Microwave-promoted total synthesis of N-(α-hydroxybenzyl)formamides using DMSO/H₂O under neutral conditions

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ABSTRACT
A total synthesis route toward N-(α-hydroxybenzyl)formamides by microwave-assisted reaction of dichloroaziridines and aqueous dimethyl sulfoxide is described. The corresponding products were obtained in excellent yields with reduced reaction times. The obtained formamides were characterized by various techniques such as Infra red (IR), Nuclear Magnetic Resonance (NMR) and mass spectroscopy data.

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1. Introduction
Formamide is the simplest naturally occurring amide. Formamides are useful intermediates in various organic syntheses as their skeletons are present in different medicinally important compounds. Due to such importance of formamides, the antimicrobial activity of these compounds has also been evaluated.
for all new formamide derivatives (1–4). In addition, formamides have been also extensively employed in organic synthesis as the protecting group of amines (5), precursor for formamidines preparation (6), an intermediate for monomethylated amines from primary amines (7) and Lewis base organocatalysts for hydroxilation of imines (8). In spite of their interesting properties, various methods have been reported for the synthesis of formamide derivatives (9–13).

Aziridines, which are extremely important synthetic building blocks, are nitrogen equivalents of epoxides. These compounds are among the most fascinating intermediates in organic synthesis, acting as precursors of many complex molecules due to the strain incorporated in their skeletons and can be opened in a stereocontrolled manner with various nucleophiles, providing access to a wide range of important nitrogen-containing products (14–17).

The organic reactions environmentally benign and aziridines can be found in natural products such as mitomycin (I) (18), porfiromycin (II) and mitiromycin which are potent antitumor and antibiotic agents (Figure 1). The antitumor and antibiotic properties of a great number of aziridine-containing compounds are of high significance among other biological properties which make them attractive synthetic targets in their own right (19–21). Because of the importance of aziridine derivatives, several of these compounds have been reported (22–24).

Microwave as a source of energy was first invented by Percey Spencer in 1947. Gedye et al. have the credit for introducing microwave in organic syntheses in 1986 (25,26). This synthetic method has shown broad applications as a very efficient way to accelerate the course of many organic reactions, producing high yields and higher selectivity, lower quantities of side products and, consequently, easier work-up and purification of the products (27–29). The microwave-induced enhancement of organic reactions is currently a focus of attention for chemists. During recent years, microwave has been extensively used for carrying out chemical reactions and has become a useful non-conventional energy source for performing organic synthesis (30–32).

In our previous studies, we have reported the preparation of novel N-(a-hydroxybenzyl)formamides under thermal conditions (33). With attention to the importance of these compounds, in continuation of our ongoing work on the synthesis of Schiff base derivatives (34–36), herein we wish to report the usage of microwave irradiation on the efficient and rapid synthesis of N-(a-hydroxybenzyl)formamides.

2. Results and discussion

In this research, total synthesis of N-(a-hydroxybenzyl)formamides has been investigated (Scheme 1). Considering this, in view of development green chemistry, Schiff base derivatives have been synthesized through solvent-free condensation reaction of benzaldehyde and aniline derivatives in the presence of montmorillonite as a heterogenous catalyst. In continuation, the preparation of gem-dichloroaziridines has been studied. There are three main routes to the gem-dichloroaziridine through in situ dichlorocarbene generation by using chloroform, hexachloroacetone or ethyl dichloroacetate under high alkaline conditions and phase-transfer catalyst (PTC) (37–41). In these methods, by using PTC, the amount of used NaOH base has been very high compared with the used base amount in our previously reported work under ultrasonic irradiation (35). Recently, Song et al. reported the synthesis of gem-dichloroaziridines through the addition reaction of dichlorocarbene generated in situ using NaOH/CHCl₃ and polyethylene glycol (PEG) as a PTC (42). In our work, gem-dichloroaziridines were synthesized without any PTC in the presence of ultrasonic irradiation (35) in excellent product yields by reducing the reaction time to 10 min and the work-up of products was easier than the same reaction by using PTC (42).

Here, the addition reaction of Schiff bases with the dichlorocarbene intermediate which was introduced in situ in the reaction medium by treatment of CHCl₃ and NaOH under ultrasonic irradiation was investigated and the desired dichloroaziridine derivatives were obtained. Hydrolysis of geminal-dihalo compounds to the corresponding carbonyl compounds often employs harsh reaction conditions, such as strong acids or strong bases under high temperature (43). In the next step of our research, hydrolysis of gem-dichloroaziridine compounds toward N-(a-hydroxybenzyl)formamides was surveyed using Dimethyl sulfoxide (DMSO)/H₂O under microwave irradiation. Because of the influence of power of microwave irradiation on the yield of N-(a-hydroxybenzyl)formamide derivatives, it is...
necessary to optimize the power of microwave irradiation. Thus, we first performed this hydrolysis with 2,2-dichloro-1,3-bis(4-chlorophenyl)aziridine (4d) as a model in DMSO/H2O under various power conditions of microwave irradiation. It was observed that the hydrolysis reaction in the presence of microwave irradiation with power 300 W afforded the best result as the product was obtained with 60% isolated yield during 15 min (Table 1, entry 3). No product was formed in the absence of microwave irradiation at room temperature even after 72 h (Table 1, entry 5).

However, under conventional heating at 70°C during 70 min, the desired product was obtained in very low yield (Table 1, entry 3 vs. entry 6).

The acceleration of the reaction by microwave exposure results directly from interactions between the material and electromagnetic field leading to the thermal and specific effects. For microwave heating, the substance must possess a dipole moment (such as water, dimethyl sulfoxide and dichloroaziridine in this reaction). A dipole is sensitive to external electric field and tries to align itself with the field by rotation. If submitted to an alternating current, the electric field is inverted at each alternate and therefore dipoles tend to move together to follow the inverted electric field. Such a characteristic induces rotation and friction of the molecules, which dissipates as internal homogeneous heating. While in conventional heating, heat is driven into the substance, passing first through the walls of the vessel in order to reach the solvent and the reactants. Thus, it is a slow and inefficient method for transferring energy into the reacting system. Reactions that usually take many hours or days, under microwave irradiation can be run in a considerably shorter time. Microwave includes the following advantages over the conventional heating: uniform heating throughout the material, increased process speed, high efficiency of heating, reduction in unwanted side reaction, purity in the final product, improved reproducibility, avoiding environmental heat loss, low wastage of heating reaction vessel and low operating cost.

In continuation to improve the yield of the reaction, optimization time of the reaction was performed (Table 2).

### 2.1. Survey the microwave irradiation effect vs. conventional heating

In microwave irradiation, the microwaves couple directly with the molecules of the entire reaction mixture, leading to a rapid rise in the temperature. Since the process is not limited by the thermal conductivity of the vessel, the result is an instantaneous localized superheating of any substance that will respond to either dipole rotation or ionic conductivity. Only the contents in the reaction vessel are heated and not the vessel itself; better homogeneity and selective heating of polar molecules might be achieved. The acceleration of chemical reactions by microwave exposure results from the interactions between the material and electromagnetic field leading to the thermal and specific (non-thermal) effects. Microwave heating plays an important role in the treatment of domestic and hazardous industrial and nuclear waste. Microwave heating can be advantageously used for waste management in areas where human exposure can cause health problems.

In conventional heating, heating reactants are slowly activated by a conventional external heat source. Heat is driven into the substance, passing first through the walls of the vessel in order to reach the solvent and the reactants. This is a slow and inefficient method for transferring energy into the reacting system. The application of microwave irradiation on the hydrolysis reaction of gem-dichloroaziridines toward N-(α-hydroxybenzyl)formamides dramatically improved the time required to obtain these compounds.

Then to ascertain the scope and limitation of the present reaction, several gem-dichloroaziridine compounds were reacted with DMSO/H2O (4.5 mL /0.8 mL) under microwave irradiation with power 300 W and desired products were prepared. The results are summarized in Table 3. As can be seen from Table 3, N-(α-hydroxybenzyl)formamides were obtained with

### Table 1. Survey effect of microwave power on synthesis of 5d by treatment of aziridine 4d with DMSO/H2O.

| Entry | Power (W) | Time (min) | Yield (%) |
|-------|-----------|------------|-----------|
| 1     | 100       | 15         | 30        |
| 2     | 180       | 15         | 45        |
| 3     | 300       | 15         | 60        |
| 4     | 450       | 15         | 60        |
| 5     | 1.5       | 4320       | 70        |
| 6     | Conventional heating | 70 | 10 |

*aReaction conditions: 4d (0.01 mol), DMSO (4.5 mL) and H2O (0.8 mL),
*bIsolated Yields.
*cHeating under 70°C.

### Table 2. Optimization time of the reaction of aziridine 4d with DMSO/H2O under microwave irradiation.

| Entry | Time (min) | Yield (%) |
|-------|------------|-----------|
| 1     | 15         | 60        |
| 2     | 25         | 70        |
| 3     | 35         | 85        |
| 4     | 50         | 98        |
| 5     | 55         | 98        |

*aReaction conditions: 4d (0.01 mol), DMSO (4.5 mL), H2O (0.8 mL) and microwave (MW) irradiation power (300 W).
*bIsolated Yields.
excellent yields without any side reaction product, easier work up, high purity and very short reaction times compared with the conventional heating conditions (33).

The structure of the products 5a–5i was confirmed by spectroscopic methods such as IR, $^1$H-NMR, $^{13}$C-NMR and mass spectroscopy. In the IR spectra, the stretching frequency of aromatic C9C bonds was observed between 1489 and 1598 cm$^{-1}$. The stretching vibration of the O–H group appeared at 3285–3340 cm$^{-1}$, and the absorption in the region between 1639 and 1659 cm$^{-1}$ was assigned to the formyl group.

Table 3. Synthesis of N-(α-hydroxybenzyl)formamides through the reaction of gem-dichloroaziridine derivatives and DMSO/H$_2$O under microwave irradiation (300 W).

| Entry | Reactant | Product | Time (h) | Yield$^a$ (%) |
|-------|----------|---------|----------|---------------|
| 1     | ![Image](image1.png) | ![Image](image2.png) | 1.8       | 93            |
| 2     | ![Image](image3.png) | ![Image](image4.png) | 1.6       | 95            |
| 3     | ![Image](image5.png) | ![Image](image6.png) | 1.6       | 94            |
| 4     | ![Image](image7.png) | ![Image](image8.png) | 0.83      | 98            |
| 5     | ![Image](image9.png) | ![Image](image10.png) | 0.83      | 98            |
| 6     | ![Image](image11.png) | ![Image](image12.png) | 1.3       | 93            |
| 7     | ![Image](image13.png) | ![Image](image14.png) | 1.3       | 94            |
| 8     | ![Image](image15.png) | ![Image](image16.png) | 0.83      | 97            |
| 9     | ![Image](image17.png) | ![Image](image18.png) | 0.83      | 98            |

$^a$Isolated yields.
In the $^1$H-NMR spectra, the CH–OH group appeared at $\delta$ (H) 5.09–5.28. The signals at $\delta$ (H) 6.99–8.23 were assigned to the aromatic protons. The H–C9O proton was observed at $\delta$ (H) 9.97–10.11, and the OH signal appeared at $\delta$ (H) 6.40–6.80. The $^1$H-NMR spectra of 5d are shown in Figure 2. In the $^{13}$C-NMR spectra, $\delta$ (C) 73.0–74.1 was assigned to the O–C–N group and $\delta$ (C) 171.0–172.0 to the H–C9O group. The mass spectra (electron ionization, EI) of all products showed the corresponding molecular ion peak.

**Figure 2.** The $^1$H NMR spectra of 5d.

**Scheme 1.** Total synthesis of N-(α-hydroxybenzyl)formamides.
2.2. The proposed mechanism for the synthesis of N-(α-hydroxybenzyl)formamides

The proposed mechanism of hydrolysis of gem-dichloroaziridine to N-(α-hydroxybenzyl)-formamides using DMSO/H₂O under microwave irradiation is presented in Scheme 2.

In conclusion, a simple method for the transformation of dihalo compounds into the corresponding formamide is described herein. The reaction can be carried out conveniently and proceeds with excellent yield, short reaction time and high purity. To the best of our knowledge, this is the first example using sulfoxide as the sole reagent to convert a gem-dihalo precursor into the corresponding aldehyde under microwave irradiation. The scope and limitations of this reaction for the preparation of other important functional groups as well as building blocks are currently under investigation.

3. Experimental

3.1. Material and apparatus

All the materials were of commercial reagent grade. The Schiff bases and 2,2-dichloroaziridines were prepared and characterized according to our previously reported procedures (34,35). IR spectra were recorded as KBr pellets on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet FTIR spectrophotometer. ¹H-NMR and ¹³C-NMR were recorded in d₆-DMSO solvents on a Bruker DRX-400 spectrometer with tetramethylsilane as the internal reference. Mass spectra were recorded on a Finnigan MAT 44S by the El mode with an ionization voltage of 70 eV. The elemental analyses (C. H. N.) were obtained from a Carlo ERBA Model EA 1108 analyzer. The Microwave conditions were carried out in a microwave oven specially designed for organic synthesis (Milestone LAVIS Basic Microwave). Melting points obtained with a Yanagimoto micro melting point apparatus are uncorrected. The purity determination of the substrates and reaction monitoring were accomplished by thin layer chromatography (TLC) on silica–gel polygram SILG/UV 254 plates (from Merck Company).

3.2. Typical procedure for the synthesis of Schiff bases 3a–3i

In this pathway, a mixture of benzaldehyde (0.21 g, 2 mmol), aniline (0.25 g, 2 mmol) and montmorillonite (0.5 g) was poured in a mortar and thoroughly grinded. The resulting mixture was placed in a flask and mechanically stirred for 10 min at room temperature conditions. The progress of the reaction was monitored by TLC. After completion of the reaction, chloroform (15 mL) was added to the reaction mixture, filtered off, with vaporization of the filtrate, and the solid product was obtained. The crude product was purified by recrystallization from petroleum ether and the pure Schiff base 3a was obtained as a white solid in 95% yield. The Schiff base products were identified by spectroscopic data (34).

3.3. General procedure for the preparation of 2,2-dichloroaziridines 4a–4i

Measured quantities of NaOH (0.075 mol, 3 g) were dissolved in 30 mL of water and the obtained solution was introduced to a 100 mL flask. The ultrasonic probe was immersed directly into the reactor. Then, the Schiff base (organic reactant; 0.028 mol, 8.2 g) dissolved in chloroform (0.07 mol, 8.3 mL) was gradually added drop wise to the NaOH solution under ultrasonic irradiation with a power of 67 W. The progress of the reaction was monitored by TLC. After the completion of the reaction in 15 min, the solution was separated and the portion of aqueous solution was extracted with diethylether. Magnesium sulfate was
also added to absorb the residual water. The organic solvent and other residues were stripped in a vacuum evaporator. The pale yellow solid, 2,2-dichloro-1,3-diphenylaziridine, was obtained in 96% yield, m.p. = 100–102°C. All of the diarylaziridine products were identified by physical and spectroscopic data (35).

3.4. A typical procedure for the synthesis of N-(4-chlorophenyl)-N-[4-(chlorophenyl)hydroxymethyl]formamide (5d)

A solution of 2,2-dichloro-1,3-bis(4-chlorophenyl)aziridine (4d; 3.3 g, 0.01 mol) in 4.5 mL (0.06 mol) of DMSO and 0.8 mL of H2O was poured into conical flask. The reaction mixture was irradiated with microwave at 300 W for 50 min (0.83 h). After completion of the reaction, the mixture was poured into cold H2O (20 mL), the resulting mixture was extracted with CHCl3 (2 x 10 mL) and the organic layer was dried with Na2SO4 and concentrated. The crude product was recrystallized from MeOH to give 5d in 98% yield in high purity. All of the other formamides (5a–5i) were similarly prepared by the same procedure. The obtained products were identified by physical and spectral data and shown as follows:

N-(Hydroxyphenylmethyl)-N-phenylformamide (5a): White solid. m.p. 120–122°C, IR (KBr): 3300 (OH), 3090, 2930, 1649 (C9O), 1509, 1590 (C9C, Ar), 1H-NMR (d6-DMSO): 5.09 (s, CH–OH), 6.48 (s, OH), 6.99–7.56 (m, 10 arom. H), 10.01 (s, CHO). 13C-NMR (d6-DMSO): 74.1, 121.0, 127.0, 128.0, 131.1, 132.0, 137.0, 138.1, 141.1, 171.0. MS: 277 (6, [M++2]), 275 (14, M+), 143 (25), 141 (56), 134 (60), 113 (85). Anal. Calcd. for C14H13NO2: C 73.99, H 4.93, N 9.79; found: C 73.50, H 5.59, N 9.99.

N-(Hydroxyphenylmethyl)-N-(4-chlorophenyl)formamide (5b): Pale-yellow solid. m.p. 135–137°C, IR (KBr): 3307 (OH), 3088, 2930, 1659 (C9O), 1514, 1588 (C9C, Ar), 1H-NMR (d6-DMSO): 5.15 (s, CH–OH), 6.50 (s, OH), 7.34–7.69 (m, 9 arom. H), 10.00 (s, CHO), 13C-NMR (d6-DMSO): 73.0, 119.4, 122.8, 128.9, 129.0, 131.9, 132.3, 136.0, 151.0, 171.0. MS: 307 (13, [M++2]), 305 (13, M+), 199 (39), 197 (39), 171 (65), 169 (65), 157 (46), 155 (46), 107 (75), 77 (100). Anal. Calcd. for C14H12ClNO2: C 62.93, H 4.93, N 9.79; found: C 63.50, H 5.59, N 9.99.

N-(4-Chlorophenyl)hydroxymethyl-N-(4-methylphenyl)formamide (5c): White solid. m.p. 127–129°C, IR (KBr): 3267 (OH), 3090, 2900, 1653 (C9O), 1599, 1492 (C9C, Ar), 1H-NMR (d6-DMSO): 5.08 (s, CH–OH), 6.47 (s, OH), 7.33–7.72 (m, 9 arom. H), 10.08 (s, CHO), 13C-NMR (d6-DMSO): 74.0, 121.8, 127.0, 128.4, 128.6, 129.0, 129.1, 137.9, 141.0, 171.8. MS: 263 (6, [M++2]), 261 (18, M+), 156 (13), 154 (35), 128 (18), 126 (55), 107 (60), 77 (100). Anal. Calcd. for C14H12ClNO2: C 64.25, H 4.62, N 5.35; found: C 64.58, H 4.92, N 5.39.

N-(4-Chlorophenyl)-N-[4-(chlorophenyl)hydroxymethyl]formamide (5d): White solid, m.p. 138–140°C, IR (KBr): 3285 (OH), 3090, 2900, 1639 (C9O), 1590, 1490 (C9C, Ar), 1H-NMR (d6-DMSO): 5.07 (s, CH–OH), 6.50 (s, OH), 7.33, 7.41, 7.52, 7.73 (2 AA'BB', JAB = 7.4, 2.9, 8 arom. H), 10.11 (s, CHO), 13C-NMR (d6-DMSO): 73.7, 121.8, 127.7, 128.6, 128.8, 132.7, 137.9, 140.1, 171.5. MS: 299 (3, [M++4]), 297 (6, [M+2]), 295 (10, M+), 156 (28), 154 (39), 143 (55), 141 (75), 125 (40), 113 (50), 111 (38), 77 (100). Anal. Calcd. for C14H13ClNO2: C 65.75, H 4.26, N 7.15; found: C 66.09, H 4.28, N 7.15.
(20), 111.5 (55), 105 (45), 91 (96), 77 (100). Anal. Calcd. for C13H14BrN2O5: C 46.34, H 5.12, N 5.08; found: C 46.05, H 5.46, N 5.14.

N-(4-Bromophenyl)-N-[hydroxy(4-nitrophenyl)methyl] formamide (5): Yellow liquid. IR (KBr): 3289 (OH), 3100, 2900, 1649 (C=O), 1589, 1488 (C=C, Ar). 1H-NMR (DMSO-d6): 6.10 (s, CH=O); 6.95, 7.0 (2 H, 10 N +2)); 7.10 (10, t, 137); 7.50 (10, M^+2)), 7.98 (18, 14.35 (55), 141.5 (70), 171 (48), 169 (48), 113.5 (45), 111.5 (60), 77 (100); Anal. Calcd. for C14H11BrN2O4: C 48.56, H 3.46, N 8.06.

Disclosure statement

No potential conflict of interest was reported by the authors.

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