CHEMISTRY JOURNAL OF MOLDOVA. General, Industrial and Ecological Chemistry. 2016, 11(2), 68-73
http://cjm.asm.md
http://dx.doi.org/10.19261/cjm.2016.11(2).02

EXTRACT OF BARBERRY AS ENTIRELY GREEN CATALYST FOR THE SYNTHESIS OF STRUCTURALLY DIVERSE 3,4,5-SUBSTITUTED FURAN-2(5*H*)-ONES

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Abstract. An eco-friendly and environmentally benign synthesis of 3,4,5-substituted furan-2(5*H*)-ones employing Iranian seedless barberry, known as Zereshk, (*Berberis integerrima* "Bidaneh", *Berberidaceae*) as a biocatalyst, was developed. For the first time, we found that the barberry juice could be effectively used for three-component condensation reaction of aldehydes, amines, and dialkyl acetylenedicarboxylates. The merits of this method include the environmentally friendly reaction conditions, simple operation, broad substrate, satisfying yields, and the generation of less waste rather than the conventional chemical reagents.

Keywords: three-component reaction, dialkyl acetylenedicarboxylates, furan-2(5*H*)-ones, aldehydes, barberry juice.

Received: August 2016/ Revised final: October 2016/ Accepted: October 2016

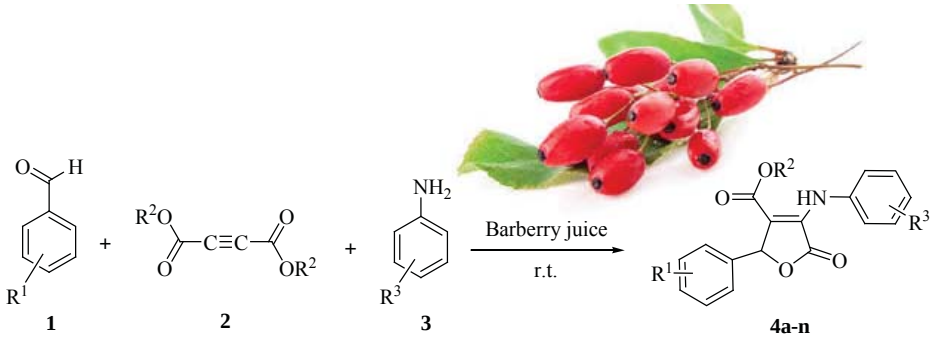
Introduction

In recent years, organic research has been mainly focused on the development of greener and eco-friendlier processes that involve the use of alternative reaction media to replace toxic and expensive catalysts or volatile and hazardous solvents like benzene, toluene and methanol, commonly used in organic synthesis. Nowadays, many organic transformations are carried out in water. Water is a unique solvent because it is readily available, inexpensive, nontoxic, and environmentally benign. The applications of an aqueous extract of different fruit juices have witnessed a rapid development. This growing interest in fruit juice is mainly rooted in its biocatalysts, nonhazardous, environmentally benign character, and cost effectiveness [1-3].

In recent years, chemical reactions using plant cell cultures and part of plants as biocatalysts received great attention [4-6]. This crescent interest is due to the wide biotechnological potential of the enzymatic reactions. The biocatalytical alteration using edible plants [7], plant root [8], plant tubers [9], edible plants [10] and plant leaves [11] extract can be applied in many organic reactions. Fruit juice is also a natural product that is used as biocatalysts in organic synthesis. Fruit juice is now being routinely used in organic synthesis as homogeneous catalysts for various selective transformations of simple and complex molecules [12].

Furanones are the five-membered heterocyclic compounds possessing lactone ring in their structures. These heterocycles are the core structures of many bioactive natural products, as well as synthetic drugs, such as rubrolide, sarcophine, benfurodil hemisuccinate, etc. The *5H*-Furan-2-one derivatives exhibit many pharmacological and biological activities including antifungal, antibacterial, anti-oxidants, anti-inflammatory, anti-microbial and anti cancer agents [13–17].

As a part of our efforts to develop new synthetic methods in heterocyclic chemistry [18-20], herein we report, for the first time, a three-component reaction of aldehydes, amines and dialkyl acetylenedicarboxylates for the synthesis of 3,4,5-substituted furan-2(5*H*)-one derivatives in barberry juice as an eco-friendly catalyst (Scheme 1). This approach offers an alternative method of construction of furanone architectures with potential biological activities based on a concise, rapid, and environmentally friendly vision.



1 **2** **3** **4a-n**

Scheme 1. Synthesis of furan-2(5*H*)-one derivatives.

Results and discussion

In continuation of our work following the principles of green chemistry [17-20], we have developed a simple, efficient, and green protocol for the preparation of 3,4,5-substituted furan-2(5*H*)-one, using an extract of barberry as a green and inexpensive catalyst and solvent. Our approach reduces the use of hazardous organic solvents and uses simple and mild conditions with inherently lower costs.

Different types of barberry are well known around the world for several benefits, such as medical, ornamental and food uses. Iranian seedless barberry (*Berberis integerrima* ''Bidaneh'') is commercially cultivated for its fruit in Iran, especially in South Khorasan province. Currently, there are over 11,000 ha of cultivated seedless barberry, annually producing more than 9200 tonnes of dried fruit [21]. Some studies have found that barberry has some important bioactive components in roots and/or fruits, such as calcium, sodium, sulphur, iron, zinc, vitamin C, carbohydrates, organic acids and alkaloids, *e.g.* berberine and palmatine [22, 23].

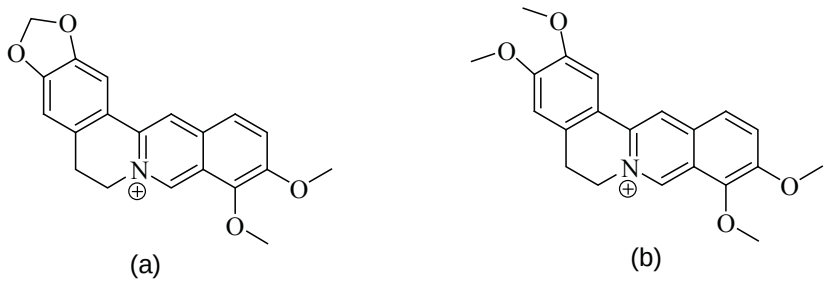


Figure 1. Chemical structures of berberine (a) and palmatine (b).

Among active components of barberry, the alkaloid berberine (Figure 1) occurring in bark of root, stem and unripe fruit, is considered the most important active constituent of the plant. Berberine, a yellow colour bitter substance [24], belongs to a large and diverse group of alkaloids called benzylisoquinolines [25]. Soluble sugars and berberine content of the barberry are one of its most important properties, since the value of the barberry is dependent on this content. The sugars were applied as an efficient and homogenous catalyst for multicomponent reaction in excellent yields [26,27].

Our further experiments have been designed to check the activities of some components of barberry juice on the synthesis of 3,4,5-substituted furan-2(5*H*)-one. For this purpose in separate experiments, components of barberry juice with the highest percentage, such as glucose, fructose, and vitamin C were selected. Investigations showed that the reaction occurred in the presence of all above components in good yield. Since the juice contains so many vitamins, ions and reducing sugars with different ratio and varieties, regarding to the mentioned experiment, it seems that a set of above agents are effective in occurring of this three component reaction.

At the beginning, a test reaction was performed using benzaldehyde **1** (1.0 mmol), aniline **3** (1.0 mmol) and dimethyl acetylenedicarboxylate **2** (1.0 mmol) in 5 mL juice of barberry at room temperature, in order to establish the real effectiveness of the catalyst/solvent, and the product was obtained in good yields.

In order to compare the strength of the juice of barberry and a catalyst with juice of various summer fruits, a model reaction was carried out between benzaldehyde **1** (1.0 mmol), aniline **3** (1.0 mmol) and dimethyl acetylenedicarboxylate **2** (1.0 mmol), using various fruit juices as catalysts at room temperature, and the results were summarized in Table 1.

Table 1

Optimization of catalyst for the synthesis of furan-2(5*H*)-ones.

Entry	*Solvent*	*pH*	*Time (h)*	*Isolated yield (%)*
1	H_2O	6.8	24	-
2	melon juice	5.8	6	50
3	blackberry juice	3.9	15	25
4	grapes juice	2.9	12	40
5	pomegranate juice	3.0	15	25
6	strawberries juice	3.2	24	-
7	peach juice	3.3	10	42
8	verjuice juice	3.5	16	38
9	**barberry juice**	**5.6**	**3**	**92**

Reaction conditions: benzaldehyde (1.0 mmol), aniline (1.0 mmol), dimethyl acetylenedicarboxylate (1.0 mmol) in various media at room temperature.

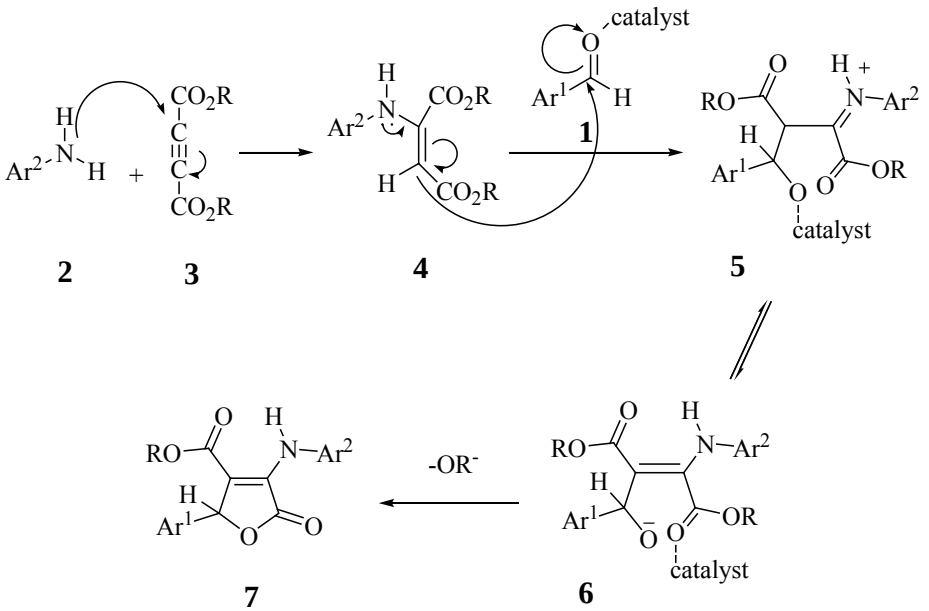
To explore the scope and generality of the presented multicomponent reaction protocol for the synthesis of 3,4,5-substituted furan-2(5*H*)-one under the optimized conditions, a variety of aromatic aldehydes containing electron donating or electron withdrawing substituents in the aromatic ring, such as -Me, -Cl, -OMe and -NO_2 were reacted with dialkyl acetylenedicarboxylate and various anilines to furnish diverse furan-2(5*H*)-one. The results were summarized in Table 3. In all cases, aromatic aldehydes with substituents carrying either electron-donating or electron-withdrawing groups reacted successfully and gave the products in high yields. It was found that aromatic aldehydes with electron-withdrawing groups reacted faster than those with electron-donating groups, as would be expected. The results were shown in Table 2.

Table 2

Synthesis of furan-2(5*H*)-one derivatives.

Entry	*R^1*	*R^2*	*R^3*	*Time (h)*	*Isolated yield (%)*	*Product*	*M.p. (°C)*	
							This work	*Lit. [Ref]*
1	Ph	CH_3	4-F-C_6H_4	2.5	70	4a	293–295	293–295 [13]
2	Ph	CH_3	4-Cl-C_6H_4	1.5	75	4b	150–152	149–152 [17]
3	Ph	CH_3	3-NO_2-C_6H_4	2.5	85	4c	178-181	179-182 [17]
4	4-NO_2-C_6H_4	CH_3	Ph	0.5	94	4d	128-130	130-131 [17]
5	Ph	CH_3CH_2	Ph	0.5	90	4e	163–165	164–166 [12]
6	4-Me-C_6H_4	CH_3CH_2	Ph	1	90	4f	119–121	120–121 [12]
7	4-OMe-C_6H_4	CH_3	Ph	1.5	67	4g	239–242	239–242 [17]
8	4-Me-C_6H_4	CH_3	Ph	2	85	4h	181–183	181–183 [13]
9	4-Cl-C_6H_4	CH_3	Ph	2	75	4i	149–152	149–152 [12]
10	Ph	CH_3	4-Me-C_6H_4	1	90	4j	284–287	284–287 [12]
11	Ph	CH_3CH_2	4-Me-C_6H_4	1	80	4k	185–186	188–191 [12]
12	Ph	CH_3CH_2	3-NO_2-C_6H_4	3	84	4l	208-210	208 [28]
13	4-Me-C_6H_4	*tert*-buthyl	Ph	2	90	4m	176-178	175-178 [28]
14	Ph	CH_3	Ph	0.5	96	4n	158-160	159–162 [13]
15	3-NO_2-C_6H_4	CH_3	Ph	1	95	4o	200–202	203–205 [13]
16	Ph	*tert*-buthyl	4-Me-C_6H_4	6	85	4p	185-187	-
17	Ph	*tert*-buthyl	4-Cl-C_6H_4	10	84	4q	169-171	-
18	Ph	*tert*-buthyl	4-F-C_6H_4	10	83	4r	170-173	-

A proposed mechanism for the discussed transformation can be combined with the nucleophilic Michael addition, iminium-enamine tautomerization and *γ*-lactonization [16-20] (Scheme 2).



Scheme 2. Suggested mechanism for synthesis of furan-2(5*H*)-ones.

The structures of new compounds presented in Table 3 were deduced on the basis of IR, ^{1}H and ^{13}C NMR spectroscopy, mass spectrometry, and elemental analysis. The mass spectrum of compound *tert*-butyl 4-(*p*-tolylamino)-2,5-dihydro-5-oxo-2-phenylfuran-3-carboxylate **4p**, displayed the molecular ion peak at *m/z* = 365 that is consistent with the proposed structure. The ^{1}H NMR spectrum of this product exhibited a singlet at δ = 1.35 ppm and singlet at δ = 2.25 ppm for *tert*-butyl protons of the carboxylate group and one sharp singlet arising from benzylic proton at δ = 5.63 ppm. The aromatic protons of product were observed at δ = 7.06-7.35 ppm. A broad singlet for the NH group at δ = 9.09 ppm indicated intra-molecular hydrogen bond formation with the vicinal carbonyl group. The ^{13}C NMR spectrum of this product showed 16 distinct resonances consistent with the proposed structure. The IR spectrum indicated one sharp peak at 3305 cm^{-1} for NH within the product. To compare the applicability and efficiency of barberry juice with the reported catalysts in the literature for the synthesis of 3,4,5-substituted furan-2(5*H*)-ones, we have tabulated the results of these catalysts in Table 3. As shown in Table 4, barberry juice can act as an efficient catalyst in terms of reaction time and yield of products.

Table 3

Comparison of using barberry juice and the reported catalysts for the synthesis of 3,4,5-substituted furan-2(5*H*)-ones 4n.

Entry	*Product*	*Catalyst*	*Time (h)*	*Yield (%)*	*Reference*
1	**4n**	nano-ZnO	2.5	94	[16]
2		$Al(HSO_4)_3$	8	84	[15]
3		$SnCl_2.2H_2O$	6.5	90	[14]
4		$[Bu_4N][HSO_4]$	5	92	[19]
5		PPA/SiO_2	1	90	[18]
6		sucrose	9	97	[27]
7		barberry juice	0.5	96	This work

Conclusions

In conclusion, we have found that the aqueous extract of barberry is as an efficient, economical, and environmentally-friendly catalyst for the synthesis of 3,4,5-substituted furan-2(5*H*)-ones. The high yield of products in a short reaction time with high purity, mild reaction conditions, and a simple workup procedure make this procedure attractive. The use of barberry juice both as a solvent and biodegradable catalyst is the attractive feature of this protocol. Furnishing pure products by simple filtration makes an aqueous approach possible for large scale preparation of furan-2(5*H*)-ones.

Experimental

Melting points and IR spectra of all compounds were measured on an Electrothermal 9100 apparatus and a JASCO FTIR 460 Plus spectrometer respectively. The ^{1}H and ^{13}C NMR spectra were obtained on Bruker DRX-400 Avance instruments with $CDCl_3$ as a solvent. Mass spectra were recorded on an Agilent Technology (HP) spectrometer operating at an ionization potential of 70 eV. Samples of fruits were blended using a laboratory electrical blender (Model 32BL79, Waring, USA). All reagents and solvents obtained from Fluka and Merck were used without further purification**.**

Plant material

Eight samples of fruits were purchased from the local commercial market, collected in the period between July and August (2013) in the region of the South of Sistan and Baluchestan and South Khorasan, Iran. The purchased samples were washed and drained. The skins of them except blackberry and barberry were peeled and the rest of the fruit was cut into 3 cm cubes. The seeds were then removed and all flesh parts were blended using a laboratory electrical blender (Model 32BL79, Waring, USA). The obtained juice was vacuum- filtered and then transferred into a beaker**.**

General procedure for the synthesis of furan-2(5H)-one derivatives

The mixture of aldehyde (1.0 mmol), amine (1.0 mmol), dialkyl acetylenedicarboxylate (1.0 mmol) and 5 mL juice of barberry were stirred at room temperature. After completion of the reaction (monitored by thin-layer chromatography, TLC), the reaction products were collected by filtration. The products were washed with water/ethanol (1:1, 3×2 mL) to give the corresponding pure compounds. The catalyst remained in the water/ethanol filtrate.

Methyl 2,5-dihydro-5-*oxo*-2-phenyl-4-(phenylamino)furan-3-carboxylate (4n). White solid: 0.296 g (96 %); m.p. 158-160 °C; IR (KBr): 3260, 3208, 1702, 1661 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ, ppm: 3.77 (s, 3H, OCH_3), 5.76 (s, 1H, $H_{benzylic}$), 7.13 (t, J = 7.3 Hz, $1H_{aromatic}$), 7.24-7.31 (m, 6H), 7.52 (d, J = 8 Hz, 2H, $H_{aromatic}$), 8.90 (br, 1H, NH).

***Tert*-butyl 4-(*p*-tolylamino)-2,5-dihydro-5-*oxo*-2-phenylfuran-3-carboxylate (4p).** Colorless solid; 0.287 g (85 %); m.p. 185-187 °C; IR (KBr): 3305, 3030, 1690, 1666 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ, ppm: 1.35 (s, 9H, CH_3), 2.25 (s, 3H, CH_3), 5.63 (s, 1H, $H_{benzylic}$), 7.06-7.35 (m, 9H, $H_{aromatic}$), 9.09 (br, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) δ, ppm: 165.1 and 162.9 (CO of ester), 157.0, 135.6, 135.4, 133.7, 129.5, 128.4, 128.5, 127.6, 122.5, 114.3 (10 $C_{aromatic}$), 83.2 (C-O), 61.8 ($C_{benzylic}$), 27.9 (3 CH_3), 20.9 (CH_3); MS m/z (%): 57 (42), 77 (25), 102 (39), 130 (100), 158 (42), 175 (24), 263 (29), 291 (39), 309 (52), 365 (M^+, 28).

***Tert*-butyl 4-(4-chlorophenylamino)-2,5-dihydro-5-*oxo*-2-phenylfuran-3-carboxylate (4q).** Colourless solid; 0.268 g (84 %); m.p. 169-171 °C; IR (KBr): 3315, 3095, 2975, 1690, 1655 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ, ppm: 1.36 (s, 9H, CH_3), 5.64 (s, 1H, $H_{benzylic}$), 7.18-7-47 (m, 9H, $H_{aromatic}$), 9.36 (br, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) δ, ppm: 165.0, 162.9 (C_{ester}), 156.6, 134.96, 134.95, 131.0, 129.0, 128.68, 128.60, 127.5, 123.3, 114.6 (10 $C_{aromatic}$ and C_{vinyl}), 83.5 (C-O), 61.6 ($C_{benzylic}$), 27.9 (CH_3); MS m/z (%): 57 (48), 77 (34), 102 (46), 130 (100), 158 (32), 175 (76), 284 (36), 329 (92), 311 (45), 385 (M^+, 33), 387 (M^++2, 12), 389 (M^++4, 0.5).

***Tert*-butyl 4-(4-fluorophenylamino)-2,5-dihydro-5-*oxo*-2-phenylfuran-3-carboxylate (4r).** Colourless solid; 0.232 g (83 %); m.p. 170-173 °C; IR (KBr): 3260, 3145, 2970, 1689, 1650 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ, ppm: 1.35 (s, 9H, CH_3), 5.61 (s, 1H, $H_{benzylic}$), 6.94-7.43 (m, 9H, $H_{aromatic}$) 9.37 (br, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) δ, ppm: 165.1, 162.9 (CO_{ester}), 160.2 (d, J_{CF} =245.9 Hz), 156.8, 135.0, 132.3 (d, J_{CF} =3.0 Hz), 128.6, 128.5, 127.6, 124.4 (d, J_{CF} =8.4 Hz), 115.8 (d, J_{CF} =22.6 Hz), 114.5 (10 $C_{aromatic}$ and C_{vinyl}), 83.3 (C-O), 62.0 ($C_{benzylic}$), 27.9 (CH_3); MS m/z (%): 57 (76), 77 (30), 102 (43), 130 (100), 158 (41), 175 (76), 268 (42), 295 (29), 313 (88), 361 (51), 369 (M^+, 29).

Supplementary information

Supplementary data are available free of charge at http://cjm.asm.md as PDF file.

Acknowledgments

We gratefully acknowledge the financial support from the Research Council of the University of Sistan and Baluchestan.

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