**Three-component reaction of β-keto esters, aromatic aldehydes and urea/ thiourea promoted by caffeine: a green and natural, biodegradable catalyst for eco-safe Biginelli synthesis of 3, 4-dihydropyrimidin-2-(1*H*)-ones/tiones derivatives under solvent-free conditions**

FARZANEH MOHAMADPOUR a,\*, MOJTABA LASHKARI b

*a Young Researchers and Elite Club, Shiraz Branch, Islamic Azad University, Shiraz, Iran*

*b Faculty of Science, Velayat University, Iranshahr, Iran*

\**Corresponding author. Mohamadpour.f.7@gmail.com*

*Abstract:* Caffeine is found to be a natural and green and biodegradable catalyst for one-pot, three-component condensation Biginelli reaction of β-keto esters, aromatic aldehydes and urea/ thiourea to afford the corresponding 3, 4-dihydropyrimidin-2-(1*H*)-ones/tiones derivatives under solvent-free conditions. The remarkable features of this green procedure are high yields, short reaction times, simplicity of operation and work-up procedures, the availability and easy to handle of this solid catalyst, avoidance of hazardous or toxic catalysts and organic solvents and economic availability of the catalyst.

 *Keywords:*

Caffeine; Natural and green biodegradable catalyst; Biginelli reaction; 3, 4-dihydropyrimidin-2-(1*H*)-ones/tiones derivatives; Eco-safe procedure; Solvent-free conditions.

INTRODUCTION

In recent years, organic chemists have focused their attention towards green chemistry for the synthesis of heterocyclic compounds by using multi-component domino reactions (MCRs)1-6 due to a broad range of notable advantages such as non-toxic substrate and environmental friendly. Atom economy, reduction in byproduct, number of steps in organic synthesis, energy cost, produced waste, use of non-hazardous reagents in catalytic protocols are one of the most important goals of green chemistry. Furthermore, organic reactions under solvent-free conditions for green and clean synthesis of organic compounds have attracted much interest in organic chemists.

Structures containing the pyrimidinones have attracted organic chemists because of their biological activities. Literature reports have already established pyrimidinones as calcium channel blockers, α-1a-antagonists,7 antihypertensive effects,8 anticancer,9 anti HIV agent,10 antibacterial and antifungal,11 antiviral,12 antioxidative,13 and anti-inflammatory.14 Also some of the alkaloids found have dihydropyrimidine unit in their structure are shown in Fig 1.



Fig. 1. Batzelladine alkaloids contained the dihydropyrimidine units.

In view of the great importance of pyrimidinone derivatives, recently, the synthesis of 3, 4-dihydropyrimidin-2-(1*H*)-ones/tiones derivatives has been reported using multi-component reactions in the presence of different catalysts such as Calcium Fluoride [15], copper(II)sulfamate,16 bakers,yeast,17 hydrotalcite,18 hexaaquaaluminium (III) tetrafluoroborate,19TBAB,20 and copper (II) tetrafluoroborate,21 [Btto][*p*-TSA],22 triethylammonium acetate,23 *p*-dodecylbenzenesulfonic acid.24 Each of the these methods has its own merits, but some of these methods are limited in terms of the use of expensive catalysts, low yields, long reaction times, tedious work-up procedures and hazardous or toxic catalysts and organic solvents with column chromatographic separation. Consequently, there is a need to develop alternative methods for the synthesis of 3, 4-dihydropyrimidin-2-(1*H*)-ones/tiones derivatives under mild, green and eco-safe conditions.

Caffeine (tri[methylxanthine](https://en.wikipedia.org/wiki/Methylxanthine) [alkaloid](https://en.wikipedia.org/wiki/Alkaloid)) (Fig 2) is chemically related to the [adenine](https://en.wikipedia.org/wiki/Adenine) and [guanine](https://en.wikipedia.org/wiki/Guanine) bases of [deoxyribonucleic acid](https://en.wikipedia.org/wiki/DNA) (DNA) and [ribonucleic acid](https://en.wikipedia.org/wiki/RNA) (RNA). It is found in the seeds, nuts, or leaves of a number of plants native to South America and East Asia and helps to protect them against predator insects and to prevent germination of nearby seeds.It is the world's most widely consumed [psychoactive drug](https://en.wikipedia.org/wiki/Psychoactive_drug) including [Parkinson and Alzheimer's disease](https://en.wikipedia.org/wiki/Parkinson%27s_disease).25-27 There are several known [mechanisms of action](https://en.wikipedia.org/wiki/Mechanism_of_action) to explain the effects of caffeine, for example caffeine augments the antidepressant-like activity of mianserin and agomelatine in the forced swim and tail suspension tests in mice.28The most prominent is that it reversibly blocks the action of [adenosine](https://en.wikipedia.org/wiki/Adenosine) on its receptor and consequently prevents the onset of drowsiness induced by adenosine. Caffeine also stimulates certain portions of the [autonomic nervous system](https://en.wikipedia.org/wiki/Autonomic_nervous_system).29



Fig. 2. Structure of Caffeine

Caffeine has emerged as natural, green, cheap and efficient catalyst in various organic transformations.30, 31In continuation of our work on the development of useful synthetic methodology for preparation of these biologically active heterocyclic compounds,32 we wish to report herein a simple and highly efficient procedure for one-pot three-component Biginelli synthesis of 3, 4-dihydropyrimidin-2-(1*H*)-ones/tiones derivatives using caffeine as a green, eco-safe and availability solid catalyst under solvent-free conditions.

The advantages of caffeine as solid catalyst in organic compounds synthesis are friendly environment, mild, inexpensive, non-toxic and biodegradable. Also, caffeine can be successfully used in the type of carbon-carbon bonds as green and naturally biodegradable catalyst in organic synthesis. Finally, in this procedure, an eco-friendly, simple and mild one-pot approach for synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones/thiones derivatives using caffeine as a green and cost effective catalyst *via* three-component Biginelli reaction between *β-*keto esters, aryl aldehyde derivatives and urea/thiourea under solvent-free conditions with excellent yields is reported.

EXPERIMENTAL

*Chemicals and apparatus*

Melting points all compounds were determined using an Electro thermal 9100. 1H NMR spectra were recorded on a Bruker DRX-400 Avance instrument with DMSO-d6 as solvents. All reagents and solvents were purchased from Merck, Fluka and Acros chemical companies were used without further purification.

*General procedure for preparation of 3,4-dihydropyrimidin-2-(1H)-ones/thiones derivatives (****4a- w****)*

A mixture of aldehyde derivatives (**1**, 1.0 mmol) and urea/thiourea (**2**, 1.5 mmol), ethyl/methyl acetoacetate (**3**, 1.0 mmol) was heated under solvent-free conditions at 80 °C for appropriate time in the presence of caffeine (15 mol %). After completion of the reaction (by thin layer chromatography TLC) the mixture was cooled to rt and cold water was added and the precipitated was separated by filtration and recrystallized from ethanol to afford the pure products (4a- w). Supporting Information associated with this article can be found, in the online version.

RESULTS AND DISCUSSION

In pursuit of our continued interest in the development of solvent-free and green synthetic procedures, we decided to explore the use of caffeine catalyst for synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones/thiones derivatives *via* Biginelli condensation in high to excellent yields at 80 °C under solvent-free conditions. Initially the reaction between Benzaldehyde (1.0 mmol), urea (1.5 mmol) and ethyl acetoacetate (1.0 mmol) as the model reaction was examined in the presence varying amount of caffeine as a catalyst and the results are presented in Table I. The best result was achieved by carrying out the reaction with 0.03 g of catalyst (Table I, entry 4). Use of a higher amount of catalyst did not improve the yield while a decrease in the amount of catalyst decreases the yield (Table I). In the absence of catalyst the reaction did not proceed even after a long reaction time (Table I, entry 1). Also the effect of temperatures was studied by carrying out the model reaction at different temperatures under solvent-free conditions (rt, 40, 60, 70, 80, 90 °C) and the best results were obtained at 80 °C (Table I, entry 4).

To study the generality of this process, a good range of aryl aldehyde derivatives (**1**, 1.0 mmol), urea/ thiourea (**2**, 1.5 mmol) and ethyl/methyl acetoacetate (**3**, 1.0 mmol) were condensed to the corresponding 3,4-dihydropyrimidin-2-(1*H*)-ones/thiones derivatives in the presence of catalytic amount of caffeine and the related 3,4-dihydropyrimidin-2-(1*H*)-ones/thiones derivatives derivatives were obtained without observation of any by-product in high to excellent yields (Table II) (Scheme 1). The structure of the products was characterized by their melting points and nuclear magnetic resonance (1H NMR) spectral data, which were then compared with those of authentic samples.



Scheme 1. Synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones/thiones derivatives.

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| TABLE I.Optimization of the reaction condition on the synthesis of **4a***a* |
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| Yield*b*, % | *τ* / min | *t* / °C | Catalyst [amount, g]  | Entry |
| Not product | 360 | 80 | Catalyst free | 1 |
| 47 | 60 | 80 | 0.01 | 2 |
| 73 | 35 | 80 | 0.02 | 3 |
| **91** | **25** | **80** | **0.03** | **4** |
| Not product | 360 | rt | 0.03 | 5 |
| 38 | 70 | 40 | 0.03 | 6 |
| 56 | 45 | 60 | 0.03 | 7 |
| 69 | 30 | 70 | 0.03 | 8 |
| 91 | 25 | 90 | 0.03 | 9 |
| 92 | 25 | 80 | 0.04 | 10 |
| *a* Reaction conditions: benzaldehyde (1.0 mmol), ethyl acetoacetate (1.0 mmol), urea (1.5mmol) and caffeine was heated under various temperatures for the appropriate time.*b* Isolated yield. |

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| TABLE II. Caffeine catalyzed synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones/thiones derivatives |
| Entry | Ar | R | X | Product | *τ* / min | Yield*a*, % | M.p.°C | Lit. M.p.°C |
| 1 |  |  | O | **4a** | 25 | 91 | 198-200 | 200-20219 |
| 2 |  |  | O | **4b** | 30 | 82 | 221-223 | 220-22316 |
| 3 |  |  | O | **4c** | 25 | 89 | 223-225 | 225-22721 |
| 4 |  |  | O | **4d** | 30 | 84 | 203-205 | 205-20617 |
| 5 |  |  | O | **4e** | 35 | 81 | 165-167 | 163-16621 |
| 6 |  |  | O | **4f** | 30 | 80 | 193-195 | 191-19316 |
| 7 |  |  | O | **4g** | 25 | 84 | 209-210 | 207-20919 |
| 8 |  |  | O | **4h** | 30 | 87 | 200-202 | 202-20318 |
| 9 |  |  | O | **4i** | 35 | 79 | 230-231 | 230-23220 |
| 10 |  |  | O | **4j** | 30 | 81 | 215-217 | 214-21518 |
| 11 |  |  | O | **4k** | 30 | 87 | 257-259 | 255-25718 |
| 12 |  |  | O | **4l** | 25 | 88 | 204-206 | 204-20517 |
| 13 |  |  | O | **4m** | 25 | 93 | 172-174 | 174-17620 |
| 14 |  |  | O | **4n** | 30 | 84 | 251-253 | 248-25216 |
| 15 |  |  | O | **4o** | 25 | 92 | 275-277 | 274-27721 |
| 16 |  |  | O | **4p** | 30 | 84 | 206-208 | 205-20623 |
| 17 |  |  | O | **4q** | 25 | 89 | 215-217 | 214-21619 |
| 18 |  |  | O | **4r** | 35 | 83 | 244-246 | 245-24616 |
| 19 |  |  | S | **4s** | 25 | 92 | 210-212 | 208-21020 |
| 20 |  |  | S | **4t** | 30 | 86 | 193-195 | 191-19515 |
| 21 |  |  | S | **4u** | 25 | 88 | 208-209 | 208-21019 |
| 22 |  |  | S | **4v** | 30 | 85 | 151-153 | 150-15219 |
| 23 |  |  | S | **4w** | 30 | 83 | 195-197 | *b* |
| *a* Isolated yield.*b*The new compound is synthesized in this work. |

Proposed mechanistic route of 3, 4-dihydropyrimidin-2-(1*H*)-ones/tiones synthesis in the presence of caffeine are shown in scheme 2. In this mechanism, The reaction of aldehydes (1) and urea (2) generates an acylimin intermediate (A), which further reacts with the activated 1,3-dicarbonyl compound (B) (the caffeine catalyzed *β*-keto esters to activated 1,3-dicarbonyl compound (B) that is presented in Scheme 2) after passing a few steps producing an open-chain ureide (C) undergoing subsequent cyclization and dehydration to give the major product (4).33



Scheme 2. Proposed mechanism for the synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones/thiones.

Also a comparison of catalytic ability some of catalysts reported in the literature for synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones/thiones derivatives are shown in Table III. This study reveals that caffeine has shown its extraordinary potential to be an alternative green, natural, biodegradable and cost effective catalyst for the Biginelli reaction. In Addition, the use of solvent-free conditions with excellent yields and short reaction times in the reaction with both urea and thiourea are the notable advantages this present methodology.

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| TABLE III. Comparison of catalytic ability some of catalysts reported in the literature for synthesis of **4a*a*** |
| *τ* /Yield, % | Solvent/*T* / °C | Catalyst  | Entry |
| 1440 min/8417 | Room temperature | bakers, yeast | 1 |
| 35 min/8418 | Solvent-free, 80 °C | hydrotalcite | 2 |
| 1200 min/8119 | MeCN, Reflux | [Al(H2O)6](BF4)3 | 3 |
| 30 min/9021 | Room temperature | Cu(BF4)2.xH2O | 4 |
| 30 min/9622 | Solvent-free, 90 °C | [Btto][*p*-TSA] | 5 |
| 45min/9023 | Solvent-free,70 °C | triethylammonium acetate | 6 |
| 180 min/9424 | Solvent-free, 80 °C | *p*-dodecylbenzenesulfonic acid | 7 |
| 25 min/91*b* | Solvent-free, 80 °C | Caffeine | 8 |
| *a*Based on reaction of benzaldehyde, ethyl acetoacetate and urea.*b* This work. |

CONCLUSIONS

In conclusion, we have demonstrated that a naturally green and biodegradable catalyst, caffeine, can be used as a highly efficient catalyst for one-pot Biginelli synthesis of 3,4-dihydropyrimidin-2-(1*H*)-ones/thiones under solvent-free conditions. Use of the inexpensive and easy to handle caffeine as a mild natural green catalyst, high to excellent yields, short reaction times, high catalytic efficiency, straightforward work-up with no column chromatographic separation, environmentally benign nature procedure and solvent-free conditions are the notable advantages of this eco-safe and simple protocol.

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**Appendix A. Supporting Information**

Supporting Information associated with this article can be found, in the online version.

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