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REVIEW ARTICLE

Zinc Oxide Nanostructures in the Textile Industry

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Abstract

Background: The zinc oxide nanostructures have been using in the textile industry since the early 2000s. However, the efficiency of dye removal, antibacterial and UV protection were enhanced by researchers using different techniques. **Objective:** This review focuses on the latest research of dye removal, UV protection and antibacterial activity with mechanisms, to discover the most efficient methods to apply ZnO nanostructures effectively for the textile industry. **Findings:** The photocatalytic activity and photosensitivity of ZnO nanostructures were enhanced by doping materials such as Cu, Fe_2O_3, Co, Ce, Al, and Mn. The UV protection of ZnO nanostructures was efficient even with low loading percentages and effectively retained in the textiles. However, the dye removal efficiency was increased with pH due to the high concentration of hydroxyl radicals in the media. Although the UV protection ability of ZnO nanostructures in the textile industry was hindered by photocatalytic activity, scientists had overcome this issue by doping impurities such as SiO_2, cobalt, and manganese. The antibacterial properties of ZnO nanostructures were changed according to the loaded amount of zinc oxide nanostructures on the textile surface. In most of the research, *Escherichia coli* and *Staphylococcus aureus* were used as test organisms to study the antibacterial property of ZnO nanostructures. The green synthesis of ZnO nanostructures is more favorable for all applications due to their nontoxicity and eco-friendly nature.

Keywords: Zinc oxide; Nanomaterials; Antibacterial; UV protection; Dye removal; Textile industry

1 Introduction

In the textile industry nanostructures have been used to attain various extraordinary properties such as self-cleaning surfaces, stain resistance, water repellency, electrical conductivity, antimicrobial resistance, hydrophobicity or controlled hydrophobicity, wrinkle resistance, anti-static, anti-odor, fire resistance, UV radiation protection,

abrasion resistance, shrink resistance. In the health sector, healthcare-associated infections that caused due to spreading of bacteria and virus can be eliminated by the usage of textiles incorporated with nanostructures such as silver, titanium, and ZnO [1]. Numerous nanomaterials are used in the textile industry such as titanium, silver, and zinc oxide (ZnO) nanostructures. However, ZnO nanostructures have gained much interest due to their superior properties when compared to other materials. The ZnO nanostructures are widely used in most industries because of their favorable features such as nontoxicity, stability, cost-effectiveness, and ease of synthesis. This review presents applications of ZnO nanostructures in the textile industry because of their desirable properties such as nontoxicity, stability, cost-effectiveness, and ease of synthesis [2]. Although there are research that have addressed dye removal, UV protection and antimicrobial activity of textiles individually, this review focuses on the latest research on each section with a comprehensive explanation of the mechanisms involved. Furthermore, this will help researchers to discover the most efficient methods to apply ZnO nanostructures effectively for the textile industry.

1.1 Dye removal

Color plays a vital role in the attraction of the fabric towards customers. Therefore, dyes are widely used in the textile industry to obtain the required color for fabrics [3]. The main hazardous waste of the textile industry is reactive dyes which are discharged into the environment during the dyeing process [4, 5]. Chromophore and auxochromes are two main groups of compounds in organic dyes. Frequently used acid dyes that are used in the coloring process of wool, nylon, and silk, consist of sulfonic acid salts, carboxylic, or phenol acids [4, 6, 7]. Most used dyes have ring structures that make them stable and resist biodegradation. Furthermore, most of them are carcinogenic and toxic not only to animals but also to the whole environment [4, 8, 9]. Dyes are mainly water pollutants and even a small quantity can hinder the photosynthesis process of aquatic vegetation [6, 10]. In addition, dyes make water sources colored and it results in less penetration of sunlight. Furthermore, it reduces dissolved oxygen level in the water and lead to water pollution which is a serious environmental problem in the world [9, 10]. There are many highly toxic compounds in dyes such as sulphur, naphthol, nitrates, acetic acid, soaps, enzymes chromium compounds, and heavy metals like copper, arsenic, lead, cadmium, mercury, nickel, and cobalt. Therefore, removing organic dyes from effluents of the textile industry has become a matter of grave concern among researchers [11]. The pollution intensity due to a dye depends on several factors such as the type of fabric, the chemical structure of dye, dyeing equipment, and the used liquor ratio [4, 12].

ZnO which is a n- type semiconductor is capable of producing electron and hole pairs under both visible and ultra violet irradiation [13]. These generated electrons react with adsorbed oxygen and water to produce O_2^- and OH, respectively. Finally, the dyes are oxidized by formed O_2- and OH and mineralized into water, carbon dioxide and inorganic compounds [14].

$ZnO + hv \rightarrow h^+ + e^-$

$h^+ + OH^-$ or $H_2O \rightarrow OH$

$e^- + O_2 \rightarrow O_2^-$

OH^- or O_2^- + azo dyes $\rightarrow CO_2 + H_2O$ + inorganic molecules

1.2 UV protection

Nowadays textiles are designed to protect the human body from various environmental conditions. The Ultra Violet (UV) radiation can cause skin cancers and alter the function of the immune system [15]. The solar radiation which reaches the earth's surface contains several regions such as ultraviolet, visible (VIS), and infrared (IR) radiation which ranges from 280 nm to 3000 nm. Furthermore, the UV region can be categorized into three as very high energy UV- C rays, high energy UV –B rays, and low energy UV –A rays [16]. The high-energy UV – C rays do not reach the earth's surface while a small amount of UV – B rays reach the earth and able to cause acute and chronic illnesses including erythema, photocarcinogenesis, and photoaging [17-20].

The UV protection properties of textiles are characterized by a factor named ultra protection factor (UPF) which determines by the transmission of UV light through the textile material. UPF can be calculated by the following equation,

$$UPF = \frac{\Sigma E(\lambda).\varepsilon(\lambda).\Delta\lambda}{\Sigma E(\lambda).(\lambda).\varepsilon(\lambda).\Delta\lambda}$$

$E(\lambda)$ = the solar irradiation [Wm-2 nm-1]

$\varepsilon(\lambda)$ = the erythema action spectrum

$\boxtimes(\lambda)$ = the spectral transmittance through specimen at wavelength λ

$\Delta\lambda$ = the wavelength interval of the measurement [nm]

The transmission of UV light through textiles depends on several factors such as the specific fiber material, moisture content, structural characteristics of the fabric, the color and dyeing intensity, presence of optical brightening agents, laundering

conditions of the garment, and specific finishing products such as UV absorbers. Moreover, when selecting fabrics to enhanced UV protection ability it is necessary to consider fabric types that are worn in summer [19]. Inorganic UV absorbers are more preferable to organic UV absorbers because of their properties such as chemical stability and nontoxicity under exposure to high temperatures or UV radiation [21, 22].

To overcome these issues, garments with UV protection are designed by incorporating nanomaterials [23] especially metal oxides such as ZnO and titanium oxide (TiO_2) due to their high-temperature tolerance and stability [24]. The TiO_2 can absorb +the light around 310 nm to 400 nm and does not cover the entire UV region. However, ZnO nanoparticles have a wide bandgap (3.3 e.V. corresponding to 376 nm) that gives them unique electro-optical properties. Therefore, ZnO nanoparticles have super UV blocking capacity because they absorb light with energy same or exceeding its bandgap [25]. Zinc oxide is a very useful semiconductor, which exhibits unique properties such as photocatalytic, electric, and optical properties [26]. Especially, ZnO effectively blocks radiation in the UV –A region [27].

1.3 Antibacterial activity

The modern health and hygiene concerns have been built a greater demand up for smart/ anti-microbial textiles. The antimicrobial function of textile is mainly two-fold. It protects the wearer from microorganisms for hygiene and medical reasons and protects the textile from biodeterioration caused by bacteria and fungi [28].

Micro-organisms can grow on textiles during their use and storage, causing fabric discoloration, formation of spots with color alteration, and reducing fabric strength while generating some bad odors [29]. The textile products, especially are made of natural fibers are the best environments for microorganisms to grow, due to their moisture content and large surface area [30]. For an instance, cotton fabrics are very susceptible to attack by microorganisms, because of cellulose biodegradation [31]. Fiber absorbent and easy stain by liquids are caused by the abundant water-absorbing hydroxyl groups on the cotton fabric, which leads to the formation of microbial colonies [32]. To overcome these issues, many researchers have been looked into adding some anti-microbial properties to textile materials with aiming to protect them from microbial damage. Meanwhile, the researchers have observed that anti-microbial textiles have the ability to protect the users against some pathogenic micro-organisms [33].

Recently various diseases due to microorganisms have risen rapidly [34] while increasing the usage of different antibiotics as medicine, leading the microorganisms are becoming resistant to medicine (ex. antibiotics) with the occurrence of superbugs. Hence, the feature of protecting the wearer from micro-organisms leads to create a remarkable demand for anti-microbial textiles. For an instance, antimicrobial textiles have been used in preparing hospital gowns, patient clothes and bed covers to prevent transferring harmful pathogens [35-39] while reducing the bioburden in clinical wards and it helps to reduce the risk of hospital-acquired infections. [30, 35, 40]. Biocides and biostatic are key in anti-bacterial textile finishes as they kill the microorganisms or inhibit their growth respectively while retaining optimum material protection. However, biostats are more preferred to use in the textile industry as they preserve the natural microbial flora of human skin [41].

Antibacterial materials which are used in the textile industry can be categorized broadly into two major groups such as organic (aldehydes, amines, and phenols) and inorganic (metal ions, oxides, nanostructures, and photocatalysts) materials. However, inorganic materials such as metal oxides have to gain more attention than organic materials due to their ability to withstand harsh environmental conditions and be safer for humans [33, 42]. These inorganic materials kill micro-organisms through various mechanisms, such as by binding to intracellular proteins and inactivating them, generation of reactive oxygen species, and direct damage to cell walls [43, 44]. The feature of protecting the wearer than minimizing textile degradation of anti-microbial textiles had led to create a turning point in the smart textile industry [30]. The application of nanostructures as inorganic compounds to textile materials has been gained advantages towards (antimicrobial fabrics) smart textiles, which are like ordinary textiles but they feature some extraordinary functionalities for specific applications such as antimicrobial [45-47], UV-protection [48, 49], self-cleaning (passive smart textiles), water-resistant ability, thermoregulation (active smart textiles) and the multidisciplinary properties such as sensing and actuation (Ultra smart textiles) [50]. Silver nanoparticles are the widely used antimicrobial agent in textiles so far. However, its toxicity and potential environmental impact lead to limit its usage. As a result, researchers tend to use nanoparticles of metal oxides such as ZnO, CuO, and MgO in textiles due to their superior antimicrobial properties and low toxicity in low concentrations [44, 51-53].

2 Methodology

2.1 Dye removal methods

There are physical, chemical, physicochemical, and biological methods to remove dye from wastewater. Some of them are coagulation, reverse osmosis, ion exchange, precipitation; photodegradation, biological treatment, filtration, chemical oxidation, adsorption, and membrane process [4, 7]. The most popular physicochemical treatment method is adsorption among these methods [4, 12] which is more effective, safe, and applicable than other methods. Various adsorbents such as natural polymeric materials (chitosan) [4], activated carbons, clays, synthetic polymers, hydrotalcite, and zeolites have been used to remove dye from wastewater [54]. The use of activated carbon is an efficient method that is able to remove most of the pollutants in water due to its high surface area and porosity. Nevertheless, the application of activated carbon limits because of high cost, regeneration, and disposal [55].

Recently, Advanced Oxidation Processes (AOPs) have been used to treat dyes in wastewater mainly via photocatalytic processes [56, 57]. This method is widely used to degrade compounds that are resistant to degradation. Complete mineralization can be obtained using this process. In this process, highly reactive hydroxyl radicals are used to degrade a wide range of pollutants [58, 59]. Before the photocatalytic process, the sonochemical process was used as an advanced oxidation process. However, consumption of a large amount of energy and incomplete mineralization leads to a sonocatalytic process that suitable catalysts are used with ultrasound irradiation [60. 61].

Photocatalysis initiates chemical reactions by activating catalysts using light irradiation. This process is used for the degradation of organic pollutants discarded from industries [62]. photocatalytic degradation has been paid more interest for the decolorization of organic dyes in waste [63, 64]. Photocatalytic degradation is aided by using semiconductors. When molecules absorbed energy, excited electrons are transferred more to the conductive band forming hole-electron pair, which leads to degrade textile dyes like organic molecules by a redox reaction [65]. Due to abundance, chemical and photochemical stability, and low toxicity, ZnO has been used as one of the photocatalysts in textile dye degradation processes [66, 67].

2.2 Synthesis of ZnO nanostructures for dye removal purposes

There are several methods of synthesizing ZnO nanostructures for dye removal purposes. In the study done by (Nassar et al., 2016), zinc oxide nanoparticles were synthesized by thermally decomposing (400 °C, 1 h) hydrozincite (Zinc hydroxide carbonate), which was synthesized using the hydrothermal method. As precursors zinc salts (Sulfate, acetate, and nitrate) and ammonium carbonate were used to synthesis hydrozincite nanospheres and nanoflowers. Furthermore, organic templates such as surfactants, block copolymers, ionic liquids are used in the synthesis of nanoparticles [68]. Cetyltrimethylammonium bromide (CTAB), ethylene glycol, diisopropylamine, polyethylene glycol, and triethylamine are some of the widely used organic templates in nanoparticle synthesis [69, 70]. However, apart from chemicals naturally occurring templates such as sugars, proteins and carbohydrates were used for the synthesis of nanoparticles. In the study done by [71], the egg white was used to assist the sol-gel synthesis of zinc oxide nanoparticles. Moreover, several studies used egg white for the synthesis of ZnO nanoparticles [71]. Song et al. has used 18.5 g of zinc nitrate which was dissolved in 50 ml of distilled water and 50 ml egg white solution for the synthesis [72].

Nanobiotechnology has become an interesting area for the synthesis of nanostructures among researchers due to its low cost, nontoxicity, simplicity, and Eco-friendliness. In the biological synthesis of ZnO nanostructures, scientists used various plant extracts such as Vitextrifolia L.(leaf) [73, 74], Zingiber officinale (rhizome) [75], Nepheliumlappaceum L. (fruit peel) [76], Eucalptus globules (leaf) [77] Cappariszeylanica [78], Musa acuminata peel extract [79], ananas comosus [75]. Jamdagni et al. [80] used Nyctanthes arbor-tristisdivaricate flower extract to synthesize ZnO NPs in the particle size range of 12-32 nm. Golmohammadi et al. [81] synthesized ZnO NPs from $Zn(NO_3)_2$.6H2O using the aqueous extract of jujube fruit as a reducing agent and stabilizer.

Although there are many dyes used in the textile industry, about half of the dyes used are azo dyes [14]. Therefore, the photocatalytic activity of ZnO nanostructures has been studied using azo dyes. Nassar et al., 2016 tested the photocatalytic activity of Reactive Black 5 (RB5) dye and the absorbance of the supernatant was measured at 598 nm wavelengths. After, the remaining concentration of the dye was obtained using a calibration curve. Another comprehensive study has done by Chen et al in 2017 using ZnO nanostructures synthesized by the sol-gel method. Here Methyl Orange (MO) was used as an azo dye and various factors affecting the efficiency of dye removal were studied [14].

Pudukudy and Yaakob, 2015; used egg white to synthesize ZnO nanostructures in their study by adding 50 ml of egg white to 18.5 g of zinc acetate. Nanoparticles with high purity resulted from this method and those nanoparticles were used to study photocatalytic activity against different types of textile dyes (10 mg/ L) such as malachite green (MG), crystal violet (CV), orange

II (OII), methyl orange (MO) and congo red (CR). However, in 2020 Molaei and Rahimi-Moghadam varied ratios of egg white to zinc nitrate as 1:1, 2:1, and 3:1 and ZnO structures with 48, 42, and 29 nm [72].

2.2.1 The Green synthesis of ZnO nanostructure for dye removal

The attention towards the green synthesis of ZnO nanostructures is much more popular than chemical synthesis since it is an environmentally friendly process. Aminuzzaman et al., in 2018; the Photocatalytic activity of biologically synthesized ZnO nanoparticles using Garcinia mangostana was studied using malachite green (MG) dye by introducing 50 mg of ZnO nanoparticles to 50 ml, 10 mg l-1 dye solution. Before exposing to sunlight the suspension was stirred with a magnetic stirrer for 30 minutes to reach adsorption-desorption equilibrium. The degree of photodegradation was monitored by measuring the absorbance at 615 nm that is the highest wavelength value of MG. The photodegradation activity of synthesized ZnO particles was carried out on a sunny day between 11 a.m. and 2 a.m., where a minimum fluctuation of sunlight is available for the reaction to happen [82].

The photocatalytic performance of biosynthesized ZnO NPs using jujube fruit was investigated under direct sunlight for the degradation of Methylene Blue (MB) and eriochrome black-T (ECBT). For that process, 15 mg of ZnO NPs were dispersed into an aqueous solution of the dye (15 ml, 100 ppm) and stirred in the dark for 30 minutes to achieve the adsorption-desorption equilibrium [83]. Kazeminezhad and Sadollahkhani have studied the photocatalytic activity of ZnO nanostructures synthesized using the co-precipitation method against Eriochrome black- T dye [84]. Karnan and Selvakumarin in 2016 used the "Rambutan" peel for the synthesis of ZnO nanostructures and its effectiveness of photocatalytic activity has been tested using methyl orange (MO) [76]. Similarly, [83] used ZnO nanostructures synthesized from Cappariszeylanica also loaded to 150 ml of methylene blue solution were kept in dark to reach adsorption-desorption equilibrium for 30 minutes. Also, an absorption spectrum peak at 665 nm was used to study extend of photodegradation of MB using ZnO nanostructures [78]. Siripireddy and Mandal, 2017 used the plant Eucalyptus globules as a capping agent in the synthesis of ZnO nanostructures and their efficiency in photocatalytic activity has been tested using methylene blue and methyl orange dyes [77]. Barzinjy and Azeez in 2020 prepared ZnO nanostructures using the same plant Eucalyptus globules and precursor with some differences in the synthesizing method. Thus, the temperature of the plant extract was maintained at 60 °C until the solution converted to a yellow paste, while in previous research there were no concerns about the temperature of the extract [85].

In some researches, photocatalytic activity has been studied under UV irradiation [73, 85]. However, the study done by Anbuvannan et al in 2015 has used visible light irradiation for the degradation of MB dye in the textile effluent. [75]. In another study that used leaf extract of Vitextrifolia for the synthesis of ZnO nanostructures, were performed photocatalytic activity against MB under UV irradiation for different time intervals [73].

2.2.2 Doped ZnO nanostructures for dye removal

Photocatalytic activity and photosensitivity of zinc oxide nanoparticles were enhanced by doping elements such as Zr [86], Cd [87], Ce [87], Eu [87], Nd [88], Sm [89] and Sn [90]. The large bandgap of ZnO requires a large amount of energy for photoexcitation. When doping elements to the ZnO nanostructures the dopant has incorporated as a solution in the used synthesis method (91).[Table 1]

The effectiveness of Mn-doped ZnO nanoparticles over pure ZnO was emphasized by many researchers [92]. Del Gobbo et al., in 2020 doped aluminum and cerium with ZnO nanorods to study the effectiveness of the photocatalytic activity [93]. Rodwihok et al., in 2020 studied the photocatalytic activity of only cerium-doped ZnO nanoparticles by doping different percentages of Ce into ZnO nanoparticles [94]. Moreover, photodegradation of methylene blue under UV irradiation has been studied using Cobalt (Co) loaded ZnO nanostructures in different concentrations [95].

Shah et al., 2020 used Copper (Cu) doped ZnO nanorods to study the efficiency of photodegradation in methylene blue and methyl orange [96]. Although most of the studies were based on single element doping with ZnO nanostructures, Rahmah et al., in 2020 doped Fe_2O_3 to study the photocatalytic activity of doped ZnO under visible light irradiation. They found that when Fe_2O_3 doped with ZnO nanoparticles it forms a metal oxide nanoparticle heterojunction barrier that facilitates the reduction of recombination rate by transferring photogenerated electrons of Fe_2O_3 in the conduction band to the ZnO conduction band. The availability of more surface charge carriers on Fe_2O_3-doped ZnO nanostructures; unpaired photogenerated electrons in the conduction band and holes produce superoxide anion radicals and hydroxyl radicals (react with water) respectively. Moreover, results showed significantly enhanced degradation percentages of 80.6, 83.8, 85.5, and 92% for doped ZnO, respectively, as compared to 73.8% for undoped ZnO [97]. Photocatalytic activity of Indium doped ZnO was studied by Yu et al., in 2020. According to their results of Indium doped acts as an effective catalyst to remove methylene blue and methyl blue from water effluents. Also, the efficiency of removing methylene blue (96.84%) was higher than methyl blue (90.05%)[98]. Moreover, Samarium [99] and Strontium [100] were used to enhance the photocatalytic activity of ZnO.

Table 1. Doped materials with pure ZnO in the textile industry

Doped material	Precursor reagents	Synthesized method	Used textile dye	References
Ag (Silver)	Zn $(NO_3).6H_2O$ $Ag(NO_3).6H_2O$	Sol gel	Methylene blue	[7]
	$ZnCl_2$ $Ag(NO_3).6H_2O$ $H_2C_2O_4$	Thermal decomposition	Basonyl Violet	[166]
Dy (dysprosium)	$DyN_3O_9.6H_2O$ $ZnCl_2$ NaOH	Sonochemical method	Acid Red 17	[167]
Gd (gadolinium)	Gd($NO_3)_3.6H_2O$ C_2H_5OH Zn $(CH_3COO)_2$. $2H_2O$	Sonochemical method	Acid Orange 7	[168]
Pr (praseodymium)	C_2H_5OH. $4H_2O$ Pr $(NO_3).6H_2O$ $ZnCl_2$ NaOH	Sonochemical method	Acid Red 17	[169]
Chitosan	CH_3COOH NaOH	Sol gel	Reactive Black HN Reactive Magenta HB	[12]
Mn	Zn $(CH_3COO)_2$ $2H_2O$), $NH_4)_2CO_3$, $Mn(CH_3COO)_2{\cdot}4H_2O$, KOH	Chemical Precipitation	brilliant green	[92]
Aluminum and Cerium	Anhydrous zinc acetate	Chemical Precipitation	methyl orange	[93]
Cerium	Zinc nitrate hexahydrate Cerium nitrate hexahydrate ($Ce(NO_3)_3.6H_2O$) and sodium hydroxide (NaOH)	one-pot hydrothermal technique	methyl orange (MO)	[94]
Cobalt	$Zn(CH_3COO)_2.2H_2O$, $Zn(NO_3).6H_2O$, $C_6H_{12}N_4$, $Co(Ac)_2\bullet$ $4H_2O$ powders	hydrothermal technique	Methyl blue (MB)	[95]
Cu	Copper acetate (Cu $(OAc)_2$, zinc acetate-dihydrate($ZnC_4H_6O_4$), ammonia solution, and ethanol (C_2H_5OH,	hydrothermal technique	methylene blue and methyl orange	[170]
Mg and La	$Zn(CH_3COO)_2.2H_2O$, $La(NO_3)_3.6H_2O$, $Mg(NO_3)_2.6H_2O$	Sol-gel	Rhodamine B	[96]
Fe2O3	Zinc nitrate hex-hydrate Zn (NO_3). $6H_2O$ Iron (III) nitrate Fe $(NO_3)_3.9H_2O$ NaOH	hydrothermal technique	Methyl blue	[97]
In	Zn $(NO_3)_2.6H_2O$ NaOH	hydrothermal technique	Methylene blue (MB) and methyl orange (MO)	[98]
Samarium	zinc acetate dihydrate and sodium hydroxide	hydrothermal technique	Methylene blue (MB)	[99]
Sr	ZnO powder Sr carbonate powder ($SrCO_3$) Stearic acid (CH_3 $(CH_2)_{16}COOH$)	-	Methylene blue (MB)	[100]

2.3 UV protection

This review focused on the main application of zinc oxide nanoparticles for UV protection in the textile industry. There are various studies where only ZnO nanostructures were used without modifications. In some studies, ZnO nanoparticles were purchased without synthesizing. *G.Broasca* and his research group in 2013 prepared an inorganic-organic material by incorporating purchased ZnO microparticles into polyester textiles. Woven polyester fabrics were prepared to 5 cm $^{\times}$10 cm strips and ZnO powder was used to prepare dispersions in methanol (99.8%) at 1%, 3%, 5%, and 7% concentrations. The solutions prepared were stirred for 15 minutes and 4 drops of vitexol and 80 g/l apretan were added to avoid foaming and to ensure bonding respectively [101]. Although *G. Broasca* purchased ZnO powders, *W. Sricharussin* and his research group in 2007 prepared ZnO nanoparticles in three shapes as ZnO multi petals [102], ZnO rods, and ZnO spherical particles and were coated onto cotton fabrics using pad dry cure process. Zinc acetate, β – CD (it forms H bonds with ZnO in different planes), and NaOH were used as precursor reagents for the synthesis of multi petal ZnO nanoparticles [103]. For the synthesis of ZnO, nanorods ammonia was used as a reagent while ethanol was used for the preparation of spherical nanoparticles. In 2020 ZnO nanostructures with different morphologies were synthesized using the liquid precipitation method by M.A.Mousa and M.Khaity. They varied experimental media like water, methanol, and ethylene glycol which resulted in star, rods, and sphere-shaped nanostructures respectively [104].

The researcher Katja Jazbec in 2015 presented an environmentally friendly solution to achieve excellent UV protection properties of cotton fabric by chemically modifying the textile substrate using low-pressure oxygen plasma created by an electrodeless radiofrequency. In this study before coating ZnO nanoparticles on cotton fabrics, they were treated in low oxygen pressure inductively coupled radiofrequency plasma system for different periods [105].

As previously mentioned preparation of stable ZnO dispersions that last a long period on textile surfaces was a challenge for the textile industry. This challenge was successfully overcome by using inorganic-organic hybrid polymers which were based on 3-glycidyloxy propyl trimethoxy silane (GPTMS). These polymers filled with ZnO nanoparticles were applied on cellulosic cotton (100%) and cotton/polyester (65/35%). The effectiveness of finished products was evaluated by UV-Vis spectroscopy and ultraviolet protection factor. In the synthesis of ZnO nanoparticles, zinc acetate and isopropanol were used as precursors and lithium hydroxide was used for the hydrolysis of the precursors. Moreover, prepared ZnO and GPTMS sols were mixed in different ratios and 1- methyl imidazole (0.5 ml/10 ml GPTMS) was used to catalyst the cross-linking reaction of the epoxy group of the GPTMS. The formulations obtained after the preparation process were applied on fabrics using a laboratory padder to a wet pick-up of 100% [19]. Although ZnO nanoparticles are effective UV blocking agents their activity is hindered by their inherent photocatalytic activity. This issue is addressed by Rosalie Hocking in his study in 2012 by modifying the surface of ZnO by impurity doping such as SiO_2, cobalt, and manganese [106]. Xue et al., in 2011 reduced the photocatalytic activity of ZnO nanoparticles by coating them with SiO_2 shells. Here successive deposition of polyelectrolytes was followed by SiO_2 to cover the ZnO completely [107].

The green synthesis of nanostructures is a much more interesting topic among researchers due to the less impact on the environment. In 2021 David Asmal-Campos and his research group synthesized ZnO nanoparticles using both chemical and green methods to evaluate the UV protection activity of different types of textiles. In the green synthesis, Coriandrum sativum extract was used as a reducing agent where NaOH was used as the reducing agent in the chemical method [108].

2.4 Antibacterial activity

2.4.1 Pad dry cure coating method

The effectiveness of pad dry cure method was more than the spin coating method when used to enhance the antibacterial property in textiles [173]. Previously research was done to ascertain the high effectiveness of ZnO nanostructures over their bulk counterpart. Rajendra et al (2010) incorporated ZnO-NPs to 100% cotton fabric using the pad dry cure method. For the synthesis of nanoparticles zinc nitrate and sodium hydroxide were used as precursors and soluble starch in different concentrations was used as the stabilizing agent. First, soluble starch was dissolved in 500ml of distilled water in different concentrations (0.1%, 0.5%, and 1.0%) by using a microwave oven. Then 14.874 g of zinc nitrate was dissolved in the previously prepared solution. After complete dissolution of zinc nitrate, 0.2M sodium hydroxide was added under constant stirring drop by drop touching the walls of the container. Then the prepared mixture was kept overnight to complete the reaction and the supernatant solution was discarded. Thereafter, the remaining solution was centrifuged at 10,000 rpm for 10 minutes and the supernatant was discarded and synthesized nanoparticles were washed several times to remove impurities (mainly excess starch bound with nanoparticles) following dried at 80 °C for overnight. Then synthesized ZnO-NPs were coated on fabric using the pad dry cure method. In there the cotton fabric was cut to 30 x 30 cm pieces and was immersed in a solution containing ZnO nanoparticles. The assessment of antibacterial activity was performed using the agar diffusion method, parallel streak method, percentage reduction tests, etc. After, the topographical analysis of the fabric was done using a scanning electron microscope. Also, the wash durability of fabric was tested by washing using neutral soup at 40 °C [37].

Although Rajendra et al., (2010) used the simple pad dry cure method to fabricate ZnO nanostructures on textiles. Tania and Ali (2021) have produced anti-microbial fabrics by treating the normal fabrics with ZnO-NPs following the pad-dry-cure method with and without an acrylic-based binder. In this study, synthesis of ZnO-NPs (65 nm) was performed with stirring by mixing the stock solution of 0.2 M $ZnSO_4$ (pH 5.3) and 4.0 M NaOH (pH 13.8). The final mixture was fixed at pH 13 because it is suitable for the direct preparation of wurtzite-type ZnO crystals [109, 110]. The antimicrobial property of treated samples is determined by ASTM E2149-01, 2001 method [111].

Eskani et al (2020) treated fabrics (batik) with ZnO-NPs following pad-dry-cure method to investigate the effect of ZnO concentration (1% and 2%), temperature (25°C and 80°C), and order of the ZnO-NPs application (before and after the batik process). Antibacterial activity of the treated fabrics was evaluated against gram-positive bacteria *Staphylococcus aureus* and gram-negative bacteria *Escherichia coli* using the agar diffusion method [112].

The effect of size of ZnO nanoparticles, fabricating method, loading percentage, and calcination temperature of ZnO nanoparticles for the effectiveness of antibacterial activity of ZnO fabricated textiles have been studied by researchers. The effect of loading percentage of ZnO nanoparticles was studied by Dural Erem et al in 2011. Dural Erem et al (2011) prepared ZnO loaded Polyamide PA6nanocomposites and their antibacterial activity has been investigated against *Staphylococcus*

aureus (gram-positive bacterium) and *Klebsiella pneumonia* (gram-negative bacterium). PA6/ZnO nanocomposites have been produced by mixing polymer granules with ZnO nanoparticles in a 15 ml micro compounder. ZnO nanoparticles have mixed in different weight percentages as 0, 0.5, 1, 3, 5 % and particle effect on polymer matrix has identified using thermal and mechanical tests (ASTM E1131 – 08, ASTM D7426-08, ASTM D3822-07) [40].

2.4.2 In situ coating methods

The fiber can be coated using different methods such as the pad dry cure method, ultrasonic irradiation, and sol-gel method. Previously, the simple pad dry cure method was used by the researchers, and later various in situ techniques were invented to coat nanoparticles on fabric surfaces. ZnO-NPs have incorporated into 100% cotton fabric using one-step in situ sol-gel syntheses by d'Água et al (2018). This method allows the coating of ZnO at a very low temperature (50 °C). Therefore, this eliminates the wastage of chemical reagents, energy, and time. Antimicrobial activity of bacteria which tend to cause nosocomial infections, community-associated skin infections, and bacteria responsible for unpleasant odors in textiles has been tested. For the synthesis, 1M NaOH solution has introduced at a constant rate, to 0.08 M Zn $(CH_3COO)_2.2H_2O$ solution with 4x4 cm^2 cotton fabric sample, at 50 °C was stirred at 800 rpm. Finally, the cotton fabric has dried. *Escherichia coli* and *Pseudomonas aeruginosa* were used as Gram-negative bacteria and *Staphylococcus aureus*, *Enterococcus faecalis*, *Staphylococcus epidermidis*, and *Propionibacterium acnes* were used as Gram-positive bacteria. The agar diffusion method [113] was used to analyze the antibacterial activity [29].

Shaban et al (2018) have successfully used the sol-gel method to coat cotton fiber with nano zinc oxide. The significance of this study is due to the creation of fabric with both antibacterial and superhydrophobic surfaces. First, the cotton fabric was washed with acetone and ethanol to remove impurities under ultrasonication at 80 °C for 10 minutes. Zinc acetate dehydrates and methoxy ethanol was used as precursors while monoethanolamine was used as the stabilizer. Also, the pH of the prepared solution was adjusted by adding sodium hydroxide or acetic acid. Magnesium acetate tetrahydrate was used as the dopant material. The prepared ZnO nanoparticles by the sol-gel method were spin-coated at the rate of 1100 rpm for 60 seconds [32].

MahmoudiAlashti et al (2021) modified a cotton fabric for antibacterial activity by deposition of ZnO-NPs using in situ method. They used zinc acetate dihydrate as precursors and sodium hydroxide, with and without starch as a capping agent. For the experiment, first, the cotton fabric (2.5×2.5cm) was immersed in 0.01 M NaOH at 90°C for 1 hour and then washed with distilled water. To deposit nanoparticles, the cotton fabric was immersed in the precursors of ZnO-NPs with and without starch at 90°C for 2 hours and then washed with distilled water followed by drying at 37°C. Re-prepare the fabric was not required in this method. The antimicrobial activity of the modified fabric was determined using agar diffusion and the absorption method against *E. coli* (ATCC 8739) at 37 °C for up to 24 h (ISO 20645:2004) [114].

In this study, 13 treads/ cm^2 density cotton bandage (10 ×10 cm, 0.7 g) was added to a solution containing 1 mM $Zn(Ac)_2.H_2O$ in ethanol: water (10:1) in a 100 ml sonication flask and NH_3XH_2O was used to adjust the pH of the solution. Then the reaction mixture was irradiated for 30 minutes in 20 kHz, 750 W at 70% efficiency under a flow of argon. The sonication flask was kept at 30 °C during the reaction. Here the color of the fabric was not changed and the fabric was thoroughly washed with water and ethanol to remove traces of ammonia followed by vacuum dry. The antibacterial test was performed against *Staphylococcus aureus* and *Escherichia coli*
[38].

In another study (Shaheen et al., 2016), the antimicrobial property of ZnO-NPs coated textiles has studied against *Staphylococcus* aureus and *Escherichia coli*. ZnO-NPs were synthesized using hexamethyltriethylene (HMTETA) and instantaneously coated onto the cotton fabric by immersing in a mixture of amine and Zn $(NO_3)_2$. Then synthesized zinc hydroxide on fabric was converted to ZnO-NPs by water evaporation. The HMTETA facilitated an alkaline medium for the conversion of $Zn(OH)_2$ to ZnO-NPs. HMTETA also acted as a stabilizing agent during the synthesis process of ZnO-NPs [115].

Lai et al (2021) prepared an antimicrobial superhydrophobic fabric and their anti-microbial property was investigated against *Escherichia coli* and *Staphylococcus aureus*. In this study, 0.05 g of ZnO was dispersed in 100 ml ethanol and then 3.5 g of polydimethylsiloxane (PDMS) was added to the mixture. A clean, Ar plasma-activated polyester fabric was then immersed in the above-prepared mixture at ambient temperature and then heated in an oven at 70 °C for 10 min to uniformly cover the fibers by a ZnO-PDMS layer [116].

Pintarić et al (2020) modified cellulose materials with ZnO-NPs by dip-coating methodology using 3-glycidyloxypropyltrimethoxysilane and bactericidal and fungicidal activity of treated fabric were determined by the disc diffusion method, as well as growth inhibition studies against both Gram-positive (*Staphylococcus aureus*) and Gram-negative (*Escherichia coli*) bacteria. The results revealed that the amount of the ZnO-NPs on fabric depends on the concentration of the reagents, ultrasound irradiation power, and the time of the sonication. Furthermore, for obtaining a homogenous,

antimicrobial, and effective surface, ultrasonic irradiation is a key parameter[117]. Javed et al (2021) treated textiles to obtain antimicrobial property by in situ growth of pure hexagonal wurtzite crystalline ZnO-NPs on 100 % cotton fabric (with aerial density 145 g/m^2,27 ends/inch and 23 picks/inch). They used $Zn(NO_3)_2{\cdot}6H_2O$ and NaOH as precursors, which were aided by ultrasonic acoustic waves. Following is a suggested mechanism for the formation of ZnO-NPs on textile.

$$Zn(NO)_3.6\,H_2O \rightarrow Zn^{2+} + 2NO_3^- + H_2O \quad (1)$$

$$NaOH \rightarrow Na^+ + OH^- \quad (2)$$

$$Zn^{2+} + 4OH^- \rightarrow Zn(OH)_4^{2-} \quad (3)$$

$$Zn(OH)_4^{2-} \rightarrow ZnO + H_2O + 2OH^- \quad (4)$$

The antibacterial efficiency was examined against Gram-negative *E. coli* and Gram-positive S. aureus following the colony count method (AATCC 100-2012) [118].

Souza et al (2018) grew ZnO-NPs on textile by the sonochemical process [119] using ($Zn(NO_3)_2.6H_2O$) and KOH as precursors and observed a strong antibacterial activity of the treated fabrics following the Japanese Industrial Standard (JIS Z 2801: 2010) against *Staphylococcus aureus* (ATCC 25923) and *Pseudomonas aeruginosa* (ATCC 27853). The halo size of treated cotton fabrics after 0 h, 1 h, and 2 h of reaction were 1.2 ± 1.0 mm, 2.5 ± 0.8 mm, and 2.1 ± 0.2 mm against *S. aureus* while 0.8 ± 0.4 mm, 2.5 ± 0.5 mm, and 2.4 ± 0.2 mm against *P. aeruginosa*, respectively [120]. Textiles that were previously carboxymethylated and then treated with titanium isopropoxide (7%), citric acid (4%), and ZnNPs size between (14-19 nm), showed a good antibacterial activity [121] against both Gram-positive and Gram-negative bacteria.

Mousa and Khairy (2020) treated different fabrics of cotton, polyester, and 50/50 wt% polyester/cotton with ZnO-NPs to acquire antibacterial properties. ZnO-NPs were synthesized with the sol-gel method using Zn acetate dihydrate and NaOH (122). The fabrics were coated with ZnO-NPs following a simple dip-cure method and antimicrobial properties were determined against Gram-negative bacteria (*Escherichia coli*), Gram-positive bacteria (*Staphylococcus aureus*), and diploid fungus (*Candida albicans*)(123). Furthermore, ZnO-NPs have produced as in situ on textiles by pulsed laser ablation (PLA) method (fundamental wavelength of pulsed Nd: YAG laser of 1064 nm and 7 ns pulse width in deionized water) by placing the fabric sample near the Zn target in the ablation container [124].

Many metal oxide nanoparticles such as silver, copper, gold, ferric, and palladium have synthesized using green synthesis methods, with excellent physical, chemical, and structural properties. Moreover, these synthesis methods did not require any harmful chemicals as reducing or oxidizing agents because only plant extracts are used in the synthesis. Also, it produces nontoxic, eco-friendly, clean nanoparticles for large-scale production.

2.4.3 Green synthesized ZnO nanostructures for antibacterial property in textiles

Karthik et al (2017) have been synthesized ZnO-NPs using zinc acetate as the precursor and leaf extract of Acalyphaindica as a chelating and capping agent. The chelating and capping ability of Acalyphaindicawas is due to a chemical component in the leaf extract named "polyol". It has stabilized the formation of nanoparticles. The Acalyphaindicahas many uses in day today life such as curing diseases, wound healing in pets, etc. In this study, fresh, cleaned leaves of Acalyphaindicahave shade dried for 15 days and a leaf extract has prepared by stirring 2 g of dried boll milled leaf for 2 hours at a constant temperature of 70 °C. Finally, the mixture has cooled to room temperature and filtered. For the synthesis of nanoparticles, the prepared 50 ml of leaf extract has added to 0.5 g of 1M zinc acetate under stirring for 1 hour at 60 °C. Then obtained precipitate has washed with double distilled water and finally, heated in a hot air oven at 80 °C for 2 hours. ZnO nanoparticles have been obtained after calcination temperatures at 100, 300, and 600 °C. Ultimately the prepared ZnO nanoparticles have dissolved in a prepared chitosan solution and have coated to fabric using the pad dry cure method. The prepared ZnO nanoparticles at three different calcination temperatures were characterized using X-ray diffraction, particle size analyzer, scanning electron microscope [30].

The green synthesis of nanoparticles is more preferred because it does not result in toxic substances for the ecosystem. Fouda et al (2018) synthesized ZnO-NPs using a bioactive molecule secreted by Aspergillus terreus and treated them onto cotton fabrics at a safe dose to gain anti-microbial properties with textiles. In this study, the *Aspergillus terreus* was grown up

in a 250 ml Erlenmeyer flask containing 100 ml CzapekDox (fermentive broth medium) at pH 6.0, 28 °C temperature and shaking 150 rpm for 36 hours. Thereafter, 15 mg of fungal biomass was collected from incubated fungus and it was separated using Whatman filter paper and washed with sterile distilled water. Then, the collected biomass was suspended in 100 ml sterile distilled water in an orbital shaker at 150 rpm, at 28 °C temperature for 48 hours. The cell-free filtrate was mixed with different ratios of zinc acetate and incubated at 28 °C on an orbital shaker (150 rpm) for 24 hours to synthesize ZnO-NPs. The particle size of the nanoparticles was determined by an incubation period, zinc acetate concentration, and pH of the medium (Fouda et al., 2018).

El-Naggar et al (2018) studied the antibacterial property of cotton fabrics that were treated with ZnO-NPs. In this study, a bio extract of date seeds was used as a stabilizing agent in the synthesis of ZnO-NPs through the in-situ formation from zinc acetate and sodium hydroxide precursors. Treatment of cotton fabric was done according to the pad dry method and conversion of zinc hydroxide was done during the curing step[31].

The antimicrobial textiles produced must be durable or resist washing. There are several methods to pre-treat fabric, such as chemical and plasma activation which need harsh processing conditions. Petkova et al (2016) used enzymes as an activation tool for textile surface which needs mild processing conditions. This enzymatic pre-treatment improves the adhesion between ZnO-NPs and fabric surface and ensures the anti-microbial characteristics of fabric even after few washing steps too. In this study, the zinc oxide particles were generated via the in-situ method using zinc acetate in water or ethanol alkaline solutions and they were deposited on fabric using ultrasound irradiation[35].

2.4.4 ZnO nanocomposites for antibacterial activity of textiles

Although, many researchers have used ZnO-NPs to improve the antibacterial properties of textiles, the usage of nanocomposites of zinc oxide[174] nanostructures for that purpose is rare. Barani (2014b) has used ZnO/SiO_2 hybrid nanocomposites to coat fabric surfaces. ZnO-NPs were synthesized on cotton fabric using two different sol-gel methods. Initially, the sole solution was prepared with tetraethyl orthosilicate (TEOS), ethanol, water, and ammonia. TEOS was used as the precursor. The dip-coating method was used to prepare the sol by stirring a mixture of 3 ml TEOS, 60 ml ethanol, 8.7 ml water, and 31.2 ml ammonia for 4 hours at 25 °C. In the first method (A), the fabric sample was immersed in the sol solution while in the second method (B) zinc acetate was added to the sol solution. Although a higher amount of Zn^{2+} was bound to cotton fabric due to surface activation by method A, it resulted in nanoparticle agglomeration. However, that issue was overcome by method B as it contained a lower amount of Zn and a higher amount of Si comparatively. The broad and lower intense peaks from the XRD pattern confirmed the silicon network formed between zinc oxide nanoparticles. According to the results, there was a large inhibition zone for *Staphylococcus aureus* and *Escherichia coli* from method B when compared with method A(125).[Figure 1]

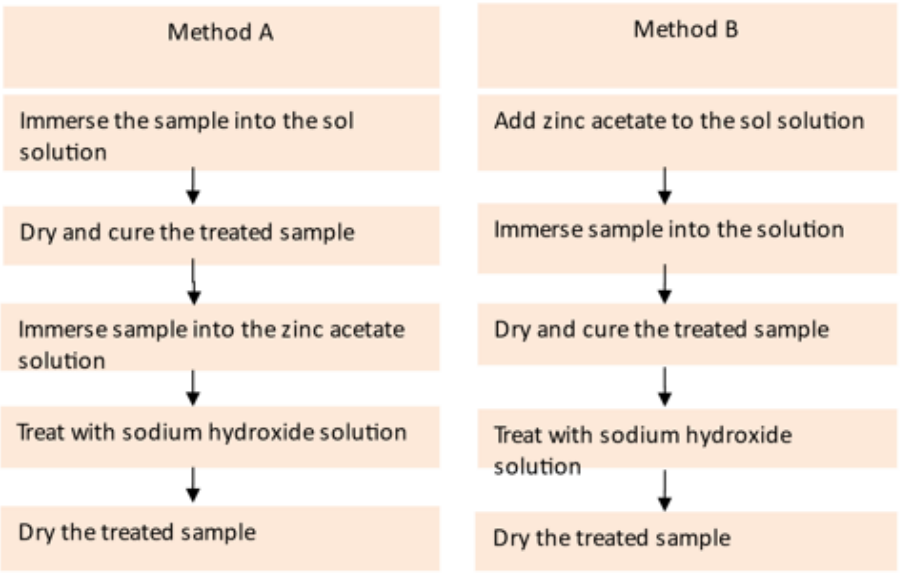

Fig 1. A & B methods of fabricating ZnO nanostructures, Barani (2014)

Barani (2014a) studied the antibacterial properties of cotton fabric treated with ZnO/Ag nanocomposites and compared them with un-activated cotton fibers. However, nanoparticles were tended to agglomerate when un-activated fibers are treated. Therefore, in this study, silver nanoparticles have been successfully used to activate cotton fibers before loading zinc oxide nanoparticles on the fabric surface. Further, the effect of silver nanoparticle activation and synthesis temperature of ZnO-NPs were studied. There are two different methods to make immobilize nanoparticles on fabric surfaces such as coating pre-synthesized nanoparticles on fabric surfaces and in situ synthesis of nanoparticles on fabric surfaces. First selected cotton fabrics were scoured with Triton which is a nonionic surfactant and it was rinsed with tap water and immersed in acetone for 15 minutes. Thereafter, those samples were dried and immersed into a sensation solution ($SnCl_2$, HCl) at 25 °C for 30 minutes. At this activation step, the sample was introduced to a solution of $AgNO_3$ and NH_3 at 25 °C for 30 minutes. Here tin chloride was used as a reducing agent for the synthesis of zinc oxide nanoparticles. After, the cotton fiber was introduced to a solution containing $(CH_3COO)_2Zn$, $NaPO_2H_2$, and $C_6H_8O_7$ and treated at 25 °C for 30 minutes. Then, the sample was introduced to a NaOH solution at 25 °C for 45 minutes. At last, the sample was rinsed with distilled water. The sample was activated at two temperatures as 25 °C and 70 °C. The sample activated at 70 °C was covered with a dense layer of ZnO than other samples [126].

3 Results

3.1 Dye removal efficiency Factors

- Size of ZnO nanostructures

Before utilizing ZnO nanostructures, in the early 2000s scientists used ZnO suspensions to remove dye from effluents. For instance, in 2004 H.T. Yatmaz used ZnO suspensions and waste water was reacted in a slurry batch reactor to remove Razamol red textile dye. However, in the late 2000s, the application of nano-photocatalysts was immerged resulting in high removing efficiency due to the minimum band gap of small nanoparticles than that of bulk particles. When the sizes of nanoparticles are reduced it decreases the band gap of ZnO due to resulted strong quantum confinements. Ultimately, the surface area to volume ratio of nanoparticles increased. The efficiency of dye removal percentage of ZnO nanostructures was affected by calcined temperatures and calcined temperature of ZnO nanostructures were in the order of 400 °C > 500 °C > 600 °C > 300 °C. The reason for having a high removing efficiency of 99.70% of methyl orange, was the smallest sized 22.56 nm ZnO particles obtained under the calcination temperature of 400 °C [14]. In 2014, 50 ml egg white and 18.5 g $Zn(NO_3)_26H_2O$ were successfully used for the synthesis of ZnO nanostructures. However, in 2020 ratios of egg white to zinc nitrate were varied as 1:1, 2:1, and 3:1, and ZnO structures with 48, 42, and 29 nm have obtained respectively. According to the results, it concludes that the nucleation, growth, and morphology of ZnO were controlled by egg white concentration used for the synthesis. In all studies, the smallest-sized ZnO nanoparticle had the highest removing efficiency. In contrast, this study 42 nm particle had better results than 29 nm particle [72].

- Catalyst and initial dye concentration

The amounts of active sites formed are increased with the increment of catalysts concentration which leads to forms higher amounts of radicals to react with dye molecules. The dye removing efficiency of textile dyes was increased with the loading percentage of the ZnO nano-catalysts [127]. For instance when ZnO nanoparticle concentrations were increased from 0.1 g/L to 0.8 g/L the dye removing efficiency of methyl orange, congo red and direct black 38 were increased from 68.00, 56.49, and 49.25% to 99.70, 99.21, and 99.45%, respectively [14]. Further, in 2014 complete degradation of MB and MG with 10 g/L has resulted within 50 minutes in presence of 0.05g ZnO nanoparticles [8].

Moreover, the dye removing efficiency was inversely proportional to the initial dye concentration. The photocatalytic performance of biosynthesized ZnO NPs using jujube fruit was investigated under direct sunlight for the degradation of 100 ppm Methylene Blue (MB) and Eriochrome Black-T (ECBT) using 15 g ZnO nanoparticles. Dye degradation efficiencies of ZnO NPs on MB and ECBT were 92% and 86% within 5 h, respectively [83]. Kazeminezhad et al. [84] reported on the catalytic activity of ZnO NPs in the degradation of ECBT dye at various conditions. When the initial dye concentration is 20 ppm, the dye removal efficiency was maximum (85%). Davar et al. [128] achieved almost 100% methylene blue degradation using a novel ZnO photocatalyst, In which, initial dye concentration was lower (5 ppm) as well as catalyst loading was high.

- pH

Furthermore, the dye degradation rate is increased with increasing catalyst loading [129] as well as with increasing the pH of the solution [4, 84]. The dye removing efficiency is increased with the pH, because it facilitates the reaction between holes in the

valance band and hydroxyl groups, resulting in a high concentration of hydroxyl radicals in the reaction medium, According to the study done by Kazeminezhad and Sadollahkhani the highest photodegradation rate of Eriochrome black – T dye was reported at pH 11 than other pH values [84].

Amongst synthesis methods of ZnO nanostructures, green synthesis occupied a special place because of its environmentally friendly nature. The researchers who used many chemical species for nanoparticle synthesized have replaced them with green materials which can be found easily in the environment. Furthermore, scientists used various plant extracts for the green synthesis of ZnO nanostructures. Table (01) summarizes different types of bio extract, precursor chemicals used, features of nanostructures and relevant references. [Table 2]. Karnan and Selvakumarin in 2016 used the "Rambutan" peel for the synthesis of ZnO nanostructures and its effectiveness of photocatalytic activity has been tested using methyl orange (MO). According to the results, 83.99% of decoloration efficiency was recorded for MO within 120 minutes that confirmed by the diminished absorbance at the wavelength of maximum absorbance (464 nm) for the dye. [76]. Similarly, [83] used ZnO nanostructures synthesized from Capparis zeylanica also loaded to 150 ml of methylene blue solution were kept in dark to reach adsorption-desorption equilibrium for 30 minutes. Also, an absorption spectrum peak at 665 nm was used to study extend of photodegradation of MB using ZnO nanostructures[78]. Siripireddy and Mandal, 2017 used the plant Eucalyptus globules as a capping agent in the synthesis of ZnO nanostructures and their efficiency in photocatalytic activity has been tested using methylene blue and methyl orange dyes. Ultimately, the color of MB and MO has disappeared within 50 minutes and 60 minutes respectively, which was further confirmed by the diminished absorbance values at the wavelength of maximum UV absorbance for MB (665 nm) and MO (460 nm)within the above time interval [77]. Barzinjy and Azeez in 2020 prepared ZnO nanostructures using the same plant Eucalyptus globules and precursor with some differences in the synthesizing method. Thus, the temperature of the plant extract was maintained at 60 °C until the solution converted to a yellow paste, while in previous research there were no concerns about the temperature of the extract. Furthermore, according to the results, the synthesized ZnO nanoparticles were thermally stable and able to absorb both dyes and heavy metals in the aqueous solution [85].

Table 2. Different types of bio extract, precursor chemicals used features of nanostructures and relevant reference.

Bio extract	precursor Chemicals used	Size/ shape of ZnO nanoparticles	References
Capparis zeylanica	Zinc acetate dehydrates	Spherical with sizes range from 32 to 40 nm.	[78]
Musa acuminata peel extract (Banana peel)	Zinc acetate dehydrates	15.3 nm crystallite size	[79]
Eucalptus globules	Zinc nitrate hexahydrate	spherical-shape 27 and 35 nm.	[85]
	Zn $(NO_3)_3$. $6H_2O$	spherical zinc oxide nanoparticles mean particle size of 11.6 nm	[77]
Nephelium lappaceum	Zinc nitrate hexahydrate	25-40 nm	[76]
Vitex trifolia	30 nm	spherical in Shape however some of the particles are hexagonal. The average size of particles is 28 nm	[73]
extract of jujube fruit	$Zn(NO_3)_2.6H_2O$	Mean size of 29 $\pm$ 8 nm.	[81]
Garcinia mangostana (fruit pericarp)	Zinc acetate dehydrates	Mostly spherical, average size 21 nm	[82]

In some researches, photocatalytic activity has been studied under UV irradiation [73, 85]. However, the study done by Anbuvannan et al in 2015 has used visible light irradiation for the degradation of MB dye in the textile effluent. It was reported that ZnO nanostructures synthesized using Phyllanthusniruri plant leaves showed good photocatalytic activity, where the UV peak (664 nm) of MB was disappeared within 30 minutes of irradiation [75]. In another study that used leaf extract of Vitextrifolia for the synthesis of ZnO nanostructures, were performed photocatalytic activity against MB under UV irradiation for different time intervals. The total decolorization was recorded from 90 minutes and new peaks in the UV spectra were not detected proving that there were no formations of intermediate products [73].

3.2 Doped ZnO nanostructures for dye removal

To further increase the efficiency of textile dye removal from the effluent researchers incorporated doped materials into ZnO nanoparticles. The bandgap of pure ZnO nanoparticle is 3.36 eV, this bandgap was successfully reduced using various doped

materials. The dosages required for the removal of effluent dyes were smaller than that of pure ZnO nanoparticles. Before doping the minimum dosage required for efficient removal of dye was about 0.8 g/L. However, scientists could minimize the amount of ZnO nano-catalysts after doping with other materials. According to author Nithya et al., 2020, the effectiveness can be further enhanced by loading Mn-doped ZnO with cotton stalk activated carbon (CSAC) at a catalytic dose of 0.20 g/L. The photocatalytic activity of Mn-doped ZnO/ CSAC increased because of the reduction of particle size, deduction of the bandgap, high surface area, and high pore volume [92]. According to the results, the photocatalytic activity of aluminum-doped ZnO nanorods was higher than cerium-doped ZnO nanorods. The author Del Gobbo concluded that photocatalytic activity becomes more efficient with heteroatoms smaller than zinc, such as aluminum, rather than with larger atoms such as cerium [93] Rodwihok et al., in 2020 studied the photocatalytic activity of only cerium-doped ZnO nanoparticles by doping different percentages of Ce into ZnO nanoparticles. In this case, the formed bi–metal heterojunction reduces the recombination of electrons and increases the photocatalytic activity of ZnO (pure) from 69.42% to 94.06% under 60 min under fluorescent lamp illumination [94]. Photodegradation of methylene blue under UV irradiation has been studied using Cobalt (Co) loaded ZnO nanostructures in different concentrations. The rate of degradation increased as 67,72, 78, and 80%, with 0, 3, 5, and 7 mol% Coloading respectively.

Shah et al., 2020 used copper (Cu) doped ZnO nanorods to study the efficiency of photodegradation in methylene blue and methyl orange. They suggested that the shape of ZnO nanorods does not alter even after Cu doping. However, it had taken 180 minutes to achieve the degradation of 57.5% and 60% for methyl orange and methyl blue respectively. Khanizadeh et al., in 2020 synthesized ZnO nanoparticles from co-doped Mg and Co using the sol-gel method. They presented that the Mg 5% - La 5%/ZnO as the most suitable ratio (91.18 % efficiency) [96].

Although most of the studies were based on single element doping with ZnO nanostructures, Rahmah et al., in 2020 doped Fe_2O_3 to study the photocatalytic activity of doped ZnO under visible light irradiation. They found that when Fe_2O_3 doped with ZnO nanoparticles it forms a metal oxide nanoparticle heterojunction barrier that facilitates the reduction of recombination rate by transferring photogenerated electrons of Fe_2O_3 in the conduction band to the ZnO conduction band. The availability of more surface charge carriers on Fe_2O_3-doped ZnO nanostructures; unpaired photogenerated electrons in the conduction band and holes produce superoxide anion radicals and hydroxyl radicals (react with water) respectively. Moreover, results showed significantly enhanced degradation percentages of 80.6, 83.8, 85.5, and 92% for doped ZnO, respectively, as compared to 73.8% for undoped ZnO [97]. Photocatalytic activity of Indium doped ZnO was studied by Yu et al., in 2020. According to their results of Indium doped acts as an effective catalyst to remove methylene blue and methyl blue from water effluents. Moreover, the efficiency of removing methylene blue (96.84%) was higher than methyl blue (90.05%) [98].

3.3 UV Protection

3.3.1 Shape of ZnO structures

In most of the research, spherical shaped nanostructures had high efficiency of UV protection than other shapes. The UV transmission spectra, untreated cotton fabric indicated transmission around 100% while spherical shaped ZnO coated fabric showed the lowest transmission which indicated the best UV blocking property for the cotton fabric. However, ZnO nanorods were less efficient in UV blocking properties than the other two shapes of ZnO nanoparticles [103]. In 2021 it was found that nanostructures (97.77 nm, spherical shaped) obtained from the green method were more efficient in UV blocking property than chemically synthesized ZnO structures (113 nm). According to this study, green synthesized spherical nanoparticles highly adhered to textiles with high cotton content [108]. Although Sricharussin in their study reported spherical ZnO nanostructures as the best UV blocking structure, according to the results of the M.A.Mousa study the UPF value of treated cotton fabrics with rod shapes nanostructures obtained the highest value [104].

3.3.2 Loading percentage

According to the study done by Broasca SEM images and reflectance percentage, the optimum level of ZnO loading was identified as 3-5% which resulted in good homogeneity and dispersion of ZnO microparticles in the textile surface [101]. In the study done by Jazbec and his research group the cotton, samples were fabricated with 3% ZnO nanoparticles and results showed that the longer plasma treatment caused the higher adsorption of ZnO nanoparticles to the cotton substrate due to increment of oxygen-containing which enhanced the surface roughness of the textile surface. Jazbec et al., 2015 mentioned that the UV absorbing activity of coated fabric was at a broad range in the region from 225 nm to 380 nm and the UV blocking properties were unchangeable after 60 minutes of washing in 33 liters of water [130]. Moreover, a higher concentration of nano ZnO caused turbidity and stability for nearly 8 hours while the lower concentration of ZnO was stable for about 12 to 13 hours. Moreover, the sol-gel finishing of fabrics was able to increase the absorbance of UV light from the fabrics when compared with untreated fabrics. Therefore, according to researchers this hybrid polymer modification of textiles is a promising approach for

the development of UV protecting textiles [19].

3.3.3. Washing durability and mechanical properties

According to the study done by Broasca mechanical properties were unchanged with 3% to 5 % loading percentage of ZnO nanostructures [101]. Moreover, in the research done by Mousa in 2020, there was no significant difference between tensile and elongation values were found compared with untreated cotton fabrics [104].

Sricharussin in his study mentioned that there was a decrease in adhesion of ZnO nanoparticles after 10 washing cycles[103]. However, in 2021 the treated fabrics with cellulose nanofibrils exhibited a UPF higher than 50 even after 30 standard washing cycles and the permeability of cotton fabrics was decreased 30% than that of untreated fabric [131].

3.3.4 Reduction of photocatalytic activity

According to obtained results, an effective reduction of photocatalytic activity of ZnO nanoparticles was obtained by SiO_2 coated ZnO samples compared to uncoated samples. Moreover, cobalt was less efficient than SiO_2 however, efficient than manganese which resulted in lower results [106]. The fading of textile dyes can be considered as a drawback of the photocatalytic activity of ZnO when used as a UV absorber. The chromophores differently interact with reactive oxygen species produced by ZnO nanostructures. These were studies using benzopyran, anthraquinone, and azo chromophores and according to results anthraquinone had the highest fading rate [132].

3.4 Antibacterial activity

Furthermore, this study demonstrated a higher antibacterial activity of treated fabrics against *S.aureus* and *E.coli* and, the effectiveness of using ZnO-NPs rather than bulk ZnO. Moreover, wash durability was increased using a high concentration of ZnO-NPs. The SEM analysis confirmed the entrapment of ZnO nanoparticles in the fabric [37]. The results revealed that the coated fabrics with acrylic-based binders have high anti-bacterial activity than treated fabrics without binders. Further, it provides a bacterial reduction of 92% against *Staphylococcus aureus* and 86% against *Escherichia coli* [111].

The inhibition area against *Staphylococcus aureus*-coated fabric is approximately 60% of the inhibition zone of the antibacterial drug (chloramphenicol), slightly greater than the inhibition zone against E. coli bacteria. To maintain the colorfastness to washing, the ZnO should be applied to the fabric before the batik process as ZnO nanoparticles increase the dye affinity towards the fabrics. However, in the order of ZnO-NPs treatment (before and after the batik process), the difference in ZnO-NPs concentration and temperature does not significantly affect the antibacterial activity [112].

Overall, ZnO nanoparticles had not made any significant influence on the tensile and thermal properties of the nanocomposites. The antibacterial effect of PA6 nanocomposites against *Staphylococcus aureus* and *Klebsiella pneumonia* was effective at the loading percentage of 5% wt. Moreover, ZnO nanoparticles were homogeneously dispersed in the PA6 matrix [40]. In the study done by Agua, microbial activity has tested using hydrogen peroxide, and results obtained from that were the same as results obtained from ZnO-NPs. Furthermore, this study hypothesized hydrogen peroxide as a reactive oxygen species responsible for the antimicrobial property of zinc oxide nanoparticles [2].

This study presented a fabric that exhibits both antibacterial activities against different species of bacteria; especially *K.pneumonia* and superhydrophobic water contact angle (154 °) at the optimum conditions [32]. The results showed that both the samples treated with and without starch showed a good antimicrobial activity [114] ensuring the same results of in situ synthesis and sol-gel method [133] and using the pad-dry-cure method [42] in modified fabrics.

The antibacterial test was performed against *Staphylococcus aureus* and *Escherichia coli* and the result revealed that 0.75 wt% performs the excellent antibacterial activity. Furthermore, it explained the sensitivity towards oxyradicals as the different responses of bacterial strains for the antibacterial test. As S.aureus contains a large number of carotenoid pigments it showed resistance to oxidative stress which resulted from oxyradicals generated by ZnO-NPs [38].The antibacterial property of the treated fabric against Escherichia coli and Staphylococcus aureus before washing were 99.89% and 99.85%, respectively. Even after 100 washing cycles, the antibacterial rates were 99.36% and 99.17%, respectively ensuring the long-lasting anti-microbial property of the fabrics [116].

The results indicated a 79.82 % reduction on *Staphylococcus aureus* and a 75.41 % reduction on *Escherichia coli* at 0.5 h sonication time. Furthermore, 100 % reduction on both *Escherichia coli* and *Staphylococcus aureus* bacteria at 1 h and 1.5 h sonication time [118].

However, more effective inhibition on S. aureus (peptide poly glycogen cell wall; less barrier) than E. coli (cell wall is consisted of lipopolysaccharide, lipoprotein, and phospholipids, and has potential barrier against foreign molecules) [134-138].

The results revealed that the antimicrobial activity depends on the morphological structure and the particle size of ZnO-NPs. Thus, the antimicrobial activity increases with decreasing the particle size of ZnO-NPs. The cotton fabric treated with 26 nm

non-spherical-ZnO-NPs showed the highest antimicrobial efficiency with values of 91.4%, 86.8%, and 84.7% for *Staphylococcus aureus, Escherichia coli*, and *Candida albicans*, respectively. Further, the usage of ZnO-NPs is more beneficial because of their biocompatibility, environmental friendliness, and nontoxicity [123]. The amount of ZnO-NPs on textile is increased with the number of laser pulses. However, this amount could be decreased following the washing of the fabric. Yazdanpanah et al (2020) observed that ZnO-NPs of fabric were decreased to 1442.1 ppm and 917.51 ppm from 1809 ppm after 5 and 20 washing cycles, respectively. The treated fabrics showed strong antibacterial activity against *Staphylococcus Aureus* (ATCC 25923) even after 20 washing cycles. This activity is due to the size of ZnO-NPs but mostly depends on their concentration [124].

The nanoparticles calcined at 600 °C have a small particle size and the highest surface area. Furthermore, the highest antibacterial activity against *Escherichia coli* and *Staphylococcus aureus* has been shown by the nanoparticles calcined at 600 °C. According to the authors, the high calcination temperatures play a vital role in nanoscale crystallization, increased resistivity for washing treatment, and microbial protection in fabrics [30].

In this method, ZnO-NPs with an average size of 10 – 45 nm were synthesized and they were able to successfully inhibit the growth of pathogenic bacteria such as *Staphylococcus aureus, Bacillus subtilis, Pseudomonas aeruginosa* and *Escherichia coli* [139].

Here, the date seed extract plays a vital role to prevent the agglomeration of nanoparticles. This extract contains dominantly phenols, polysaccharides and other materials which are rich in bearing many hydroxyl groups. These hydroxyl groups and divalent zinc ions interacted by ionic dipolar interactions between hydrogen atoms and oxygen in zinc oxide [31].

This single step sono-enzymatic process is an industrial attractive technology that offers a 100% reduction of *E.coli* bacteria and 50% of S.aureus after intensive washing cycles [35]. According to the results, the reaction temperature increased the ZnO deposition rate on the fabric surface. Furthermore, the inhibition zone of *S.aureus* and *E.coli* were wide in the sample treated with ZnO/Ag nanocomposite at 70 °C [126].[Table 3]

4 Discussion

When the egg white is vigorously stirred it gets denatured due to hydrophobic and hydrophilic amino acids. Also, proteins present in egg white have a high affinity with metal ions and able to interact with zinc ions present in the solution forming organo-inorganic zinc complexes [140]. Those formed complexes are adsorbed onto planes of crystals and the growth of nanoparticles on that plane are retarded and less spherical nanoparticles are formed [71]. In the green synthesis of ZnO nanostructures extracts of plant, materials were used as a reducing agents and stabilizers.

In dye removing experiments, before exposing to sunlight the suspension was stirred with a magnetic stirrer for 30 minutes to reach adsorption-desorption equilibrium. The photodegradation activity of synthesized ZnO particles was carried out on a sunny day between 11 a.m. and 2 a.m., where a minimum fluctuation of sunlight is available for the reaction to happen [82].

In the doping process of ZnO nanoparticles the absorbed energy of the electron dissipated due to vibrations and light generation which is named as "recombination" phenomenon. This rapid recombination process diminishes the photocatalytic activity of ZnO. This barrier can be overcome by doping elements as electron scavengers. Doping narrows the bandgap by promoting absorption improves the conductivity of ZnO (mobility of charge carriers) and alters the positions of the conduction band (CB) and valence band (VB). Therefore, doping reduces the recombination and separates photogenerated electrons and holes efficiently [62].

Furthermore, metal Co-facilitated the breakage of chemical bonds in methyl blue by decreasing the bandgap and by increasing the active sites for oxygen adsorption and hydroxylions [95]. They found that when Fe_2O_3 doped with ZnO nanoparticles it forms a metal oxide nanoparticle heterojunction barrier that facilitates the reduction of recombination rate by transferring photogenerated electrons of Fe_2O_3 in the conduction band to the ZnO conduction band. The availability of more surface charge carriers on Fe_2O_3-doped ZnO nanostructures; unpaired photogenerated electrons in the conduction band and holes produce superoxide anion radicals and hydroxyl radicals (react with water) respectively [97].

A major limitation of using ZnO nanoparticles to treat textile is the poor adhesion of nanoparticles to the textile substrate. Typically wet chemical methods are followed to fabricate ZnO nanostructures on the textile surface [141, 142]. Here fabrics are impregnated with a wet pick up and simply by treating the fabric in a bath for 10 minutes using a magnetic stirrer, drying, and curing [101, 143]. Fabricated textiles using a simple wet chemical method do not successfully bind ZnO nanoparticles to the textile surface. Therefore, researchers had to use binding agents such as acrylic [132] and epoxy binding agents to increase the adsorption of ZnO nanoparticles [144-147]. However, a large amount of ZnO nanoparticles remained in the bath due to poor adsorption to the textile substrates after which tend to increase the environmental pollution caused by the textile industry.

The zinc oxide nanoparticles (ZnO-NPs) are capable of suppressing both gram-negative and positive bacterial growth. The antimicrobial property of ZnO-NPs has been explained in different mechanisms such as (i) direct attachment of ZnO-NPs on the bacterial surface via electrostatic forces and destructing bacterial cell integrity [148-150], (ii) liberation of antimicrobial

Table 3. The summary of antibacterial applications of ZnO nanostructures in the textile industry

Description	Microorganisms	References
PA6/ ZnO	*Staphylococcus aureus Klebsiella pneumonia*	[40]
ZnO precursors: Zinc acetate, Sodium hydroxide	*Staphylococcus aureus Escherichia coli*	[29]
ZnO : Hexamethyltriethylene	*Staphylococcus aureus Propionibacterium acnes Escherichia coli Pseudomonas aeruginosa*	[115]
ZnO: Zinc acetate dehydrate, Methoxy ethanol	*Klebsiella pneumonia*	[32]
ZnO: Ultrasound irradiation	*Staphylococcus aureus Escherichia coli*	[38]
Green synthesis		
1. Acalypha indica (Polyol) leaf extract	*Staphylococcus aureus Escherichia coli*	[30]
2. Bioactive molecule secreted by Aspergillus terreus	*Staphylococcus aureus Escherichia coli Bacillus subtilis Pseudomonas aeruginosa*	[139]
3. Bio extract of Data seed Precursors: Zinc acetate, Sodium hydroxide	*Staphylococcus aureus Escherichia coli*	[31]
ZnO/SiO_2 hybrid nanocomposites to coat fabric surfaces	*Staphylococcus aureus Escherichia coli*	[125]
ZnO-NPs: zinc acetate, NH_4OH (Water : ethanol 1:9)	*Staphylococcus aureus Escherichia coli*	[44]
ZnO-NPs: $Zn(CH_3COO)_2(H_2O)_2$, NH_3(aq) (Water:ethanol 10 :1)	*Staphylococcus aureus Escherichia coli*	[159]
ZnO-PDMS coating	*Staphylococcus aureus Escherichia coli*	[116]
ZnO-NPs, 3-glycidyloxypropyltrimethoxysilane	*Staphylococcus aureus* (ATCC 29213) *Escherichia coli* (ATCC 10536) methicillin-susceptible *S. aureus* (MSSA MFBF 10663) methicillin-resistant *S. aureus* (MRSA MFBF 10679)	[117]
ZnO-NPs zinc acetate dihydrate sodium hydroxide With and without starch as a capping agent	*E. coli* (ATCC 8739)	[114]
pure hexagonal wurtzite crystalline ZnO-NPs, $Zn(NO_3)_2{\cdot}6H_2O$, NaOH	*Escherichia coli Staphylococcus aureus*	[118]
ZnO-NPs, $Zn(NO_3)_2.6H_2O$, KOH	*Staphylococcus aureus* (ATCC 25923) *Pseudomonas aeruginosa* (ATCC 27853)	[120]
ZnO-NPs, treated before and after the batik process	*Staphylococcus aureus Escherichia coli*	[112]
ZnO-NPs,Zn acetate dihydrate, NaOH	*Escherichia coli Staphylococcus aureus Candida albicans*	[123]
ZnO-NPs,$ZnSO_4$, NaOH	*Staphylococcus aureus Escherichia coli*	[111]
ZnO-NPs, pulsed laser ablation (PLA), Zn plate	*Staphylococcus aureus* (ATCC 25923)	[124]

ions mainly Zn^{2+}ions [151-153] and (iii) cell membrane penetration and production of intracellular reactive oxygen species (ROS) such as superoxide anion(O2*), hydrogen peroxide (H_2O_2) and hydroxyl radicals (*OH) which are responsible for the antibacterial activity [154-158]. Further, it has also been reported that ZnO is photocatalytic and can be activated by UV and visible light to generate ROS [159]. The toxicological effects are associated with a ROS-dependent mechanism, by the interaction of NPs and ions with mitochondria, or by direct contact with the DNA, due to their permeation across nucleopores [160] [Figures 2 and 3]

When higher energy than binding energy (3.3 e.V.) between the conduction band and valance band is provided the energy is absorbed and electrons pass to the conduction band from the valance band. This phenomenon initiates series of photoreactions. The free electrons of the conduction band react with dissolved oxygen molecules and transformed into superoxide radical anions (O^{2-}) that formed HO_2 radicals. After HO_2 radicals combine with electrons and hydrogen peroxide is formed (H_2O_2). However, formed superoxide radical anions and HO^{2-} radicals unable to penetrate due to the negative charge of the bacteria cell

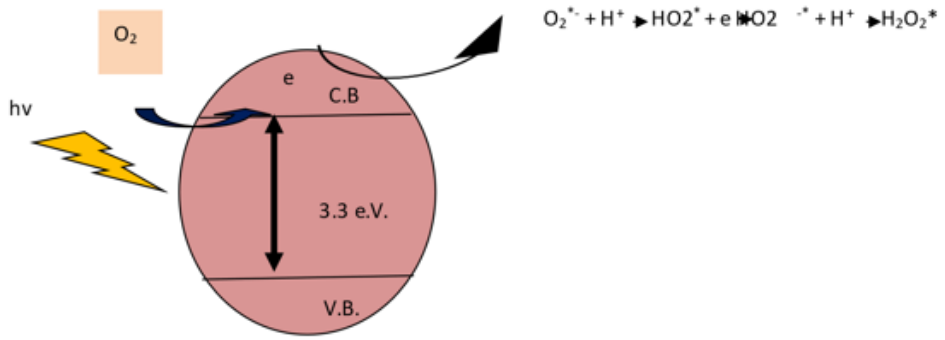

Fig 2. The formation of reactive oxygen species by zinc oxide nanoparticles

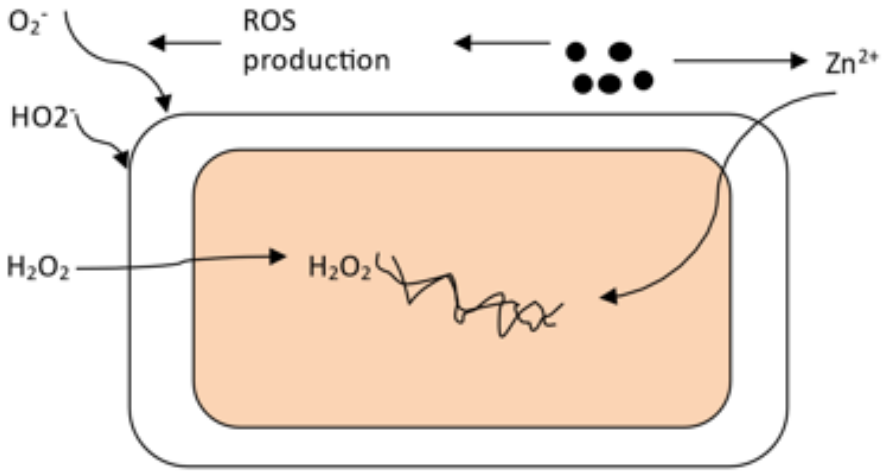

Fig 3. The mechanism of ZnO against bacteria

wall and hydrogen peroxide penetrates the bacteria cell and triggers cell death. There might be Zn^{2+} due to partial dissolution of ZnO nanoparticles when in a solution. The ZnO nanoparticles remain intact in neutral and biological pH values while rapidly dissolved in acidic pH levels. Therefore, in lysosomes of bacteria ZnO nanoparticles dissolved and release Zn^{2+} and combine with biomolecules to inhibit the growth [161].

There are only a few methods normally used to coat ZnO nanoparticles into fabrics such as the pad dry cure method, radiation, and thermal treatment[162-165]. Perelshtein et al (2009) emphasized the advantages of ultrasound irradiation over other traditionally used methods in ZnO-NPs coating on the fabric. The aforementioned traditional coating methods require several stages and a stabilizing agent to obtain small nanoparticles as well as the results in impurities in the final product. The ultrasound irradiation immobilizes the ZnO nanoparticles on fabric in a one-step reaction which is a green approach. Furthermore, a low coating of ZnO can make excellent antibacterial properties using ultrasound irradiation.

5 Conclusion

In conclusion, the efficiency of all UV protection, dye removal and antibacterial property were increased with the reduction of nanoparticle size. In 2021 ZnO nanostructures were successfully coated using binders on textile surface. Furthermore, in latest research ZnO nanoparticles were incorporated in the Bathik process. Always doped ZnO had superior properties than undoped ZnO nanoparticles.

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