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Green chemical approach for facile one-pot synthesis of 2,4,8-trisubstituted-1,5-benzothiazepines and their dioxides under microwave irradiation

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RESEARCH ARTICLE

Green chemical approach for facile one-pot synthesis of 2,4,8-trisubstituted-1,5-benzothiazepines and their dioxides under microwave irradiation

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2,4,8-Trisubstituted-1,5-benzothiazepines (synthesized '*in situ*') have been selectively oxidised to the corresponding sulfones via a one-pot procedure in 9 min. with 91% yield under microwave irradiation coupled with clay-supported ferric nitrate.

Keywords: 2,4,8-Trisubstituted-1,5-benzothiazepines; Selective oxidation; Microwave irradiation; Clayfen

1. Introduction

The immense chemotherapeutic applications of 1,5-benzothiazepines [1, 2], especially that of Diltiazem in the treatment of cardiovascular ailments, has generated great interest in this class of compounds.

Sulfoxides are also valuable reagents in the application of organosulfur compounds in organic synthesis [3]. Sulfoxidation of various heterocycles has been extensively studied, *e.g.* 1,4-benzothiazines [4], phenothiazines, benzothiazepine [5] derivatives as potent psychotropics, anticonvulsants, hypolipidemics, and pesticides. Given the enhanced bioactivity of sulfonyl derivatives of benzothiazepine derivatives, the sulfide oxidation of some of the title compounds was investigated so as to explore their medicinal properties.

Conventionally, oxidative access to sulfoxides/sulfones of 2,3-dihydro-1,5-benzothiazepines [6] and its derivatives [7] has been reported using $KMnO_4$, H_2O_2, +AcOH/oxalic acid, conc. HNO_3, *m*-chloroperbenzoic acid, $NaIO_4$ and dimethyldioxirane. The procedures often involve prolonged refluxing in volatile solvents, acidic catalysts with tedious workup procedures and a further need of solvents for the purification process, eventually affording the required product in lower yield. Microwave irradiation (MWI) promotes the synthesis of

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various compounds [8], accelerating chemical reactions through the selective absorption of microwaves by polar molecules. Recently, the coupling of MWI with solid supports under solvent-free conditions has attracted much attention due to reduced reactions times, ease of workability, and the opportunity to work with open vessels under scale up conditions. Solvent-free $NaIO_4$-catalyzed oxidation of sulfide into sulfone under microwaves has also been reported by Varma *et al* [9].

Among the various solid supports, clay has been used extensively in view of its Lewis acid character [10]. Clay doped with Fe(III) nitrate (Clayfen) has been reported by Varma *et al*. for the oxidation of a wide variety of sulfides [11] (acyclic, cyclic and aromatic), selectively to the corresponding sulfoxide under solvent-free conditions. In addition to this favorable chemoselective aspect, this solid-state reaction process is also applicable to long-chain aliphatic sulfides, which are normally insoluble in polar solvents and are therefore difficult to oxidize by conventional methods. Although Clayfen has been used in several organic reaction transformations, its use for exploring the oxidation of heterocyclic sulfides has received no attention.

2. Results and discussion

In continuation to our endeavors in this general area of reactions that are accelerated by microwave irradiation [12] we have developed a one-pot procedure for the synthesis of 2,4,8-trisubstituted-1,5-benzothiazepine-1,1-dioxides by varying different parameters of inorganic solid supports (table 1, scheme 1). The key intermediates, *i.e.* 1,5-benzothiazepines required for the oxidation reactions were prepared *in situ* under microwave irradiation in quantitative yields. For comparison, the reaction was also tried conventionally, which required a three-step procedure involving prolonged reflux in volatile organic solvents and corrosive acids (HCl/AcOH).

The relative amount of support:substrate:ferric nitrate was optimized, with the best results obtained for 0.001 mole of $Fe(III)NO_3$ catalyst in 2 g of the support, keeping the molar ratio of the substrate constant, *i.e.* 0.001 mole. In another experiment, a 1:3 molar ratio of substrate:ferric nitrate also afforded sulfones, but in lower yields due to concomitant formation of byproducts that, however, could not be separated and identified.

In conclusion, a simple, rapid and high yielding MW accelerated method for the selective oxidation of heterocyclic sulfides to sulfones has been developed that occurs under solvent-free conditions using $Fe(III)NO_3$ impregnated on clay, avoiding the drastic conditions used in earlier reactions [6, 7].

Synthesis of new key intermediates (**6a**–**g**) was confirmed on the basis of spectral studies [13]. IR spectra (ν, cm^{-1}) of **6a**–**g** did not reveal absorption bands due to carbonyl and

Table 1. Comparative results for the synthesis of **7a** ($X = OC_2H_5$).

Entry	Method	Solvent/support	Time (min.)	Temp[a] · (°C)	Yield[b] (%)
1	Δ	gl. AcOH + H_2O_2	720	Reflux	42
2	MW	H_2O_2	20	60	–
4	MW	H_2O_2 + montmorillonite KSF	15	105	65
6	MW	KSF + $Fe(III)NO_3$	9	110	91
7	MW	K10 + $Fe(III)NO_3$	6	125	88
8	Δ	KSF + $Fe(III)NO_3$	9	110	nil
9	Δ	KSF + $Fe(III)NO_3$	60	110	10

[a]Final temperature measured at the end of microwave irradiation by introducing a glass thermometer in the reaction mixture.
[b]Yield of the isolated products.

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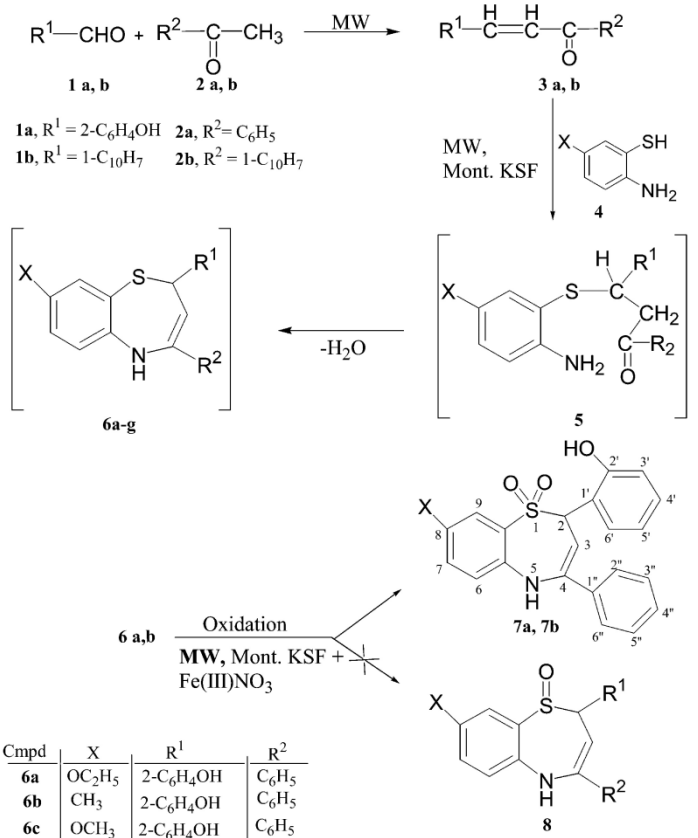

Cmpd	X	R^1	R^2
6a	OC_2H_5	2-C_6H_4OH	C_6H_5
6b	CH_3	2-C_6H_4OH	C_6H_5
6c	OCH_3	2-C_6H_4OH	C_6H_5
6d	Cl	2-C_6H_4OH	C_6H_5
6e	Br	2-C_6H_4OH	C_6H_5
6f	OCH_3	1-$C_{10}H_7$	1-$C_{10}H_7$
6g	CH_3	1-$C_{10}H_7$	1-$C_{10}H_7$
7a	OC_2H_5	2-C_6H_4OH	C_6H_5
7b	CH_3	2-C_6H_4OH	C_6H_5

SCHEME 1

primary amino group and showed characteristic absorptions in the range 3500–3600 (OH), 3350–3320 (NH), indicating the formation of **6** instead of the Michael adducts **5**. ^{1}H NMR spectra (δ, ppm) of **6a**–**g** show a pair of doublets at 6.22–6.65 (d, 1H, C-2-H, $J = 6$ Hz) and at 6.69–7.05 (d, 1H, C-3-H, $J = 6$ Hz) along with aromatic protons at 6.25–7.15 [(m, Ar-H, 12H (**6a**–**e**), 17H (**6f**, **g**)] and N–H proton at 4.06 (bs, exchangeable with D_2O).

The ^{13}C NMR spectrum (δ, ppm) of **6c** shows signals at 41.2 (C_2), 56.2 (OCH_3), 106.7 (C-3), 108–130.7 (16 aromatic ring carbons), 158.5 (C-4–N), 161.0 (Ar-C_8OCH_3), 168.3 (Ar-C-2-OH). The mass spectrum of **6e** shows m/z at 409 $[M]^+$ (14.1%), which corresponds to the molecular weight of the compound. Other peaks appear at 407 (24.8%), 384 (8.1%), 301 (100%), 300 (75.2%), *etc*.

Further oxidation of **6a**, **b** to give exclusively the corresponding sulfones **(7a, b)** was also confirmed by spectral studies. IR (ν, cm^{-1}) spectra show characteristic bands in the region 1395–1240, 560–520 and 1160–1125 analogous to three fundamental absorption bands in the sulfur dioxide molecule [14]. Other characteristic N–H and C–S absorption bands appear at 3420–3400 and 1110–1095, respectively, involving shifts to a higher frequency region as compared with starting materials (**6a, b**). ^{1}H NMR spectra (δ, ppm) of **7a**, **b** show signals for aromatic and NH protons in the expected range along with a pair of doublets at 6.80–6.82

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(d, 1H, C-2-H, $J = 6$ Hz) and at 7.32–7.35 (d, 1H, C-3-H, $J = 6$ Hz). The downfield shift of C-2 and C-3 protons signals further supports the oxidation of **6a**, **b** into their corresponding sulfones.

The ^{13}C NMR spectrum (δ, ppm) of **7a** further confirms the formation of sulfones. Signals appear at 14.3 ($OCH_2\underline{C}H_3$), 42.9 (C_2), 66.1 ($O\underline{C}H_2CH_3$), 99.6 (C-3) along with usual signals due to aromatic carbons at 110–146.7. A shift in the IR spectral pattern and the pronounced downfield shift of C-2 and C-3 in ^{13}C NMR spectrum of **7a**, as compared with that in **6a**, can be explained by the strong electron-withdrawing nature of SO_2 group [15]. The formation of sulfone was also confirmed by the mass spectrum of **7a**, which exhibits a molecular ion peak at m/z 407 $[M]^+$ (49.6%), corresponding to the molecular weight of the sulfone, and other relevant peaks appear at 408 $[M + 1]^+$ (75%), 395 (17.0%), 384 (37.6%), 377 (0.8%), 304 (100%), 300 (82%), 168 (68.3%).

3. Experimental

Melting points were determined in open glass capillaries and are uncorrected. IR spectra (ν_{max} in cm^{-1}) were recorded on a Perkin-Elmer infracord spectrophotometer (Model-577) in KBr pellets. ^{1}H and ^{13}C NMR spectra [$CDCl_3$ + DMSO-d_6] were taken on a Jeol FX 90Q spectrophotometer at 89.55 and 22.45, MHz respectively, using TMS as an internal reference. Mass spectra were recorded on a Jeol D-300 spectrometer at an ionization potential of 70 eV. Elemental analyses for C, H and N were performed on a Heraeus Carlo Erba 1108 analyzer. Microwave-assisted reactions were carried out using a BPL BMO domestic oven operating at 700 W, generating a frequency of 2450 MHz.

Clayfen [16] and 5-substituted-2-aminobenzenethiols (**4a-e**) were synthesized by reported methods [17].

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3.1 *1-(1-Naphthyl)-3-(1-naphthyl)-2-propenone (3b)*

An equimolar mixture of **1b** and **2b** (0.01 mol) was dissolved in the minimum quantity of alcohol required to make slurry. To this was added 1 pellet of KOH and the contents were irradiated inside a microwave oven for 7 min. Yellow crystals appeared on cooling, which were filtered off, and washed with water to give pure crystalline compound **3b**. Mp 141°C, yield = 94%. TLC: R_f (benzene-ethyl acetate, 8:2) 0.56. Found: C, 89.51, H 5.21, $C_{23}H_{16}O$ required C, 89.58, H, 5.29. IR ν(cm^{-1}): 1675 (C=O) 1615–1600 (C=C); HMR (δ, ppm): 6.25 (d, 1H, C-2-H, $J = 18$ Hz), 6.90 (d, 1H, C-3-H, $J = 18$ Hz), 6.40–7.15 (m, 14 H, Ar-H).

3.2 *2-Hydroxybenzalacetophenone (3a)*

3a was prepared following the same procedure, mp 154°C; yield = 98%. (Lit. [17] mp 156°C, 71%).

3.3 *8-Ethoxy-2-(2-hydroxyphenyl)-4-phenyl-2,5-dihydro-1,5-benzothiazepine (6a)*

This was prepared by two routes:

A. Conventional synthesis. An equimolar mixture of 2-amino-5-ethoxybenzenethiol (**4a)** (0.001 mol, 0.169 g) and 2-hydroxybenzalacetophenone (**3a)** (0.001 mol, 0.224 g) in dry ethanol (20 mL) was saturated with dry hydrogen chloride gas until boiling and the mixture was then refluxed for 6 h (TLC). It was then cooled and concentrated to obtain a solid, which on crystallization from methanol gave **6a**.

B. MW-induced dry media synthesis. An equimolar mixture of **3a** and **4a** (0.001 mol) was adsorbed on a suitable solid support (KSF) (20% by weight of the reactants) *via* a solution of ethanol. The solvent was evaporated in a rotary evaporator and the dry free-flowing powder thus obtained was irradiated inside a MW oven for an appropriate time at 640 W (TLC). The

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Table 2. ^{1}H NMR spectral (δ ppm) and analytical data of **6a–g** and **7a–b**.

Compd	Yield (%) Δ/MW	Mp (°C)	R_f^a	CH	NH/OH[b] (s, b)	X	Ar–H(m)	Elemental analyses (calcd./found) C	H	N
6a	65/88	172	0.70	6.60 (C-2, 1H, d, $J = 6$ Hz), 6.70 (C-3, 1H, d, $J = 6$ Hz)	4.35/10.8	1.42 (t, 3H, $J = 6$, $\underline{CH_2}$) 4.09 (q, 2H, $J = 6$, $\underline{CH_3}$)	6.34–7.40 (m, 12 H)	73.45/74.23	5.64/5.74	3.73/3.68
6b	67/82	120	0.68	6.67(C-2, 1H, d, $J = 6$ Hz), 6.71 (C-3, 1H, d, $J = 6$ Hz)	4.28/10.9	2.72 (s, 3H, $\underline{CH_3}$)	6.25–7.35 (m, 12 H)	76.49/77.29	5.54/5.60	4.05/4.00
6c	63/78	90	0.75	6.65(C-2, 1H, d, $J = 6$ Hz), 6.69 (C-3, 1H, d, $J = 6$ Hz)	4.20/11.4	3.73 (s, 3H, $\underline{OCH_3}$)	6.20–7.30 (m, 12 H)	73.01/72.52	5.30/5.18	3.88/3.79
6d	62/80	131	0.74	6.68(C-2, 1H, d, $J = 6$ Hz), 6.70 (C-3, 1H, d, $J = 6$ Hz)	4.30/11.2	–	6.10–7.15 (m, 12 H)	68.94/69.69	4.41/4.36	3.83/3.89
6e	67/90	102	0.68	6.66(C-2, 1H, d, $J = 6$ Hz), 6.70 (C-3, 1H, d, $J = 6$ Hz)	4.38/12.5	–	6.00–7.20 (m, 12 H)	61.47/61.90	3.93/4.00	3.41/3.45
6f	42/68	138	0.66	6.25(C-2, 1H, d, $J = 6$ Hz), 7.12 (C-3, 1H, d, $J = 6$ Hz)	4.00/–	3.55 (s, 3 H, $\underline{OCH_3}$)	6.15–7.55 (m, 17 H)	80.89/79.76	5.16/5.24	3.14/3.12
6g	54/72	152	0.54	6.22(C-2, 1H, d, $J = 6$ Hz), 7.17 (C-3, 1H, d, $J = 6$ Hz)	4.15/–	2.67 (s, 3 H, CH_3)	6.15–7.50 (m, 17 H)	83.91/85.33	5.36/5.32	3.26/3.22
7a	48/91	142	0.74	6.82(C-2, 1H, d, $J = 6$ Hz), 7.35 (C-3, 1H, d, $J = 6$ Hz)	5.15/13.8	1.25 (t, 3 H, $J = 7$, $\underline{CH_2}$) 3.48 (q, 2 H, $J = 7$, $\underline{CH_3}$)	7.05–8.01 (m, 12 H)	67.79/68.59	5.19/5.28	3.44/3.48
7b	51/89	168	0.76	6.80(C-2, 1H, d, $J = 6$ Hz), 7.32 (C-3, 1H, d, $J = 6$ Hz)	4.98/12.9	2.59 (s, 3 H, $\underline{CH_3}$)	7.10–8.15 (m, 12 H)	70.00/68.95	5.07/4.99	3.71/3.78

[a]Using solvent system benzene–ethyl acetate (8:2).
[b]NH and OH are D_2O exchangeable.

resultant mixture was eluted with methanol to give pure **6a** in quantitative yield. Since 100% conversion of reactants was observed with formation of single product, it was used as such for further reaction. In some trials, **6** was isolated for analytical and spectral data (table 2). Compounds **6b–g** were prepared in a similar manner by microwave-induced synthesis.

3.4 *8-Ethoxy-2,5-dihydro-1,5-benzothiazepine-1,1-dioxide (7a)*

7a was synthesized by two routes.

A. Conventional synthesis. To **6a** (0.001 mole; 0.375 g) dissolved in glacial acetic acid (15 ml), 30% hydrogen peroxide (30 mL) was added and the mixture was boiled reflux for 10–12 h (TLC). Excess solvent was removed by distillation under reduced pressure. The solution was poured into ice-cold water and the residue so obtained was filtered off, washed thoroughly with water, dried and crystallized from ethanol to give **7a**.

B. MW-assisted synthesis.

(1) A solution of ferric nitrate (0.001 mol) was added to **6a** (synthesized *in situ*). Solvent was then evaporated in a rotoevaporator and the mixture was irradiated inside a MW oven at 90% power (640 W) for 9 min (TLC). The resultant product was extracted by eluting with ethanol to give pure crystalline compound **7a**. Compound **7b** was prepared in a similar manner under microwave irradiation.

(2) **6a** (0.001 mol, 0.375 g) was adsorbed on Clayfen (prepared from 2 g clay + 0.001 mol $Fe(III)NO_3$) with the help of ethanol, and then dried and irradiated inside a microwave oven for the appropriate time (6 min, TLC). The product was extracted in a similar manner to that above to give **7a**.

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