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NaOH/PEG-400: An eloquent system for the synthesis of new thienyl benzo[*b*]1,4-diazepines

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ABSTRACT

A simple and eloquent procedure for the synthesis of a new series of thienyl benzo[*b*]1,4-diazepines is reported. They were synthesized by the condensation of *o*-phenylenediamine (*o*-PDA) with distinct hetero chalcones using NaOH in polyethylene glycol (PEG-400) as green and alternative reaction solvent. The significances of this present method are shorter reaction time, easy work-up, high yields, and mild reaction conditions. Furthermore, this method is environment friendly and without use of an expensive catalyst. The all newly synthesized compounds are characterized by the spectroscopic methods.

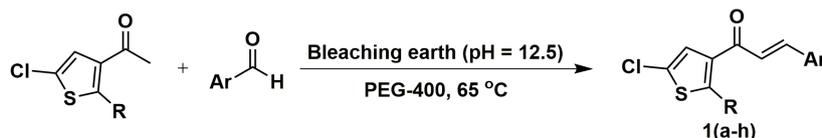
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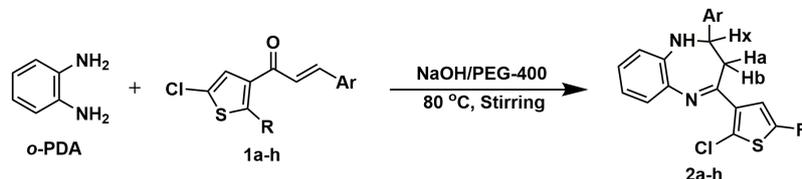
1. Introduction

Benzodiazepines and their derivatives are a very significant class of bioactive compounds because of their diverse pharmaceutical properties. They are widely used as antidepressants, anticonvulsant, analgesic, hypnotic, and sedative [1]. This compound possesses antimicrobial [2], antioxidant [3], and anticancer activity [4]. It acts as an inhibitor of respiratory syncytial virus [5]. 1,4-Benzodiazepine analogs have been demonstrated as anticonvulsants, muscle relaxants, blood pressure lowering, and Central Nervous System (CNS) depressant agents [6]. In addition to this, 1,5-benzodiazepines are also useful synthons for the preparation of various fused ring compounds such as triazolo, oxazino, oxadiazolo and furano benzodiazepines [7,8]. On the other hand, sulfur and nitrogen heterocycles having pharmaceutical activities are widely found in nature in the form of alkaloids, vitamins, pigments, and as constituents of plant and animal cells [9].

They are commonly prepared by the classical condensation reaction of *o*-phenylenediamine and α,β -unsaturated carbonyl compounds, β -haloketones. There are various methods for the preparation of 1,5-benzodiazepines reported in the literature such as BF_3 -etherate [10], NaBH_4 [11], SiO_2 [12], Amberlyst-15 [13], $\text{Yb}(\text{Otf})_3$ [14], MgO/POCl_3 [15], $\text{Al}_2\text{O}_3/\text{P}_2\text{O}_5$ [16], CH_3COOH in MWI, TiCl_4/THF [17], [bbim] ionic liquid [18] Silica-gel [19], and $\text{CeCl}_3/\text{Silica-gel}$ [20]. Recently, the synthesis of benzodiazepines has also been reported using different solid acid catalysts such as sulfated zirconia, $\text{Al}_2\text{O}_3/\text{P}_2\text{O}_5$, $\text{Ag}_3\text{PW}_{12}\text{O}_{40}$, PVPFeCl_3 , and zeolite catalysts [21-25]. However, many of these reported methods have some limitations such as use of expensive catalysts, long reaction time, high catalyst loading, low selectivity, requirement of special apparatus, and side reactions. These factors stimulate us for the search of new methodology with simple catalyst under the framework of green chemistry at mild reaction conditions.



Scheme 1. Synthetic route for preparation of hetero chalcones.



Scheme 2. Synthetic route for the preparation of thienyl benzo[b]1,4-diazepines.

2. Experimental

2.1. Instrumentation

The melting points were uncorrected and determined by an open capillary method. IR spectra were recorded (in KBr pellets) on Shimadzu FTIR-8400S spectrophotometer. ^1H NMR spectra were recorded (in $\text{DMSO-}d_6$) on Bruker Avance-300 MHz spectrometer using tetramethylsilane (TMS) as an internal standard. The mass was recorded on EI-Shimadzu QP 2010 Plus GC-MS spectrometer. The elemental analyses were performed on a Carlo Erba 106 Perkin-Elmer model 240 analyzer. The required chemicals and solvents used were purified and laboratory grade.

2.2. General procedure for the synthesis of benzo[b]1,4-diazepines (2a-h)

A mixture of substituted chalcone **1** (1 mmol), *o*-phenylenediamine (1.5 mmol), and solid NaOH (0.1 g) in polyethylene glycol (PEG-400) (15 mL) was stirred on a magnetic stirrer at 80 °C for the time period as shown in Table 1. After completion of the reaction (monitored by thin layer chromatography, TLC), the reaction mixture was cooled and poured in 100 mL ice-cold water. The obtained solid crude product was filtered and washed with 2×5 mL water. The crude product was recrystallized by the suitable solvent to give pure product (Schemes 1 and 2).

4-(5-(Benzylthio)-2-chlorothiophen-3-yl)-2-(4-chlorophenyl)-2,3-dihydro-1H-benzo[b][1,4]diazepine (2a): Color: Pale yellow. Yield: 90%. M.p.: 132-134 °C. FT-IR (KBr, ν , cm^{-1}): 1608 (C=N), 3065 (Ar-CH), 3272 (NH). ^1H NMR (300 MHz, $\text{DMSO-}d_6$, δ , ppm): 3.10-3.35 (m, 2H, Ha and Hb), 4.09 (s, 2H, -SCH₂-Ph), 4.3 (m, 1H, Hx), 4.8 (s, 1H, NH), 6.95-7.40 (m, 14H, Ar-H). MS (EI, m/z (%)): 494 (M⁺, 100), 496 (M+2), 499 (M+4). Anal. calcd. for $\text{C}_{26}\text{H}_{20}\text{N}_2\text{S}_2\text{Cl}_2$: C, 63.03; H, 4.07; N, 5.65. Found: C, 63.13; H, 4.11; N, 5.56%.

4-(5-(Benzylthio)-2-chlorothiophen-3-yl)-2-(4-fluorophenyl)-2,3-dihydro-1H-benzo[b][1,4]diazepine (2b): Color: Yellow. Yield: 86%. M.p.: 150-152 °C. FT-IR (KBr, ν , cm^{-1}): 1610 (C=N), 3061 (Ar-CH), 3265 (NH). ^1H NMR (300 MHz, $\text{DMSO-}d_6$, δ , ppm): 3.15-3.38 (m, 2H, Ha and Hb), 4.15 (s, 2H, -SCH₂-Ph), 4.36 (m, 1H, Hx), 4.86 (s, 1H, NH), 7.05-7.51 (m, 14H, Ar-H). MS (EI, m/z (%)): 478 (M⁺, 100), 480 (M+2). Anal. calcd. for $\text{C}_{26}\text{H}_{20}\text{N}_2\text{S}_2\text{FCl}$: C, 65.19; H, 4.21; N, 5.85. Found: C, 65.11; H, 4.28; N, 5.74%.

4-(5-(Benzylthio)-2-chlorothiophen-3-yl)-2,3-dihydro-1H-benzo[b][1,4]diazepin-2-yl)-N,N-dimethylaniline (2c): Color: Yellow. Yield: 85%. M.p.: 138-140 °C. FT-IR (KBr, ν , cm^{-1}): 1615 (C=N), 2998 (Ar-CH), 3268 (NH). ^1H NMR (300 MHz, $\text{DMSO-}d_6$, δ , ppm): 2.89 (s, 6H, N(CH₃)₂), 3.15-3.36 (m, 2H, Ha and Hb),

4.11 (s, 2H, -SCH₂-Ph), 4.31 (m, 1H, Hx), 4.78 (s, 1H, NH), 7.11-7.65 (m, 14H, Ar-H). MS (EI, m/z (%)): 503 (M⁺, 100), 505 (M+2); Anal. calcd. for $\text{C}_{28}\text{H}_{26}\text{N}_3\text{S}_2\text{Cl}$: C, 66.71; H, 5.21; N, 8.34. Found: C, 66.62; H, 5.12; N, 8.43%.

4-(5-(Benzylthio)-2-chlorothiophen-3-yl)-2-(4-methoxyphenyl)-2,3-dihydro-1H-benzo[b][1,4]diazepine (2d): Color: Pale Yellow. Yield: 90%. M.p.: 146-148 °C. FT-IR (KBr, ν , cm^{-1}): 1612 (C=N), 3026 (Ar-CH), 3276 (NH). ^1H NMR (300 MHz, $\text{DMSO-}d_6$, δ , ppm): 3.11-3.34 (m, 2H, Ha and Hb), 3.41 (s, 3H, OCH₃), 4.16 (s, 2H, SCH₂-Ph), 4.38 (m, 1H, Hx), 4.82 (s, 1H, NH), 7.09-7.68 (m, 14H, Ar-H). MS (EI, m/z (%)): 490 (M⁺, 100), 492 (M+2). Anal. calcd. for $\text{C}_{27}\text{H}_{23}\text{N}_3\text{O}_2\text{S}_2\text{Cl}$: C, 66.04; H, 4.72; N, 5.70. Found: C, 66.13; H, 4.81; N, 5.62%.

5-Chloro-4-(2-(4-chlorophenyl)-2,3-dihydro-1H-benzo[b][1,4]diazepin-4-yl)thiophene-2-sulfonamide (2e): Color: Light Brown. Yield: 86%. M.p.: 157-159 °C. FT-IR (KBr, ν , cm^{-1}): 1610 (C=N), 3051 (Ar-CH), 3245 (NH₂), 3326 (NH₂). ^1H NMR (300 MHz, $\text{DMSO-}d_6$, δ , ppm): 3.09-3.35 (m, 2H, Ha and Hb), 4.31 (m, 1H, Hx), 4.76 (s, 1H, NH), 5.26 (s, 2H, NH₂), 7.05-7.76 (m, 9H, Ar-H). MS (EI, m/z (%)): 451 (M⁺, 100), 453 (M+2), 455 (M+4). Anal. calcd. for $\text{C}_{19}\text{H}_{15}\text{N}_3\text{O}_2\text{S}_2\text{Cl}_2$: C, 50.45; H, 3.34; N, 9.29. Found: C, 50.52; H, 3.46; N, 9.21%.

5-Chloro-4-(2-(4-fluorophenyl)-2,3-dihydro-1H-benzo[b][1,4]diazepin-4-yl)thiophene-2-sulfonamide (2f): Color: Light Brick red. Yield: 88%. M.p.: 173-175 °C. FT-IR (KBr, ν , cm^{-1}): 1615 (C=N), 3038 (Ar-CH), 3238 (NH₂), 3315 (NH₂). ^1H NMR (300 MHz, $\text{DMSO-}d_6$, δ , ppm): 3.05-3.28 (m, 2H, Ha and Hb), 4.36 (m, 1H, Hx), 4.68 (s, 1H, NH), 5.28 (s, 2H, NH₂), 7.09-7.86 (m, 9H, Ar-H). MS (EI, m/z (%)): 435 (M⁺, 100), 437 (M+2). Anal. calcd. for $\text{C}_{19}\text{H}_{15}\text{N}_3\text{O}_2\text{S}_2\text{FCl}$: C, 52.35; H, 3.47; N, 9.64. Found: C, 52.28; H, 3.56; N, 9.55%.

5-Chloro-4-(2-(4-(dimethylamino)phenyl)-2,3-dihydro-1H-benzo[b][1,4]diazepin-4-yl)thiophene-2-sulfonamide (2g): Color: Brick red. Yield: 82%. M.p.: 154-156 °C. FT-IR (KBr, ν , cm^{-1}): 1610 (C=N), 3051 (Ar-CH), 3246 (NH₂), 3309 (NH₂). ^1H NMR (300 MHz, $\text{DMSO-}d_6$, δ , ppm): 2.85 (s, 6H, N(CH₃)₂), 3.09-3.35 (m, 2H, Ha and Hb), 4.31 (m, 1H, Hx), 4.75 (s, 1H, NH), 5.19 (s, 2H, NH₂), 7.05-7.81 (m, 9H, Ar-H). MS (EI, m/z (%)): 460 (M⁺, 100), 462 (M+2). Anal. calcd. for $\text{C}_{21}\text{H}_{21}\text{N}_4\text{O}_2\text{S}_2\text{Cl}$: C, 54.71; H, 4.59; N, 12.15. Found: C, 54.78; H, 4.66; N, 12.06%.

5-Chloro-4-(2-(4-methoxyphenyl)-2,3-dihydro-1H-benzo[b][1,4]diazepin-4-yl)thiophene-2-sulfonamide (2h): Color: Brown. Yield: 89%. M.p.: 160-162 °C. FT-IR (KBr, ν , cm^{-1}): 1612 (C=N), 3046 (Ar-CH), 3262 (NH₂), 3312 (NH₂). ^1H NMR (300 MHz, $\text{DMSO-}d_6$, δ , ppm): 3.11-3.38 (m, 2H, Ha and Hb), 3.51 (s, 3H, OCH₃), 4.38 (m, 1H, Hx), 4.82 (s, 1H, NH), 5.25 (s, 2H, NH₂), 7.09-7.91 (m, 9H, Ar-H). MS (EI, m/z (%)): 447 (M⁺, 100), 449 (M+2). Anal. calcd. for $\text{C}_{20}\text{H}_{18}\text{N}_3\text{O}_3\text{S}_2\text{Cl}$: C, 53.63; H, 4.05; N, 9.38. Found: C, 53.51; H, 4.14; N, 9.31%.

Table 1. Physico-chemical data of synthesized thienyl benzo[b]1,4-diazepines **2a-h**.

Entry	R	Ar	Time (h)	Yield (%)	M.p. (°C)
2a	SCH ₂ -Ph	4-Cl-C ₆ H ₄	2.5	90	132-134
2b	SCH ₂ -Ph	4-F-C ₆ H ₄	3	86	150-152
2c	SCH ₂ -Ph	4-N-(CH ₃) ₂ -C ₆ H ₄	3	85	138-140
2d	SCH ₂ -Ph	4-OMe-C ₆ H ₄	2.5	90	146-148
2e	SO ₂ NH ₂	4-Cl-C ₆ H ₄	2.5	86	157-159
2f	SO ₂ NH ₂	4-F-C ₆ H ₄	2.5	88	173-175
2g	SO ₂ NH ₂	4-N-(CH ₃) ₂ -C ₆ H ₄	3	82	154-156
2h	SO ₂ NH ₂	4-OMe-C ₆ H ₄	2.5	89	160-162

Table 2. Physical-chemical data of synthesized chalcone derivatives **1a-h** [40].

Entry	R	Ar	Time (h)	Yield (%)	M.p. (°C)
1a	SCH ₂ -Ph	4-Cl-C ₆ H ₄	1.5	92	148
1b	SCH ₂ -Ph	4-F-C ₆ H ₄	1.0	92	142
1c	SCH ₂ -Ph	4-N-(CH ₃) ₂ -C ₆ H ₄	1.5	88	156
1d	SCH ₂ -Ph	4-OMe-C ₆ H ₄	1.5	90	134
1e	SO ₂ NH ₂	4-Cl-C ₆ H ₄	1.5	89	127
1f	SO ₂ NH ₂	4-F-C ₆ H ₄	1.5	90	168
1g	SO ₂ NH ₂	4-N-(CH ₃) ₂ -C ₆ H ₄	1.5	89	130
1h	SO ₂ NH ₂	4-OMe-C ₆ H ₄	1.0	90	126

Table 3. Effect of solvent on the reaction of 1-(2-(benzylthio)-5-chlorothiophen-3-yl)-3-(4-chlorophenyl) prop-2-en-1-one (**1a**) with *o*-phenylenediamine (*o*-PDA) using NaOH.

Entry	Solvent	Time (h)	Yield (%)
1	EtOH	10	55
2	DCM	9	58
3	CH ₃ CN	8	62
4	Acetic acid	9	58
5	PEG-400	2.5	90

3. Results and discussion

The replacement of toxic solvents with environmentally benign solvents is the broad focus area of green chemistry. The utility of alternative reaction solvents includes water [26], ionic liquid [27], flouros [28], supercritical media [29], and polyethylene glycol (PEG) [30] is rapidly growing. Liquid polymers have emerged as an alternative green reaction media in organic synthesis. Polyethylene glycol (PEG-400) competed reactions [31-35] have attracted attention towards organic chemists due to their solvating ability and aptitude to act as a phase transfer catalyst, negligible vapor pressure, easy recyclability, ease of work-up, eco-friendly nature and low cost.

As a part of our continuous work towards the exploration of polyethylene glycol (PEG-400) [36-39] as a green reaction solvent for the preparation of biologically active compounds, herein we report the synthesis of some new thienyl benzo[b]1,4-diazepines **2a-h** by the condensation reaction of *o*-phenylenediamine (*o*-PDA) with distinct hetero chalcones using NaOH in polyethylene glycol (PEG-400) as green reaction solvent. The starting compounds (hetero chalcones) **1a-h** were prepared by our previously reported method (Scheme 1, Table 2) [40].

Initially, we started the reaction of 1-(2-(benzylthio)-5-chlorothiophen-3-yl)-3-(4-chlorophenyl) prop-2-en-1-one (**1a**) with *o*-phenylenediamine (*o*-PDA) using NaOH (catalyst) in polyethylene glycol (PEG-400) as green reaction solvent. The reaction was completed within 2.5 hours and the corresponding product (**2a**) was obtained in 90% yield. To optimize the reaction conditions, we studied the same above reaction in different solvents such as ethanol, dichloromethane, acetonitrile, acetic acid, and PEG-400 (Table 3). We found that PEG-400 as an efficient reaction medium in terms of reaction time as well as yield (90%). Next, we moved our attention towards the different substituted chalcones. In all cases, the reaction was smoothly proceeded in high yields at 80 °C using PEG-400 as an alternative reaction solvent (Scheme 2, Table 1). Furthermore, these newly synthesized compounds were characterized by the IR, ¹H NMR, and Mass spectroscopic methods.

The IR spectra of benzo[b]1,4-diazepines were showed disappearance of bands at 1660-1640 cm⁻¹ due to

transformation of >C=O of 3,5-diaryl prop-2-en-1-ones (chalcones) into products. The characteristic bands at near 1600-1610 and 3100-3330 cm⁻¹ due to -C=N and -NH stretching, respectively, in products. Besides these bands, 1050-1150 cm⁻¹ are observed due to C-S and 680-800 cm⁻¹ due to C-Cl stretching.

In the ¹H NMR spectra of the products, the three hydrogen atoms attached to the C-2 and C-3 carbon atoms of the heterocyclic ring gave an ABX spin system proved the benzodiazepines structure. The CH₂ protons of the benzodiazepines showed as multiplet at δ 3.09-3.21 (Ha), δ 3.41-3.54 (Hb) and δ 4.0-4.3 ppm triplet 1H of Hx. A characteristic singlet of NH was observed at δ 4.7-4.8 ppm. The singlet observed at 4.1-4.2 ppm due to -SCH₂-Ph moiety, while the corresponding aromatic and aliphatic protons were observed at excepted regions. These findings are in agreements with those observed by different researchers. The mass spectra (EI-MS) of the synthesized benzo[b]1,4-diazepines were in agreement with their molecular formula weight.

4. Conclusion

In conclusion, we have developed a new, efficient and environmentally benign reaction methodology towards the synthesis of novel thienyl benzo[b]1,4-diazepines derivatives by the treatment of chalcones with *o*-phenylenediamine using solid NaOH as catalyst in polyethylene glycol as green reaction solvent is described. The advantages of the present protocol are the shorter reaction time, simple reaction workup, high yields of products, and avoidance of expensive catalysts. Therefore, the present study is more beneficial in the synthesis of some new benzo[b]1,4-diazepines derivatives as medicinal drugs. In the future, these synthesized compounds can be used for medicinal investigation against bacterial and fungal diseases as drug molecules.

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Disclosure statement 

Conflict of interests: The authors declare that they have no conflict of interest.

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