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(RESEARCH ARTICLE)

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Synthesis of Triphenyl Imidazole by Green chemistry approach

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Abstract

Synthesis of 2,3,5- Triphenyl imidazole is carried out by conventional method/ traditional method as well as green chemistry in the present study. Green chemistry is also known as micro-wave assisted synthesis. Green chemistry is the design of chemical products and process that eliminates the use and generation of hazardous substances. Using green solvent, like water, synthesis of biologically active moiety with high percentage yield as well as purity. The Percentage yield of 2,3,5- Triphenyl imidazole obtained by Green Chemistry Approach is 90.90% whereas Conventional/Traditional method produces only 69.60% of 2,3,5- Triphenyl imidazole constant concentration of all organic reagents. Hence, we concluded that green chemistry approach is environment friendly.

Keywords: 2,3,5- Triphenyl imidazole; Conventional method; Green chemistry; Percentage yield; UV spectrophotometer

1. Introduction

Micro-wave assisted synthesis is defined as invention, design, development, application of chemical products and processes to reduce or to eliminate the use and generation of substances hazardous to human health and environment. A microwave is a form of electromagnetic energy that falls at the lower frequency of the electromagnetic spectrum in the range of 300 to 300000MHz. Within in this region of electromagnetic energy only molecular rotation is affected not the molecular structure. However, for their use in laboratory reaction a frequency of 2.45GHz is preferred because it has the right penetration depth for laboratory reaction condition [1].

1.1. Principles of Microwave assisted reaction

Preventing trash is more effective than treating or cleaning it up after it has occurred. Synthetic approaches should include all components employed throughout the procedure into the finished result. Wherever practicable synthetic methodologies should be designed to use and generate substances that pose little or no toxicity to human health and the environment. Microwave irradiation has gained popularity in the last decade as a powerful tool for rapid and efficient synthesis of a variety of compounds because of selective absorption of microwave energy by molecules. This phenomenon is dependent on the ability of a specific material to absorb microwave energy it into heat. Microwave passes through material and causes oscillation of molecule which produces heat. Micro wave heating process heat in the entire material in the same rate and the same time at a high speed and at a high rate of reaction. Micro wave heating is the best process due to the microwave couple directly with the molecule that are present in the reaction mixture, leading to fast rising temperature, faster reaction and cleaner chemistry. The microwave chemistry is also called as green chemistry because it does not produce any hazardous material like gas, fumes, heating etc. [2].

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1.2. Green approaches in organic synthesis

The concept of green chemistry introduced the environmentally benign synthetic protocols for the synthesis of heterocycles that has had a significant impact in many fields, such as the use of green solvents, solvent-free synthesis, sustainable catalytic materials, reduced energy consumption, improved atom economy, optimized reaction yields, the use of alternative energy sources, the introduction of multicomponent reactions ionic liquids and the design of high-efficiency and time-saving reactions that work at ambient temperatures. Pollution and an increase in energy demands prompted the design of novel synthetic protocols to fulfil the requirements of green and sustainable chemistry to promote the synthesis of organic products in an ecofriendly environment [3].

1.3. Mechanism of Microwave Heating

All the materials are not susceptible to microwave heating as response of various materials to microwave radiation is diverse. Microwave absorbing materials (e.g. water) are of utmost important for microwave chemistry and three main different mechanisms are involved for their heating namely [4].

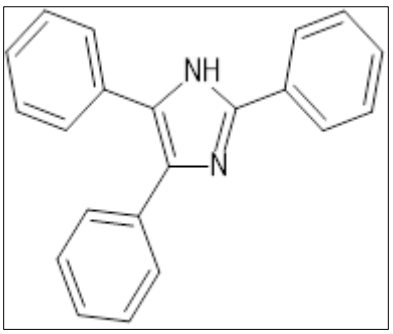
- **Dipolar polarization:** For a substance to be able to generate heat when irradiated with microwaves it must be a dipole. i.e. its molecular structure must be partly positively charged. Since the microwave field is oscillating field. This alignment causes rotation, which results in friction and ultimately in heat energy [5].
- **Ionic conduction:** During ionic conduction dissolved (completely) charged particles (usually ions) oscillate back and forth under the influence of microwave irradiation. This oscillation causes collisions of the charged particles with neighboring molecules or atoms, which are ultimately responsible for creating heat energy [6].
- **Interfacial polarization:** The interfacial polarization method can be considered as combination of both the conduction and dipolar polarization mechanisms. It is important for heating systems that comprise a conducting material dispersed in a non-conducting material [7]

2. 2, 4, 5 -TRIPHENYL IMIDAZOLE

Imidazole is a heterocyclic compound, and these activities are important for biological activity. Imidazole is synthetic with naturally occurring derivatives. Derivative of imidazole is used for anti- cancer, anti-inflammatory action. Triphenyl imidazole was discovered in 1877. The crystal structure of 36 ancestral lophine is known; the three phenyl rings are bonded to imidazole, and they are not coplanar. This phenyl ring attached to the 2,4,5 position of imidazole ring [8].

2.1. Chemical structure

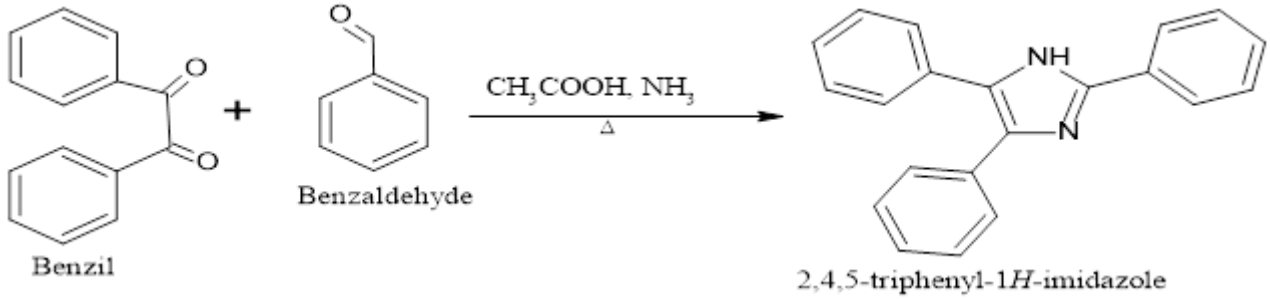
- The 2,4,5-triphenylimidazole molecule is present in 42 bonds these molecules were divided in the Following way [9].
- 1 Five-membered ring
- three six-membered rings
- 23 aromatic bonds
- 3 rotatable bonds
- 23 multiple bonds



2,4,5-triphenyl-1*H*-imidazole

Molecular Formula:	$C_{21}H_{16}N_2$
Formula Weight:	296.36514
Composition:	C(85.11%) H(5.44%) N(9.45%)
Molar Refractivity:	92.55 ± 0.3 cm^3
Molar Volume:	256.8 ± 3.0 cm^3
Parachor:	681.1 ± 4.0 cm^3
Index of Refraction:	1.640 ± 0.02
Surface Tension:	49.4 ± 3.0 dyne/cm
Density:	1.153 ± 0.06 g/cm^3
Dielectric Constant:	Not available
Polarizability:	36.69 ± 0.5 $10^{-24}cm^3$
RDBE:	15
Monoisotopic Mass:	296.131349 Da
Nominal Mass:	296 Da
Average Mass:	296.3651 Da
M+:	296.1308 Da
M-:	296.131897 Da
[M+H]+:	297.138625 Da
[M+H]-:	297.139722 Da
[M-H]+:	295.122975 Da
[M-H]-:	295.124072 Da

2.2. Preparation of 2,4,5-triphenylimidazole by traditional method from Benzil and Benzaldehyde [10]



2.2.1. Procedure by traditional method [11]

Place 2.5 gm of Benzil, 1.5 ml of Benzaldehyde and 5 gm of Ammonium acetate to a 250 ml round bottom flask add 40 ml of glacial acetic acid heat the reaction mixture on water bath at 100°Cfor 5-24 hours by connecting water condenser. Cool the flask to room temperature and pour the reaction mixture into 100 ml of cold water and filter the product under suction to remove an insoluble by-product. Recrystalize the product with methanol.

2.2.2. Calculation of percentage yield by conventional method

- Molecular weight of Benzil = 210.23 gm/mol
- Molecular weight of 2,3,5-Triphenyl imidazole = 296.4 gm/mol
- Weight of reactant taken (Benzil) = 2.5 gm
- Practical yield = 2.45 gm

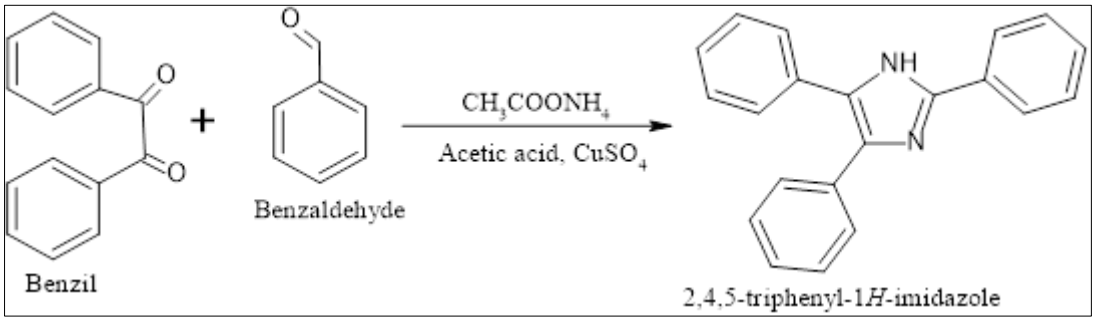
$$Theoretical\ yield = \frac{Molecular\ weight\ of\ Triphenyl\ imidazole\ X\ Weight\ of\ Benzil\ taken}{Molecular\ weight\ of\ Benzil}$$

$$= \frac{296.4\ gm/mol\ X\ 2.5\ gm}{210.23\ gm/mol} = 3.52\ gm$$

$$Percentage\ yield = \frac{Practical\ yield}{Theoretical\ yield}\ X\ 100$$

$$= \frac{2.45}{3.52}\ X\ 100 = 69.60\%$$

2.3. Preparation of 2,4,5-triphenylimidazole by green chemistry approach from Benzil, Ammonium acetate and Benzaldehyde [12]



2.4. Procedure by green chemistry approach [13]

In a 250 ml round bottom flask place 2.5 gm of Benzil, 1.5 ml of Benzaldehyde and 5 gm of Ammonium acetate and add 40 ml of glacial acetic acid. 2% Copper sulphate was prepared separately and 2.5 ml was added to the flask. The mixture in flask was stirred in circumfluence for 2 hours. Reaction mixture was cooled to 50-60℃. And pour the reaction mixture into 125 ml of water and mix carefully. Allow the reaction mixture to stand for 15 minutes. And then filter the product under suction to remove an insoluble by-product. Recrystallise the product with methanol.

2.5. Calculation of percentage yield by green chemistry approach

- Molecular weight of Benzil = 210.23 gm/mol
- Molecular weight of 2,3,5-Triphenyl imidazole = 296.4 gm/mol
- Weight of reactant taken (Benzil) = 2.5 gm
- Practical yield = 3.20 gm

$$Theoretical\ yield = \frac{Molecular\ weight\ of\ Triphenyl\ imidazole\ X\ Weight\ of\ Benzil\ taken}{Molecular\ weight\ of\ Benzil}$$

$$= \frac{296.4\ gm/mol\ X\ 2.5\ gm}{210.23\ gm/mol} = 3.52\ gm$$

$$Percentage\ yield = \frac{Practical\ yield}{Theoretical\ yield}\ X\ 100$$

$$= \frac{3.20}{3.52}\ X\ 100 = 90.90\%$$

3. Identification and characterization

The synthesized compounds were characterized by using following techniques to confirm the formation of the product [14].

3.1. Melting point determination

The melting point of organic compounds were determined by open capillary tube method. Melting point is a valuable criterion of purity for an organic compound as a pure crystal is characteristic of having definite and sharp melting point. The purity should not be assumed but must establish by observation of any changes in the melting, when the compound is subjected to purification by recrystallization. Melting point of the compounds were reported in the table 1.

3.2. Solubility

The solubility of synthesized compounds was tested in various solvents. Compounds were soluble in Methanol.

3.3. Thin layer chromatography

Chromatography is an important technique to identify the formation of new compounds and also to determine the purity of the compound. The Rf value is characteristic for each of the compound [15].

Preparation of solvent system and saturation of chamber

The solvent system used for the development of chromatogram was prepared by mixing, Methanol: Chloroform: Acetic acid glacial (7:2:1)

3.4. Development of Chromatogram

Plates were developed by ascending technique when solvent front had reached a distance of 8-10 cm; they were taken out and dried at room temperature.

3.5. Detection of spots

The developed spots were detected by using UV Chamber.

3.6. Calculation of R_f values

The R_f values of compounds were calculated using the formula.

R_f value = Distance travelled by sample/Distance travelled by solvent front.

In all the cases the distance travelled by the sample was found to be different from that of the parent compound spotted along with it. Thus, confirming the fact that the compounds formed is entirely different form that of the parent compound. Since all the sample gave a singles spot, the compounds were taken to be free from impurities. The R_f values of compounds were reported in table.

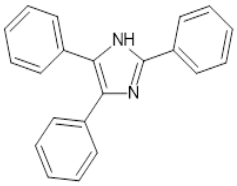
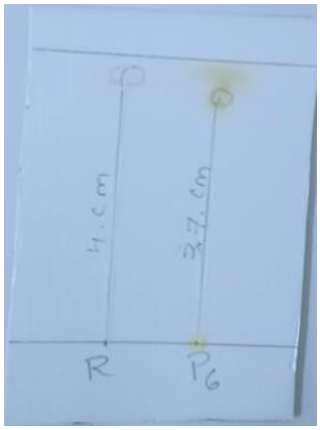
3.7. Ultra-violet and Visible Spectroscopy (UV)

UV-Visible spectroscopy is an analytical technique that measures the amount of discrete wavelengths of UV or visible light that are absorbed by or transmitted through a sample in comparison to a reference or blank sample. This property is influenced by the sample composition, potentially providing information on what is in the sample and at what concentration. The UV spectra of synthesized compounds were recorded by using methanol as solvent in the range of 200-800 nm and observed their absorbance and wavelength listed in the table 2 and UV spectra is shown in Figure 1.

4. Result and discussion

Table 1 Physico-chemical properties of 2,4,5-triphenyl-imidazole by traditional method

2,4,5-triphenyl-imidazole by traditional method	
Chemical name	2,4,5-Triphenyl-1H-imidazole
Solubility	Soluble in Methanol
Molecular Formula	$C_{21}H_{16}N_2$
Molecular Weight	296.4 g/mol
Melting Point	272-274℃
Theoretical yield	3.52 gm
Practical yield	2.45 gm
Percentage yield	69.60%
TLC Solvent system	Methanol: Chloroform: Acetic acid glacial (v/v/v 7:2:1)
R_f value	$Rf = \frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$ R_f of Reactant (Benzil) = 4.5/4.8=0.93 R_f of Product (triphenyl-imidazole) = 4.1/4.8=0.85

	
Physical state	Yellowish white (Crystaline)
2,4,5-triphenyl-imidazole by green chemistry approach	
Structure	
Melting Point	276℃
Theoretical yield	3.52 gm
Practical yield	3.20 gm
Percentage yield	90.90%
TLC Solvent system	Methanol: Chloroform: Acetic acid glacial (v/v/v 7:2:1)
R_f value 	$Rf = \frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$ R_f of Reactant = 4.0/4.9=0.81 R_f of Product = 3.7/4.9=0.75
Physical state	Yellowish white (Crystaline)

4.1. Analytical data of synthesized compounds

4.2. UV Spectra

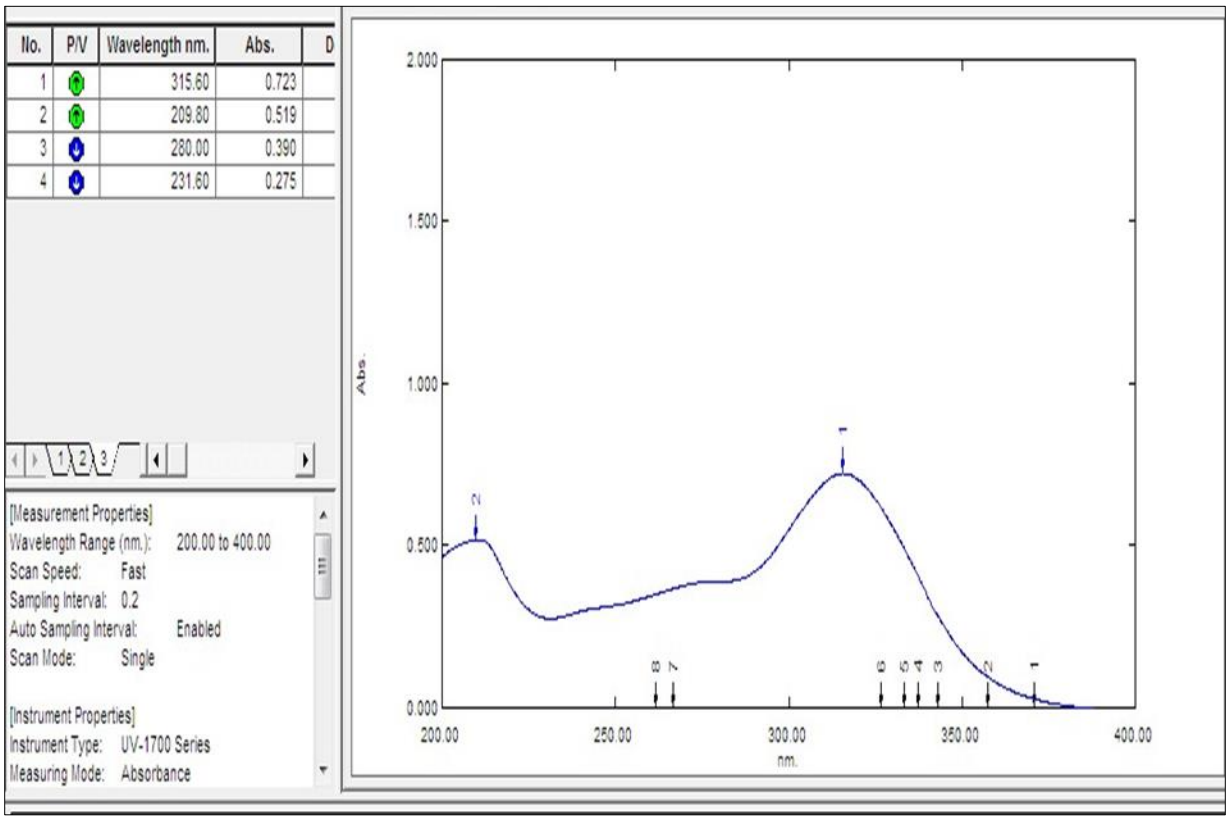


Figure 1 UV Spectra of 2,4,5-triphenyl-imidazole

4.3. UV-Data

Table 2 UV Data of 2,4,5-triphenyl-imidazole

Wavelength in nm	Absorbance
315.60	0.723

5. Summary and conclusion

The present work is to synthesize Triphenyl-imidazole by conventional method and micro-wave assisted method and comparison of results obtained from two different route of synthesis of Triphenyl-imidazole.

The synthesized compounds were characterized by various physico-chemical parameters and by spectral studies, which includes solubility, physical state, color, odour, melting point, thin layer chromatography and UV Spectrophotometer.

Triphenyl-imidazole is soluble in methanol, physical state is crystalline, color- Yellowish white, odour is pungent, melting point found at 272-274°C, Rf value is found at 0.85 and UV data showing absorbance 0.723 at wavelength 315 nm. The Percentage yield of Triphenyl-imidazole obtained by Green Chemistry Approach is 90.90% whereas Conventional/Traditional method produces only 69.60% Triphenyl-imidazole constant concentration of all organic reagents. Hence, we concluded that green chemistry approach is environment friendly.

Triphenyl-imidazole is synthesized by application of principle of green chemistry with high purity profile as well as having safety by omitting the use of ethanol. Water a green solvent is used instead of ethanol in synthesis of Triphenyl-imidazole. There is reduction in time and ultimately cost as compare to conventional procedure of synthesis of Triphenyl-imidazole. Thus, we conclude that the synthesized compound has potential in the green chemistry.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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Author's short Biography

Ms. Vasantha T S is graduated from Rajiv Gandhi University of Health Sciences, Bangalore, India and post graduated from Adichunchanagiri University, B G Nagara, taken specialization in Pharmaceutical Chemistry, completed master thesis in "Design, Synthesis and Pharmacological Evaluation of Benzimidazole- Methylamine Bridged Phenyl-1,3,4-Thiadiazolamine Derivatives". Currently working as an Assistant Professor at Varadaraja Institute of Pharmaceutical education and Research, Tumkur. She also guiding Under graduate pharmacy students.