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Microwave Irradation Outperforms Traditional Methods: Improved Synthesis of Azidobenzohydrazide Schiff Bases

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Abstract

An efficient synthesis of organic compounds including Azidobenzohydrazide derivatives, is important for industrial, agricultural, and medical applications. Synthesizing bioactive compounds need various procedures, however, simple methods are highly preferred. In the present study, four different synthetic techniques, including stirring, reflux, ultrasonic, and, microwave irradiation, were employed to synthesize series of azidobenzohydrazide derivatives. Each procedure take place with their optimum conditions to yield highest and purest products. In this study, we aimed to compare different synthetic approaches, and highlighting the most effective one among the others. The results showed that the microwave irradiation is the most efficient approach among the others, since it's faster and more product yields. This comparative analysis highlights the importance of microwave techniques over the conventional methods, providing insights into how to optimize synthesis procedures for these significant chemicals.

1. Introduction:

Organic compounds are an important source in different fields and various applications, such as agriculture [1], industry [2], [3] and medicinal chemistry [4]. Among the organic compounds, hydrazide Schiff bases possess a vital role in various applications, due to the presence of unique functional groups. This compound can be a useful building block in organic synthesis [5], especially in pharmaceutical chemistry [6], [7]. Accordingly, several benzohydrazide derivatives have been synthesized with dual functionality, particularly possessing imine functional groups. Having these two functional groups will enhance the biological potential of the products [8]. These compounds have earned great attention because of their diverse applications, including anticancer activity [6], [9], antimicrobial activity [10], and fabric science [6].

Because of their high reactivity, azides are a significant family of intermediates that are frequently employed to create amine and heterocyclic compounds. However, some azides also have considerable biological activity and are utilized as drugs [11]. Synthesizing novel and new compounds need new procedure; however, several common compounds have been resynthesized using modern and new techniques [12], to overcome the challenge that faced the scientists in traditional protocols, such as stirring [13] and reflux [14].

Alongside these two conventional methods, ultrasonic techniques [15] are reliable strategies; however, in a variety of synthetic approaches, they require long reaction times and the use of solvents [16], [17]. Microwave irradiation technique is considered a modern protocol for the synthesis of various organic products, by means of high product yields and short reaction time [18].

Benzohydrazide Schiff bases are prepared by direct con-

densation of benzohydrazide derivatives with different aldehydes or ketones under reflux conditions, with heating techniques [19]. This method often involves long reaction times, high temperatures, and large amounts of organic solvents, which are less environmentally friendly and have a higher carbon footprint. In addition, the method can result in moderate yields and may require additional purification steps, which can limit its applicability for large-scale synthesis [20], [21].

The advent of microwave-assisted organic synthesis (MAOS) has revolutionized the field of synthetic chemistry by offering a rapid, efficient, and environmentally benign alternative to conventional heating methods [22]. Microwave (MW) irradiation allows effective thermal management, fast heat transfer, and high product yield. These advantages make MW accelerate reaction kinetics and improve product selectivity [23].

Recent studies have demonstrated the superiority of microwave-assisted synthesis in preparing various Schiff base derivatives. The Schiff bases [24] were prepared with aromatic aldehyde and aromatic amine using cashew shell extract as a catalyst. This reaction is rapid, efficient, and solvent-free and involves the one-pot synthesis of Schiff bases under microwave irradiation. The application of MW in benzohydrazide synthesis has shown promising results, with pyrazole, triazole based benzohydrazones in the presence of acetic acid catalyst demonstrating enhanced efficiency [25].

Despite the advantages, comprehensive comparative studies evaluating the efficiency of microwave irradiation versus traditional methods specifically for benzohydrazide Schiff base synthesis remain limited. The optimization of reaction parameters such as power settings, temperature profiles, reaction time, and solvent systems requires systematic investigation to fully harness the potential of this technology [22], [23]. Additionally, the mechanistic understanding of how microwave irradiation influences the condensation reaction pathway and product distribution in benzohydrazide Schiff base formation warrants detailed examination [26].

The present study assesses the efficacy of the microwave-assisted technique in comparison to the traditional methods, including stirring, reflux, and ultrasound, to produce azidobenzohydrazide Schiff bases. Through seriously comparative analysis, our objective is to determine the most efficient and possible approach for furnishing these vital compounds.

2. Material and Methods:

2.1 Reagents and instrumentation:

All the chemicals were purchased from Sigma Aldrich, Merk, and Fluka, and used without further purification. An Electrothermal 9100 apparatus was used to measure the melting point of the products. The IR spectra have been recorded by FTIR spectroscopy (Schimadzu IRAffinity-1) at the Salahaddin University-Erbil, Kurdistan Region of Iraq. Varian Inova Nuclear Magnetic Resonance for ^{1}H-NMR and ^{13}C-NMR with 400 MHz and 125 MHz, respectively, at the University of Pavia, Italy, when the TMS was used as reference and DMSO-d_6 as solvent. High-resolution mass spectra were determined on an AEI MS-9 using electrospray ionization (ESI) and a time-of-flight (TOF). The parts per million (δppm) units are utilized for chemical shifts; the abbreviations of peaks are (s=singlet, d=doublet, t=triplet, and m=multiplet). Microwave irradiation was carried out with an oven (1000 W).

2.2 Synthesizing 4-azidobenzohydrazide from 4-aminobenzoic acid:

Synthesizing 4-azidobenzohydrazide required 3 steps that are mentioned in [5]. Briefly, 4-aminobenzoic acid is mixed with sodium nitrite in the presence of an acidic (HCl) medium and refluxed for 1 h, followed by the addition of sodium azide and refluxing for an additional 1h. The reaction yielded 4-azido benzoic acid. Later, the product [2] has been acidified with sulfuric acid and then ethanol added with refluxing for another three hours, to esterify the product and form ethyl 4-azidobenzoate [3]. Compound [3] has been mixed with 0.1 moles of hydrazide hydrate in an ethanolic (30 mL) solution and refluxed for 6 hours. The resulting mixture was cooled down to room temperature, and the targeted solid product was separated from the solution by filtration and washed with cold water, followed by recrystallization with ethanol. The spectroscopic data are available in [5].

2.3 Synthesis of Azidobenzohydrazide Schiff Bases by Stirring Method (A):

To a stirred solution of substituted benzaldehyde (0.01 mole) and 4-azidobenzohydrazide (0.01 mole) in 10 mL dry Ethanol, DMEA (0.1 mL) as a catalyst was added. The reaction mixture was stirred (8 hours) at room temperature. After the complete renovation of the benzaldehydes as examined by TLC. The products have been purified by filtration. The filtrate was cleaned with distilled water and recrystallized from absolute ethanol, dried well.

2.4 Synthesis of Azidobenzohydrazide Schiff Bases by Reflux Method (B):

A solution of 4-azidobenzohydrazide (0.01 mole) in dry Ethanol (5 mL) has been added in a solution of substituted benzaldehyde (0.01 mole) in dry Ethanol (5 mL), and then DMEA (0.1 mL) as a catalyst added. The mixture was heated at reflux temperature and stirred for 45 minutes. The obtained products have been filtered to be isolated from the unwanted reagents and then recrystallized from Ethylacetate to obtain the title compound.

2.5 Synthesis of Azidobenzohydrazide Schiff Bases by Ultrasonic Technique Method (C):

A mixture of different substituted benzaldehyde (0.01 mole), with 4-azidobenzohydrazide (0.01 mole) has been dissolved

in 10 ml dry ethanol in a conical flask, and DMEA (0.1 mL) added was into the ultrasonic for (30 min – 8 hours). After cooling, the solid product was recrystallized from ethanol to provide (57%-70%) of the desired compound.

2.6 Synthesis of Azidobenzohydrazide Schiff Bases by Microwave Irradiation Method (D):

A mixture of different substituted benzaldehyde (0.01 mole), was mixed with 4-azidobenzohydrazide (0.01 mole) in 10 ml of dry Ethanol in a conical flask, then DMEA (0.1 mL) was added. The mixture was put into the microwave oven and irradiated for (1-10 min.) (output power at 40%). After cooling, the solid product was recrystallized from ethanol to provide (85%-87%) of the desired compound.

4-azido-N′-benzylidenebenzohydrazide (6a) 90% yield (238 mg), Pale yellow solid, m.p.: 115-117 ^{o}C. ^{1}HNMR (400 MHz, DMSO-d6): δ 11.87 (s, 1H, –NH), 8.47 (s, 1H, –CH), 8.00 – 7.27 (m, 9H, Ar.). ^{13}CNMR (125 MHz, DMSO-d_6), δ 162.60 (C=O), 148.26 (C-N_3), 143.38 (N=CH), 134.80 (C–C=O), 130.55 - 119.57 (Ar. C). FTIR (cm^{-1}): 3221 (NH, str.), 3070 (C-H, Ar. str.), 2926 (C-H, Aliph. str.), 2117 and 2083 (N_3), 1647 (O=C-NH, str.), 1600 (C=N, str.), 1556 (C=C, str.). HRMS (ESI) *m/z*: calcd for $C_{14}H_{11}N_5O$ [M+Na]$^+$: 288.0856; found: 288.0853.

4-Azido-N′-(4-Chlorobenzylidene) Benzohydrazide (6b). 85% yield (254 mg), pale yellow solid, m.p.: 165-167 ^{o}C. ^{1}HNMR (400 MHz, DMSO-d_6), δ 12.09 (s, 1H, NH), 8.87 (s, 1H, CH), 8.03 – 7.28 (m, 8H, Ar.). ^{13}CNMR (125 MHz, DMSO-d_6), δ 162.63 (C=O), 144.09 (C-N_3), 143.56 (N=CH), 133.67 (C-Cl), 132.06 – 119.60 (Ar. C). FTIR (cm^{-1}): 3203 (NH, str.), 3174 (C-H, Ar. str.), 3062 (C-H, Aliph. Str.), 2119 and 2085 (N3), 1647 (O=C-NH, str.), 1656 (C=N, str.), 1560 (C=C, str.), 754 (C-Cl, Str.). HRMS (ESI) *m/z*: calcd for $C_{14}H_{11}N_5OCl$ [M+Na]$^+$: 322.0466; found: 322.0461.

4-Azido-N′-(4-Methoxybenzylidene) Benzohydrazide (6c). 89% yield (263 mg), yellow solid, m.p.: 174-176 ^{o}C. ^{1}HNMR (400 MHz, DMSO-d_6), δ 11.74 (s, 1H, NH), 8.41 (s, 1H, CH), 7.99 – 7.02 (m, 8H, Ar.), 3.82 (s, 3H, OCH3). ^{13}CNMR (125MHz, DMSO-d_6) δ 162.43 (C=O), 161.32 (OCH3), 148.-16 (C-N_3), 143.23 (N=CH), 130.43 (C–C=O), 129.96 –114.80 (Ar. C), 55.75 (CH_3). FTIR (cm^{-1}): 3236 (NH, str.), 2127 and 2092 (N3), 1654 (O=C-NH, str.), 1600 (C=N, str.), 1571 (C=C, str.). HRMS (ESI) *m/z*: calcd for $C_{15}H_{13}N_5O_2$ [M+Na]$^+$: 318.0961; found: 318.0954.

4-Azido-N′-(4-(Dimethylamino)Benzylidene) Benzohydrazide (6d). 91% yield (281 mg), yellow solid, m.p.: 172-174 ^{o}C, ^{1}HNMR (400 MHz, DMSO-d_6), δ 11.56 (s, 1H, NH), 8.31 (s, 1H, CH), 7.97 – 6.76 (m, 8H, Ar.), 2.99 (s, 6H, N(CH_3)$_2$). ^{13}CNMR (125 MHz, DMSO-d_6): δ 162.14 (C=O), 151.99 (C-N), 149.13 (C-N_3), 143.03 (N=CH), 130.69 (C-C=O), 129.88 – 112.27 (Ar. C), 40 (N-CH_3). FTIR (cm^{-1}): 3205 (NH, str.), 3003 (CH, Ar. str.), 2920 (CH, Aliph, str.), 2127 and 2090 (N3), 1635 (O=C-NH), 1600 (C=N, str.), 1558 (C=C, str.). HRMS (ESI) *m/z*: calcd for $C_{16}H_{16}N_6O$ [M+Na]$^+$: 331.1278; found: 331.1277.

4-Azido-N′-(3-Phenylallylidene) Benzohydrazide (6e). 80% yield (240 mg), yellow solid, m.p.: 154-156 ^{o}C, ^{1}HNMR (400 MHz, DMSO-d_6), δ 11.76 (s, 1H, NH), 8.25 (dd, 1H, CH), 7.96 (m, 2H, –HC=CH–), 7.65 – 7.07 (m, 9H, Ar.). ^{13}CNMR (125 MHz, DMSO-d_6), δ 167.20 (C=O), 154.97 (C–N_3), 148.11 (N=CH), 144.27 (C–C=C), 141.13 (C–C=C), 134.79 (C–C=O), 134.06 – 130.90 (Ar. C), 124.31 (=CH–CH=N). FTIR (cm^{-1}): 3255 (NH, str.), 3064 (CH, Ar. str.), 2976 (CH, Aliph. str.), 2125 and 2086 (N3), 1676 (O=C-NH), 1624 (C=N, str.), 1600 (C=C, str.). HRMS (ESI) *m/z*: calcd for $C_{16}H_{13}N_5O$ [M+Na]$^+$: 314.1012; found: 314.1011.

4-Azido-N′-(4-Nitrobenzylidene) Benzohydrazide (6f). 93% yield (288 mg), yellow solid, m.p.: 223-225 ^{o}C, ^{1}HNMR (400 MHz, DMSO-d_6), δ 12.17 (s, 1H, NH), 8.57 (s, 1H, N=CH), 8.33 – 7.29 (m, 8H, Ar.). ^{13}CNMR (125 MHz, DMSO-d_6): δ 162.84 (C=O), 148.31 (C–NO_2), 145.67 (C–N_3), 143.68 (N=CH), 141.11 (C–CH=N), 130.18 (C–C=O), 129.87 – 119.-63 (Ar. C). FTIR (cm^{-1}): 3257 (NH, str.), 3068 (CH, Ar. str.), 2624 (CH, Aliph. str.), 2123 and 2094 (N_3), 1651 (O=C–NH), 1604 (C=N), 1575 (C=C, str.), 1541 and 1344 (NO_2, str.). HRMS (ESI) *m/z*: calcd for $C_{14}H_{10}N_6O_3$ [M+Na]$^+$: 333.0707; found: 333.0699.

3. Results and discussion:

In the present study, four different methods have been tried to synthesize the azidobenzohydrazide Schiff bases. The products have been fully characterized via IR, ^{1}HNMR, ^{13}CNMR, and HRMS and published [5]. In all FTIR spectra, a peak near 1600 appeared, which is attributed to the azomethine functional group, proving a new functional group that was absent in the previous one. Additionally, peaks 8.87 – 8.25 in ^{1}HNMR and 143.03 – 148.11 in ^{13}CNMR, confirming the formation of Schiff bases; furthermore, their chemical structures were employed to HRMS. These indications are acceptable for the products to ensure their synthesis and purity. All the other peaks are appeared at their normal and usual positions with some shifting, shielding, or deshielding, that can be explained due to attachment of some other groups on the structure of the products, such as N_3, N, Cl, OCH_3, etc.

The reaction is four step process Figure 1. In the first step, replacing the amine group with azide, an acidic medium is used, followed by the addition of sodium azide to afford product [2]. Second step required a stronger acidic medium to change the carboxylic acid to an ester functional group and heating to reflux temperature to synthesize product [3]; which is further treated with hydrazide hydrate in ethanolic

solvent and refluxing temperature for a long time (six hours), to replace the ester with hydrazide and afford product [4].

The final step is synthesizing the Schiff bases (6a-f); which is the main idea for the present study. In these step four techniques were employed (regular stirring, reflux, ultrasonic bath, and microwave irradiation). For all the reactions, the conditions have been stabilized and unified (100 microliters of dimethylethylamine as catalyst and ethanol as solvent). Among the traditional methods, the ultrasonic technique is the highest performing in the production of the compounds, since a shorter time is needed to furnish a higher yield of the targeted products, besides the addition of DMEA as catalyst [27], [28]. As shown in Figure 2, the ultrasonic approach can yield the products 65% within one hour, while this percentage is observed after seven hours of stirring and three hours by reflux.

Additionally, none of stirring and reflux is able to reach more than 65 and 75 percent, respectively, when DMEA is used as catalyst and ethanol as solvent. Product 6a was taken as a model compound to compare the reaction conditions. Ultrasonic protocol has a great impact on the synthesis of the azidobenzohydrazide Schiff bases, and the method is green and safe; however, when compared with the microwave-assisted procedure, the microwave irradiation is more efficient and powerful [29], since the reactions occur within a very short period of less than 10 minutes. Therefore, the production of the Schiff bases via microwave irradiation protocol is significantly more effective than the other procedures, as shown in Table 1. In the microwave irradiation procedure, three minutes are enough to furnish 85% of the product, which is equivalent to 420 minutes to synthesize the same amount of the product via ultrasonics.

Table 1. Comparison between microwave irradiation protocol and ultrasonic technique.

Microwave-Assisted Technique		Ultrasonic Technique	
Time (min)	Yield (%)	Time (min)	Yield (%)
1	65	30	50
2	78	60	65
3	85	120	65
4	90	180	70
5	90	240	75
6	94	300	80
7	96	360	80
8	96	420	85
9	96	480	85

4. Conclusion:

Traditional methods can produce a variety of chemicals with a broad spectrum of biological and industrial applications; however, they require a large amount of solvent, a long period of time, and may not yield sufficient products. Therefore, scientists innovated new techniques to overcome these problems. One of the best methods to synthesize organic products is microwave-assisted approaches. In this study we utilized four different techniques to illustrate the best one among them. Our results concluded that, synthesizing azidobenzohydrazide Schiff bases (6 a-e) utilizing the microwave irradiation protocol needed less than 10 minutes to synthesize them, and recorded the highest yield percentage among other techniques, including reflux, stirring, and ultrasonic.

5. Acknowledgment:

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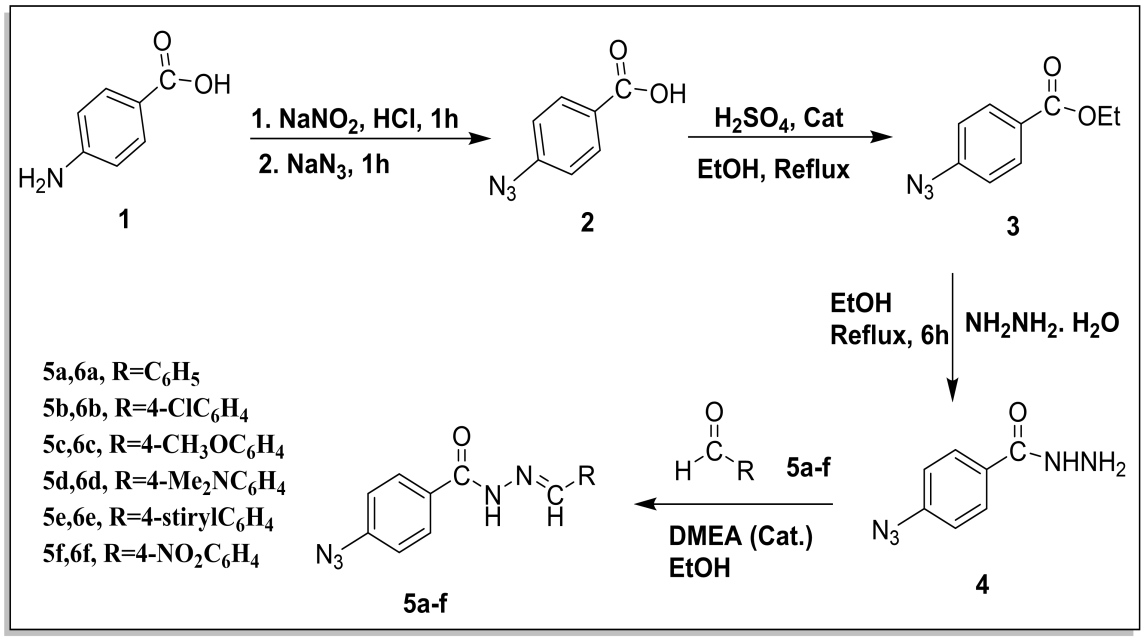

Figure 1. General reaction for the synthesis of azidobenzohydrazide Schiff bases.

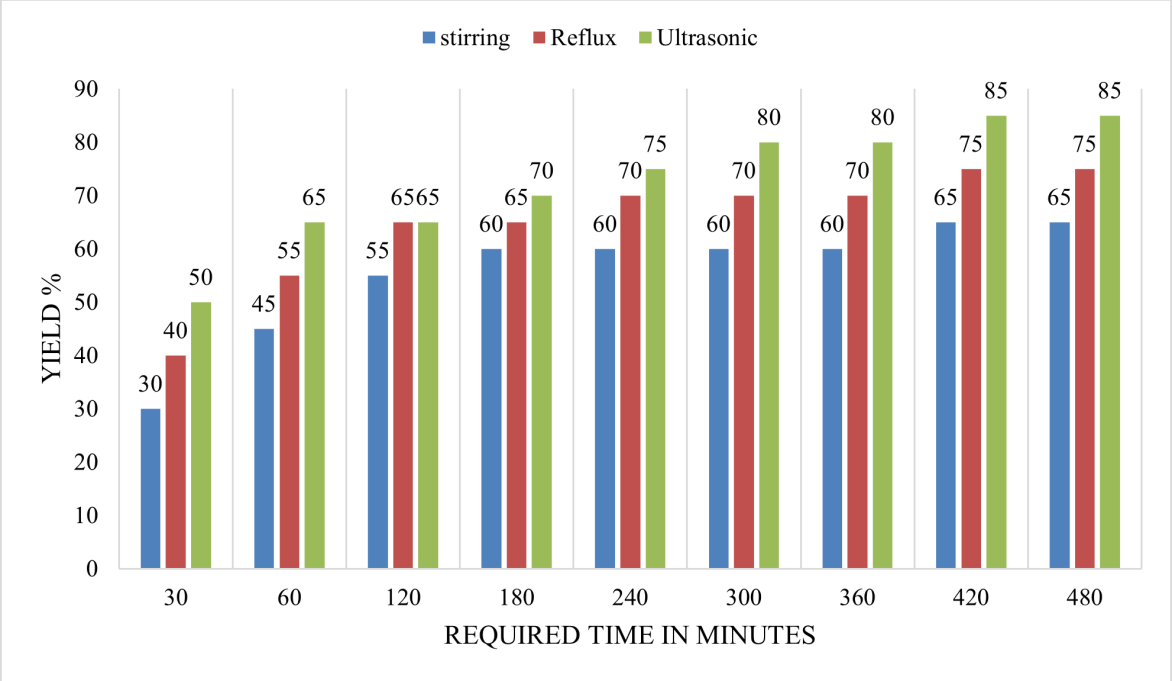

Figure 2. Comparison between traditional methods (stirring, reflux, and ultrasonic).

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Data Availability Statement: All of the data supporting the findings of the presented study are available from the corresponding author on request.

Declarations:
Conflict of interest: The author declare that he has no conflict of interest.

Ethical approval: This study did not involve human participants or animals; therefore, ethical approval was not required.

Author Contributions: Muslih Saber Hamasharif led all aspects of the research, including field data acquisition, data analysis, and interpretation of results. He was solely responsible for drafting the manuscript and ensuring the accuracy and clarity of the final version through comprehensive proofreading.

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إشعاع الميكروويف يتفوق على الطرق التقليدية: تحسين تحضير قواعد شف ازايدوبنزوهيدرازيدية

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الخلاصة

التحضير الفعال للمركبات العضوية المتضمنة مشتقات ازايدوبنزوهيدرازيد هو أمر مهم لتطبيقاتها في مجال الصناعة والزراعة والطب. تم التحقق من تركيب المركبات المصنعة عن طريق نقطة الانصهار، وكروماتوغرافيا الطبقة الرقيقة، والأشعة تحت الحمراء، والرنين النووي المغناطيسي، وطيف الكتلة. تقوم هذه الدراسة باختبارأربعة طرق تحضيرية مختلفة (الإشعاع الميكروويف، والموجات فوق الصوتية، التسخين الإرجاعي والتحريك). وقد توصلت نتائج الدراسة الى أن الإشعاع بالميكروويف هو الطريقة الأكثر كفاءة بين الطرق الأخرى، لأنه أسرع ويحقق إنتاجية أكثر. ان هذا التحليل المقارن يبرز المزايا التي تحققها التقنية الميكروويف، مما يوفر رؤى حول كيفية تحسين طرق التحضير لهذه المواد الكيميائية المهمة.

الكلمات الدالة : بنزوهيدرازيدات ؛ التحليل المقارن؛ الإشعاع بالموجات الدقيقة؛ قواعد شيف.

التمويل: لايوجد.

بيان توفر البيانات: جميع البيانات الداعمة لنتائج الدراسة المقدمة يمكن طلبها من المؤلف المسؤول.

اقرارات:

تضارب المصالح: يقر المؤلف أنه ليس لديه تضارب في المصالح.

الموافقة الأخلاقية: لم يتضمن هذا البحث اي تجارب على البشر او الحيوانات بالتالي لم يكن من الضروري الحصول على الموافقة الاخلاقية.

مساهمات المؤلفين: تولى مصلح صابر حمةشريف جميع جوانب البحث، بما في ذلك جمع البيانات الميدانية، وتحليل البيانات، وتفسير النتائج، وكان مسؤولاً بشكل كامل عن إعداد مسودة البحث وضمان دقة ووضوح النسخة النهائية من خلال المراجعة الشاملة والتدقيق اللغوي.