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A green protocol for one pot synthesis of benzosuberone tethered thiadiazolopyrimidine-6-carboxylates using PEG-400 as potent anti-proliferative agents

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ABSTRACT

A new concise and facile method was explored to synthesize a collection of new benzosuberone based thiadiazolo [3,2-*a*] pyrimidine-6-carboxylates using polyethylene glycol (PEG), which could be regarded as the derivatives of the hybrid scaffolds of bioactive natural benzosuberone and heterocyclic thiadiazolo [3,2-*a*]pyrimidine. The structures of the synthesized compounds were characterized by ^{1}H, ^{13}C NMR, MS and IR; and their anti-proliferative activity was evaluated against four human cancer cell lines; A549, SKNSH, HeLa and MCF-7. Among the tested compounds, compound **8k** showed the most prominent activity against all the cell lines and these results may lay the foundation for further design of novel anti-proliferative agents.

The interest in the anticancer properties of benzosuberone has been increased since 1984, when Colchicine [1] (**I**) was discovered. Benzosuberone, which constitute the core for various natural products plays a unique role in drug discovery program. The related moieties of benzosuberone have historically been incredible therapeutic agents such as cytotoxic, anticancer [2–5], high CB1 receptors [6], anti malarial [7] and has potent antagonistic activity [6]. Here, some new benzosuberone analogues [8–9], (**II** to **V**) shown in Fig. 1 are proved to be potent tubulin polymerization inhibitors. Furthermore, heterocyclic sulfur compounds are of special interest in modern medicinal chemistry. For example, oxadiazole and thiadiazole (Fig. 1) derivatives are a well-known class of biologically active basic compounds for a large number of new drugs [10–13a]. On the other hand, pyrimidine derivatives and heterocyclic annulated pyrimidines have attracted a great deal of interest owing to their medicinal activities [15b–d].

Construction of thiadiazolo[3,2-*a*]pyrimidins [14,15] is also attractive and imperative, because numerous compounds of marine origin bear this thiadiazolo[3,2-*a*]pyrimidins scaffold [16–17], and they have exhibited great potential for medicinal applications and this type of motif is a privileged core structure of many bioactive molecules (Compounds **VI** and **VII**) [18]. In this view, we anticipated that a rational fusion of two known separated scaffolds (benzosuberones and thiadiazolo[3,2-*a*]pyrimidins) possessing bioactivity as a new hybrid, leading to an unprecedented skeleton of benzosuberone based thiadiazolo[3,2-*a*]pyrimidins, may generate novel biologically active leads. Furthermore, picking up these moieties and locking them together to increase the biological activities has been the target of our current endeavour.

Our recent studies have been focused on the development of new synthetic pathways for the preparation of cyclic compounds, which was based on the use of cascade or one-pot reactions [19]. Multi component reactions are useful for the creation of chemical library of drug-like compounds with levels of molecular complexity and diversity [20–22]. In recent years, the development of eco-friendly organic synthesis is gaining considerable interest in both industrial and academic research [23]. Hazardous, toxic, and volatile organic solvents are being continuously replaced by the use of solvent-free techniques [24], water [25], phase-transfer catalysts, or ionic liquids [26]. The use of polyethylene glycol (PEG) as a

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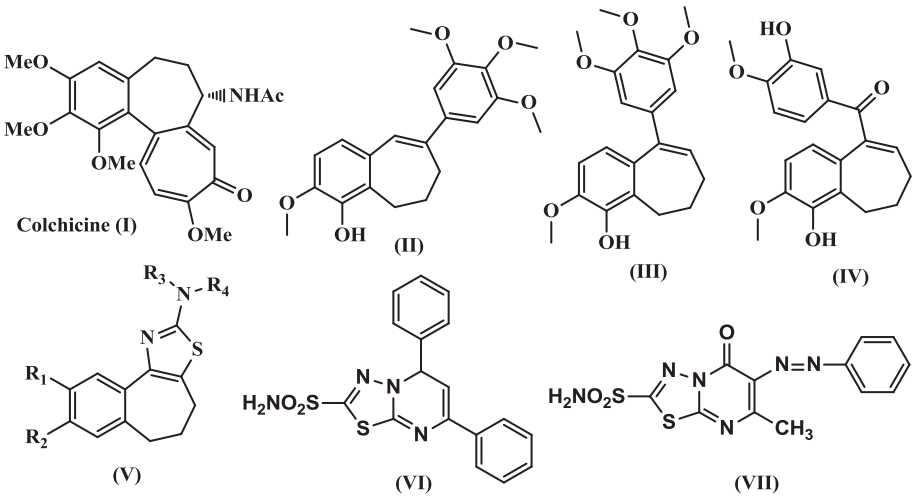
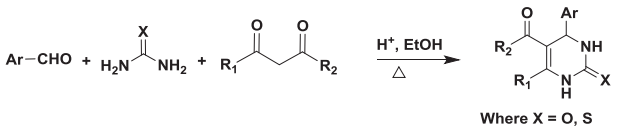


Fig. 1. Biologically active compounds containing thiadiazolo[3,2-*a*]pyrimidines.

reaction medium is highly beneficial as the system remains neutral, which helps in maintaining a number of acid and base-sensitive functional groups which remains unchanged [27]. In short, green chemistry has become a major inspiration for organic chemists to develop environmental routes for synthesis of organic compounds of biological values and to enhance the reaction efficiency from both economical and ecological points has received great attention.

As we know, a widely used multicomponent reaction (MCR), of Biginelli type (Scheme 1), involved the cyclocondensation of an aldehyde, urea or thiourea and β-ketoester in the presence of concentrated hydrochloric acid to give the corresponding 3,4-dihydropyrimidin-2-(1*H*)-ones (DHPMS) [28].

The use of toxic materials is highly undesirable, particularly when the procedure has to be prepared in parallel fashion to generate a library of targets. The overall sequence reported in literature (Scheme 1) often resulted in low yield of the desired thiadiazolo[3,2-*a*]pyrimidine-6-carboxylates. In contrast, we report a highly efficient one-pot protocol to prepare benzosuberone based thiadiazolo[3,2-*a*]pyrimidine-6-carboxylates that often does not require any chromatographic purification.

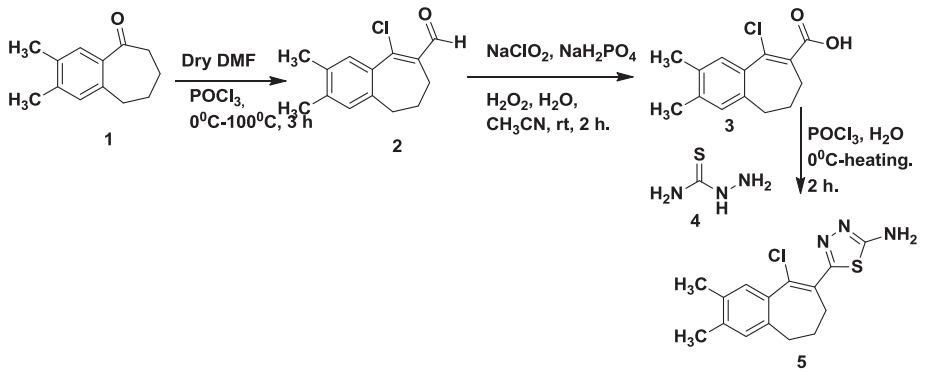


Scheme 1. Biginelli condensation reaction.

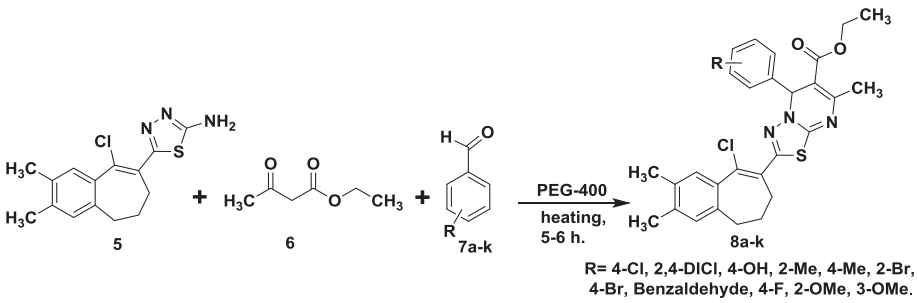
The synthetic routes used to prepare the derivatives (**8a–k**) used in this study are described in Schemes 2 and 3. The initial starting point for our work was the benzo [7]annulenyl thiadiazol-2-amine (**5**), which was prepared by heating of 9-chloro-2,3-dimethyl-6,7-dihydro-5*H*-benzo[7]annulene-8 carboxylic acid (**3**) [29], *N*-aminothiourea (**4**) at 75 °C for 0.5 h (Scheme 2).

In continuation of our investigation [30] in the development of new synthetic methodologies for carbon-heteroatom bond formation, herein we report an efficient, eco-friendly protocol and convenient one-pot promoted synthesis of benzocycloheptenone bearing thiadiazolo-pyrimidine carboxylate derivatives (**8a–k**). The compounds were obtained in good yields through an one-pot, three component condensation reaction of benzo[7]annulenyl thiadiazol-2-amine with various structurally divergent aromatic aldehydes and ethylacetoacetate in the presence of polyethylene glycol (PEG-400) at 80 °C temperature (Scheme 3). Therefore, the developed MCR may provide a valuable practical tool for the synthesis of novel physiological active agents containing the title core fragment (Fig. 2).

Synthesized derivatives **8a–k** were prepared in good to excellent yields (Scheme 2). The compounds **8a–k** obtained by using various aromatic aldehydes (**7a–k**), ethylacetoacetate and benzosuberone containing 1,3,4-thiadiazol-2-amine with higher yields. The structures of the products **8a–k** were established from their spectral data IR, ^{1}H NMR, ^{13}C NMR and Mass spectroscopic analysis. The formation of compound **8a** was evident from the



Scheme 2. Synthesis of 5-(9-chloro-2,3-dimethyl-6,7-dihydro-5*H*-benzo[7]annulen-8-yl)-1,3,4-thiadiazol-2-amine.



Scheme 3. Multicomponent condensation reaction of benzosuberone with 1,3,4-thiadiazol-2 amine (**5**), ethylacetoacetate (**6**) and substituted benzaldehyde (**7a**–**k**).

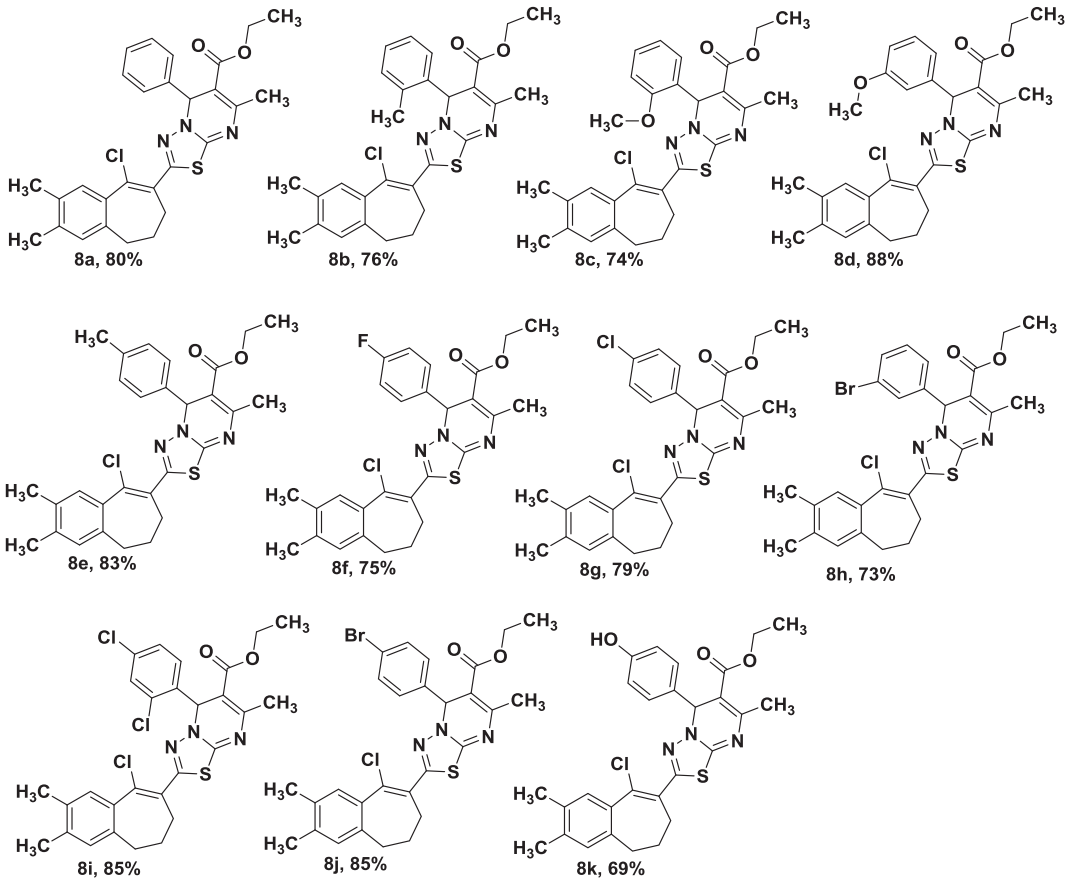


Fig.2. Benzosuberone functionalized thiadiazolo[3,2-*a*]pyrimidine-6-carboxylate derivatives (**8a**–**k**).

appearance of $[M+H]^+$ peak at *m*/*z* 505 in mass spectrum $(ESI)^+$ and the appearance of characteristic C—H proton as singlet at δ 6.36 in ^{1}H NMR.

To further confirm the structure by X-ray analysis (Crystal data for **8c**, as **AZ91M**: yellow coloured crystals, $C_{29}H_{30}ClN_3O_3S$, M = 536.07, monoclinic, space group *P*-1, a = 8.9021(5) Å, b = 11.0062(6) Å, c = 14.6357(8) Å, α = 80.7090(10)°, β = 72.7690(10)°, γ = 78.9010(10)°, V = 1335.64(13) Å^3, ρ_{calc} = 1.333 g/cm^3). Data were collected at room temperature using a Bruker Smart Apex CCD diffractometer with graphite monochromated MoKα radiation (λ = 0.71073 Å) with ω-scan method [31]. Preliminary lattice parameters and orientation matrices were obtained from four sets of frames. Unit cell dimensions were determined using 9838 reflections. Integration and scaling of intensity data were accomplished using SAINT program [31]. The structures were solved by Direct Methods using SHELXS97 and refinement was carried out by full-matrix least-square technique using SHELXL-2014/7 [32]. Anisotropic displacement parameters were included for all non-hydrogen atoms. All H atoms were positioned geometrically and treated as riding on their parent C atoms with C—H distances of 0.93–0.97 Å, and with $U_{iso}(H) = 1.2U_{eq}$ (C) or $1.5U_{eq}$ for methyl atoms. The structure is shown in Fig. 3.

Subsequently, to further demonstrate the potential activities of these synthesized compounds (**8a-k**), were evaluated *in vitro* against four different human cancer cell lines; A549 (lung adenocarcinoma), SKNSH (neuroblastoma), HeLa (cervical) and MCF-7 (mammary gland adenocarcinoma) by the MTT-based assay using the commercially available broad-spectrum anticancer drug of doxorubicin as a positive control, and their IC_{50} concentration is depicted in Table 1. The results demonstrated that most of the compounds showed considerable cytotoxicities to these cell lines and showed comparable activity to the positive control of

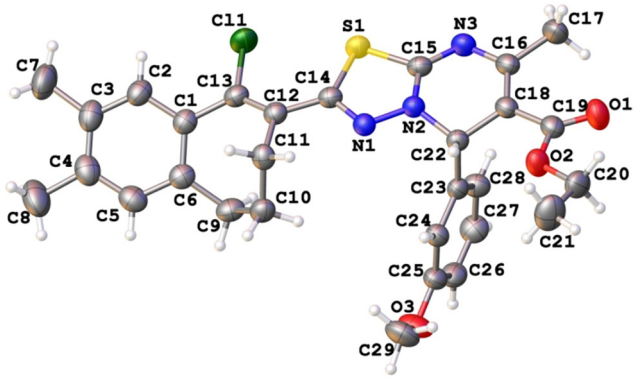


Fig. 3. X-ray crystal structure **AZ91**. Displacement ellipsoids are drawn at 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Table 1
IC_{50} (μM)[a] values of the tested compounds against four human cancer cell lines.

Compound. No.	IC_{50} concentration (μM)			
	A549	SKNSH	HeLa	MCF7
8a	4.4 ± 0.16	21.76 ± .86	9.51 ± 0.42	13.15 ± 1.54
8b	94.27 ± 5.41	55.64 ± 3.24	32.46 ± .41	10 ± 1.07
8c	66.64 ± 2.16	12.08 ± 0.92	31.48 ± 0.86	11.33 ± 1.42
8d	19.26 ± 0.93	25.22 ± 1.68	25.57 ± 1.85	5.67 ± 1.62
8e	6.42 ± 0.75	21.12 ± .46	22.38 ± 0.96	6.46 ± 1.38
8f	38.69 ± 3.62	71.87 ± .19	52.01 ± 3.51	7.38 ± 0.81
8g	34.91 ± 1.42	81.46 ± 3.41	9.21 ± 0.47	5.98 ± 0.96
8h	85.24 ± 2.41	44.36 ± .97	28.23 ± .49	14.24 ± 2.31
8i	75.24 ± 2.14	84.54 ± 3.24	37.42 ± .63	15.21 ± 1.42
8j	42.34 ± 1.41	54.20 ± .21	42.98 ± 2.41	20.48 ± 3.51
8k	4.38 ± 0.54	15.15 ± 1.28	5.96 ± 0.48	2.26 ± 0.45
Doxo[b]	2.14 ± 0.13	7.43 ± 0.97	3.46 ± 0.34	1.43 ± 0.62

[a] Each data represents mean ± SD of at least three independent experiments.
[b] Doxorubicin was used as a positive control.

doxorubicin. The results also indicated that synthesized benzosuberone based thiadiazolo[3,2-*a*]pyrimidine-6-carboxylate scaffolds may be useful leads for further biological screenings (Fig. 4).

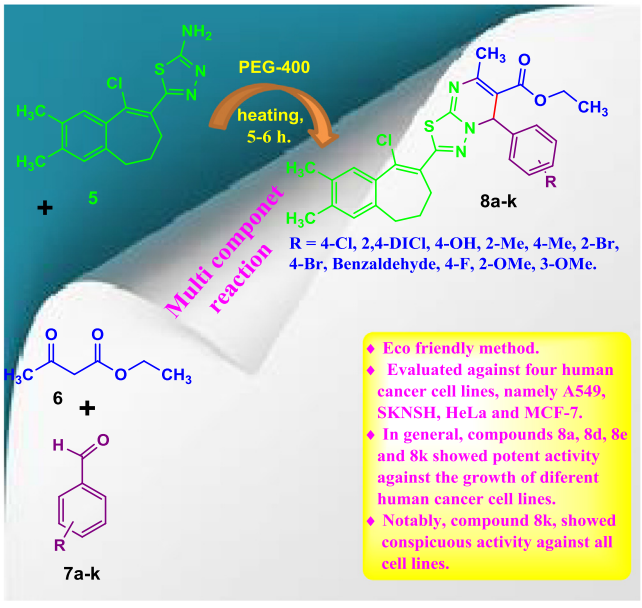


Fig. 4. Design strategy for benzosuberone based thiadiazolo[3,2-*a*]pyrimidine-6-carboxylate derivatives (**8a–k**).

Based on Table 1, the synthesized compounds **8a-k** showed prominent to moderate cancer cell growth inhibition with IC_{50} values ranging from **2.26** to **94.27** μM. Among all the compounds **8a**, **8d**, **8e** and **8k** showed potent activity against the growth of different human cancer cell lines. Particularly, the compounds **8a**, **8e** and **8k** showed promising anti-proliferative activity against A549 with IC_{50} values of **4.4**, **6.42** and **4.38** μM respectively. In addition, compounds **8a**, **8g** and **8k** displayed promising anti-proliferative activity against HeLa with IC_{50} values of **9.51**, **9.21** and **5.96** μM while compounds **8d**, **8e**, **8f**, **8g** and **8k** exhibited promising anti-proliferative activity against MCF-7 with IC_{50} values of **5.67**, **6.46**, **7.38**, **5.98** and **2.26** μM respectively. Additionally, the above results suggested that compound **8k** (IC_{50} values ranging from **2.26** to **15.15** μM) exhibits its potent anti-proliferative activity against all the cell lines. The results in Table 1 revealed that, compounds **8a** (Simple H), **8d** (*p*-OCH_3), **8e** (*p*-CH_3) and **8k** (*p*-OH) bearing a benzosuberone was active against four human cancer cell lines. In comparison, the *p*-OH substituent attached to the thiadiazolo [3,2-*a*]pyrimidine-6-carboxylate of benzosuberone scaffold (**8k**) which may exhibit strong electron donating property was more active than the remaining compounds.

In summary, we have described a polyethylene glycol (PEG)-mediated facile one-pot synthesis of benzosuberone based thiadiazolo[3,2-*a*]pyrimidine-6-carboxylates under green reaction conditions with excellent yields and short reaction times. A valuable feature of this method was the design of new hybrid architectures through the adequate fusion of these subunits thiadiazoles or pyrimidines with benzosuberone, generating biological active leads. These synthesized compounds were evaluated against four human different cancer cell lines. Notably, compound **8k** showed prominent activity against all the cell lines. Moreover, efforts are also in progress to improve the antitumor activities of these potential leads, and other biological activity evaluation including antibacterial and antiviral activities are also underway in our laboratory.

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A. Supplementary data

Supplementary data (experimental section and copies of the ^1H NMR, ^{13}C NMR, HRMS and IR spectra for some of the important compounds) associated with this article can be found, in the online version, at https://doi.org/10.1016/j.tetlet.2018.06.068.

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