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Green Synthesis of Zinc Oxide Nanoparticles and Their Application in Ring Opening of Epoxides

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ABSTRACT

Epoxides are molecules with extensive usages in various products synthesis. They are capable of reacting with different nucleophiles and their potential in the ring opening of epoxides adds to their importance as pioneers in the synthesis of organic compounds. In the present research, a green and easy method for the synthesis of zinc oxide nanoparticles as a catalyst was introduced. Synthesized zinc oxide nanoparticles were characterized by UV-Vis spectroscopy, Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The UV-Vis spectrum of the nanoparticles showed an absorption peak at 327 nm corresponding to the ZnO nanoparticles. X-ray diffraction analysis also showed a high degree of crystallinity of the synthesized nanoparticles with the hexagonal structure. Zinc oxide nanoparticles were studied as an effective catalyst for epoxide ring- opening. The reactions were done by aliphatic and aromatic amines and alcohols as a nucleophile at room temperature and under microwave conditions. The results of the reactions showed that the presence of zinc oxide nanoparticles catalyzed the ring-opening of the epoxides well. The ZnO nanocatalyst ledto the synthesis of products with high efficiency and good regioselectivity in a short period of time.

Highlights

- ZnO NPs were synthesized by the green method for the first time from violet flower Extract.
- Ring-opening of the epoxides was done by nucleophile by using ZnO nanoparticles.
- The results showed that zinc oxide nanoparticle is an effective catalyst in the ring-opening of epoxides.

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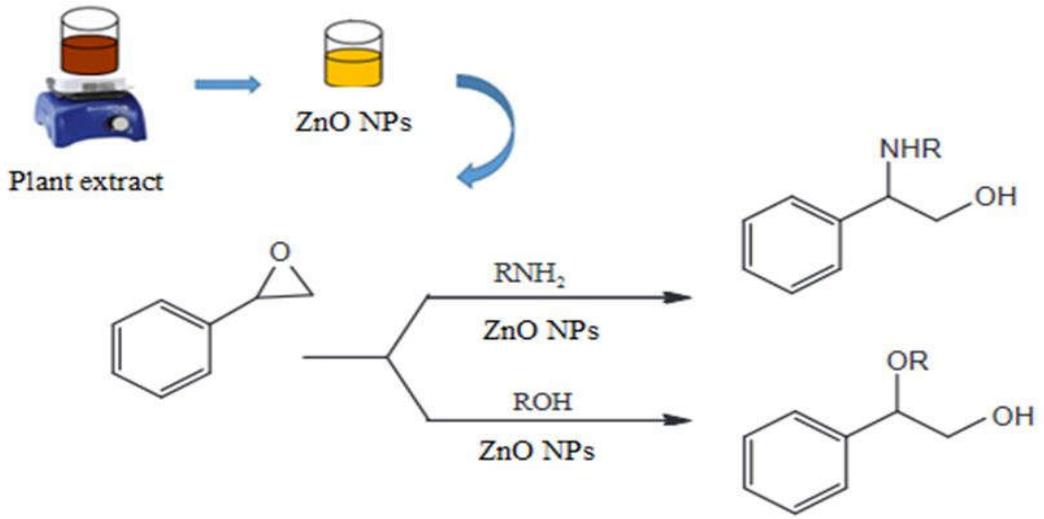
GRAPHICAL ABSTRACT



Introduction

Epoxides or oxiranes are one of the greatest intermediates in organic reactions. The high polarity and the tensile of the ring make the epoxides susceptible to react with a variety of nucleophiles, acids, bases, reducing and oxidizing agents. Epoxides have a tendency to ring opening reaction [1, 2]. Different nucleophiles such as azides, amines, alcohols, esters, cyanides, and halogens can be used for the ring-opening of epoxides [3, 4]. Thus, epoxides are useful structural blocks in organic synthesis that can be applied in industry. Ring opening can be achieved in neutral, acidic and bases solutions, but the presence of acids accelerates it. In recent years, various studies have been conducted on the opening of epoxide in the presence or absence of catalysts [5, 6]. These studies were based on the high-efficiency and low-time methods to synthesize different products. Several metal oxides like Al_2O_3, ZnO, and Fe_2O_3 show lewis acid characters and they increase the rate of reactions [7]. Past research shows that the ring-opening of the epoxide under neutral or alkaline conditions proceeds according to the SN_2 mechanism but in acidic conditions, a borderline SN_2 has been done [8]. The application of nano catalysts shows good yields and selectivity in organic reactions. Recent researches display that ZnO nanocatalyst has received significant attention due to their non-toxic and environmental properties. For example, the synthesis of pyranopyrazole derivatives in the presence of a heterogeneous zinc oxide nanocatalyst has been reported [9]. In addition, they have been used in various syntheses such as the Manich and Knoevenagel reaction and synthesis of coumarins [10]. Also, ZnO was employed in different fields such as ring opening of epoxides with acetyl chloride. Recently, the synthesis of nanoparticles has attracted much attention because of their catalytic usages in various sciences and a high surface area [11-14]. Chemical procedures for the synthesis of nanoparticles have been applied, but green methods are more important due to their non-use of harmful compounds and eco-friendly effects [15, 16]. Bio-organisms such as fungi, bacteria, yeast, or microorganisms like plants and algae can be used for the synthesis of nanoparticles [17, 18]. Zinc oxide nanoparticles have been widely used because of their photocatalytic, electrical, electronic, optical, and antibacterial properties [19]. Different ways have been offered for the synthesis of them like deposition methods [20], combustion synthesizes [21], and sonochemical methods [22]. These methods have problems like the purification of nanoparticles from the surfactant, the presence of organic solvents and toxic

substances. For this reason, green methods of producing zinc oxide nanoparticles are expanded [23]. Based on previous studies, the type of plant extract can affect the size and shape of the synthesized nanoparticles [24].

In this study, zinc oxide nanoparticles (ZnO NPs) were produced using violet flower by a simple and green technique and used as a catalyst in epoxide reactions. Ring-opening of epoxides was performed in the presence of nitromethane solvent and solvent-free conditions. The catalyst can easily open the epoxide rings and produce the high-efficiency.

Experimental

Material and methods

Styrene oxide, Propylene oxide, nitromethane, $ZnSO_4$, NaOH and all other chemicals were purchased from Sigma-Aldrich and Merck. Fourier transform infrared spectrometer was recorded in KBr on Perkin Elmer spectrum 100. Ultraviolet-visible spectrometer was recorded on AGILENT8454-GERMANY. Scanning electron microscopy (HITACHIS-4160) and X-ray diffraction (D8_BURKER_2002) were used to identify synthesized nanoparticles. The ^{1}H NMR and the ^{13}C NMR were recorded on a Bruker Avance instrument to identify synthesized compounds. Thin-layer chromatography (TLC) was used to monitoring the progress of the reactions.

Preparation of Iranian violet flower extract

Iranian violet flower (Viola odorata) was collected in spring from the mountains around Quchan city, Khorasan Razavi, and then identified by the Research Center of Natural for plant sciences, Ferdowsi University of Mashhad (Iran) with the number E1037-FUMH. Flowers and leaves were excised and rinsed several times with water. It was dried and powdered at room temperature to be used for the extraction. 5 g powder of plant was boiled with 200 ml distilled water for 15 minutes at 70 °C under magnetic stirring. The extract was filtered through Whatman No1 filter paper.

Synthesis of zinc oxide nanoparticles

20 ml of the plant extract was mixed with 300 ml of 0. 4 M $ZnSO_4$ solution. Then, 10ml of 1M NaOH solution was added until the pH of the solution reached12. The color of the mixture changed to yellow. The precipitate was separated by centrifugation (3500 rpm for 5 minutes) and was washed with ethanol and distilled water. The collected nanoparticles were dried at 100 °C for 6 h [25]. The formation of zinc oxide nanoparticles (ZnO NPs) was investigated by ultraviolet-visible absorption spectroscopy(UV-Vis), Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and scanning electron microscopy (SEM) [26].

Ring opening of epoxide by ZnO NPs catalyst in room temperature

0. 01 g of ZnO NPs, 3 mmol nucleophile, and 1 mmol epoxide in the presence of 37 mmol nitromethane were mixed and stirred for 2 h at room temperature with a magnetic stirrer. Thin-layer chromatography (Hexane -Ethyl acetate) was applied for monitoring the progress of the reaction. The reaction was quenched with increasing water and the products were extracted with dichloromethane. The products were purified by column chromatography (Ethyl acetate- Hexane) and then were characterized by comparison of $^{13}CNMR$, $^{1}HNMR$, and FTIR spectroscopy with those of authentic samples [27].

Ring opening of epoxide by ZnO NPs catalyst using microwave

0. 03 g of zinc oxide nanoparticles as a catalyst, 2 mmol nucleophile and 2 mmol epoxide and 37 mmol nitromethane were mixed and then subjected to 400 W microwave for 2 min. The reaction progress was monitored by thin-layer chromatography. Then separation and purification of products were performed [28].

Results and discussion

In this study, biosynthesis of zinc oxide nanoparticles was performed using Iranian violet flower extract. Synthesis of ZnO NPs was characterized by various identification techniques. Initially, the formation of nanoparticles was confirmed by a color change in the reaction vessel. Further characterization was accomplished by UV-Vis, FTIR, XRD, and SEM.

Colour change

The observed color change from olive green to pale yellow and finally hard yellow is the first sign of the formation of zinc oxide nanoparticles (Figure 1). Change in coloris due to vibrations of surface plasmon in nanoparticles [29]. The presence of antioxidant compounds such as flavonoids, proteins, anthocyanins and alkaloids can cause the extract of the Iranian violet flower to act as a good reducing agent and produce zinc oxide nanoparticles. These results were consistent with other studies. Nasser et al. , have reported these changes in the synthesis of zinc oxide nanoparticles from fistula and Melia azedarach extract [24].

UV-Vis

In metal oxide nanoparticles, the optical properties are affected by factors such as the size of the nanoparticles, their spacing, and the refractive index of the surrounding environment. Peak displacement, peak intensity change, and color are factors that are dependent on the size of the nanoparticles. The ultraviolet spectrum of zinc oxide nanoparticles was obtained by dissolving the nanoparticles in deionized water (Fig. 2). Zinc oxide nanoparticles synthesized by the violet flower have an absorption peak at 381 nm [30]. The absorption peak between 300 and 400 nm confirms the presence of zinc oxide nanoparticles. This could be related to the electron transition from the valence band to the conduction band. Also, the peak presence at 327 nm is related to nanoparticles below the gap band wavelength. This was consistent with the results of Aldalbahi et al. [31].

Infrared Spectroscopy (FT-IR)

FTIR spectrometry is used to recognize the functional groups of molecules. The FT-IR spectra of violet flower extract and synthesized zinc oxide nanoparticles are shown in Figure 3. In the spectrum of zinc oxide nanoparticles, a strong band is observed around 500 cm^{-1}, which is related to the frequency of the ZnO bond [32]. Rajendran et al., obtained similar results in the synthesis of zinc oxide nanoparticles from R. fairholmianus root extract [33]. Absorption bands of Iranian violet flower extract were found at 3400, 2900, 1400, and 1000 cm-1, respectively. An absorption band that shows the presence of O-H stretching vibrations appeared in 3400 Cm^{-1}. Aromatic and aliphatic C-H stretching vibrations and (NH)-C-O stretching vibrationare possible groups in the structure of the violet flower extract [34].

Fig 1. The color changes in the production process of zinc oxide nanoparticlesa) Extract from Iranian violet flower b) Forming nanoparticles c) Zinc oxide Nanoparticles

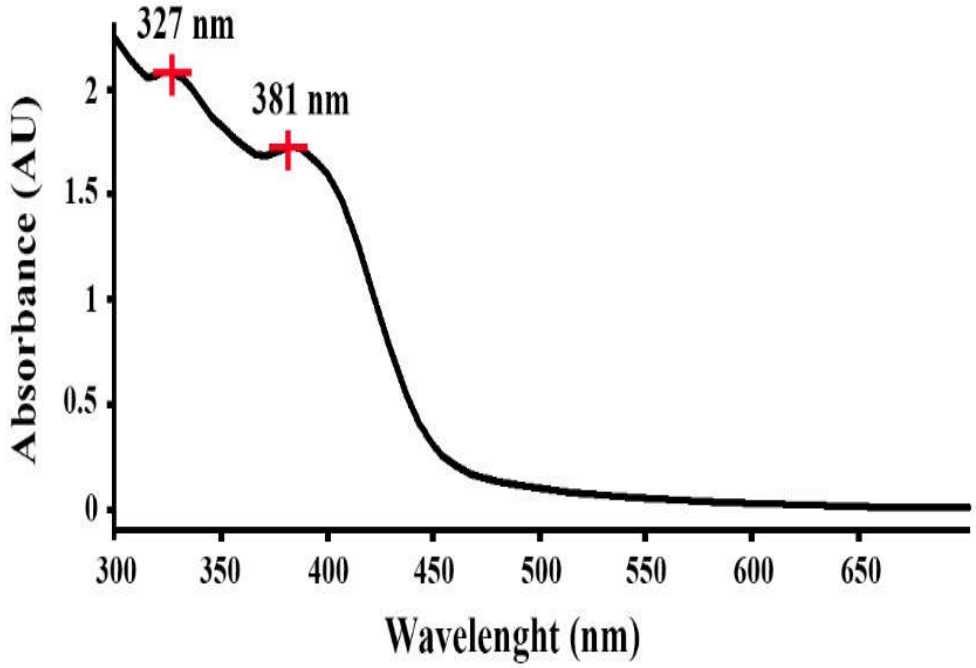


Fig. 2. UV-Vis spectrum of ZnO NPs

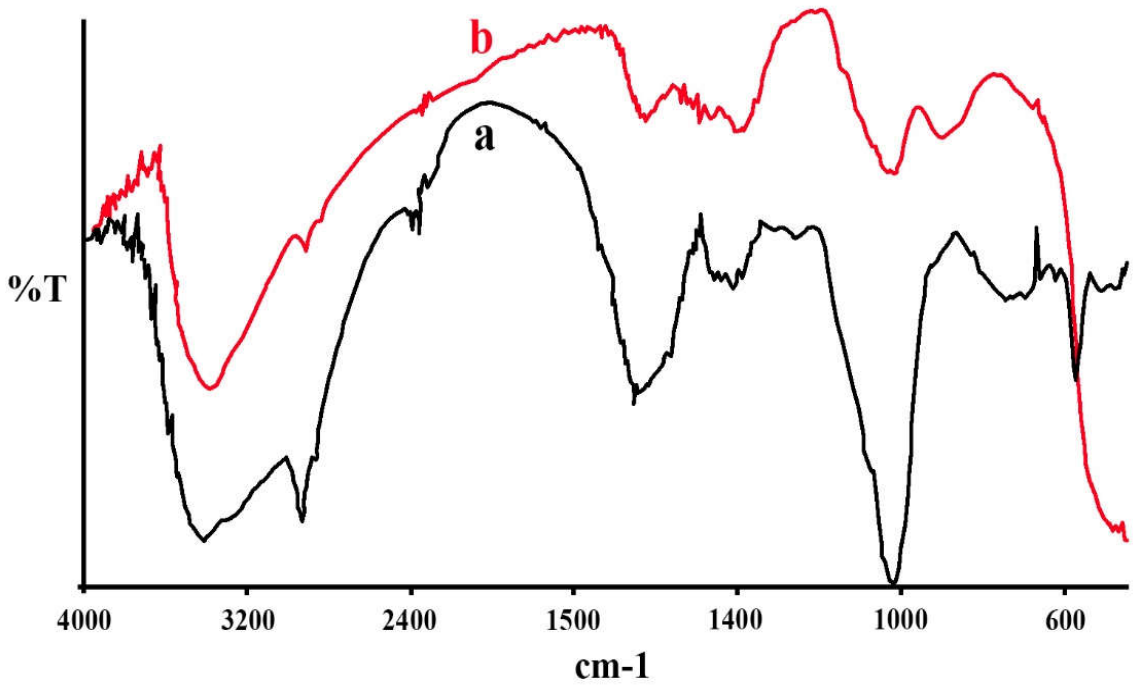


Fig. 3. FT-IR spectrum a) Violet flower extract and b) Zinc oxide nanoparticle

X-ray diffraction (XRD)

The crystal structure of zinc oxide nanoparticles was investigated using X-ray diffraction analysis. The XRD spectra of the synthesized nanoparticles are shown in Figure 4. By Debye-Scherrer equation (equation 1), the approximate size of crystals (D) can be calculated as:

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

Where D, β, θ and λ are the size of the crystals, the peak width at half the maximum intensity (in radians), the Bragg angle of the peak (in radians), and the x-ray wavelength (in angstroms), respectively.

The XRD pattern appears with peaks degrees of 31. 11, 34. 40, 36. 32, 47. 45, 56. 50, 63. 11, 66. 15, 66, 67, 69. 02 in the index (100), (002), (101), (102), (110), (103), (200), (112), (201), and data were in good agreement with the standard pattern of zinc oxide JCDP [NO: 89-7102]. Observation of this spectrum shows the absence of impurities as well as the size of the synthesized ZnO nanoparticles. Based on the Debye-Scherrer equation, the size of the synthesized nanoparticles is 55 nm and their structure is hexagonal [35]. Modi and Fulekars also studied the synthesis of zinc oxide nanoparticles by using garlic skin extract and the Crystallite size calculated was 12. 61 nm [36].

Scanning Electron Microscopy (SEM)

The shape and size of the particles were examined by scanning electron microscopy. The image of homogeneous nanoparticles with an average size of 50-60 nm corresponded to the size obtained from the XRD spectrum as shown in Figure 5 [37].

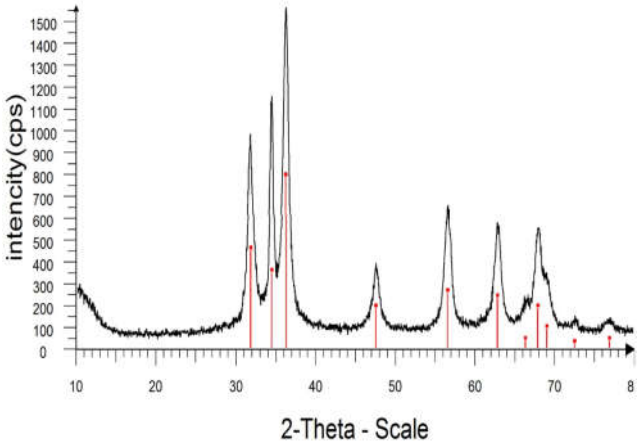


Fig. 4. XRD spectrum of synthesized ZnO nanoparticles

Ring opening of epoxide in presence of nanocatalyst

In this work, we applied ZnO NPs catalyst in the epoxide ring-opening. Reactions were evaluated in different conditions at room temperature and under microwave conditions. In the absence of solvent, the reaction could not proceed satisfactorily, so nitromethane as a polar solvent was selected for this reaction. In our work, synthesized zinc oxide nano catalyst is cheaper in comparison to the previous research, and can improve the yield of the reaction. In this condition, epoxides react with high regioselectivity. Ring opening of aryl epoxide takes place at the benzylic position. All reactions were monitored by TLC and FTIR. Products were confirmed by comparison of ^{1}H NMR and ^{13}C NMR with those of authentic samples. Zinc oxide catalysts assisted the reaction in a short time with high efficiency. We also examined reactions under catalyst-free conditions. These reactions can be accomplished without the presence of catalysts for extended periods of time [38]. In the

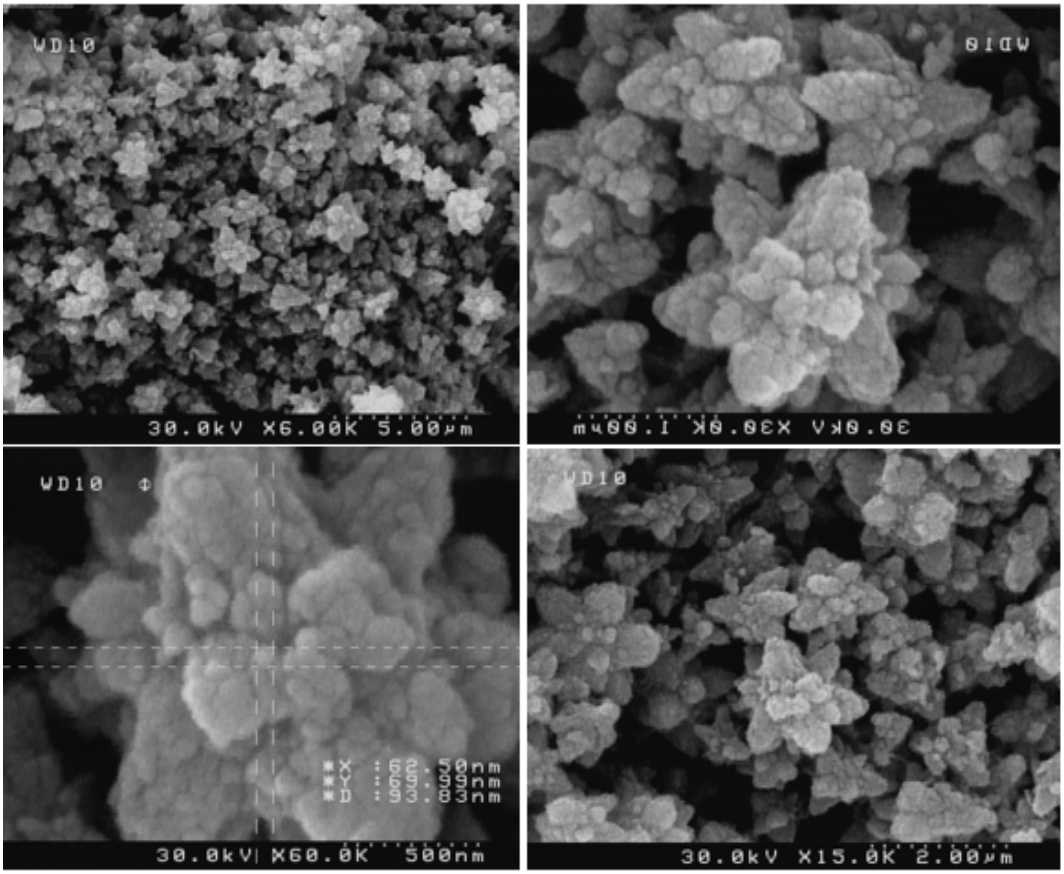


Fig.5. Scanning electron microscopy of zinc oxide nanoparticles

FTIR spectra of all products, the hydroxyl group was observed to indicate the epoxide ring-opening reaction. The absorption peak of this O-H group was observed in 3200-3500 cm^{-1}. In products 2, 3, 4, 5, 9, 10, 11, 12, N-H tensile absorptions appeared in the same range. In compounds with the aromatic rings, tensile vibration C-H aromatic was seen in 3100-3200 cm^{-1}, while the tensile vibration of C-H aliphatic compounds was observed in the range of 2800-300 cm^{-1}. Azizian et al. , have developed epoxide ring-opening in the presence of Nano catalyst ZrO_2[39]. In 2021, regioselectivity ring-opening in aliphatic and aromatic epoxides also was done in the presence of Co/Ni-catalyzer [40]. Some results from spectroscopy are given below:

Compound 7: IR(KBr) (3500, 2985. 5, 1737. 3, 1563. 7, 1440. 7, 1378. 1, 1233. 4, 1043. 0, 932. 1, 842. 9, 635. 6, 609. 1)MS(EI) M/Z 139(M+).

Compound 9: ^{1}HNMR ($CDCl_3$, 300 MHz) δ(ppm): 7. 17-7. 47 (m, 10H), 3. 77(d ,1H), 3. 96(d,1H) 3. 95, (s, 1H), 4. 54 (s, 1H).

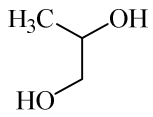
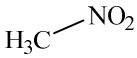
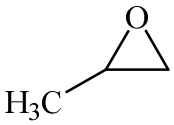
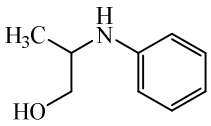
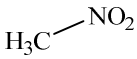
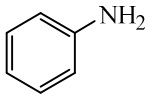
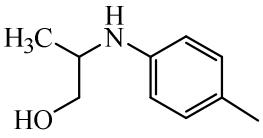
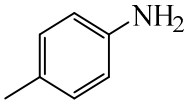
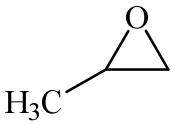
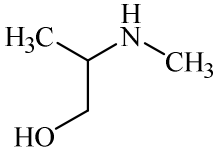
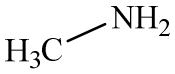
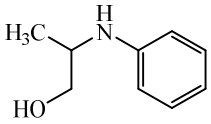
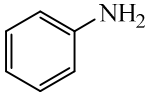
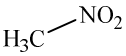
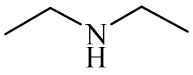
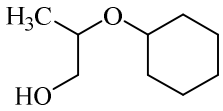
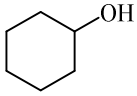
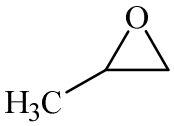
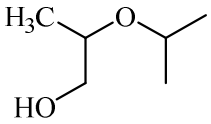
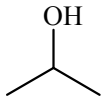
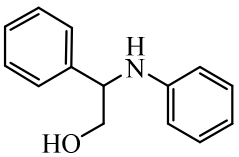
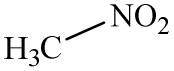
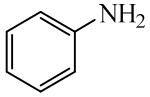
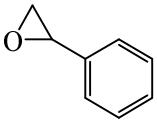
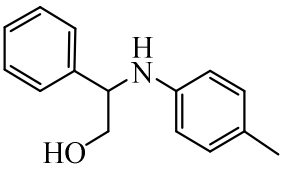
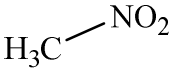
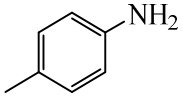
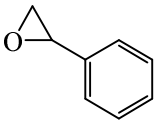
^{13}CNMR (CDCl3, 125 MHz) δ(ppm): (59. 98,67. 73,113. 67,113. 99, 117. 96,118. 32, 126. 96,126. 85,127. 16,128. 73,129. 20, 129. 41,140. 88, 147. 36)

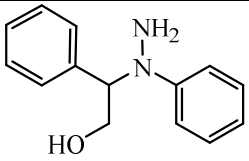
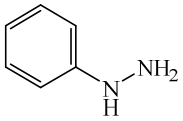
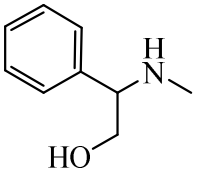
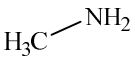
Compound 10: IR(KBr) (3450, 2971. 0, 2077. 3, 1720, 1638. 4, 1464. 8, 1378. 1, 1247. 9, 1158. 7, 1045. 4, 951. 4 cm^{-1}). MS(EI) M/Z 228(M+).

Compound 12:

^{13}CNMR ($CDCl^3$, 125 MHz) δ(ppm): (56. 82, 67. 36, 84. 50, 126. 93, 128. 03, 128. 47, 128. 54,138. 37)

Table 1. Reaction of ring opening of epoxide by nucleophiles in the presence of zinc oxide nanoparticles

Time min	Efficiency (%)	Product	Solvent and conditions	Nucleophile	Epoxide	Entry
2	89		 Nitromethane Microwave 400w	H_2O		1
2	84		 Nitromethane Microwave			2
2	86		Microwave 400w Nitromethane			3
2	90					4
120	88		Nitromethane R. T			5
-	-	-	 Nitromethane			6
100	85		R. T Solvent free R. T			7
45	92		Solvent free R. T			8
140	86		 Nitromethane R. T			9
300	89		 Nitromethane			10

240	93		R. T		11
120	84				12

Conclusion

In recent years, metal nanoparticles have extensive application in various fields such as their use as catalysts in organic reactions. Several techniques like chemical and physical methods have been used for the synthesis of nanoparticles. The synthesis of nanoparticles by green chemistry has led to the development of non-toxic and eco-friendly methods. In this study, zinc oxide nanoparticles were produced via violet flower extract in a short time and environmentally method. The morphology of the synthesized nanoparticles was distinguished by using UV–Vis, FTIR, SEM, and XRD analysis. The regioselectivity for ring opening of epoxide was studied in presence of zinc oxide nanoparticles. Epoxide rings were opened at the appropriate time with high efficiency and were confirmed by TLC, FTIR, ^{13}CNMR, and^{1}HNMR, spectroscopy. Due to the high performance of ZnO nanoparticles in these reactions, they can be used as catalysts in other organic reactions. On the other hand, their green production methods are both economically and biologically cost-effective.

Declarations of interest: none

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