures, and therefore chemical methods are not appropriate. Out of different methods, adsorption is found to be most convenient and economical.

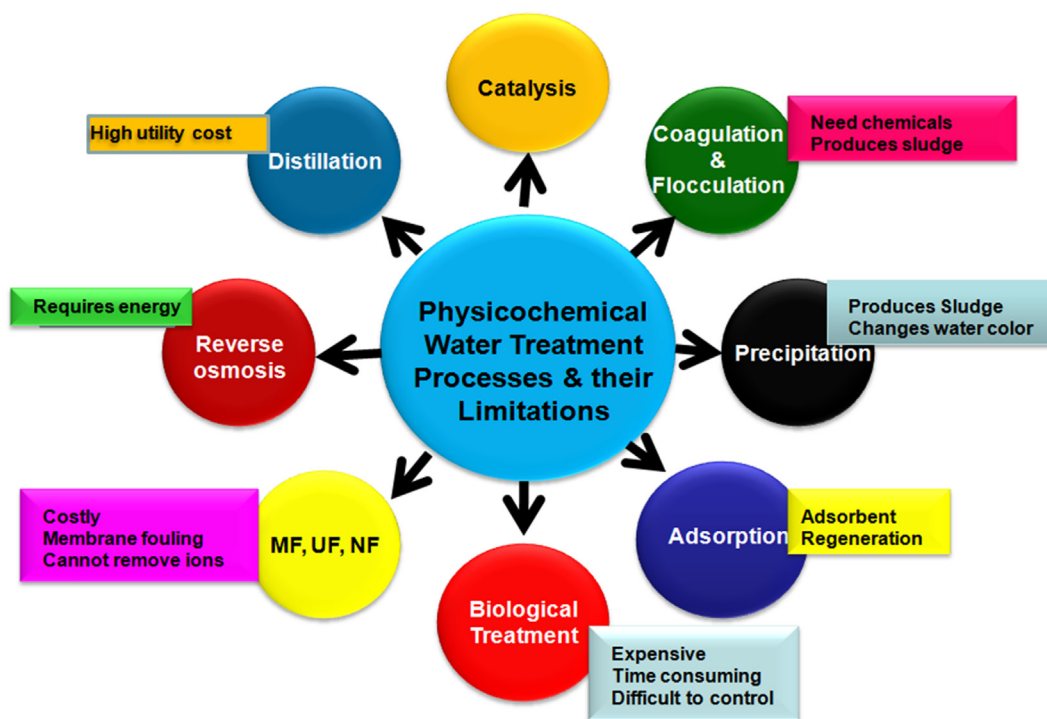


FIGURE 11–1 Different methods of water purification and their merits and demerits.