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Comparison Study of Synthesis of some Aromatic Compounds Using Conventional and Microwave Method

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Abstract:
In this study, four aromatic compounds—benzylidene aniline, acetanilide, and (N)-cinnamylidene aniline-benzimidazole—were prepared and their physical properties (Molecular weight, Melting point, Density, Solubility, Reaction time, and Yield). The conventional solvent method was evaluated for benzylidene aniline yielded 88% with a reaction time of 30 min, while the microwave method significantly improved the efficiency, producing a 97% yield in just 2 min, with no changes in melting point or density. Infrared spectra confirmed the presence of characteristic C–H, C=C, and C=N vibrations. For acetanilide, the conventional method yielded 86% in 20 min, while microwave irradiation increased the yield to 95% with a shorter reaction time of 3 to 5 min. The melting point and density were almost identical in both methods, confirming the purity of the product. Infrared spectral data supported the successful formation of acetanilide through the characteristic stretching vibrations of N–H, C–H, and C=O. For N-cinnamylidene aniline, the conventional solvent-based method achieved a yield of 77%, with a melting point between 175 and 176.5 °C and a density of 0.93 g/cm^3. Vibrations were found at characteristic bands for the benzamidazole compound at 1620 cm^{-1} (C=N) and 1600–1450 cm^{-1} (aromatic C=C), the yield was 75% using the conventional method, while the yield was 91% using the microwave method, with a reduced time of only 15 minutes. The IR spectrum showed two peaks at 3400–3300 cm^{-1} (N–H) and 1620–1600 cm^{-1} (C=N), confirming the formation of the expected product. Overall, the results demonstrate that microwave-assisted synthesis outperforms conventional solvent-based methods, providing higher yields and shorter reaction times while maintaining similar physical and spectral properties of the final products.

Keywords: Benzimidazole, Microwave, Conventional, Melting Point, Benzylidene Aniline.

دراسة مقارنة لتحضير بعض المركبات العطرية باستخدام الطريقة التقليدية والميكروويف

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الملخص
في هذه الدراسة، تم تحضير أربع مركبات عطرية - بنزيليدين أنيلين، وأسيتانيليد، و(N)-سيناميليدين أنيلين-بنزيميدازول - وتم تقييم خصائصها الفيزيائية (الوزن الجزيئي، ونقطة الانصهار، والكثافة، والذوبان، وزمن التفاعل، والعائد). وقد أعطى المذيب التقليدي الذي تم تقييمه لبنزيليدين أنيلين 88٪ مع زمن تفاعل قدره 30 دقيقة، بينما حسّنت طريقة الميكروويف الكفاءة بشكل كبير، حيث أنتجت عائدًا بنسبة 97٪ في دقيقتين فقط، دون أي تغييرات في نقطة الانصهار أو الكثافة. أكدت أطياف الأشعة تحت الحمراء وجود اهتزازات مميزة لـ C–H وC=C وC=N. بالنسبة للأسيتانيليد، أعطت الطريقة التقليدية 86٪ في 20 دقيقة، بينما زاد إشعاع الميكروويف من العائد إلى 95٪ بوقت تفاعل أقصر من 3 إلى 5 دقائق. كانت نقطة الانصهار والكثافة متطابقة تقريبًا في كلتا الطريقتين، مما يؤكد نقاء المنتج. دعمت بيانات طيف الأشعة تحت الحمراء نجاح تكوين الأسيتانيليد من خلال اهتزازات التمدد المميزة لـ N–H وC–H وC=O. بالنسبة لـ N-سيناميليدين أنيلين، حققت الطريقة التقليدية القائمة على المذيبات عائدًا بنسبة 77%، مع درجة انصهار تتراوح بين 175 و176.5 درجة مئوية وكثافة 0.93

غ/سم^{3}. وُجدت اهتزازات عند نطاقات مميزة لمركب البنزاميدازول عند 1620 سم^{1} (C=N) و1450-1600 سم^{1} (C=C عطري)، وبلغ العائد 75% بالطريقة التقليدية، بينما بلغ 91% بطريقة الميكروويف، مع وقت أقل قدره 15 دقيقة فقط. أظهر طيف الأشعة تحت الحمراء قمتين عند 3300-3400 سم^{1} (N–H) و1600-1620 سم^{1} (C=N)، مما يؤكد تكوين المنتج المتوقع. بشكل عام، تُظهر النتائج أن التركيب بمساعدة الموجات الدقيقة يتفوق على الطرق التقليدية القائمة على المذيبات، حيث يوفر إنتاجية أعلى وأوقات تفاعل أقصر مع الحفاظ على خصائص فيزيائية والطيفية مماثلة للمنتجات النهائية.

الكلمات المفتاحية: بنزيميدازول، ميكرويف، تقليدية، درجة انصهار، بنزيليدين انبلين.

Introduction

Aromatic compounds are among the most important organic molecules, characterized by their resonance stability and structural robustness, and are widely used as solvents and raw materials in various chemical and biochemical industries [1,2]. Heterocyclic compounds, which contain one or more non-carbon atoms in the ring, play an important role in biological processes such as vitamins, nucleic acids, and coenzymes, and are also essential components of many pharmaceuticals, pesticides, and industrial polymers [3,4].

Schiff bases, first discovered by Hugo Schiff in 1864, are compounds characterized by a carbon-nitrogen double bond and are typically formed by the condensation of primary amines with carbonyl compounds [5]. Their ease of synthesis and versatility in structure make them effective ligands for transition metal complexes. Schiff bases and their metal complexes have been used in diverse fields, including organic and biocatalysis, fungal and bacterial photochemical reactions, the development of anticancer agents, and environmental applications such as selective sensors for organic pollutants [5, 6, 7, 8].

In the context of green chemistry, there is growing interest in synthesis methods that reduce chemical waste and use environmentally friendly methods, such as microwave-assisted synthesis, which can provide economic and environmental benefits [1,3,5].

Methods

In the present study, three organic compounds of Schiff bases were synthesized: (Benzylidene Aniline, Acetanilide, and N-(3-phenylallyl) aniline). And one organic compound was Benzamidazole.

Compound I: Synthesis of Benzylidene Aniline.

Benzylidene aniline is a yellowish-brown compound with the chemical formula $C_{12}H_{12}N$ and a molecular weight of 181.23 g/mol.

Experimental Procedure:

Benzylidene aniline was prepared using two methods:

A. **Solvent-free synthesis:**
   4 mL of benzaldehyde was pipetted and placed in a small bottle pipette and placed, then 4 mL of aniline was added. The mixture was gently stirred with a glass rod and heated in a water bath for 30 minutes. The reaction mixture was then cooled in an ice bath to obtain the crude product. The product was weighed and recrystallized by dissolving in ethanol and left for 12 hours to allow crystal formation. After recrystallization, the product was weighed again and some of its physical properties were measured.
B. **Microwave-assisted synthesis:**
   Using the same quantities of benzaldehyde and aniline, the mixture was stirred and placed in a microwave for 1.9 seconds. The sample was then cooled, recrystallized, weighed, and its physical properties were recorded. The yield of the product was also determined.

Compound II: Synthesis of Acetanilide (C_8H_9NO)

Acetanilide is an odorless solid organic compound with the molecular formula **C_8H_9NO** and a molar mass of **135.17 g/mol**. It appears as a white to light gray crystalline solid, slightly soluble in water but readily soluble in organic solvents such as ethanol and ether. It has a melting point in the range of **114–116 °C**, indicating good stability under normal conditions.

Experimental Procedure:

A. **Conventional method:**
   A **3 mL** portion of aniline was placed in a round-bottom flask, followed by the addition of **5 mL of acetic anhydride** and **5 mL of glacial acetic acid**. A reflux condenser was attached to the flask, and the mixture was heated over a Bunsen flame for **20 minutes**. Upon completion, the contents were allowed to cool and then poured into a beaker containing **30 mL of sodium hydroxide solution** with continuous stirring,

resulting in the formation of white acetanilide crystals. The crystals were collected by filtration using a **Büchner funnel** and recrystallized from **50 mL of distilled water**. Finally, the physical properties of the product were measured, and the percentage yield was determined.

B. Microwave-Assisted Method:

3 mL of aniline was placed in a porcelain crucible, and 5 mL each of acetic anhydride and glacial acetic acid were added. The mixture was covered with a microwave- safe lid and heated in a microwave oven at medium power (≈ 350 W) for 3–5 minutes. After cooling to room temperature, the reaction mixture was added to 30 mL of sodium hydroxide solution with continuous stirring, resulting in the formation of white acetanilide crystals. The crystals were filtered using a Buchner funnel and purified by recrystallization from 50 mL of hot distilled water to afford pure acetanilide.

Compound III: Synthesis of N-[(E)-3-phenylprop-2-en-1-ylidene] aniline.

Common name is Cinnamylideneaniline ($C_{15}H_{13}N$, M.wt. 207.27 g/mol, red crystalline Schiff base with low water solubility and good solubility in organic solvents.

Experimental Procedure:

A- Conventional method:

10 mL of cinnamaldehyde was placed in a 100 mL round-bottom flask, and 25 mL of absolute ethanol was gradually added with vigorous stirring. Five drops of glacial acetic acid were then added, followed by the dropwise addition of 10 mL of aniline with continuous stirring to ensure homogeneity of the reaction mixture. Dark red crystals were formed, which were collected by filtrate ion, dried, and recrystallized.

B- Microwave Synthesis:

10 ml of cinnamaldehyde were placed in a suitable beaker, and 25 ml of absolute ethanol were added while stirring with a magnet to ensure homogeneity. Five drops of glacial acetic acid were then added, followed by 10 ml of aniline. The reaction time was 5–6 minutes at a temperature of 40–50°C. Dark red crystals formed, which were collected by filtration and dried.

Compound IV- Synthesis of Benzimidazole.

Benzimidazole is a unique compound in medicinal chemistry. It is a highly crystalline substance with a melting point of 169-171°C. It is a non-regenerating bicyclic compound, resulting from the fusion of a benzene and an imidazole ring. It has amphoteric (acidic and basic) properties and is applicable in polar solvents such as alcohols and aqueous solutions of strong acids and alkalis.

Experimental Procedure.

A- Conventional method:

O-phenylenediamine (1.9 g) was mixed with formic acid (7.5 mL) in a 100 mL round-bottomed flask equipped with a magnetic stirrer and a reflux condenser. The mixture was heated to 100–105 °C under continuous stirring for 2 hours. After the reaction was complete (monitored by thin-layer chromatography (TLC) and the disappearance of the starting material), the mixture was allowed to cool to room temperature and then diluted with 40 mL of distilled water. The medium was gradually adjusted with saturated sodium bicarbonate solution until the evolution of carbon dioxide gas ceased and the medium was nearly neutral (pH ≈ 7). The resulting precipitate was filtered by vacuum, washed with cold water, dried, and recrystallized in a 3:1 ethanol/water mixture to yield yellowish-white crystals of benzimidazole.

B- Microwave-Assisted Method:

o-phenylenediamine (1.9 g) was weighed and placed in a microwave reactor flask. Formic acid (10 mL) was added, the mixture was stirred, the flask was sealed with a lid, and the microwave was operated for 20 minutes at 120–150°C. When the reaction was complete, the flask was cooled to room temperature and then carefully opened under a vacuum. Ice water was slowly added to the mixture to dilute and neutralize. The pH was adjusted by adding dilute $NaHCO_3$ to pH ~7 and a precipitate formed. The solidified product was collected by filtration over a Buchner filter, washed with cold water, and then with a small amount of cold methanol. It was dried at ≤ 50°C until constant weight.

Results And Discussion:

In this study, three organic compounds belonging to Schiff bases were synthesized, namely benzylideneaniline, **Acetanilide**, and N-(3-phenylallyl) aniline. For each compound, the molecular weight, melting point, and density at room temperature were determined. In addition, the reaction time, physical appearance (color), and solubility of the obtained products were also evaluated.

From the table 1, it can be seen that the yield obtained using the microwave-assisted method was higher than that of the conventional method. In addition, the reaction time was significantly shorter, requiring only 2 minutes with the microwave method compared to 30 minutes using the conventional approach**,** fig. 1 &2.

The infrared spectrum (cm^{-1}) of the compound showed characteristic absorption bands at 3015 cm^{-1}, corresponding to the C–H stretching of the sp^2-hybridized carbons, 2942 cm^{-1} to the C–H stretching of the sp^3-hybridized carbons, 1627 cm^{-1} attributed to the C=C stretching, and 1591 cm^{-1} corresponding to the C=N stretching vibration.

Table 1. Comparison of Selected Physical Properties of Benzylideneaniline Prepared by the Conventional Solvent Method and the Microwave- Assisted Method.

Method	Yield (%)	Melting point, °C	Reaction time (min)	Density g\cm3
Conventional (solvent)	**88**	**52-53**	**30**	**0.832**
Microwave-assisted	**97**	**52-53**	**2**	**0.832**

This study was compared to the work of James et al. (2019, OKO), where the yield was 83.986% for N-benzylidene aniline. This result is close to the yield of our conventional method (88%). However, the use of microwave-assisted synthesis significantly improved the reaction efficiency, achieving a much higher yield of 97%, while significantly reducing the reaction time from 30 minutes using the conventional method to only 2 minutes using microwave irradiation.

The comparative results between the conventional method and the microwave-assisted approach clearly demonstrate the superiority of the latter in terms of operational efficiency and environmental friendliness. The yield increased from 86% using the conventional solvent-based method to 95% with microwave irradiation, reflecting the enhanced conversion of reactants into the desired product. Furthermore, the reaction time was significantly reduced from 20 minutes to only 3–5 minutes, highlighting the accelerated chemical process afforded by rapid and uniform microwave heating table2.

Table 2. Comparison of Selected Physical Properties of Acetanilide Prepared by the Conventional Solvent Method and the Microwave-Assisted Method.

Method	Yield (%)	Melting point, °C	Reaction time (min)	Density g\cm3
Conventional (solvent)	**86**	**115 - 116**	**20**	**1.22**
Microwave-assisted	**95**	**114 - 115**	**120**	**1.22**

Regarding the physical properties, no significant differences were observed between the two methods; melting points and densities remained comparable, confirming the chemical identity and purity of the final product. These findings support the notion that solvent- and catalyst-free microwave-assisted reactions represent an effective and environmentally sustainable approach, combining reduced time and energy consumption with high product quality.

This is consistent with the findings of Tiwari, G., Khanna, A., Mishra, V. K., & Sagar, R. (2023).and the study on microwave-assisted reactions of aniline derivatives, both of which demonstrated that solvent-free or microwave-assisted methods enhance reaction rate and product efficiency. Moreover, the comparable melting points of the products in both approaches indicate compound purity, further supporting the effectiveness of the selected method for rapid and environmentally friendly synthesis of acetanilids.

IR spectral analysis further supported the successful formation of the desired compound. The spectrum exhibited characteristic absorption bands at 3294 cm^{-1} (N–H stretching), 3083 cm^{-1} (C–H stretching of sp^2 carbons), 2899 cm^{-1} (C–H stretching of sp^3 carbons), and 1665 cm^{-1} (C=O stretching). Additional bands at 1598 cm^{-1} and 1538 cm^{-1} corresponded to C=C stretching and N–H bending, respectively, confirming the presence of the expected functional groups.

The results of this study indicate that both conventional and microwave methods produce N-cinnamylidene aniline with similar properties, as evidenced by the stability of the melting point (175–176 °C) and density (0.93 g/cm^3). However, there is a clear difference in yield and reaction time, with the microwave method producing 93% versus 77% in a much shorter time (5–6 minutes versus 50 minutes) table 3 and fig. 1 &2.

Table 3. Shows the physical properties of N-[(E)-3-phenylprop-2-en-1-ylidene] aniline.

Method	Yield (%)	Melting point, °C	Reaction time (min)	Density g\cm3
Conventional (solvent)	77	175 – 176	50	0.93
Microwave-assisted	93	175 -176	5-6	0.93

The IR spectrum of the compound (KBr, cm⁻¹) showed characteristic absorption bands at 3060–3010 (aromatic C–H), 3080–3070 (C–H of the alkene), 1620–1615 (C=N stretching), 1600–1450 (aromatic C=C stretching), and 750–700 (aromatic out-of-plane bending), confirming the formation of the expected Schiff bas. The results indicate that both conventional and microwave-assisted methods yield benzimidazoles with similar properties, as demonstrated by the stability of the melting point (170–172 °C) and density (1.20 g/cm³). However, there is a clear difference in yield and reaction time, with microwave method yielding 91% versus 75% in a much shorter time (20 minutes versus 2 hours).

Table 4. Shows the physical properties of Benzimidazole.

Method	**Yield (%)**	**Melting point, °C**	**Reaction time (min)**	**Density g\cm3**
Conventional (solvent)	**75**	**172 - 170**	**20**	**1.20**
Microwave-assisted	**85**	**172 - 170**	**3-5**	**1.20**

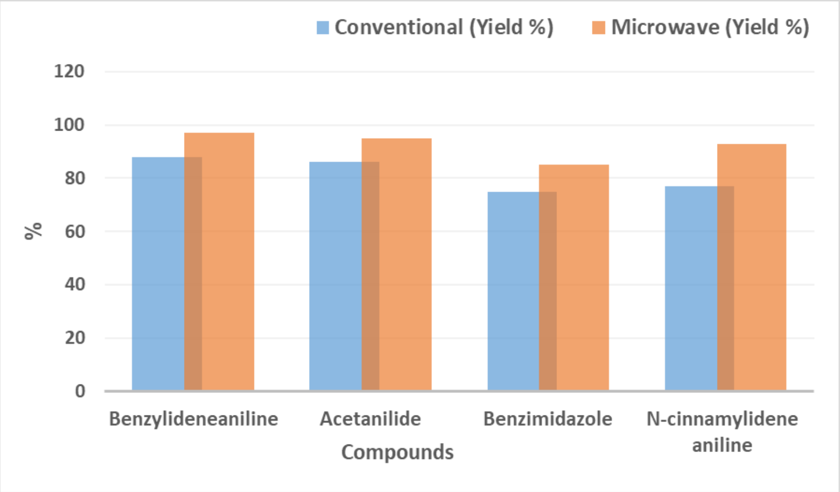

Figure 1. Comparison of Yield (%) of Selected Aromatic Compounds Prepared by the Conventional Solvent Method and the Microwave- Assisted Method.

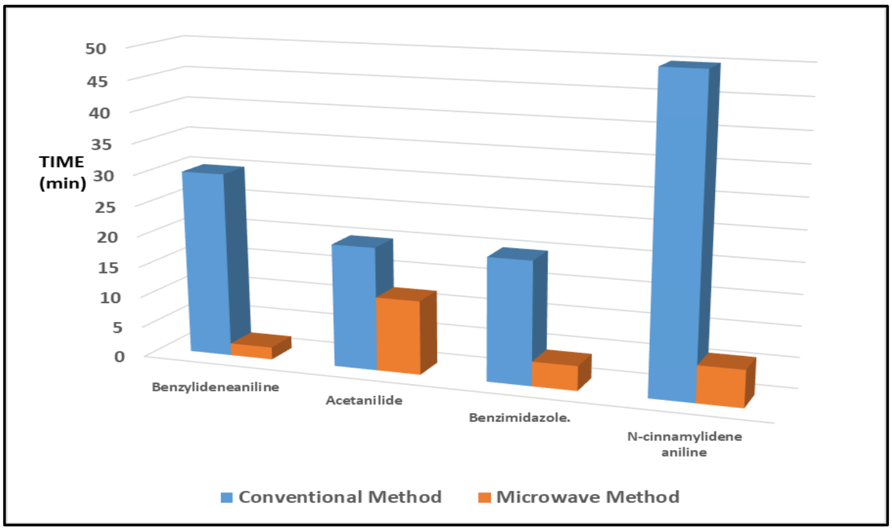

Figure 2. Comparison of Time (min) of Selected Aromatic Compounds Prepared by the Conventional Solvent Method and the Microwave- Assisted Method.

The IR spectrum of benzimidazole (KBr, cm⁻¹) exhibited characteristic absorption bands at 3400–3300 (N–H stretching), 3100–3000 (aromatic C–H), 1620–1600 (C=N stretching), 1500–1450 (C=C aromatic stretching), and 750–700 (aromatic out-of-plane bending), confirming the formation of the benzimidazole ring system.

These results are consistent with previous studies. Dubey and Moorthy (2007) showed that microwaves reduced the reaction time for the preparation of benzimidazole derivatives from 2–3 hours to less than 10 minutes, while increasing the yield from 60–70% to 80–90%. Yılmaz and Jose (2008) also confirmed that microwaves reduced the reaction time for benzimidazole and benzothiazole derivatives by 95–98%, with yield increases ranging from 10–50% depending on the substrate type. In another study, the World Journal of Pharmaceutical Research (2018) demonstrated that microwave preparation of benzimidazole was accomplished in just 4 minutes with a yield of approximately 90%, while the conventional method took more than 30 minutes with a yield of less than 75%. Microwave processing can be considered a more efficient, environmentally friendly, and time-saving approach than conventional heating.

Conclusion:

This study demonstrates that both conventional and microwave-assisted methods are effective for the preparation of aromatic compounds. Although both methods produce compounds with similar physicochemical properties, the microwave method is clearly superior in terms of reaction efficiency, yield, and time, without compromising product purity. These results highlight that microwave use is a faster, more efficient, and environmentally friendly option for the preparation of aromatic and other cyclic compounds. Overall, the microwave method offers significant advantages for laboratory and industrial applications, while promoting sustainability and resource efficiency in organic chemistry.

References:

1. Abumhdi, A. A. (2014). *Microwave-activated synthesis of potential biodynamic heterocyclic scaffolds* (Doctoral dissertation, Department of Chemistry, University of Allahabad, India).
2. Archana, S. (2013). *Synthesis and characterization of Schiff base salicylaldehyde and thiohydrazones and their metal complexes*. [Doctoral dissertation / Thesis].
3. Catalano, A.; Sinicropi, M. S.; Iacopetta, D.; Ceramella, J.; Mariconda, A.; Rosano, C.; Scali, E.; Saturnino, C.; Longo, P. (2021). A review on the advancements in the field of metal complexes with Schiff bases as antiproliferative agents. *Applied Sciences*, 11(13), 6027. https://doi.org/10.3390/app11136027
4. Cunha, S., & de Santana, L. L. (2017). *Eco-Friendly, Catalyst and Solvent-Free, Synthesis of Acetanilides and N-Benzothiazole-2-yl-acetamides.* ***Journal of the Brazilian Chemical Society, 28,*** 1137–1144.
5. Dubey, R. S., & Moorthy, H. N. (2007). Comparative studies on conventional and microwave assisted synthesis of benzimidazole and their 2-substituted derivative. *Indian Journal of Chemistry - Section B: Organic Chemistry Including Medicinal Chemistry*, 46B (5), 758–762
6. Jorge, J.; Del Pino Santos, K. F.; Timóteo, F.; Vasconcelos, R. R. P.; Ayala Cáceres, O. I.; Granja, I. J. A.; de Souza, D. M.; Frizon, T. E. A.; Di Vaccari Botteselle, G.; Braga, A. L.; Saba, S.; Rashid, H. U.; Rafique, J. (2024). Recent advances on the antimicrobial activities of Schiff bases and their metal complexes: An updated overview. *Current Medicinal Chemistry*, 31(17), 2330–2344.
7. Malav, R.; Ray, S. (2025). Recent advances in the synthesis and versatile applications of transition metal complexes featuring Schiff base ligands. *RSC Advances*, 15, 22889–22914. https://doi.org/10.1039/D5RA03626G
8. Mittal, A. (2009). Synthetic nitroimidazoles: Biological activities and mutagenicity relationships. *Scientia Pharmaceutica*, 77, 497–520.
9. Mycak, M., Doležal, R., Bílek, M., & Kolá, K. (2018). *Microwave-Aided Reactions of Aniline Derivatives with Formic Acid: Inquiry-Based Learning Experiments.* ***Journal of Chemical Education, 95***, 1231–1235.
10. Nagalakshmi, G. (2008). Synthesis, antimicrobial and anti-inflammatory activity of 2,5-disubstituted-1,3,4-oxadiazoles. *Indian Journal of Pharmaceutical Sciences*, 70(1), 49–55.
11. Tiwari, G., Khanna, A., Mishra, V. K., & Sagar, R. (2023). Recent developments on microwave-assisted organic synthesis of nitrogen- and oxygen-containing preferred heterocyclic scaffolds. *RSC Advances, 13*(54), 32858–32892. https://doi.org/10.1039/D3RA05986
12. Valverde, M. G.; Torroba, T. Sulfur–Nitrogen Molecules. *Sulfur Chemistry*, 2005, 10, 318–340.
13. World Journal of Pharmaceutical Research. (2018). Microwave assisted synthesis of benzimidazole and its characterization. *World Journal of Pharmaceutical Research*, 7(19), 1–
14. Yılmaz, A., & Jose, J. (2008). Comparative studies on conventional and microwave synthesis of some benzimidazole, benzothiazole and indole derivatives. *Molecules*, 13(4), 736–748