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ARTICLE

Microwave-assisted synthesis and luminescent activity of imidazo[1,2-*a*]pyridine derivatives

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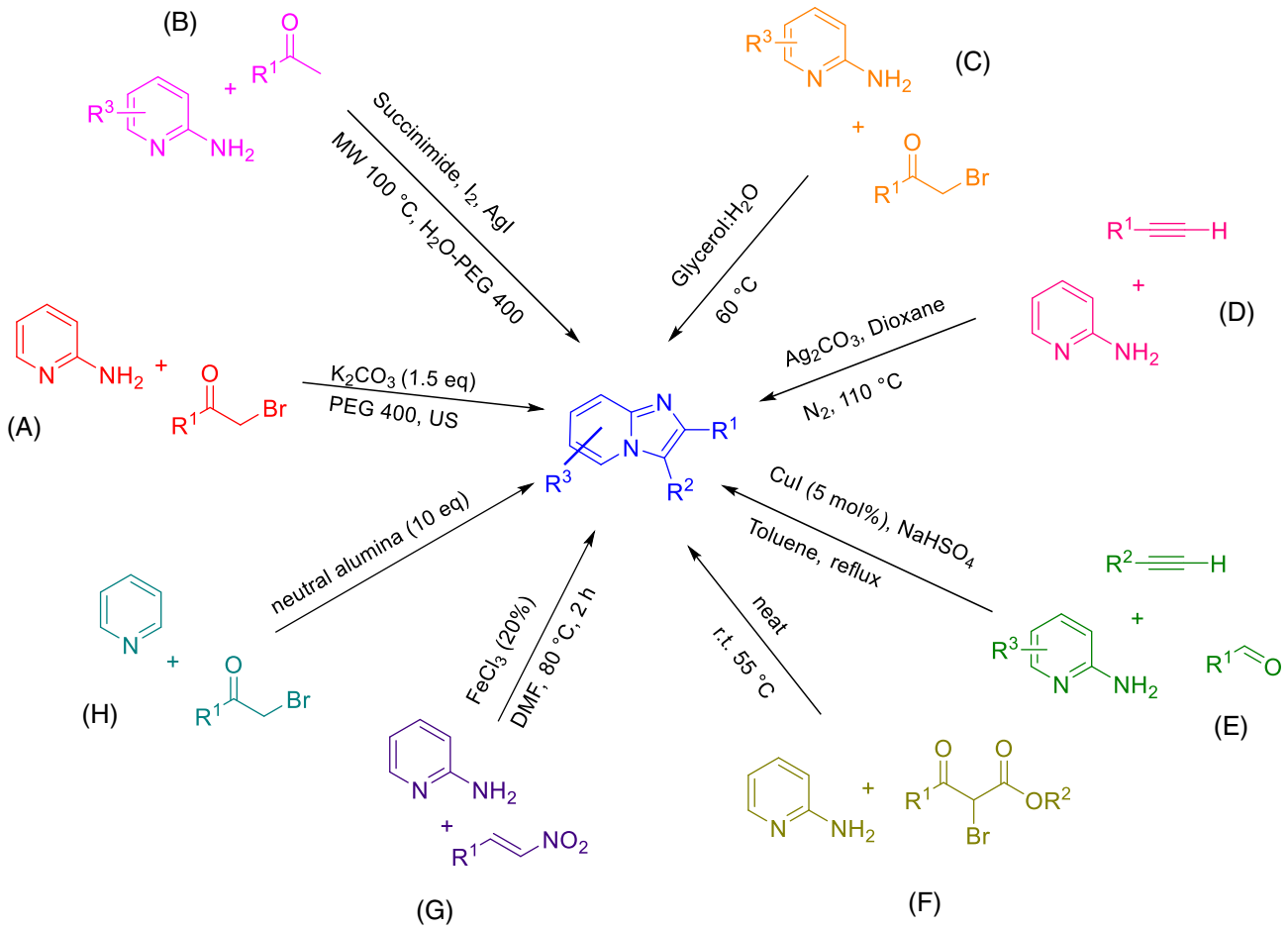
Abstract

In this work, a series of phenacyl bromide derivatives was synthesized and employed as key intermediate for the synthesis of substituted imidazo [1,2-*a*]pyridines. First, phenacyl bromide molecules were obtained from the bromination reaction of acetophenones assisted by microwave irradiation, obtaining the products **4a-v** in a 15 minutes reaction with yields in the range of 50% to 99%. Subsequently, the conjugation of these molecules with 2-aminopyridine conduced to the production of imidazo[1,2-*a*]pyridine derivatives (**7a-v**) in a 60-second reaction with yields of 24% to 99%. Improved yields were determined with respect to those obtained with more tedious methodologies like thermally and mechanically assisted routes. Intense luminescence emissions in the purple and blue regions of the electromagnetic spectra were observed under UV excitation according to the nature of the substituents. This environmentally friendly methodology is expected to constitute an important class of organic compounds for the development of biomarkers, photochemical sensors, and medicinal applications.

1 | INTRODUCTION

The chemistry of imidazopyridines has been studied since the beginning of this century. Accordingly, the most widely used and practical synthesis routes are the condensation between α-halocarbonyls and 2-aminopyridine where Vieira et al.[1] employed ultrasound as an alternative energy in a basic medium (Scheme 1A). On the other hand, Jadhav et al[2] used water and polyethylene glycol as solvent in a one-pot reaction avoiding the synthesis of the α-halocarbonyl (Scheme 1B). In the same way, Tufail et al[3] developed a green media using glycerol and water as solvent with the regioselective product in position 2 (Scheme 1C). On the other hand, He et al[4] accomplished the product by an oxidative cross-coupling/cyclization catalyzed by silver in nitrogen atmosphere (Scheme 1D), using alkynes as well. Mishra et al[5] generated in a one-pot reaction with aldehydes, 2-aminopyridine and terminal alkynes with copper iodide and a silica-supported Brønsted acid obtained the product with substitution in both 2 and 3 positions (Scheme 1E). Chunavala et al[6] reported the unexpected synthesis between α-bromo-β-keto esters and 2-aminopyridine under solvent-free conditions (Scheme 1F). Santra et al[7] used nitro olefins instead of ketones to obtain the nuclei, this reaction is catalyzed by iron (III) (Scheme 1G). Finally, Ponnala et al[8] reported the use of neutral alumina in room temperature as an efficient medium for the product to be formed (Scheme 1H).



SCHEME 1 Representative pathways for the synthesis of imidazo[1,2-*a*]pyridine with different substitution patterns

Although new synthetic routes have been developed, the condensation between α-halocarbonyls and 2-aminopyridine is nowadays the most popular strategy.[9] These molecules exhibit a wide range of medical activities such as anti-inflammatory,[10–12] antioxidant,[13,14] antimicrobial,[15,16] fungicide,[17,18] and anticancer agent.[19] In addition, other applications such as fluorescent sensor[20] and biomarkers[21] have been described.

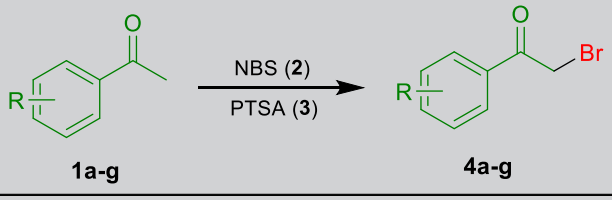
Beside the above-described applications, imidazo [1,2-*a*]pyridines and pyrimidines are also attractive due to their physicochemical properties such as the fluorescent activity.[22,23] Several studies concerning the effects of substituents on their fluorescent properties have been reported. Generally, its solid-state emission is due to the π-conjugated bicyclic structure, providing intense fluorescence emissions with high quantum yields.[24] The experimental evidence demonstrated that the substitution with phenyl or naphthyl groups in position C2 increased their fluorescence yield.[25] Electron-donating substituents also improved the luminescence performance whereas electron-withdrawing substituents led to a less intense emissions.[26] While the amino or dimethylamine substitution at the 4′-position cause a red shift of the fluorescence in polar solvents.[27] The replacement of methyl by trifluoromethyl triggers a blue shift.[28] Despite these observations, the development of more efficient fluorophores and bioactive compounds is a field of growing interest, making necessary the evaluation of new methodologies for the synthesis of imidazo[1,2-*a*]pyridine derivatives with varied substituents allowing the modulation of the physicochemical properties and therefore, their activity and applications. This work reported a novel microwave irradiation-assisted methodology for the synthesis of imidazo[1,2-*a*]pyridine derivatives in eco-friendly conditions, obtaining the phenacyl bromide precursors in 15 minutes process, and the substituted imidazo[1,2-*a*] pyridines in a 1-minute reaction. Furthermore, the photophysical evaluation described in this work provide additional insight on the relationship between structure and optical activity.

2 | RESULTS AND DISCUSSIONS

2.1 | Synthesis of phenacyl bromides

In order to obtain the desired products, two reaction steps are required. The first one is the synthesis of phenacyl bromides (**4a-v**). These compounds are considered useful precursors for organic synthesis of many molecules.[29] The synthesis was conducted by an halogenation in the alpha position of the respective acetophenone (**1a-v**), using NBS (**2**) as a brominating agent, and PTSA as a catalyst (**3**). That reaction was promoted by three different strategies: microwave irradiation (MW), mechanic interaction (mechanosynthesis), and thermal treatment (ThT) in a hot plate. The results of these reactions are grouped in Table 1. When comparing the three methodologies proposed in entries 1, 8, and 15 (compound **4a**), it is observed that the best route of synthesis is the mechanosynthesis, obtaining an excellent yield of 98%, compared with 78% (entry 1, MW) and 80% (entry 15, ThT). However, in the case of double substitution in the aromatic ring (**4g**), the yield of the mechanosynthesis decreases considerably (entry 14) until 65%, while the same compound was obtained by means of the MW and thermal paths (entries 7 and 21) with yields of 99 and 94%, respectively. In addition, the products **4b** and **4c** (entries 9 and 10) were not obtained by mechanosynthesis, while the microwave irradiation

TABLE 1 Synthesis of **4a-g** derivatives under different methodologies



1a-g 4a-g

Methodology	Entry	4	R	Time (min)	Yield (%)[a]
Microwave[b]	1	**a**	H	15	78
	2	**b**	4-Br	15	99
	3	**c**	4-NO_2	15	93
	4	**d**	4-Me	15	95
	5	**e**	4-Cl	15	95
	6	**f**	4-OMe	15	95
	7	**g**	3,4-Cl	15	99
Mechanosynthesis[c]	8	**a**	H	15	98
	9	**b**	4-Br	45	np
	10	**c**	4-NO_2	45	np
	11	**d**	4-Me	15	96
	12	**e**	4-Cl	15	95
	13	**f**	4-OMe	15	96
	14	**g**	3,4-Cl	15	65
Thermal[d]	15	**a**	H	40	80
	16	**b**	4-Br	110	98
	17	**c**	4-NO_2	120	95
	18	**d**	4-Me	40	96
	19	**e**	4-Cl	45	99
	20	**f**	4-OMe	55	90
	21	**g**	3,4-Cl	40	94

[a]Isolated yield.
[b]Reaction conditions: **1a-g** (1 eq), **2** (1.1 eq), **3** (1.5 eq), DCM (1 mL), 40°C, 15 minute.
[c]Reaction conditions: **1a-g** (1 eq), **2** (1.1 eq), **3** (0.5 eq), neat, r.t, 15 to 45 minute.
[d]Reaction conditions: **1a-g** (1 eq), **2** (1.1 eq), **3** (0.5 eq), ACN (5 mL), 85°C, 40 to 120 minute.
Note: np = the product is not formed.

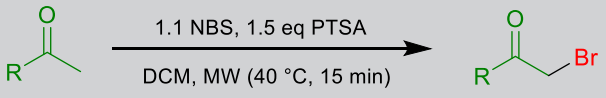
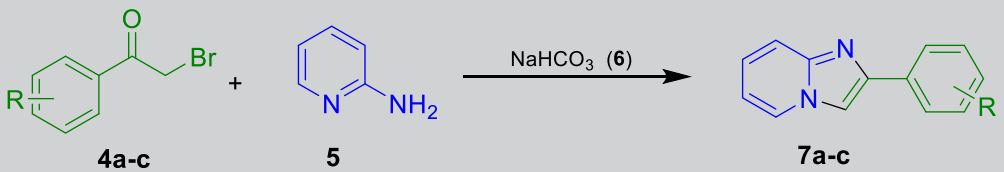


TABLE 2 Derivatives of **4h-v** obtained by the MW assisted methodology

Entry	4	R	Yield (%)[a]
1	**h**	NHPh	75
2	**i**	2-OMePh	90
3	**j**	2-ClPh	85
4	**k**	3-MePh	90
5	**l**	3-OMePh	50
6	**m**	3-ClPh	87
7	**n**	3-BrPh	99
8	**ñ**	2,5-OMePh	92
9	**o**	2,5-FPh	60
10	**p**	2,5-ClPh	97
11	**q**	2,6-OMePh	40
12	**r**	2,6-ClPh	70
13	**s**	3,4-OMePh	95
14	**t**	3,4-FPh	90
15	**u**	2,4-OMePh	90
16	**v**	2,4-FPh	97

[a]Isolated yield.

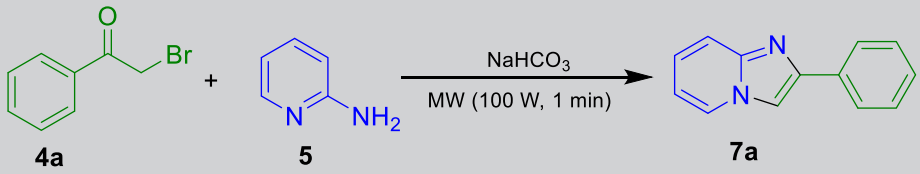
TABLE 3 Synthesis of **7a-c** under different methodologies



Methodologies	Entry	7	(R)	Time (min)	Yield (%)[a]
Thermal[b]	1	**a**	H	40	80
	2	**b**	4-Br	80	78
	3	**c**	4-NO_2	120	30
Microwave[c]	4	**a**	H	1	88
	5	**b**	4-Br	1	85
	6	**c**	4-NO_2	1	41
Mechanosynthesis[d]	7	**a**	H	45	mix
	8	**b**	4-Br	45	mix
	9	**c**	4-NO_2	45	mix

Note: mix = product and byproduct.
[a]Isolated yield.
[b]Reaction condition: **4a-c** (1 eq), **5** (1.1 eq), **6** (1.5 eq), ACN (3–5 mL), 85°C.
[c]Reaction condition: **4a-c** (1 eq), **5** (1.2 eq), **6** (2.0 eq), MeOH (1 mL), 80°C.
[d]Reaction condition: **4a-c** (1 eq), **5** (1.2 eq), **6** (2.0 eq), neat, r.t.

TABLE 4 Reaction conditions for the synthesis of **7a**



4a **5** **7a**

Entry	$NaHCO_3$ (eq)	2-aminopyridine (5) (eq)	Solvent	%R[a]
1	1	1	EtOH	80
2	1.5			76
3	2			82
4	2	1	EtOH	82
5		1.1		82
6		1.2		88
7	2	1.2	EtOH[b]	88
8			MeOH[b]	90
9			ACN[c]	88
10			DCM[d]	81
11			Neat[b]	78

[a]Isolated yield.
[b]Reaction temperatures with solvents: 80°C.
[c]Reaction temperatures with solvents: 90°C.
[d]Reaction temperatures with solvents: 50°C.

strategy gave yields of 99% and 93% (entries 2 and 3), and the ThT conduced to yields of 98% and 95% (entries 16 and 17). Accordingly, MW and ThT methodologies are the best option for the synthesis.

However, the reaction time is much shorter (15 minutes) with respect to those required in ThT (40 to 120 minutes). Thereby, the MW methodology was selected to synthesize the α-halogenation derivatives reported in Table 2, which were obtained with moderate to excellent yields.

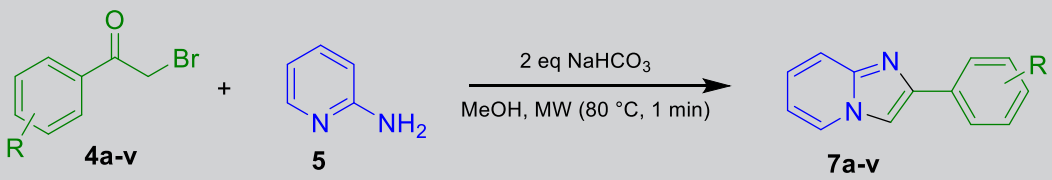
2.2 | Synthesis of imidazo[1,2-a]pyridine

Once the brominated compounds were obtained, the respective imidazo[1,2-*a*]pyridine nuclei were synthesized. Similarly, to the first step of the reaction, ThT, MW and mechanosynthesis strategies were applied to promote the condensation between 2-aminopyridine (**5**) with the phenacyl bromide molecules **4a-c** in a basic medium (Table 3). When comparing entries 1, 4, and 7, MW resulted as the best methodology, presenting a yield of 88% (Table 3, entry 4), while 80% and a mixture of byproducts were obtained by ThT and mechanosynthesis (entries 1 and 7, respectively). In general, higher yields of the reaction were obtained by means of the MW strategy (41%-88%) respect to those observed with the ThT path (30%-80%). Whereas, a mixture of the main product plus different byproducts were obtained in all the cases by means of mechanosynthesis. As for the synthesis of the precursors reported in Tables 1 and 2, the MW strategy allowed the synthesis of the imidazo[1,2-*a*]pyridine nuclei in only 1 minute. This is remarkably faster with respect to the ThT methodology, which required periods between 40 and 120 minute. This represents a clear advantage of the MW methodology in terms of yield of the reaction and time consumption.

Once the MW irradiation was selected as the most efficient energy source for the synthesis of imidazopyridine derivatives, modifications were made on the base equivalents of **5**, solvent, and reaction time in order to improve the yields of the reaction, and the results are shown in Table 4. Upon modification of the base equivalents ($NaHCO_3$) in the range of 1 to2 eq (entries 1-3), the best yield was obtained with 2 eq of the base (entry 3). Similarly, better yield was obtained when a high quantity of 2-aminopyridine equivalents were used (entry 6). Different solvents were used (entries 7-10) as well as neat reaction was tested (entry 11), resulting the lowest yield in absence of solvent (78%), and the best one in presence of methanol at 80°C (90%).

Therefore, considering the optimized conditions, a wide set of derivatives with structural diversity were

TABLE 5 Synthesis of **7a-v** according to the optimized conditions under MW irradiation



Entry	7	R	Yield (%)[a]
1	**7a**	H	88
2	**7b**	4-Br	85
3	**7c**	4-NO_2	41
4	**7d**	4-Me	86
5	**7e**	4-Cl	95
6	**7f**	4-OMe	94
7	**7g**	3,4-Cl	95
8	**7h**	NHPh	np
9	**7i**	2-OMe	24
10	**7j**	2-Cl	80
11	**7k**	3-Me	70
12	**7l**	3-OMe	50
13	**7m**	3-Cl	99
14	**7n**	3-Br	55
15	**7ñ**	2,5-OMe	90
16	**7o**	2,5-F	84
17	**7p**	2,5-Cl	99
18	**7q**	2,6-OMe	np
19	**7r**	2,6-Cl	70
20	**7s**	3,4-OMe	50
21	**7t**	3,4-F	60
22	**7u**	2,4-OMe	np
23	**7v**	2,4-F	98

Note: Reaction conditions: **4a-v** (1 eq), **5** (1.2 eq).
Note: np = the product is not formed.
[a]Isolate yield.

obtained and described in Table 5. This family of imidazo [1,2-*a*]pyridine nuclei is expected to constitute an important class of organic compounds for tracking and medicinal applications, by which their photophysical properties were evaluated.

2.3 | Photophysical characterization

The UV-Vis absorbance spectra of the different molecules were obtained in different solvents. For instance, the spectra of the product **7e** are presented in Figure 1, where narrow absorption bands were detected with maximal absorbance at 250, 253, 253, and 254 nm for MeOH, ACN, THF, and DCM, respectively. In all cases, a secondary band was observed in the range from 280 to 360 nm. Similar patterns were observed with the other synthesized molecules (Figures 47 and 48 in Data S1).

The photoluminescent behavior of the imidazo [1,2-*a*]pyridines were analyzed under 365 nm UV-excitation (Figure 2). As observed in the inset

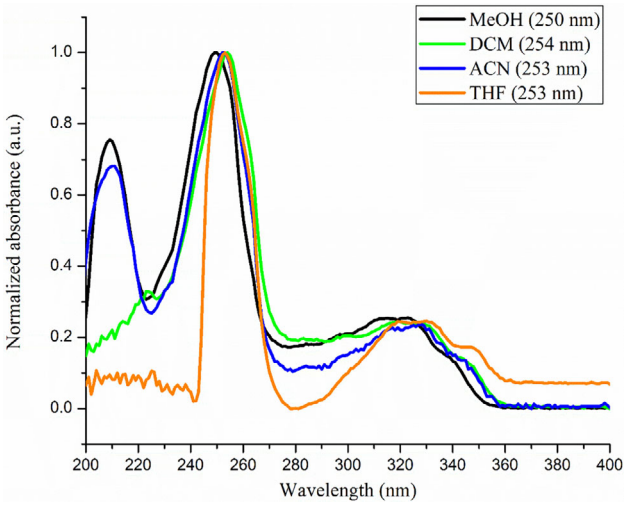


FIGURE 1 UV-Vis absorbance spectra of **7e** in different solvents

photographs, all the synthesized derivatives presented strong emission at the blue and violet regions of the electromagnetic spectrum upon dispersion in varied solvents. The wavelengths of maximal emission were registered at 395, 401, 428, 394, 398, and 400 nm for the compounds **7a**, **7d**, **7e**, **7v**, **7f**, and **7g**, respectively, while the most intense emission was presented by the methoxy-substituted derivative (**7f**). The different analyzed substitutions on the central imidazo[1,2-*a*]pyridine nuclei caused no deterioration of the fluorescent property, while important shifts were observed in the wavelength of maximal emissions, including regions from 394 until 428 nm. This strong emission was promoted by the extension of the electronic delocalization due to the presence of functionalized phenacyl substituents enclosed to the imidazo[1,2-*a*]pyridine nuclei.

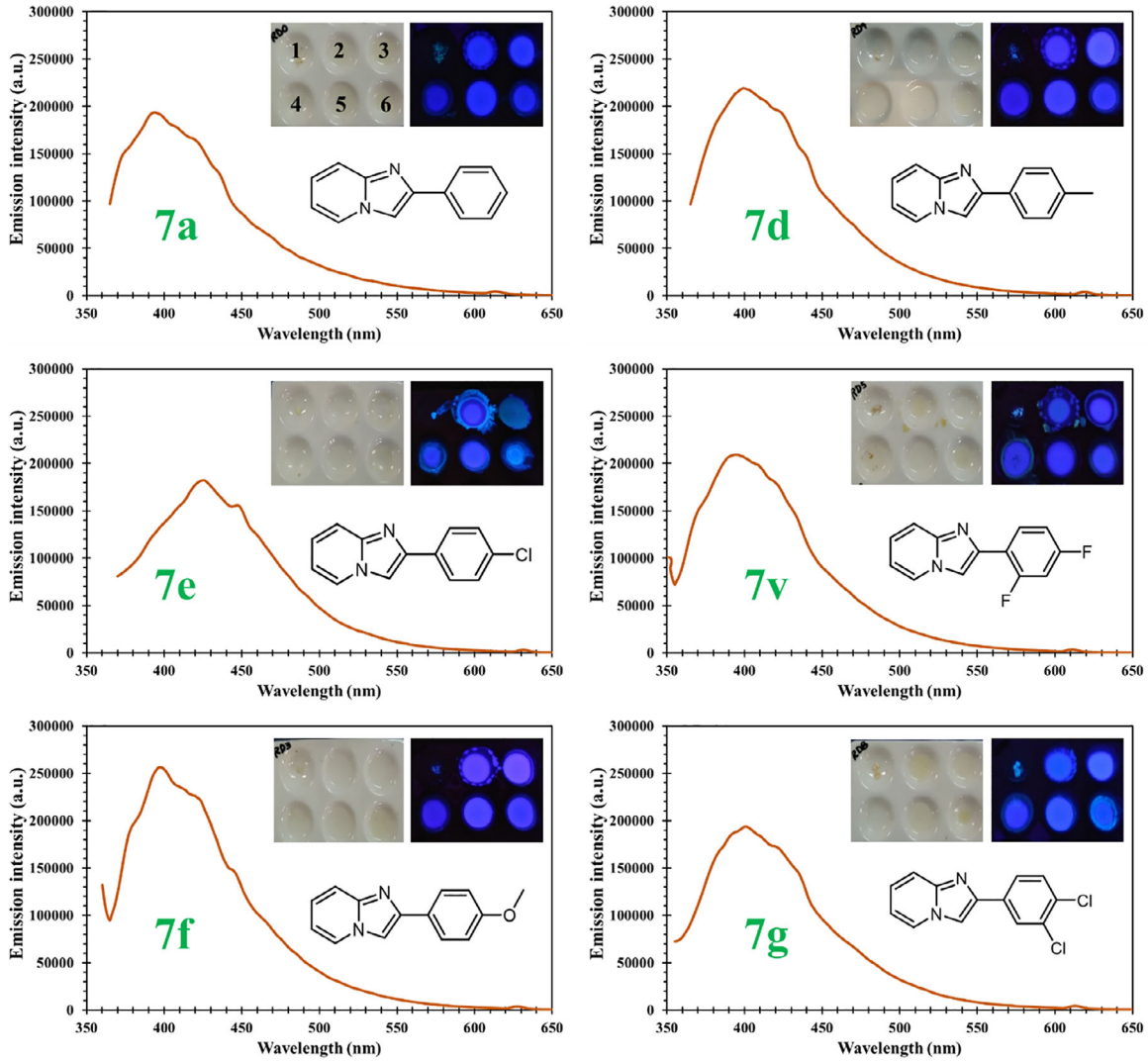


FIGURE 2 Fluorescence emission spectra of different imidazo[1,2-*a*]pyridine derivatives. The inset shows photographs of these molecules in different solvents under visible and 365 nm UV irradiation. (1) Dry compound, (2) MeOH, (3) THF, (4) Hexane, (5) ACN, and (6) DCM

3 | CONCLUSIONS

The reported method for the synthesis of imidazo [1,2-*a*]pyridine compounds assisted by microwave irradiation offers different advantages respect to the mechanosynthesis and the thermal treatment methodologies such as higher yields, and very short reaction times. In addition, both α-halogenation of ketones and condensation for the imidazopyridine derivatives production reactions assisted by microwave irradiation were obtained under more environmentally friendly conditions respect to the other evaluated methodologies. The importance of these molecules lies in their potential application as anti-inflammatory, antioxidant, antimicrobial, anti-proliferative, and fungicide agents, as well as contrast agents in the monitoring of the progression of the related diseases or the development of optical sensors by considering the photoluminescence response in the blue and purple regions.

4 | EXPERIMENTAL SECTION

4.1 | General considerations

Microwave-assisted reactions were performed in a closed vessel in a CEM Discover Microwave. The progress of the reaction was monitored by TLC (aluminum sheets, silica gel 60 F/UV254), using a hexane/ethyl acetate system as eluent. Visualization was carried out under UV light (254 and 365 nm). The products were purified by recrystallization with EtOH:H_2O. NMR spectra were obtained on a Bruker Ultrashield 500 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm, relative to tetramethylsilane (TMS) and $CHCl_3$ used as the internal standard. Melting points were determined on a digital Electrothermal 9200 melting point apparatus. Infrared spectra were recorded on a Perkin-Elmer Spectrum GX spectrophotometer by the ATR method using neat compounds. UV-vis spectra were obtained on a Jenway 7315 spectrophotometer.

4.2 | General procedure for the mechanosynthesis method synthesis of 4a-g

In a mortar were triturated acetophenone **1a-g** (1 eq), NBS (1.1 eq) and PTSA (0.1 eq) for 15 to 45 minute at room temperature. The progress of the reaction was monitored by TLC (hexane/EtOAc, 7:3). The resulting reaction mixture was extracted with CH_2Cl_2:H_2O, thus concentrated and finally dried under vacuum.

4.3 | General procedure for the thermal treatment method synthesis of 4a-g

In a ball flask were mixed NBS (1.1 eq) and PTSA (0.5 eq) in acetonitrile at 55°C, then the respective acetophenone (1 eq) was added and reacted for 40 to 120 minute. The progress of the reaction was monitored by TLC (hexane/EtOAc, 7:3). The resulting reaction mixture was extracted with CH_2Cl_2:H_2O, thus concentrated and finally dried under vacuum.

4.4 | General procedure for the microwave method synthesis of 4a-v

In a microwave tube were mixed acetophenone **1a-v** (1 eq), NBS **2** (1.1 eq) and *p*-TsOH **3** (1.5 eq) in CH_2Cl_2. The reaction mixture was exposed to microwave irradiation in a closed vessel (100 W) at 40°C for 15 minute. The progress of the reaction was monitored by TLC (hexane/EtOAc, 7:3). The resulting reaction mixture was extracted with CH_2Cl_2:H_2O, thus concentrated and finally dried under vacuum.

4.5 | General procedure for the mechanosynthesis method synthesis of 7a-c

In a mortar were triturated **4a-c** (1.0 eq), 2-aminopyridine **5** (1.1 eq) and sodium bicarbonate (2.0 eq). The reaction was triturated for 45 minute. The reaction progress was monitored by TLC in Hx:AcOEt.

4.6 | General procedure for the thermal treatment synthesis 7a-c

In a ball flask were mixed **4a-c** (1.0 eq), 2-aminopyridine **5** (1.1 eq) and sodium bicarbonate (1.5 eq) in acetonitrile as a solvent. The reaction was heated at 85°C for 40 to 120 minute. The reaction progress was monitored by TLC in Hx:AcOEt. Upon completion, the reaction mixture was extracted with CH_2Cl_2:H_2O and thus concentrated. The organic phase was recrystallized in EtOH:H_2O to obtain the respective compound.

4.7 | General procedure for the microwave method synthesis 2-phenylimidazo[1,2-*a*]pyridine (7a-v)

In a microwave tube were mixed **4a-v** (2.57 mmol), 2-aminopyridine **5** (3.08 mmol) and sodium bicarbonate (2.0 eq) in MeOH as a solvent. The reaction mixture was irradiated by microwave in a closed vessel (100 W) at

80°C for 1 minute. The reaction progress was monitored by TLC in Hx:AcOEt. Upon completion, the reaction mixture was extracted with CH_2Cl_2:H_2O and thus concentrated. The organic phase was recrystallized in EtOH:H_2O to obtain the compounds **7a-v**.

4.8 | Fluorescence and absorbance analysis

The products obtained were dissolved in methanol, THF, hexane, acetonitrile, and methylene chloride and were observed under an UV-lamp at 254 and 365 nm. 2×10^{-5} M solutions of the **7e** molecules were prepared in MeOH, THF and CH_2Cl_2, and 4×10^{-7} M in CH_3CN, and subsequently the UV-absorbance spectra obtained in a Jenway 7315 spectrophotometer. For the other products, only MeOH and CH_3CN were used at a 2×10^{-5} M concentration. The luminescence emission was analyzed with a Spectrograph Spectra Acton Pro 3500i and a R955 photomultiplier tube from Hamamatsu. The system was PC controlled with Spectra Sense software.

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