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Synthesis of Benzimidazole using Plant Assisted Zinc Sulphide by Mortar–Pestle Grinding Method

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Abstract: *Benzimidazole derivatives have the immense importance in the process of the drug discovery. Now a days these are synthesized by the various catalyst and solvent. Due to the envoirment issue we need to develop new method of the synthesis which are green and ecofriendly .in this we have developed the use of the mortar–pestle grinding technique in the presence of plant assisted zinc Suphide nanoparticles derived from euphorbiaceous family. A catalyst is described as an ecologically safe and effective method for the production of benzimidazole derivatives. The condensation of aldehyde and o-phenylenediamine was followed by a Cylisation reaction, which resulted in the synthesis of the matching benzimidazoles in up to 97-98 percent yields. This approach might be used to make benzothiazoles and benzoxazoles derivatives as well.*

Keywords: Synthesis of Benzimidazole using Plant Assisted Zinc Sulphide by mortar–pestle grinding method

INTRODUCTION

Benzimidazole and its derivatives are frequent building blocks in physiologically active and therapeutically beneficial products among the many N-containing heterocyclic compounds. They're also known for their extensive use as enzyme inhibitors, medicines, dyes, and polymers.[1] Benzimidazole compounds with broad spectrum of various biological activities, ranging from widely used human and anticancer properties [2]. The anthelminticactivity [3-5] are commonly known. Along with this, Benzimidazole derivatives with different pharmacological properties such as, anti-ulsaral [6], cardiotonic [7], antihypertensive against depression [8], antibacterial and antiviral against virus and bacteria [9], antitumor [10], antimutagent[11], antiallergenic [12] are already reported.[table 1] It also exhibits analgesic, anti-inflammatory and antipyretic activity [13]. Moreover, it also shows hypoglycaemic [14] anticalmodulin [15] and anti-aggregate [16] activities. Most common Benzimidazole containing compounds are 5,6-dimethyl-1-D-ribofuranosyl Benzimidazole, part of the structure of Vitamin B-12 and other pharmaceutical drugs. Drugs like Micardis, Vermox, and Nexium, for example, include benzimidazole core structures . As a result, benzimidazole preparation methods have gotten a lot of interest in recent years. Benzimidazoles can be made using a variety of methods that involve condensation reactions between o-phenylenediamine and carboxylic acids2 or their derivatives (nitriles, amidates, and orthoesters),[18] or aldehydes [19] in the presence of various catalysts such as 4-OMe-TEMPO, polymer-supported hypervalent iodine, iodobenzene diacetate, NaHSO4-SiO2, HCl. [20] Although various other techniques have been devised, such as the tandem carbonylation–cyclization process of o-phenylenediamine catalysed by transition metals or the hydroformylation reaction of N-alkenyl phenylenediamines catalysed by transition metals, etc [21] The majority of these procedures have one or more flaws, such as the use of strong acidic conditions, high temperatures, a stiochiometric amount of oxidants, ecologically unfriendly solvents, long reaction times, or costly metal catalysts, to name a few. On the other hand, due to environmental concerns as well as low cost, the mechano chemical approach is gaining popularity. [22] Mechanical grinding, which became a powerful tool in the paradigm of synthetic organic

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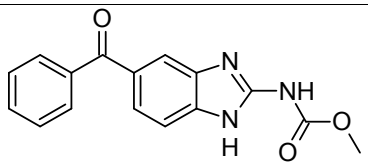
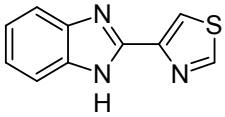
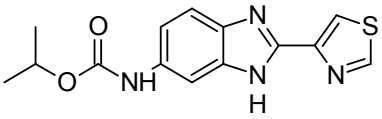
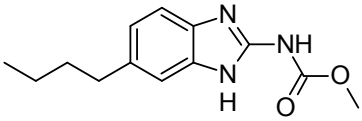
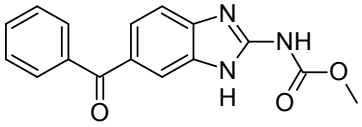
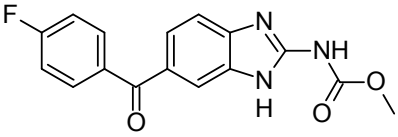
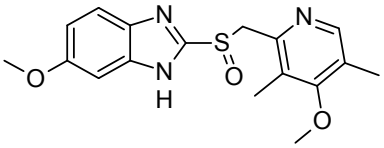
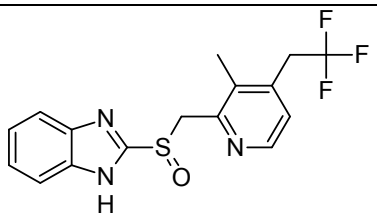
chemistry because of its simplicity of operation and clean reaction profile, as well as its potential application at a larger scale by employing adequate experimental set-up, is one of the most common techniques used in solvent-free reactions.

. **Table 1 Importance of Benzimidazole Derivatives**

Sr. No	Trade Name	Structure	Activity
1	Mebendazole		Anthelmintic
2	Thiabendazole		Anthelmintic
3	Cambendazole		Anthelmintic
4	Parbendazole		Anthelmintic
5	Albendazole		Anthelmintic
6.	Flubenzadzole		Anthelmintic
7	Omeprazole		Anti-ulcer drugs
8	Lansaprazole		Anti-ulcer drugs

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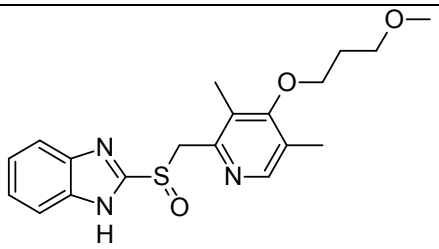
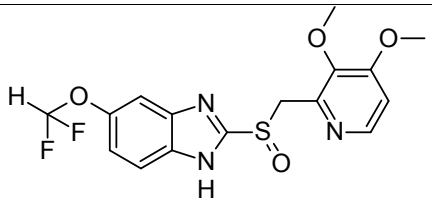
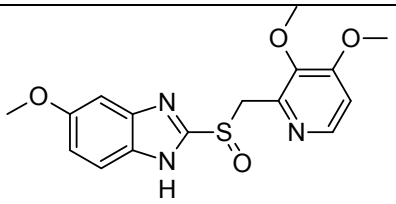
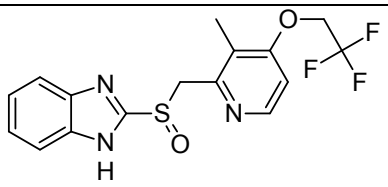
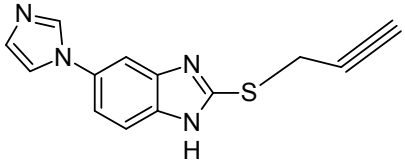
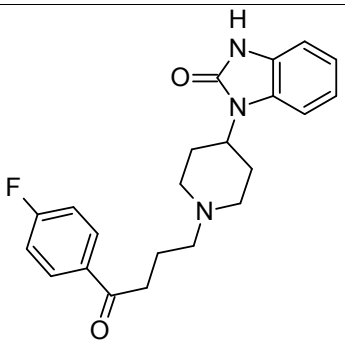
9	Rabeprazole		Anti-ulcer drugs
10	Pantoprazole		Anti-ulcer drugs
11	Esomeprazole		Anti-ulcer drugs
12	Triethoxy-pyridylBenzimidazole derivative		Anti-ulcer drugs
13	Thiophene derivatives of Benzimidazole		Anti-ulcer drugs
14	Droperidol		Anti-psychotic agents

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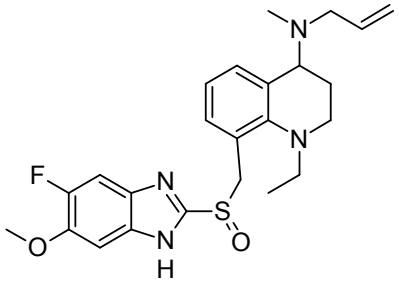
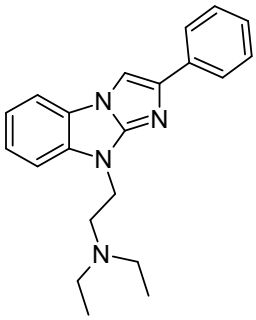
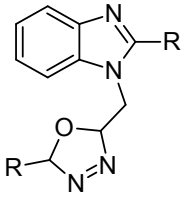
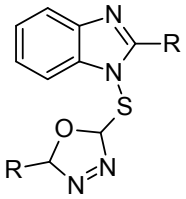
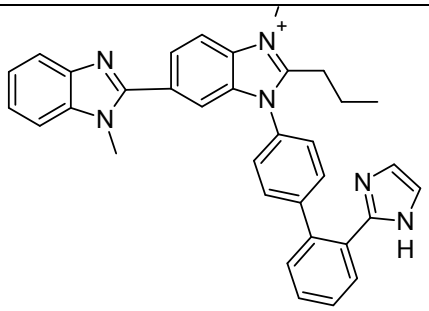
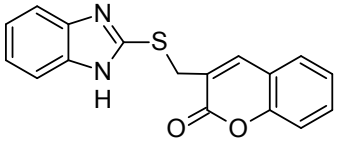
15	QuinolineBenzimidazoleAnalog		Anti-psychotic agents
16	Imidazole derivative with Benzimidazole		Anti-psychotic agents
17	Oxazolederivative with Benzimidazole		Antimicrobial activity
18	Oxazole derivative with Benzimidazoleand thio-linkage		Antimicrobial activity
19	Bibenzoimidazole derivatives		Antagonist
20	Benzimidazoleandcoumarine derivative		Antiseptic virus c activity

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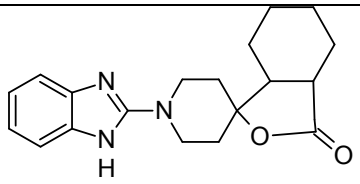
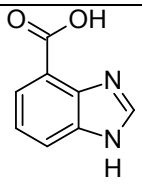
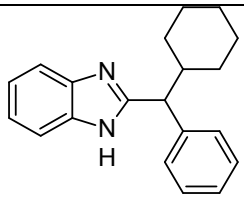
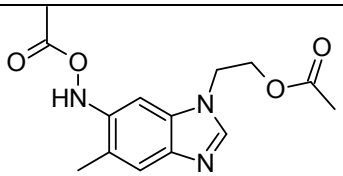
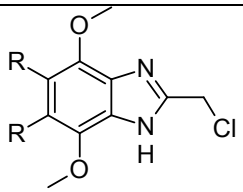
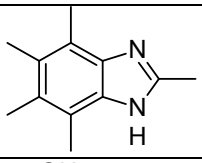
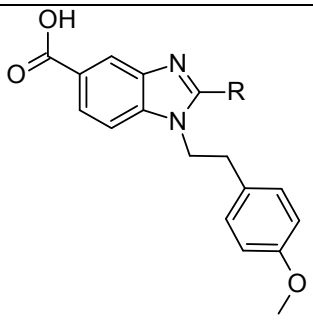
21	Spiro compound of Benzimidazole		NPY N5 Receptor Antagonist
22	4-Carboxylic acid Benzimidazole		Selective 5 HT 4 Antagonist
23	Phenylcylcohexyl derivative of Benzimidazole		Amp Activated protein kinase activator
24	Amide derivative of Benzimidazole		Anticancer activity
25	Substituted Benzimidazole		Anticancer activity
26	Alkylsubstituted Benzimidazole		Antiamoebic activity
27	Benzyl substituted carboxyl Benzimidazole		Antilukamic activity

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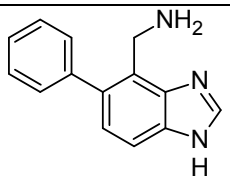
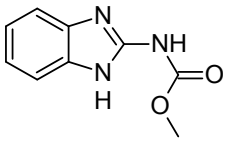
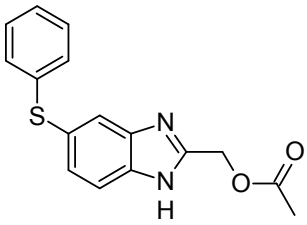
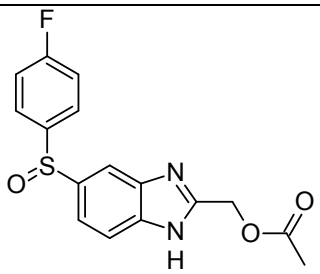
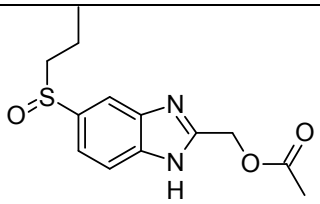
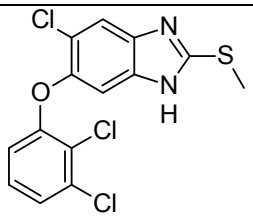
28	Phenyl amine derivative of Benzimidazole		Antidiabetic activity
29	Amide derivative of Benzimidazole		Cyticidal activity
30	Thioether derivatives of Benzimidazole		**Nematicide**and**taenicid**
31	**Oxfendazole**		Roundworms and tapeworms
32	**Ricobendazole**		Anthelmintic
33	Triclabendazole		anthelmintic

II. TYPHONIUM FLAGELLIFORME PLANT

plant is very rare plant. It has very good inflorescence at appearing alongside leaves. The leaves are 5-20cm very thin peduncle. This plant has convolute at base, its flower is globose, green, ovoid, or depressed, measures 1.5-3.5 × 1.2-2cm. This plant has the spadix sub cylindric, slightly fusiform, measures 1.7- 1.9cm × 9-11mm. This plant shows good biological activity mostly on the cure for tumor, asthma, swelling, and as a detoxicant. This plant mostly occurs Sri Lankan, Southeast Asian, Indian countries and it is edible used in these countries. Many times, juice of this plant used for drinking purpose, it is mixed with honey as ayurvedic formulations.

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Synthesis of the plant assisted Nanoparticles by conventional method

This method's basic idea is the precipitation of metal ions with the help of sulphide ions in the solution. In the presence of a plant extract of Typhonium flagelliforme leaves extract, 0.1 M aqueous solutions of sodium sulphite and zinc cacitate dehydrate were combined. First, distilled water was used to produce solutions containing 0.1 M zinc acetatedi hydrate, 0.1 M sodium sulphide, and 1% by weight of an extract of Typhonium flagelliforme leaves. To create a homogenous solution, zinc acetate and Typhonium flagelliforme leaves extract solution were combined and agitated for 30 minutes on a magnetic stirrer. This was followed by the required amount of 0.1 M Sodium Sulphide being added drop by drop while being vigorously stirred for an hour. After centrifugation and washing with distilled water, a precipitate of a white colour was produced. To get a powder sample, the precipitate was dried in an oven at 80°C for four hours. The final product was obtained by calcining the sample at 600 oC for two hours in a furnace.

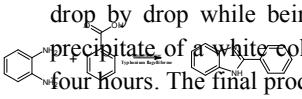

Fig. **Synthesis of the plant assisted Nanoparticles by conventional method**

ISynthesis of the plant assisted Nanoparticles by Sonication

This method's basic idea is the precipitation of metal ions with the help of sulphide ions in the solution. In the presence of a plant extract of Typhonium flagelliforme leaves extract, 0.1 M aqueous solutions of sodium sulphite and zinc cacitate dehydrate were combined. First, distilled water was used to produce solutions containing 0.1 M zinc acetatedi hydrate, 0.1 M sodium sulphide, and 1% by weight of an extract of Typhonium flagelliforme leaves. To create a homogenous solution, zinc acetate and Typhonium flagelliforme leaves extract solution were combined and agitated for 30 minutes on a magnetic stirrer. This was followed by the required amount of 0.1 M Sodium Sulphide being added drop by drop while being vigorously stirred for an hour. After centrifugation and washing with distilled water, a precipitate of a white colour was produced. To get a powder sample, the precipitate was dried in an oven at 80°C for four hours. The final product was obtained by calcining the sample at 600 oC for two hours in a furnace.

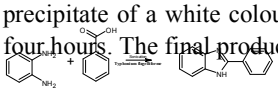

Fig. **Synthesis of the plant assisted Nanoparticles by Sonication**

Synthesis of 2-benzimidazoles derivatives using ZnS NPs as catalyst

Aldehyde (10 mmol) and o-phenylenediamine (10 mmol) were dissolved in 10 ml ethanol in a usual manner. Nanocatalyst was added to the solution along with 0.5 mol percent ZnS (4.1 mg). The mixture was grinded for 10 minutes at room temperature (25°C) using Pristle and mortar The reaction mixture was dumped into ice-cold water after the reaction was completed as shown by the TLC. The solid was filtered, and the result was washed twice with water, dried, and purified by ethanol Recrystallization.(Fig.1)

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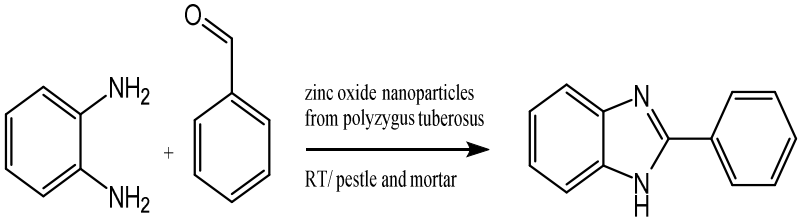

III. RESULTS AND DISCUSSION

3.1 X-ray powder diffraction

Figure 2 presents the x-ray diffraction patterns of pristle and morter were synthesised ZnS in comparison with that of commercial ZnS. The peak positions for both the ZnS particles are at values of 28.6, 47.5 and 56.3 corresponding to the (111), (220) and (311) planesθ2 respectively confirmed the crystallisation of ZnS in cubic blende structure. (Black line for the Pure ZNS nanoparticles, Red line for the plant assisted nanoparticles using the Conventional method ,and sonochemical method (blue line)

Spectra of XRD of ZnS nanoparticles.

3.2 FTIR SPECTRUM

The FT-IR spectroscopy experiments were carried out to investigate the plausible process behind the creation of these biosynthesized Zinc sulphude NPs and to learn more about the functional groups on the nanoparticles' surfaces. The presence of sulphide linkage, which could be from the oxide form of a solution, the metal precursor involved in biosynthesized Zinc sulphude NPs, is represented by the very strong absorption peaks at 3200,1560,1428,1037 and 678.Procedure of synthesis In the nanoparticle spectrum, the presence of a large peak at wavenumber 3200 reveals the e xistence of phenolic sulphidegroup (SH), which symbolizes the stretching vibration due to the existence of hydrogen bo nds from flavonoids and tannins. The presence of free catechin is shown by the peak of 678cm1, reflecting the aromatic ring CH vibrations. This shows that some polyphenolic components are attached to biosynthesized Zinc suphide NPs.

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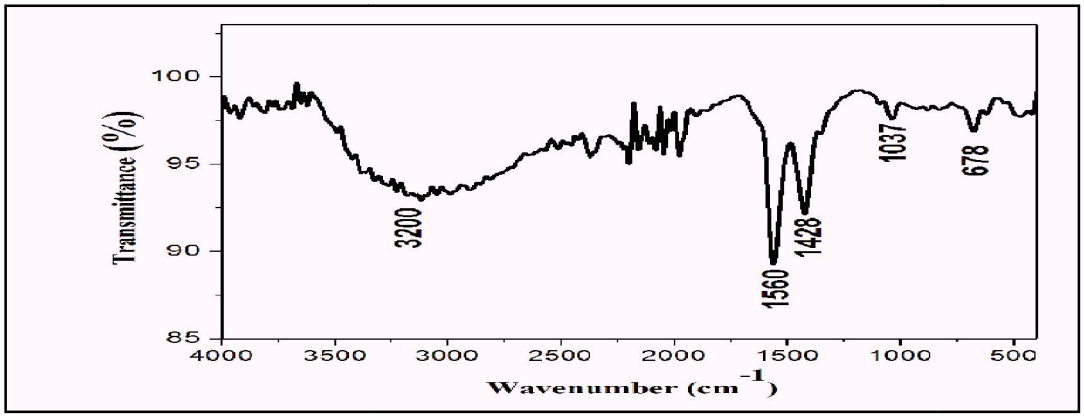

Fig.3. FTIR pattern of Zinc suphide nanoparticles

UV Spectra

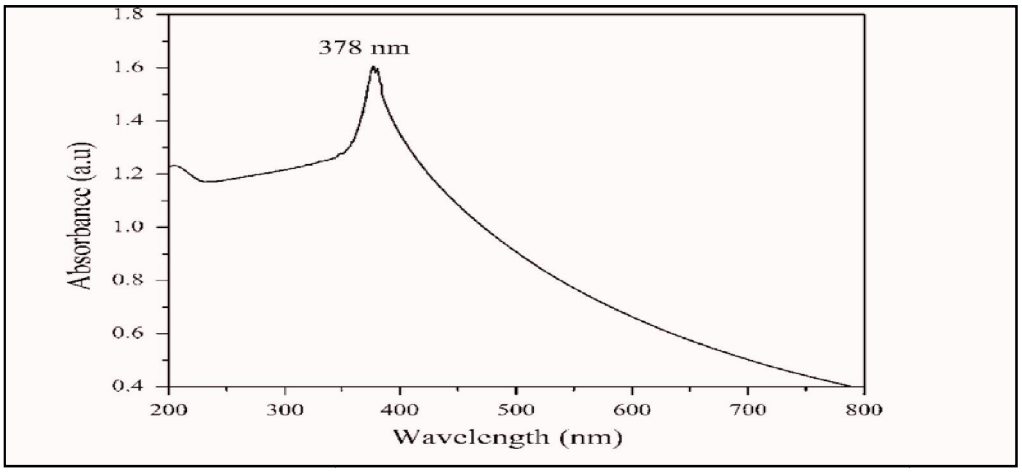

Fig.4. UV-Vizspectra of Zinc sulphude nanoparticles

UV-Vis spectroscopy was also performed to confirm the formation of Zinc suphide NPs further. The absorption spectrum of synthesized Zinc suphide NPs was shown in Fig. 4. The UV-Vis measurement was performed after the Zinc suphide NPs were dispersed in ultrapure water. The absorption peak was observed at 367 nm. Furthermore, the absorption peak of Zinc suphide NPs also confirmed the properties of Zinc suphide NPs, which are known for UV protections in sunscreens products[25].

3.4 Scanning Electron Microscope (SEM)

Scanning electron microscopy Fig. 6 was used for diffing the size of the nanoparticles. It indicates that the synthesized plant-assisted nanoparticles hasa 50-60 nm particle size.

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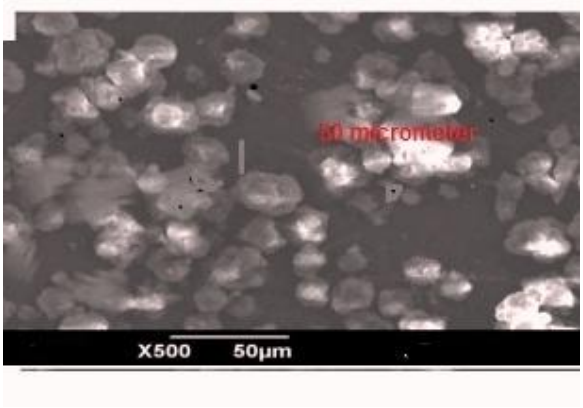

Fig.6. Image of SEM analysis

Spectral Analysis Pale yellow solid; m.p. 224 °C.
1H NMR (400 MHz, DMSO-d6) δ ppm (J, Hz): 12.86 (br s, 1H), 8.20 (d, J = 7.2 Hz, 2H), 7.67 (s, 2H), 7.27-7.02 (m, 4H), 3.83 (s, 3H);

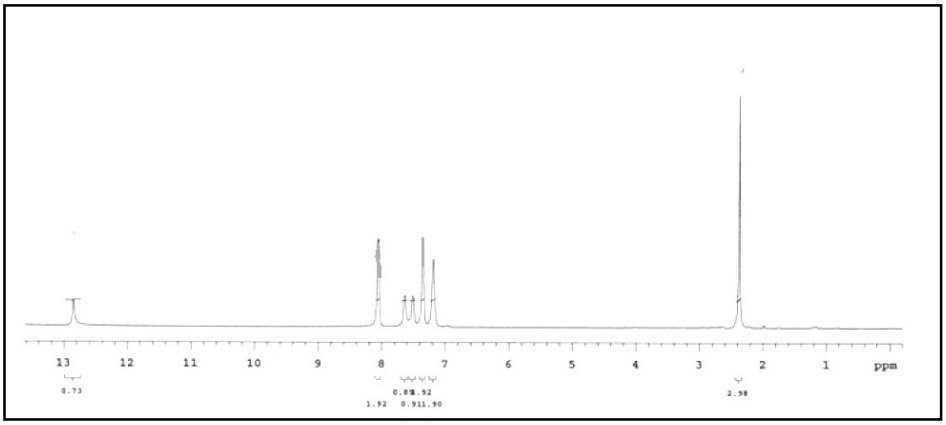

Fig.7. ^{1}H NMR Spectra of 2-(4-Methoxyphenyl)-1*H*-benzimidazole

13C NMR (400 MHz, DMSO-d6) δ ppm: 162.6, 153.4, 126.0, 121.4, 123.7, 116.4, 112.3, 109.2, 55.3, 13.70.

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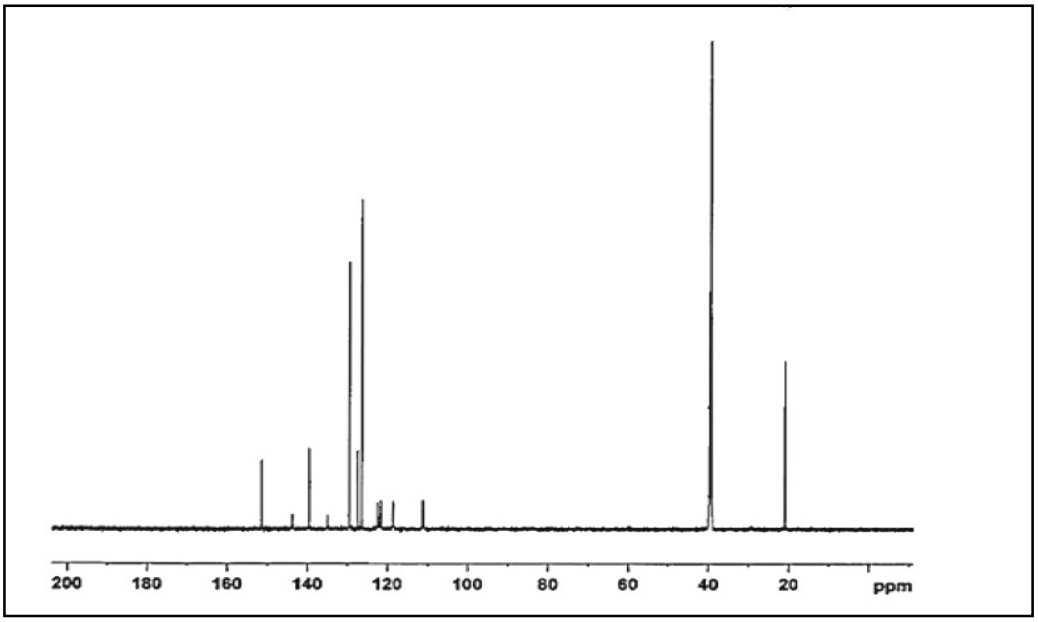

Fig.8. ^{C13} NMR Spectra of 2-(4-Methoxyphenyl)-1*H*-benzimidazole

IR Spectrum of Benzimidazole Derivative

Aromatic C-H bending occur at the 939-789 cm -1.the -OCH3 occur at the 1060.5 cm -1 .N-H Proton occur at the 3485 CM-1 at the 2934 cm-1 occur at the shows presence of ether and aromatic range. C=C bond occur at the 1654 cm -1. this indicate the presence of the Functional Groups

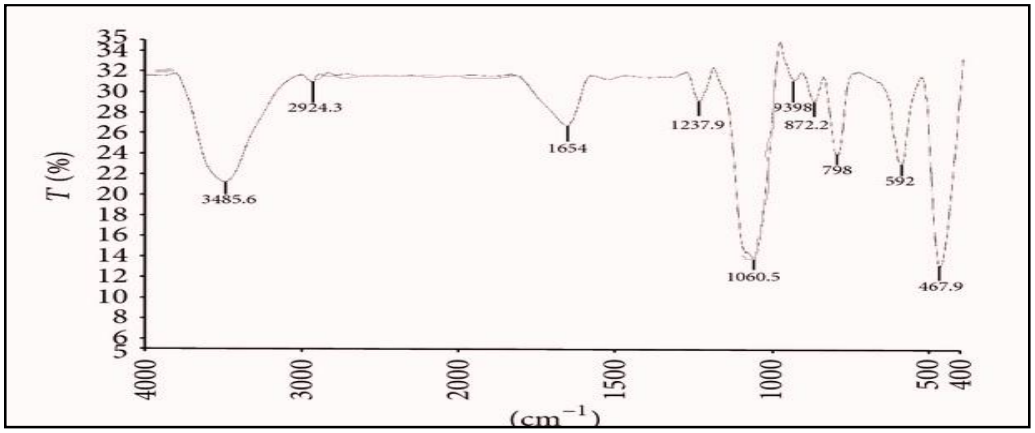

Fig.9. FTIR Spectrum of 2-(4-Methoxyphenyl)-1*H*-benzimidazole

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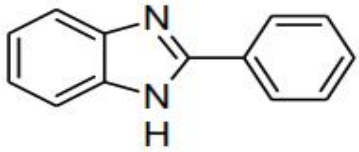
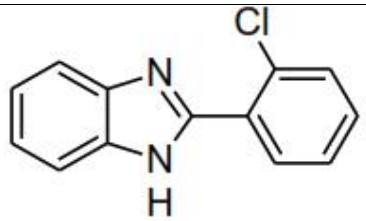
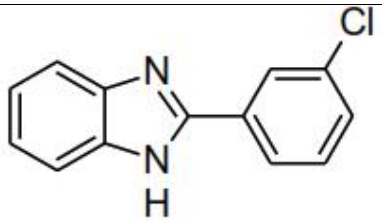
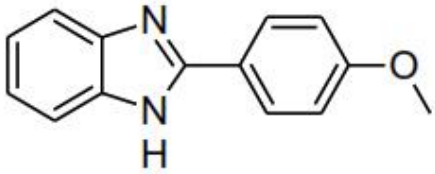
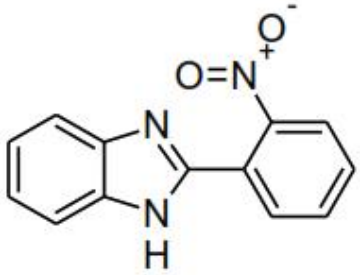
Table:2 . Reaction time,% yield and Melting point of Benzimidazole Derivatives.(Sonication Method)

Sr.No	Benzimidazole derivatives	Reaction Time (Min.)	(%) Yield	MP(^{0}C)
1		25	87	296
2		22	94	135
3		22	96	229
4		52	96	226
5		22	98	257

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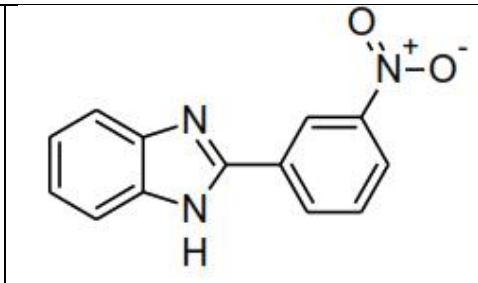
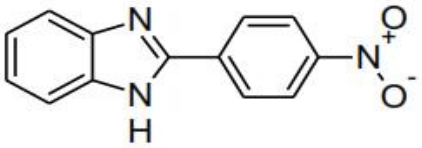
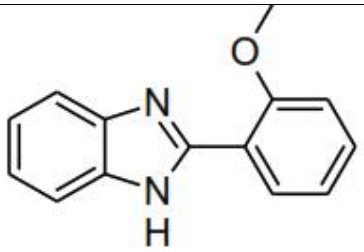
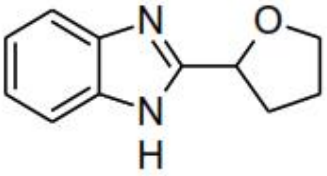
6		22	98	306
7		22	96	305
8		55	90	269
9		42	90	286

Table 3 Catalytic Activity Comparison of the Various Catalyst

Entry	Acid	Amount of acid (mL)	t (min)	Yield (%)
1	None	0	10	20
2	H2SO4	0.5	10	10
3	TsOH	0.5	10	Trace
4	HNO3	0.5	10	25
5	HCl	0.5	10	30
6	CF3COOH	0.5	10	36
7	HCOOH	0.5	10	41
8	Zinc sulphide Nanoparticles	0.2 gm	06 min	87

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IV. CONCLUSION

We investigated Benzimidazole by using plant-assisted zinc Sulphide nanoparticles to react orthophenylenediamine and different aromatic aldehydes at room temperature. This synthesis uses a variety of aromatic aldehydes. The aldehydes with electron-withdrawing groups had faster reactions than the aldehydes with electron-donating groups, according to our findings. The synthesized organic compounds were validated using NMR and IR; the study reveals that the catalyst had an effect on the synthesis of the product. We discovered that carrying out a reaction in a controlled environment without a catalyst takes longer and yields less. For the comparison, we utilize 2-nitrobenzaldehyde. This reaction takes more than five hours and yields 40%. We employed a total of 0,0.1,0.15, 0.2, and 2.5g of catalyst to adjust the amount of catalyst. Finally, keep in mind that the reaction takes the least amount of time and yields 0.2g of nanocatalyst.

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