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## Zinc oxide Nanoparticles from waste date seed Catalyzed Preparation of Benzimidazole derivatives

Gurumeet C. Wadhawa<sup>1</sup>, Manas Koli<sup>2</sup>, Shwedant Thakur<sup>3</sup>, Akash Singh<sup>4</sup>

<sup>1</sup>Assistant Professor, Veer Wajekar ASC College, Phunde, Ta. Uran, Dist. Raigad, Navi Mumbai (Maharashtra, India)

<sup>2,3,4</sup> Student, Department of Chemistry, Veer Wajekar ASC College, Phunde, Ta. Uran, Dist. Raigad, Navi Mumbai (Maharashtra, India)

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### Abstract

Value of Benzimidazole derivatives in chemical synthesis and the pharmaceutical sector is enormous. Therefore, a wide range of techniques is employed to synthesize Benzimidazole Derivatives. For the synthesis, a vast number of catalysts are employed. The production of benzimidazole derivatives employing Zinc oxide Nanoparticles from waste date seed as an effective catalyst is described in this paper. Under the sonication technique, the Zinc oxide Nanoparticles from waste date seed in ethanol solvent. This is used for the synthesis of Nanoparticles.

**Keywords:** Waste date seed, Zinc oxide, Nanoparticles, Sonication, Benzimidazole

### Introduction:

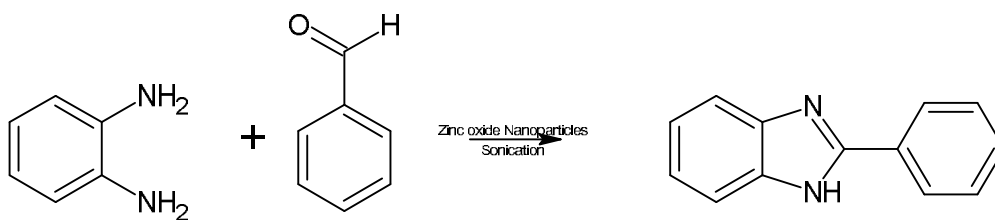
Heterocyclic structures are compounds that have one or more cyclic structures, with one or more heteroatom like N, O and S. Mostly in nature and drugs, nitrogen containing heterocyclic compounds play an essential function in the forms of proteins like purine, histidine, proline, and pyrimidine bases in the genetic material like DNA and RNA are more critical which play a vital role in life such as metabolism of all the animal and plant cells. They also play an essential role as enzymes, coenzymes and many natural products. Most biologically active molecules such as hormones, acids, enzymes, and neurotransmitters, may contain one, two or many heterocyclic rings. This most common heterocyclic compound is Benzimidazole as an essential nucleus.

Several pharmacologically active heterocyclic compounds, such as Benzimidazole, are daily used in the medical sector. In addition, many manufactured and naturally occurring heterocyclic compounds are used in pharmaceuticals, insecticides, agrochemicals, polymers, plastics, medications, and colours. Therefore, there is much room for a study that will lead to novel heterocyclic compounds with high biological activity [1-9].

Benzimidazole is one of the most significant heterocycles in medicinal chemistry. In 1950, benzimidazole-containing compounds were discovered as chemotherapy drugs for the first time. Mostly vitamin derivative a component of Vitamin B-12 and other medicinal medications, is the most prevalent Benzimidazole-containing molecule. The use of Benzimidazole and its derivatives is shown in Figure 1. Benzimidazole molecules have various biological actions, including anticancer and anti-inflammatory characteristics. Anthelmintic activity [10] is a well-known property. Benzimidazole compounds with a variety of pharmacological activities, including cardiotoxic [11], anti-ulcer [12-13], antibacterial and antiviral against virus and bacteria [14], anticancer [15], antimutagenic [16], and anti-allergenic [17], have already been identified. It also has anti-inflammatory, antipyretic, and analgesic properties [18]. It also has anti-aggregatory [19] and hypoglycaemic anti-calmodulin [20] actions.

Various approaches have been used to synthesize these benzimidazole derivatives. Condensing ortho phenylenediamines with different carboxylic acids their analog is a common approach, albeit it necessitates unsympathetic conditions like strong acid polyphosphoric acid [21] at 190 to 200°C. Another technique involves combining aldehyde and ortho phenyl phenylenediamine in the being there of several catalysts, including 190 resin Indion [22] and Boron trifluoride [23], CAN [24], and I<sub>2</sub>. Polyethylene glycol [26-27], Hydrogen peroxide and ferrous nitrate [28-29], Indium trifluoride [30-31], Zinc chloride supported on silica [32], silica-supported Na<sub>2</sub>H<sub>2</sub>SO<sub>4</sub> [33], Only polyethylene glycol [34], Ferrous nitrate 3 [28-29], In(OTf)<sub>3</sub> [30-31], (NO<sub>3</sub>)<sub>3</sub> In recent years, Ytterbium (OTf)<sub>3</sub>, KSF clay [35], metal halide supported Plane alumina [36-39], and solid support catalyst [40-44] have been used to explain solvent-free benzimidazole synthesis under microwave irradiation. However, many of these methods have downside such as the prerequisite for harsh and strong acidic conditions, elongated reaction times, small yields, time consuming set-up procedures, large volumes of reagents, and the use of toxic chemicals, catalysts, or solvents.

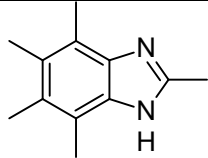
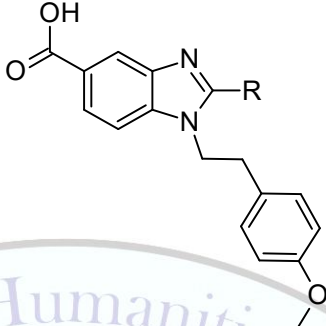
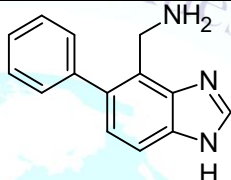
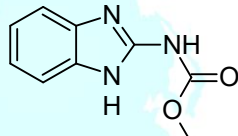
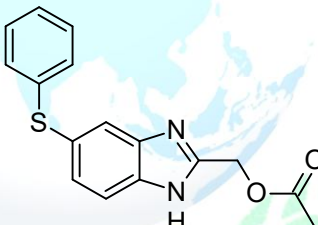
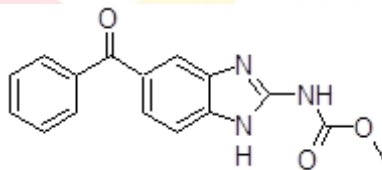
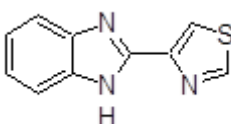
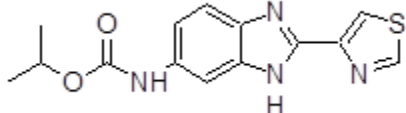
As a result, present is a considerable insist for a highly well-organized and environmentally benign method of synthesizing these heterocycles. As fraction of our research programme in creating varied synthetic methodologies, we present the synthesis of benzimidazoles by means of Zinc oxide Nanoparticles from waste date seed as an excellent catalyst Reaction shown below In the literature, the catalyst has been described as an effective catalyst for various organic reactions.

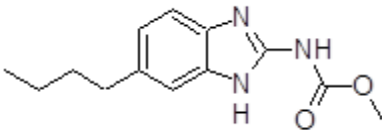
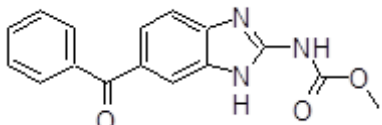
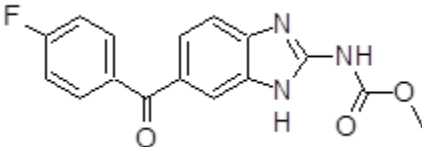
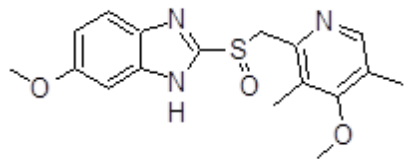
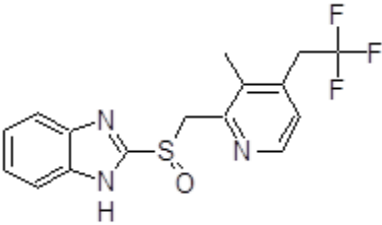
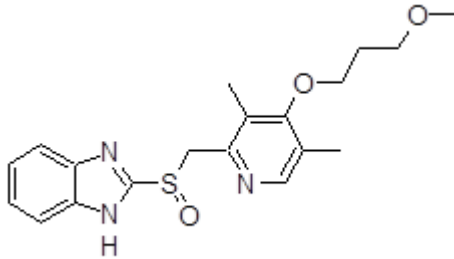
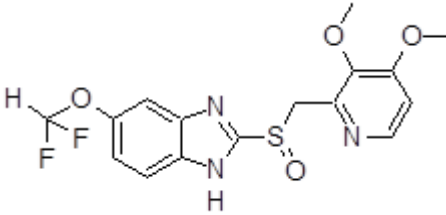


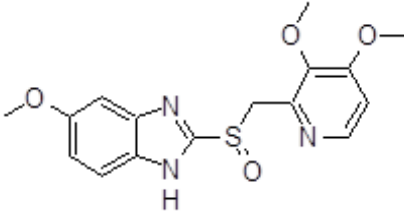
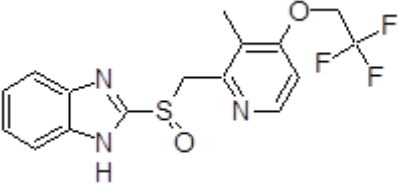
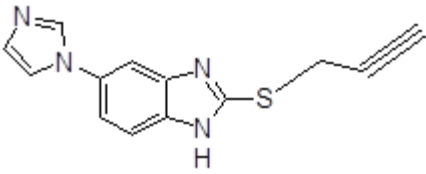
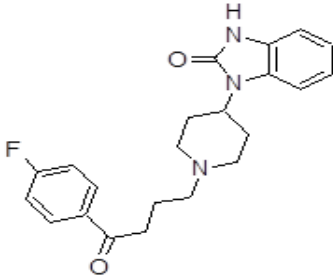
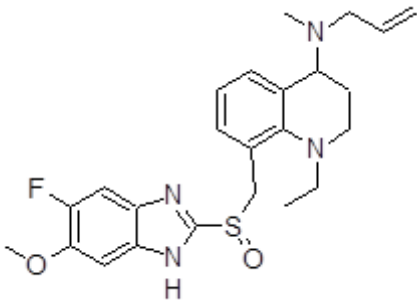
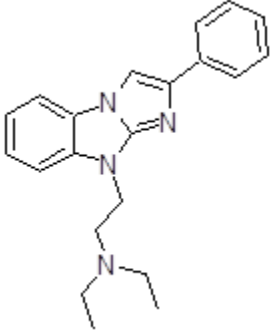
Zinc oxide Nanoparticles from waste date seed for synthesis

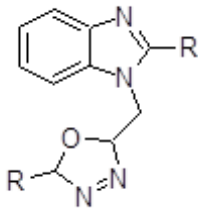
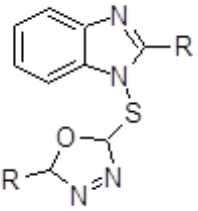
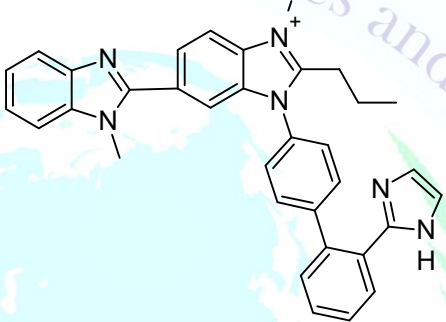
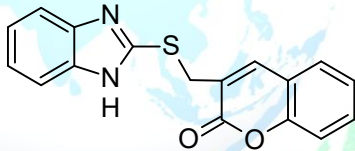
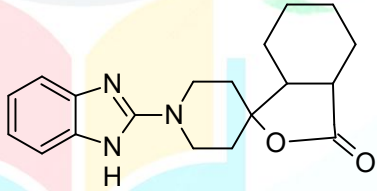
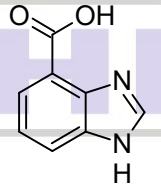
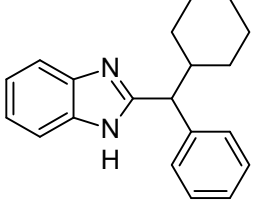
**Table 1 Different Activity of Benzimidazole Derivatives**

Trade Name	Structure	Activity
Oxfendazole		Roundworms and tapeworms
Ricobendazole		Anthelmintic
Triclabendazole		anthelmintic
Amide derivative of Benzimidazole		Anticancer activity
Substituted Benzimidazole		Anticancer activity

Alkyl substituted Benzimidazole		Ant amoebic activity
Benzyl substituted carboxyl Benzimidazole		Antileukemic activity
Phenyl amine derivative of Benzimidazole		Antidiabetic activity
Amide derivative of Benzimidazole		Cytocidal activity
Thioether derivatives of Benzimidazole		<b>Nematicide and taenicide</b>
Mebendazole		Anthelmintic
Thiabendazole		Anthelmintic
Cambendazole		Anthelmintic

Parbendazole		Anthelmintic
Albendazole		Anthelmintic
Flubendazole		Anthelmintic
Omeprazole		Anti-ulcer drugs
Lansoprazole		Anti-ulcer drugs
Rabeprazole		Anti-ulcer drugs
Pantoprazole		Anti-ulcer drugs

Esomeprazole		Anti-ulcer drugs
Triethoxy-pyridylBenzimidazole derivative		Anti-ulcer drugs
Thiophene derivatives of Benzimidazole		Anti-ulcer drugs
Droperidol		Anti-psychotic agents
QuinolineBenzimidazoleAnalog		Anti-psychotic agents
Imidazole derivative with Benzimidazole		Anti-psychotic agents

<p>Oxazole derivative with Benzimidazole</p>		<p>Antimicrobial activity</p>
<p>Oxazole derivative with Benzimidazole and thio-linkage</p>		<p>Antimicrobial activity</p>
<p>Dibenzimidazole derivatives</p>		<p>Antagonist</p>
<p>Benzimidazole and coumarin derivative</p>		<p>Antiseptic virus activity</p>
<p>Spiro compound of Benzimidazole</p>		<p>NPY N5 Receptor Antagonist</p>
<p>4-Carboxylic acid Benzimidazole</p>		<p>Selective 5 HT 4 Antagonist</p>
<p>Phenyl cyclohexyl derivative of Benzimidazole</p>		<p>Amp Activated protein kinase activator</p>

### Experimental:

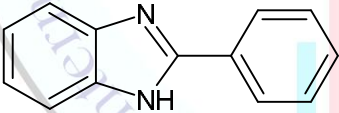
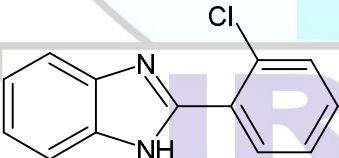
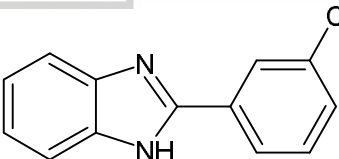
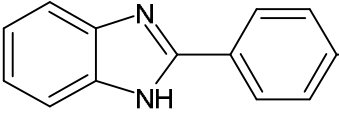
On MHz 400 Varian NMR spectrometers, all <sup>1</sup>H NMR spectra were captured. All chemical shifts are indicated as —chemicals purchased from LOBA Chemicals for the Synthesis Purchase. The paraffin technique was used to determine the melting point of the material. Because all produced compounds are novel, the comparative approach is used to characterize them.

### Zinc oxide Nanoparticles from waste date seed Used in Synthesis:

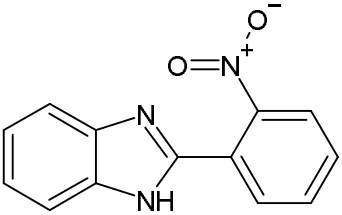
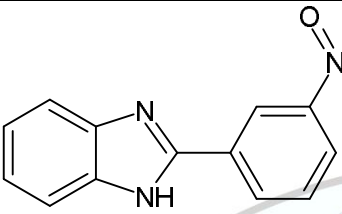
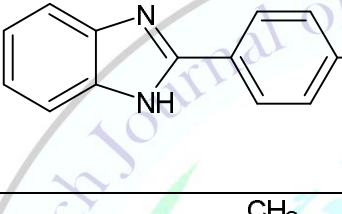
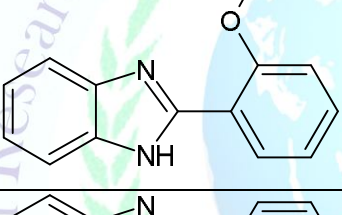
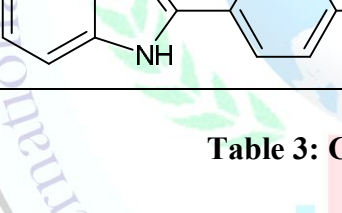
In a 100 ml round bottom flask, a combination of o-phenylenediamine (10 mmol), benzaldehyde (10 mmol), and Zinc oxide Nanoparticles from waste date seed (10 mol percent) in Ethanol (5 ml) was Sonicated for the appropriate period at 30-40 0C. TLC Hexane kept track of the reaction's progress: Chlorform and methano; (8:2). After the reaction was completed, the reaction mixture was cooled and treated with DCM dilution (20 mL). Water and brine solution was used to wash the whole organic layer, then dried on Sodium sulphate and evaporated under vacuum. Column chromatography was used to purify the crude residue, yielding 2-substituted benzimidazoles.

### Observation:

**Table 2. Reaction time,% yield and Melting point of Benzimidazole Derivatives. (Sonication Method)**

Entry	Compound	Reaction Time (Min.)	Yield(%)	MP(°C)
1.		100	88	298
2.		105	96	137
3.		110	94	227
4.		100	96	229



5.		105	95	258
6.		100	98	309
7.		105	96	302
8.		105	92	268
9.		100	92	287

**Table 3: Choice of Solvent for reaction**

Sr.No	Solvent	Temperature(° C)	Time(min)	Yield(%) <sup>a</sup>
1	CHCl <sub>2</sub>	30-40	100-110	56
2	CH <sub>3</sub> OH	30	100-110	74
3	CH <sub>3</sub> CH <sub>2</sub> OH	30	100-110	96
4	THF	30	100-110	66
5	CH <sub>3</sub> CN	30	100-110	75

**Table 4: Reaction Time**

Sr.No	Zinc oxide Nanoparticles from waste date seed (mol%)	Time (min)	Yield(%) a
1	0	100-110	18
2	5	100-110	68
3	10	100-110	96
4	15	100-110	90
5	20	100-110	80

### Spectral Analysis

**1) 2-Phenyl benzimidazole:** White Solid ;

NMR in DMSO :12.12 $\delta$  (brsinglet,1H),8.18  $\delta$ (doblet, $J$  value =7.8Hz,2H),7.70-7.71  $\delta$  (multiplet,1Hydrogen ),7.61-7.63  $\delta$  (multiple, 4H), 7.24 - 7.26  $\delta$  (multiplet, 2H)

IR( $\text{cm}^{-1}$ ):3430<sup>1</sup>,2930  $\text{cm}^{-1}$ ,2630,1640  $\text{cm}^{-1}$ ,1420,1280, 1120 ,980,740 ,

**2) 2- 2-Chlorophenyl Benzimidazole:** Light pinkSolid ;

NMR in DMSO :12.76 $\delta$  (brsinglet,1H),7.98-8.19  $\delta$ (multiplet ,1H),7.64-7.66  $\delta$ (multiplet, 3H), 7.58 - 7.62  $\delta$  (multiplet, 2H), 7.23 - 7.28  $\delta$  (multiplet , 2H);

**3) 2- 3-Chlorophenyl benzimidazole:** White Soild

NMR in DMSO :12.12  $\delta$ (brsinglet ,1H),8.30  $\delta$ (singlet ,1H),8.27 $\delta$ (doublet , $J$ =6.8Hz,1Hydrogen ),7.61- 7.72  $\delta$  (multiplet , 4H), 7.39 - 7.45 (multiplet , 2Hyderogen );

**4) 2-(4-Chlorophenyl) Benzimidazole:** White Soild;

NMR in DMSO :11.9 $\delta$  (brsinglet,1H),8.16  $\delta$ (doublet , $J$ =8Hz,2Hydrogen ),7.45-7.48  $\delta$ (multiplet ,4Hydrogen ), 7.20 $\delta$  (doublet , $J$  value =8Hz,2Hydrogen );

### Result and Discussion:

Special solvents and mole ratios of Zinc oxide Nanoparticles from waste date seed were investigated to determine the best reaction conditions. The representation reaction of o-phenylenediamine and 4-methoxy benzaldehyde was used in our preliminary research. Varying solvents can produce different yields, as seen in the Table. by means of its quick conversion, high yield, and low toxicity, Ethanol was the optimum solvent for condensation reactions. In the sonication, zinc oxide nanoparticles from waste date seed were introduced in a variety of mole ratios to Ethanol, as indicated in Table. The greatest yields were produced with 10 mol percent Zinc oxide Nanoparticles from discarded date seeds. In Table 3, the electrical effects of several substituted

aldehydes have been examined. Aldehydes with both types of the electron-donating and electron-withdrawing substituents were found to yield the required benzimidazoles well. The authenticity of the products was validated by comparing them to authentic samples Spectral data.

### Conclusions:

Finally, discarded date seed-derived zinc oxide nanoparticles were an effective catalyst for the production of Benzimidazole from aldehydes and o-phenylenediamine. This procedure is feasible, environmentally benign, and economically appealing because of the affordable and easily accessible catalyst. The proposed approach also has the advantages of a quick work-up procedure, high yields of products, and the benign nature of the catalyst.

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