



# A new approach of photocatalytic degradation of remazol brilliant blue by environment friendly fabricated zinc oxide nanoparticle

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## Abstract

The aim of present study was to fabricate zinc oxide nanoparticles (ZnO NPs) from facile and green route for wastewater treatment. Purity and crystalline character of synthesized nanoparticles from seeds extract of citrus lemon were investigated by X-ray diffraction. The average crystalline size of ZnO Nps was calculated as 51.68 nm using Debye–Scherrer formula, while the lattice parameters ( $a = 3.32$  and  $c = 4.56$ ) were calculated using cell software, while calculated volume of the unit cell for hexagonal symmetry was  $8.68 \text{ \AA}^3$ . The particle size, morphology and elemental composition of biosynthesized nanoparticles were confirmed by scanning electron microscope and energy-dispersive X-ray spectroscopy, respectively. The photocatalytic activity of the synthesized nanoparticles was examined by degradation of remazol brilliant blue, a textile dye under UV light irradiation. Maximum degradation of remazol brilliant blue was found to be 85.91% after only 60 min of incubation. ZnO Nps retained its activity after five washings and therefore can be recycled and reused. Furthermore, it is concluded and recommended that ZnO Nps synthesized from green route should be the best economical alternate for textile effluent treatment

**Keywords** Citrus lemon · Hexagonal symmetry · Elemental analysis · Photocatalytic activity · Remazol brilliant blue

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## Introduction

More than 10,000 commercially available dyes with over  $7 \times 10^5$  tones of dyestuff are produced annually across the world among which approximately 10–15% textile dyes or effluents are eliminated into water reservoir during processing and synthesis (Nogueira et al. 2014). Dyes having complex chemical structure are visible in minute amounts ( $1 \text{ mg L}^{-1}$ ), consequently very difficult constituents of wastewater to treat (Barwal and Chaudhary 2016). The degradation products of these textile dyes are very toxic; even some are carcinogenic (Asgher et al. 2012). Aesthetic problems are developed when the concentration of these dyes is raised to 10–200  $\text{mg L}^{-1}$  (Capek 2004). Therefore, treatments of textile effluents before they are discharged to the natural water bodies diminish the jeopardies posed to environmental and human's health (Soltania et al. 2015). Conventional physical and chemical methods of treatments are very costly and cannot fully eradicate xenobiotic as it transforms them into another form, posing secondary pollution (Sahay and Nath 2008). Thus, the development of environment friendly, inexpensive method for complete treatment of textile dyes is the need of time. Latterly,



photocatalysis can be expediently used for the complete deterioration of dyes without producing secondary pollutants (Fujishima et al. 2008). Metal semiconductors like ZnS, Fe<sub>2</sub>O<sub>3</sub>, CdS and TiO<sub>2</sub> are efficient, cost-effective and environment friendly photocatalysts which are conveniently used to mitigate environmental glitches (Sharma et al. 2018). Among all reported metals, photocatalysts, ZnO Nps representing *n*-type conductivity and 3.37 eV band gap, exhibit excellent efficiency for photocatalysis of different textile dyes (Greene and Wuts 2002). Complete mineralization of textile dyes into CO<sub>2</sub> and H<sub>2</sub>O can be achieved with the generation of free radicals of ·OH and ·O<sub>2</sub><sup>-</sup> by interacting electron and hole with absorbed O<sub>2</sub> and H<sub>2</sub>O on the surface of ZnO (Xie et al. 2010).

Different physicochemical methods have been used for the synthesis of ZnO nanoparticles including chemical reduction (Rizi et al. 2019), laser ablation (Soltania et al. 2015), solvothermal (Sahay and Nath 2008), inert gas condensation (Loponov et al. 2009) and sol–gel method (Gude and Narayanan 2011). Most of the methods illustrated in literature are costly and lead to hazardous by products. A number of reactions need high pressure and temperature for initiation, while some others require toxic gases like H<sub>2</sub>S, stabilizer and metallic precursors (Kuang et al. 2013).

Biological techniques (microorganisms, plant extract or plant biomass) have gained the attention of researcher and considered as alternative to traditional chemical and physical methods for the fabrication of an eco-friendly approach of nanoparticles (Lu et al. 2007). Currently, plants and plant-derived material are preferred over microbes for the fabrication of nanoparticles due to their environment friendly nature. Furthermore, microbe-mediated nanoparticles synthesis route is complex as they eliminate the complicated practice of maintaining cell cultures, intracellular production as well as several purification steps are required (Bonne-mann and Nagabhushana, 2008). Moreover, plant infusions are regarded as inexpensive source of capping agents as compared to microorganisms in nanofabrication (Li et al. 2006). The literature reported that seeds and peels of citrus lemon are usually discarded during processing though they have strong antioxidant potential (Ashraf et al. 2017). To make the fabrication process economical, seeds of citron lemon were selected as reducing agent. To the best of our literature survey, green route using seeds extract of citrus lemon has been used for the first time as a reducing material as well as surface stabilizing agent for the fabrication of zinc oxide nanoparticles. Therefore, the current investigation was carried out to synthesize, characterize and photocatalytic activity of fabricated zinc oxide nanoparticles using seeds extract of citrus lemon. The structure, phase and morphology of synthesized product were investigated by the modern conventional standard characterization techniques. This study was carried out between January and July 2015

in Department of Chemistry, University of Bahawalpur, Pakistan.

## Materials and methods

### Materials

All the chemicals were of analytical grade, purchased from Sigma-Aldrich and used as received without any further purification.

### Fabrication of ZnO Nps through green route

#### Preparation of bio-extract

Fresh seeds of citrus lemon (Fig. 1) were collected from local market of Bahawalpur and washed with simple tap water followed by deionized water to remove the dust particulate and then sun-dried to take out the residual moisture content. Seeds extract was prepared by taking appropriate amount of deionized water and thoroughly washed dry seeds of lemon, and then grinded with the help of electric grinder. The grinded mixture was boiled at 80–90 °C for 6–7 min along with stirring. The boiled mixture was cooled at room temperature and then filtered with Whatman No. 1 filter paper to remove the biomaterials. This extract was used for subsequent experiments as reducing agent (Garza et al. 2013).

#### Synthesis of nanoparticles

For the fabrication of nanoparticle, 10–20 ml of aqueous seeds extract of citrus lemon was added to 200 ml of 2 mM of zinc sulfate heptahydrate solution and heated at 60–75 °C along with continual vigorous stirring for 30–35 min. The resultant solution was kept at room temperature for the



Fig. 1 Seeds of circuits lemon

reduction of zinc ions into zinc nanoparticles for about 1 h. The formation of nanoparticles was visually identified by color change from yellow to dark brown. ZnO nanoparticles were settled down at the bottom of the beaker after 50–60 min. The filtered nanoparticles were dried after keeping at 50 °C in vacuum oven for 20–22 h; finally, grinded powder was used for characterization by various conventional techniques.

### Characterizations

To confirm the formation of nanoparticles, their purity, crystalline character, dispersity as well as their morphology, powder X-ray diffraction was performed using a X-ray diffractometer (Shimadzu, XRD-6000) with CuK $\alpha$  radiation  $\lambda = 1.5405 \text{ \AA}$  over a wide range of Bragg angles  $2\theta = 20^\circ - 80^\circ$  at a scanning rate of  $5 \text{ min}^{-1}$ . Scanning electron microscopy (SEM) analysis of fabricated zinc oxide nanoparticles was done using a Hitachi SX-650 (Tokyo, Japan) SEM machine. Elemental composition of the fabricated ZnO Nps was executed using energy-dispersive X-ray spectroscopy (EDX).

### Photocatalytic activity of fabricated nanoparticles under sun light irradiation

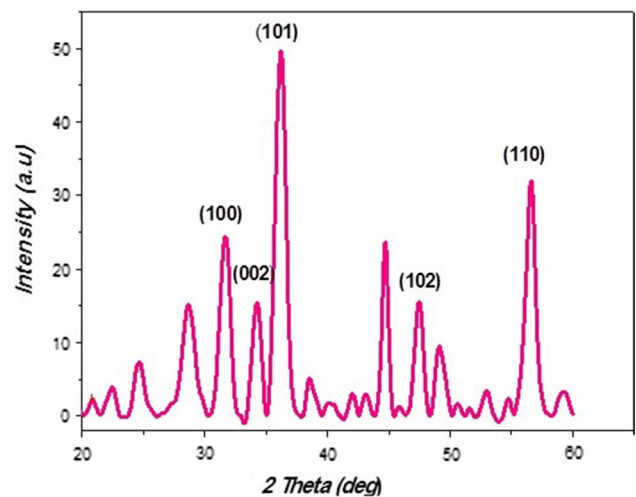
Photocatalytic degradation was performed by using 200 ml solution of 50 ppm target remazol brilliant blue textile dye and 32 mg of bio-fabricated zinc oxide nanoparticles. Control was also run without the addition of as-synthesized nanoparticles. Prior to sunlight and UV exposure, the resultant reaction mixture was well vortexed using vortex mixer for 10–15 min to establish the equilibrium. Subsequently, the degradation was carried out under natural sunlight and ultraviolet radiation. After certain definite time intervals, the sample was withdrawn and % degradation was determined by using UV–Vis spectrophotometer. Concentration of dye throughout the biodegradation was calculated at lambda max (592 nm) of remazol brilliant blue textile dye. Percentage of dye degradation was estimated by the following formula:

$$\% \text{ removal} = \frac{\text{Initial concentration of dye before treatment} - \text{Final concentration of dye after treatment}}{\text{Initial concentration of dye before treatment}} \times 100$$

## Results and discussion

### XRD Analysis of ZnO Nanoparticles

Figure 2 clearly indicates the X-ray diffraction pattern of zinc oxide particles synthesized by facile economical green route. The phase, orientation and crystalline



**Fig. 2** X-ray powder diffraction (XRD) pattern of ZnO NPs produced from seed extract of citrus lemon

character of fabricated nanoparticles of ZnO were confirmed by powder X-ray diffraction, with the use of advance X'pert PRO diffractometer with copper-K $\alpha$  as radiation source (where  $k = 1.54056 \text{ \AA}$ ) and scanned from  $20^\circ$  to  $60^\circ$  with the scanning rate of  $5.0^\circ/\text{min}$ . The major diffraction peaks with  $2\theta$  values of 31.76, 34.37, 36.26, 47.41 and 56.57 are observed and well indexed to (100), (002), (101), (102) and (110) planes. This demonstrates the characteristics XRD pattern of hexagonal zinc oxide nanoparticles. Our findings are nearly in accordance with the joint committee on powder diffraction standards (JCPDS) card no 36–1451. Almost comparable values have also been reported by Ahmad et al. (2003). Some of the diffraction peaks corresponding to the impurity were found in the XRD patterns which might be due to the presence of phenolic compounds in seeds extract of citrus lemon, while the X-ray diffraction pattern has a peak position at  $2\theta$  value of  $20.9^\circ$ , which is the typical peak of zinc phase. Similar findings were achieved by Li et al. (2006). In the above X-ray diffraction pattern, the broadening of

the peaks might be due to the small size of as-synthesized ZnO nanoparticles (Meng et al. 2014; Prabhu et al. 2013; Saleh and Gupta 2012).

The lattice parameters for ZnO hexagonal geometry with diffraction peaks corresponding to (100), (002), (101), (102) and (110) hkl values were calculated as  $a = b = 3.325 \text{ \AA}$  and  $c = 4.56 \text{ \AA}$ ,  $c/a$  1.373 using cell software which are almost

consistent with JCPDS data card of zinc oxide. While the volume of the unit cell for hexagonal symmetry was calculated as  $8.68 \text{ \AA}^3$  using following equation

$$V = \sqrt{3}a^2c/2$$

From the width of major diffractions peaks like (100), (002), (101) and (102), the grain size ( $D$ ) of ZnO nanocrystallites was calculated to be in the range of 43.87–73.10 nm using Debye–Scherrer formula.

$$D = K\lambda/\beta \cos \theta$$

where  $K$  is the Scherrer constant with value ranging from 0.9 to 1,  $D$  is grain size of ZnO nanoparticles,  $\theta$  is the Bragg angle,  $\lambda$  corresponds to wavelength of X-ray source 0.1541 nm used in X-ray diffraction, and  $\beta$  is the full width at half maximum of the diffraction peak.

### Scanning electron microscopy (SEM) and elemental analysis by energy-dispersive X-ray (EDX)

Surface morphology as well as the distributions of fabricated nanocrystallites was examined by scanning electron microscopy (SEM, Hitachi S4800 FESEM). A very thin film of fabricated nanoparticles was prepared by locating very minute amount of as-synthesized zinc oxide nanoparticles on carbon-coated copper grating; subsequently, the film was allowed to desiccate by placing the mercury lamp for 4–6 min on the SEM grid. Scanning electron microscopy has provided further insight into the morphology and size details of the synthesized nanoparticles. SEM micrographs of synthesized zinc oxide nanoparticles using the seeds extract of citrus lemon as shown in Fig. 3 clearly depicted that grains nucleate in a hexagonal-like shape. The number of the particles also looks like to agglomerate into bigger particles. The average particles size computed from scanning electron microscopy (SEM) image varied from 15 to 75 nm. The elemental composition and purity of the as-synthesized nanoparticles were attained by EDX on the same scanning electron microscopy apparatus. The energy-dispersive spectra (Fig. 4) of fabricated ZnO nanoparticles obtained from the SEM–EDX analysis confirm the presence of zinc as the primary components. Table 1 shows the elemental percentage of particles yielded of 76.04% of zinc, 21.7% of oxygen and 2.26% of carbon that provide the evidence of as-synthesized zinc oxide nanoparticles by green route is in its maximum purity with only almost 2% of carbon and also in accord with the previous investigations (Tek et al. 2007; Zhang et al. 2010). Carbon is mainly attributed to the polyphenolic compounds present in the seeds extract of citrus lemon.

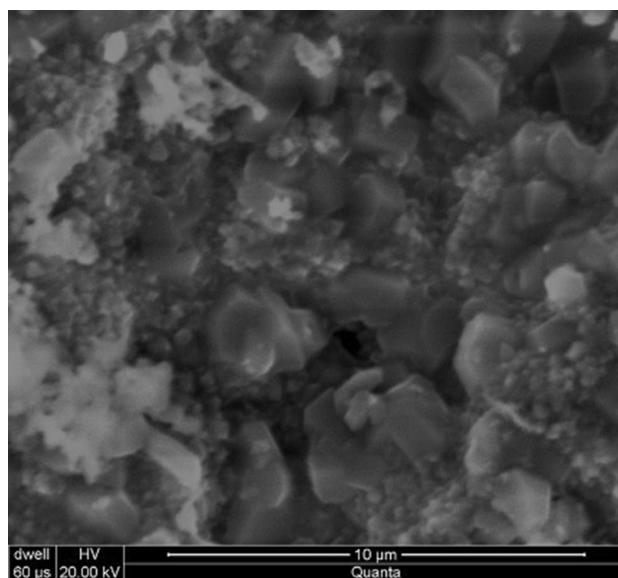
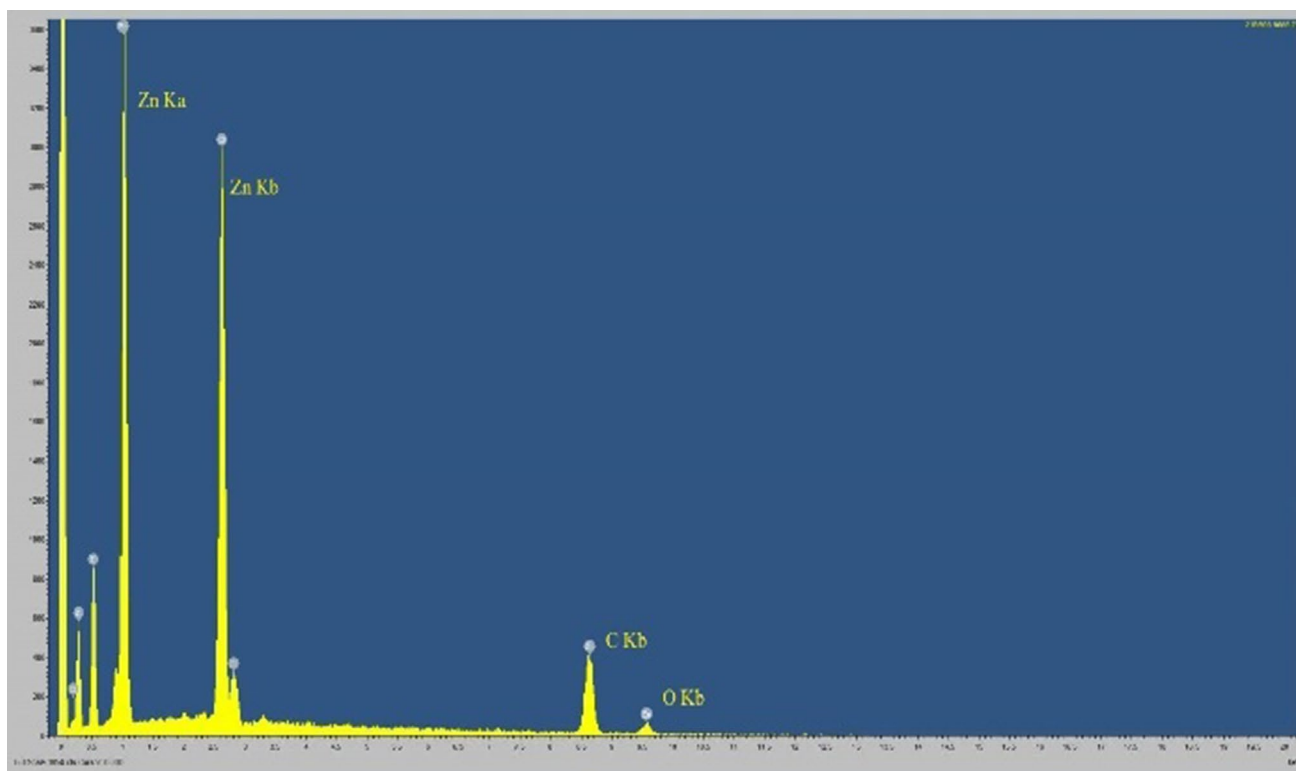


Fig. 3 Scanning electron microscopy of ZnO NPs

### Photocatalytic degradation of dye

The photocatalytic degradation was carried out in an aqueous solution using remazol brilliant blue (Aldrich,  $\lambda_{\text{max}} = 592 \text{ nm}$ ) as a target dye. The reaction mixture contains 150 ml of 10 ppm of target textile dye and magnetically stirred with 32 mg of newly as-synthesized nano-photocatalyst of ZnO and then kept in UV photoreactor equipped with xenon lamp set at UV intensity of  $2.00 \times 10^{-6} \text{ Einstein l}^{-1} \text{ s}^{-1}$ . After regular time interval, the sample was withdrawn and filtered by Millipore 0.45- $\mu\text{m}$  membrane filter and % degradation was determined by using UV–Vis spectrophotometer. The maximum degradation was found to be 85.91% (Figs. 5, 6) after 60 min of incubation. In actual mechanism, ZnO absorbs UV light and electron ( $e^-$ ) and hole ( $h^+$ ) are generated. Hydrogen and hydroxyl-free radical are formed when water combines with hole on the surface of ZnO Nps. Actually, free radicals are responsible for the degradation of target dye into various products (Fig. 7). Chen et al. (2017) reported that photocatalyst ZnO degraded 99.70% azo dye (methyl orange) in only 30 min. Different factors like size of nanoparticle, concentration of dye, dosage of photocatalyst, pH, etc., influenced the degradation capacity. Degradation of the dye should be increased in acidic pH with high doses of photocatalyst (Liu et al. 2016). The degradation capacity was much higher as compared to laccase bio-system (Palazzolo et al. 2019).





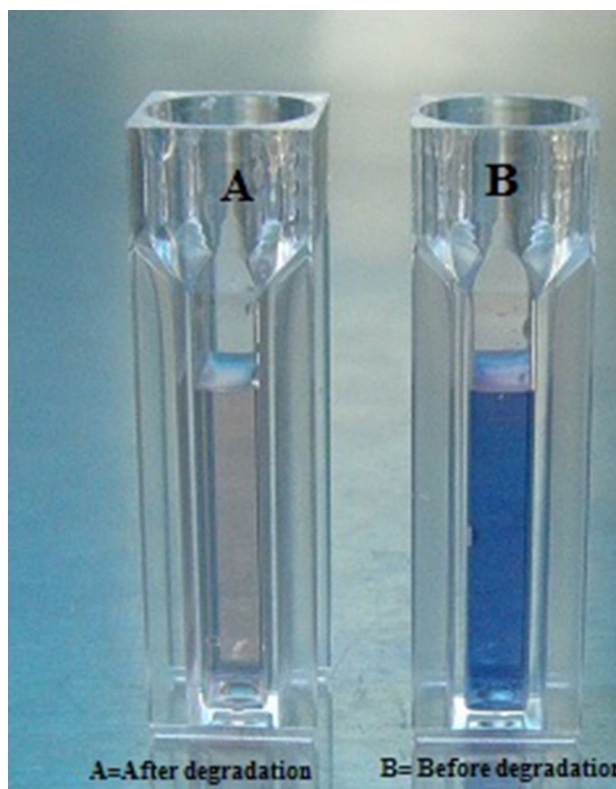
**Fig. 4** Energy-dispersive X-ray (EDX) photogram of ZnO NPs

**Table 1** EDAX analysis of ZnO nanoparticles

Sr. no.	Element	Atomic %
1	Zn K	76.04
2	O K	21.7
3	C K	2.26
4	Totals	100

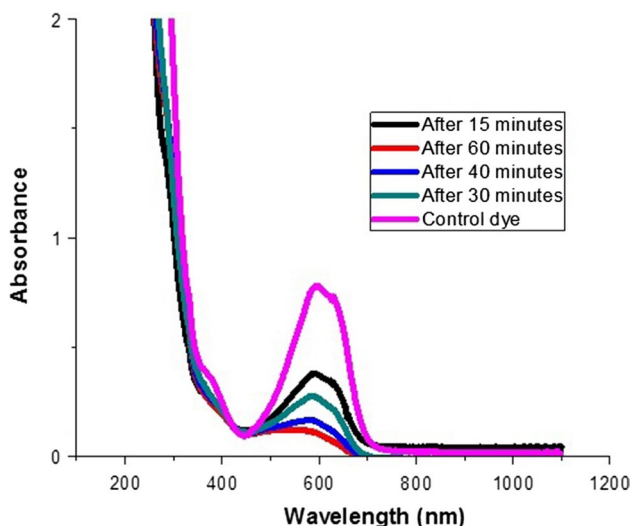
### Efficiency and reusability of biologically synthesized ZnO Nps

Cycling experiments for photodegradation of remazol brilliant blue were performed to determine efficiency, reusability and photo-stability of biogenic ZnO Nps at 100 °C using oven-drying method in reaction cycles (Khalafi et al. 2019). Negligible change in the catalytic efficiency of ZnO Nps was observed after five consecutive cycles (Fig. 8). Slight reduction in photodegradation from 85.91 to 85.83% after five runs indicated higher durability and recyclability of biogenic ZnO Nps toward remazol brilliant blue (Fig. 8). These findings are in accordance with the findings of Sen et al. (2019) and Khalafi et al. (2019). Comparative study of photocatalytic potential of biogenic ZnO Nps with chemically synthesized ZnO Nps or other Nps like  $\alpha$ - $\text{Fe}_2\text{O}_3$ ,  $\text{Mn}_2\text{O}_3$  and  $\text{Fe}_3\text{O}_4$  indicated that biogenic ZnO Nps had been potentially more effective and durable (Amini and Ashrafi 2016).



**Fig. 5** Visual degradation of remazol brilliant blue dye before and after degradation

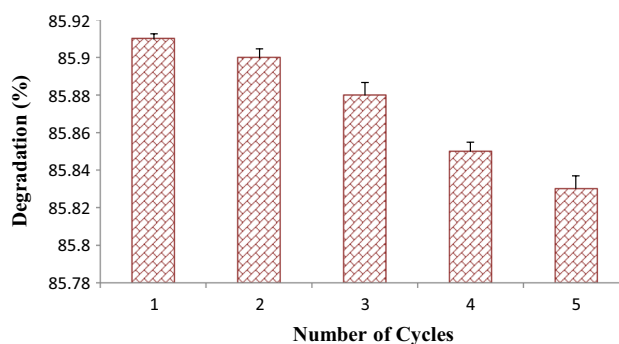




**Fig. 6** UV–Visible scanning spectrum of degradation of remazol brilliant blue textile dye in different time intervals

**Conclusion**

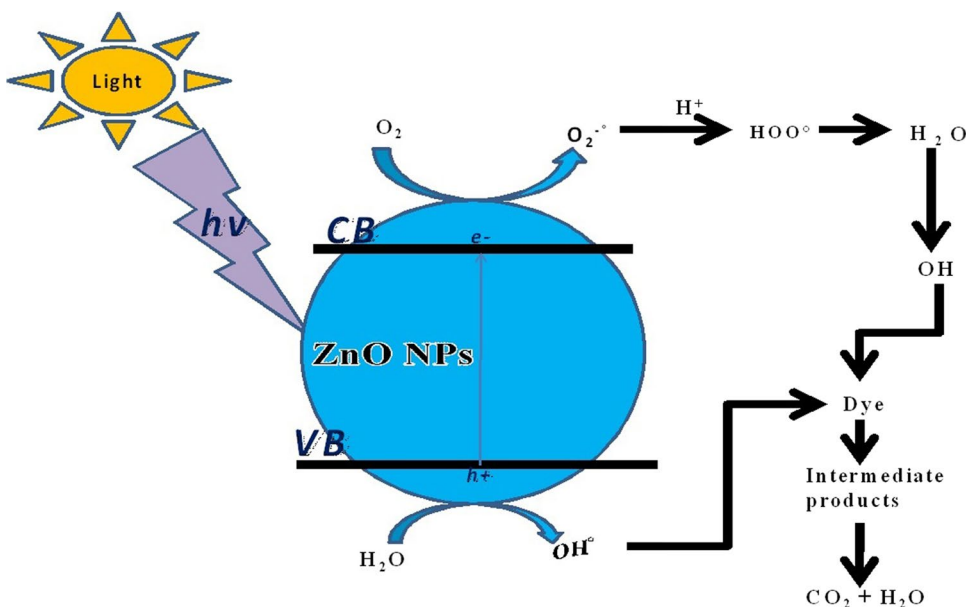
In current research effort, we reported the successful synthesis of cost-effective, durable, eco-friendly and stable ZnO Nps for the first time using electron-rich lemon seed extracts as stabilizing and reducing agent. It is clear from the present investigation that green technology is simple, eco-friendly and efficient as compared to other conventional and outdated techniques. SEM micrographs of fabricated ZnO Nps using the seeds extract of citrus lemon clearly indicated that grains nucleate in a hexagonal-like shape. The average particle size computed from scanning electron microscopy



**Fig. 8** ZnO Nps facilitated photodegradation of remazol brilliant blue in five consecutive cycles

(SEM) image varied from 15 to 75 nm. SEM–EDX analysis confirms the presence of zinc as the primary components, while X-ray diffraction peaks demonstrate the characteristics pattern of hexagonal zinc oxide nanoparticles. The photocatalytic degradation of the biogenic ZnO Nps was examined by degradation of remazol brilliant blue textile dye under UV light irradiation, and maximum degradation was found to be (85.91%) after 60 min of incubation. The rate of degradation of remazol brilliant blue remained almost constant after five consecutive cycles representing effectiveness and higher stability of biogenic ZnO Nps. Therefore, the ZnO Nps produced from seed extract of citrus Limon are projected to have wide applications in various industrial sectors and further scientific researches using more natural sources for fabrication of biogenic nanoparticles are in progress.

**Fig. 7** Proposed degradation pathway of industrial textile dye effluents using ZnO/UV radiation



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